THESIS

STUDIES ON DRYING OF SELECTIVE FOOD ITEMS USING NOVEL DRYING METHODOLOGY

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CERTIFICATE FROM THE SUPERVISORS

This is to certify that the thesis entitled **"STUDIES ON DRYING OF SELECTIVE FOOD ITEMS USING NOVEL DRYING METHODOLOGY"** submitted by **Mr. Niladri Chakraborty** who got his name registered on 23/03/2012 for the award of **Ph. D. (Engineering) degree of Jadavpur University** is absolutely based upon his own work under the supervision of **Prof. (Dr.) Rajat Chakraborty (Chemical Engineering Department, Jadavpur University, Kolkata)** and **Prof. (Dr.) Asit Kumar Saha (Principal, Haldia Institute of Technology, Haldia).** Neither his thesis nor any part of the thesis has been submitted for any degree or diploma course or any other academic award anywhere before.

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(NILADRI CHAKRABORTY)

CONTENTS

NOMENCLATURE / ABBREVIATIONS / SYMBOLS USED

AOAC: Association of Official Analytical Chemists

ARMD: Age-Related Macular Degeneration

BFDKF: blended freeze-dried kiwi fruit

BKF: blended kiwi Fruit

CA: Catla (*Catla catla*)

CS: Chela (*Chela cachius*)

CF: corn flour

CFD: conventional freeze-drying

CHD: coronary heart disease

CPMDH: copper plated multidimensional heater

DG: dried ginger powder

DHA: ω-3decosahexaenoic acid

DMA: dimethyl amine

DSC: differential scanning calorimetry

EFSA: European Food Safety Agency

EPA: ω-3eicosapentaenoic acid

FAO: Food and Agriculture Organization

FDA: Food and Drug Administration

FD: freeze-drying

FS: fructose solution

GM1: first-order Gauss-Markov process

FF: fish flesh

FS-1: fortified Shrimp and blended set-1

FS-2: fortified Shrimp and blended set-2

FSSAI (fssai): Food Safety and Standards Authority of India

GM: Gauss-Markov Process

IOMC: Inter-organization Programme for the Sound Management of Chemicals

KF: kiwi fruit

MUFA: monounsaturated fatty acids

NPN: non-protein nitrogen

OD: osmotic dehydration

ODFD: osmotic dehydration and freeze-drying

OPOD: optimized OD process

OPODFD: optimization of osmotically dehydrated freeze-drying

OS: osmotic solution

PID: proportional-integral-derivative

PUFA: polyunsaturated fatty acids

RC: rehydration capacity

RF: rice flour

RSM: response surface methodology

RTD: resistance temperature detector

SBM: sterilized brine mix

SCSH: silver coated steel heater

SCSRP: silver coated steel round plate

SG: solute gain

SH: steel heater

SPMDH: silver plated multidimensional heater

SFA: saturated fatty acids

SS: sucrose solution

STSP: Shrimp-CA-CS pulp

TMA: trimethyl amine

TMAO: trimethylamine oxide

TVBN: total volatile base nitrogen

VD: vacuum-drying

VDFS-1: final VD product corresponding to FS-1

VDFS-2: final VD product corresponding to FS-2

WHO: World Health Organization

WL: water loss

χ^2: Chai-square

$D_{eff,2}$: effective diffusivity at state 2 (at temperature T-2)

D_{eff}: effective diffusivity

$D_{eff,1}$: effective diffusivity at state 1 (at temperature T-1)

E: activation energy

$E\%$: Mean relative deviation

k, a, b, c, n: drying model constants

L: thickness of the sample kept in plate

$MR_{pre,i}$: predicted moisture ratio

$MR_{exp,i}$: experimental moisture ratio

MR: moisture ratio

M_c: moisture content at particular time

M_o: initial moisture content

N: number of observations

p: number of constants

$RMSE$: root mean square error

R^2: regression coefficient

R: universal gas constant

S/N: signal to noise ratio

T_2: temperature at state 2

T_1: temperature at state 1

t: time (in second) of vacuum drying

W_o: initial weight of sample (g),

$W_{i,w}$: initial weight of water in sample (g)

W_e: amount of evaporated water (g)

W_d: dry matter content of sample(g)

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ABSTRACT

Food needs to be stored to sustain human life. Preservation of food is utmost necessary to meet the challenges due to imbalances between food demand for consumption and supply of raw food. Worldwide, in every 40 years the population becomes doubled. So naturally, food production must also increase by the same amount every year. The starvation of people surely confirms that the demand and supply of food is not proportionately matched till date. According to the FAO's recent report, world food production per person has been steadily increasing. So, the fact is that there is both a surplus and a shortage of food; it means there is a lack of preservation of food. Preservation techniques are based on addition or removal of heat, controlling both moisture and water activity, addition of preservatives and other techniques (irradiation, pulsed electric field, high pressure etc.). Among them, the addition of heat (drying) and removal of heat (refrigeration and freezing) are mostly acceptable and have gained popularity. Drying/dehydration is the most ancient method of preservation for the removal of water by evaporation from a wet solid. Sun (natural) drying, tray drying, drum drying, vacuum drying, spray drying are also very popular. Low temperature preservation has now captured 50% of the total preservation though it is marginal costly. The idea of cold storage, cold air blast-freezing and cryogenic-freezing can short out the problems. The combination of freezing and drying (called 'freeze-drying') has also been applied but its high energy cost did not gain popularity. The vacuum-drying also gain popularity with maximum retention of nutrition. Recently, reduction of energy by different methods has gained much acceptance. The pre-processing before drying (both freeze-drying and vacuum-drying) was done such as osmotic dehydration (OD), blending, fortification with blending and blanching techniques. Different base materials, raw materials and / or additives viz. kiwi fruit, shrimp, fishes, broccoli, rice flour, corn flour, oat flour, lona salt, sugar, table salt, preservatives were used for presented research work.

Kiwi fruit [KF] was preserved through consecutive OD and freeze-drying (FD) [ODFD] retaining its high nutritional values. OD was performed using sucrose solution (SS) and optimized by "Taguchi orthogonal design" at 3 h time, at 40°B, 50°C corresponding to 60.75% water loss [WL] and 9.55% solute gain [SG] .

Notably, at optimal OD [OPOD] conditions, application of fructose solution resulted undesirable lower WL and higher SG of KF. In FD of OPOD (OPODFD) kiwi fruit, the thermal energy was supplied by an innovative silver coated steel heater (SCSH) , which was found to be much more efficient (2.5 h FD time) compared to an equal power conventional steel heater (SH) [3.82 h FD time] to achieve a final KF moisture content ≤ 5% (w/w). At both optimal OD and corresponding FD the drying kinetics were assessed using four models. The effective diffusivity (D_{eff}) for OPOD was 3.405 x 10^{-9} m^2/s. During consecutive FD, D_{eff} and activation energy (E) were 6.04 x 10^{-7} m^2/s and 23.96 kJ/mol for SCSH; while D_{eff} of 2.22 x 10^{-7} m^2/s and E of 53.55 kJ/mol for SH indicated higher energy-efficiency for SCSH. The proximate nutritional analyses are reported for fresh and dehydrated kiwi samples indicating acceptable quality.

It was already known that kiwi fruit (KF) is best fruit item among all fruits due to its maximum amount of nutrients present particularly vitamin C and potassium. It has so many health benefits for the human beings. In spite of its many health benefits, it was not reported to taste it other readily ingestible forms. So, it was value added to make a new product as a powder form. The KF is blended with several raw materials which used regularly in domestics purpose and rich in their individual nutrients by using step wise-short time addition and mixing methodology to avoid unwanted biochemical and chemical reactions of excess sourness, sweetness, bitterness, stickiness etc. and also to increase final powdery products shelf life, flavor, nutrients and retention of maximum vitamin C. The blended and less moisture contained KF was kept in silver coated steel round plate (SCSRP), freeze-dried (FD) under vacuum by heating at moderate temperature using a new designed cubic heater for sublimation energy. The FD time was found 4.5 h. The drying kinetics was studied by using four established models.

The diffusion coefficient (D_{eff}) and activation energy (E) were studied during FD. These results were compared with optimized osmotically dehydrated freeze-dried (OPODFD) KF for betterment. The FD sample was ground; vacuum dried without heat treatment and stored it for proximate analysis, physico-chemical properties analysis and sensory evaluation by 9-point Hedonic scale.

Fortification protocols for enrichment of shrimp (*Metapenaeus monoceros*) were developed employing two fishes viz. *Catla catla* (CA) and *Chela cachius* (CS). Pre-sterilized shrimps were blended with sterilized CA and CS at 2 : 1 : 1 weight ratio. Pre-blended shrimps were converted to fortified shrimp (FS) by adding corn, rice flour (FS-1)and additionally mixing with dried-ginger (FS-2) and subsequently dehydrated by vacuum-drying (VD) [using silver /copper plated multidimensional heater (SPMDH / CPMDH)] to produce vacuum-dried fortified shrimps (VDFS-1 and VDFS-2). The VD kinetics indicated faster dehydration of FS-1 compared to FS-2 in both SPMDH and CPMDH. Higher effective diffusivity (7.464×10^{-10} m^2/s) and lower activation energy (28.42 kJ /mol) was computed for FS-1 in SPMDH. The VDFS-1 exhibited superior quality due to remarkable augmentation in protein (188%), ω-3 fatty acids (20%), carbohydrate (35%), ash (151%) and other essential elements with acceptable water activity, rehydration ratio, TVBN and histamine content.

Broccoli has many health benefits (such as rich in vitamin C, B-vitamins, vitamin D, dietary fiber, many minerals viz. Fe, Ca, Cu, Mg, Mn, P, Se, K and Zn and rich source of a flavonoids) so that it is a unique vegetable as compare with other vegetables. Therefore, fresh broccoli must have to preserve by suitable technique (s) with reduction of initial moisture content and without destroying the nutrients can be alternate solution. It is found that frozen conditioned broccoli (both cooked &

uncooked) is far better vitamin C retained as compare with unfrozen conditioned broccoli (both cooked & uncooked). Therefore, after blanching always freezing is preferable. Hence, selection of FD may be beneficial for broccoli. Cold blanching technique was done as pre-processing of broccoli before FD and it was found that the FD time was less (5.5 h) as compared with non-blanching (6.5 h). The final freeze-dried product protein and ash incremented by 2 and 4 times respectively, vitamin C lost 12% to reach final moisture level 5% from 88% of raw broccoli.

Key words: Preservation, Population, Starvation, Drying, Freezing, Freeze-drying. Freeze-drying, osmotic dehydration, optimized, silver coated steel heater, effective diffusivity, activation energy, nutritional value. Kiwi fruit powder, value added product, Cubic heater, Freeze-dried, Diffusion coefficient, Activation energy, Proximate analysis and physico-chemical properties, Hedonic scale. Fortification, Shrimp, Catla, Chela, Vacuum-drying, Drying kinetics, Broccoli. Cold blanching.

CHAPTER- 1

DRYING FOR FOOD PROCESSING, AND ITS ADVANCEMENT

1.0 INTRODUCTION

Drying is one of the most ancient methods of food preservation known to mankind. Drying is a mass transfer process consisting of the removal of water or another solvent by evaporation from a solid or semi-solid. This process is often used as a final production step before selling or packaging products. Compare with drying and evaporation, the evaporation is mostly applied for the concentration of solution but drying is associated with the removal of water or moisture from the material to give a dry product. A basic difference between dehydration and drying is that dehydration means to get rid of all the water present in the material but drying involves the removal of free water completely and bound water partially from the material. A source of heat supply is to be required to remove the vapour produced by the process.

1.1 TECHNOLOGICAL OBJECTIVES OF DRYING

Unfortunately, the quality of products dried by the conventional method is inferior to that of the initial product. Drying or dehydration is the removal of water by evaporation from a solid or liquid food. The main technological objectives of drying are:

- Preservation as a result of lowering of *water activity*
- Reduction in volume and weight
- Transformation of a food to a form more convenient to store, package, transport
- Imparting to a food product a particular desirable feature such as a different flavor, crispiness, chewiness etc. i.e. creating a new product development.

The most important **engineering and technological** issues in food dehydration are the following:

- ***The kinetics of drying***: with few exceptions (such as spray drying), drying is a relatively slow process. Knowledge of the factors that affect the rate of drying is essential for the optimal design and operation of drying systems
- ***Product Quality***: removal of water is not the only consequence of most drying operations. Other important quality-related changes in taste, flavor, appearance, texture and nutritive value may occur in the course of drying

- ***Energy consumption***: most common drying processes use extensive quantities of energy at relatively low efficiency (Zeuthen and Sorensen, 2000).

1.2 MECHANISM OF DRYING

The *mechanism* of water removal by drying (or by dryer) involves two simultaneous processes: transfer of heat for the evaporation of water to the food and transport of the water vapours formed away from the food. Therefore, drying is a unit operation based on *simultaneous heat and mass transfer* which is shown in Figure 1.1.

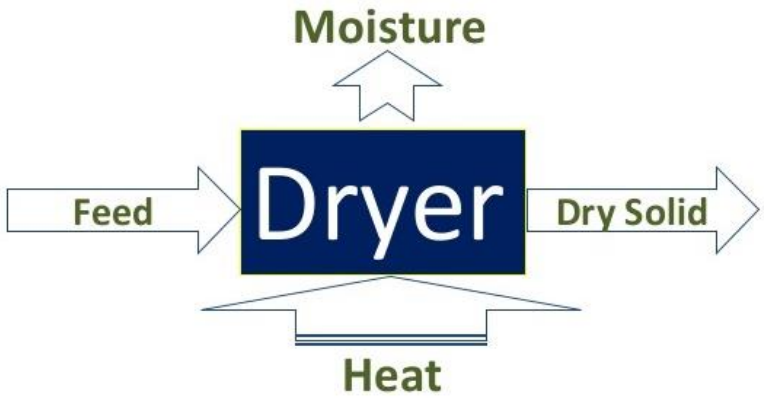


Figure 1.1: Basic block diagram of drying process.

Depending on the mode of transfer, industrial drying processes can be grouped in *two* categories: *convective drying* and *conductive drying*. In *convective drying*, hot and dry gas (usually air) is used both to supply the heat necessary for evaporation and to remove the water vapour from the surface of the food. Both heat and mass exchanges between the gas and the particle are essentially convective transfers, although conduction and radiation may also be involved to some extent. Air-oven drying, *spray drying* etc. are the examples of this type of drying. In *conductive drying*, the moist food is brought into contact with a hot surface. The water in the food is boiled-off. *Vacuum drying*, drum drying and drying in superheated steam are the examples of this mode of drying.

1.3 FEED STOCK FOR DRYING PROCESS

- Wet solids
- Slurry
- Solution
- Suspension

1.4 TYPES OF DRYERS

1.4.1 On the basis of heating mode

- Conductive
- Convective
- Radiation

1.4.2 On the basis of contact with heating media / element

- Direct contact dryers
- Indirect contact dryers

1.4.3 On the basis of processing mode

- Batch dryers
- Continuous dryers

1.5 SELECTION OF DRYING EQUIPMENT

It depends upon:

- Physical / chemical properties of materials
- Production capacity / throughput (kg / h)
- Initial moisture content of raw material and final moisture content of end products
- Particle size distribution
- Temperature and drying characteristics
- Explosion and toxicological characteristics

1.6 GENERAL CONSIDERATION FOR SELECTION OF DRYER

- Dryer kept in the room must be well designed for particular dryer and the room will be well ventilated, cleaned and maintained all hygienic conditions
- Energy consumption must be minimized
- Dryer must operate smoothly, safely and economically
- Dryer must be operated by a technical sound person
- Controlling parameters of that dryer must be previously known
- Operating and maintenance cost must not be excessive
- Environmental Pollution must be controlled

1.7 DRYING EQUIPMENTS

- Tray Dryers
- Tunnel Dryers
- Roller or Drum Dryers
- Fluidized Bed Dryers
- Spray Dryers
- Pneumatic Dryers
- Rotary Dryers
- Trough Dryers
- Bin Dryers
- Belt Dryers
- Freeze-dryers
- Vacuum-dryers

In the food industry so many diversified and extensive drying operations are performed, it would be expected that a great number of different types of dryer would be in used (Ramaswamy and Marcotte, 2006; Rao, 2010; Pare and Mandhyan, 2011). The principles of drying may be applied to any type of dryer, but it should help the understanding of these principles if a few common types of dryers are described. The general applications on the food items (selective) of the above twelve drying equipments (dryers) are shown in Table 1.1.

The major problem in calculations on real dryers is that conditions change as the drying air and the drying solids move along the dryer in a continuous dryer, or change with time in the batch dryer. The principles of mass and heat balances are the basis and the analysis is not difficult once the fundamental principles of drying are understood.

1.7.1 Tray Dryers

In tray dryers, the food is spread out, generally quite thinly, on trays in which the drying takes place. Heating may be by an air current sweeping across the trays, by conduction from heated trays or heated shelves on which the trays lie, or by radiation from heated surfaces. Most tray dryers are heated by air, which also removes the moist vapours. Tray or cabinet dryers are frequently found in rural installations where they are used for drying fruits (grapes, dates, apples), vegetables (onion, cabbage) and herbs (parsley, basil, mint). Air inlet temperatures are usually in the range of 60-80°C. Air velocity is between 2-3 ms^{-1}. Depending on the product and the conditions, the duration of a batch is generally 2 to 10 hours. Most cabinet dryers design to recirculate the air (to save energy).

Limitation (s):

- Due to the intensive use of labour (the large amount of manual work is involved to operate them), the operating cost is high.
- The product is dried non-uniformly, since the hot air current not reached to the product in different surfaces.

1.7.2 Tunnel Dryers

These may be regarded as developments of the tray dryer, in which the trays on trolleys move through a tunnel where the heat is applied and the vapours removed. In most cases, air is used in tunnel drying and the material can move through the dryer either parallel or counter current to the air flow. Sometimes the dryers are compartmented, and cross-flow may also be used.

Limitation (s):

- Though the limitation (s) of tray dryer can be removed by it easily, but the area of installation and operation are more.

1.7.3. Roller or Drum Dryers

In these the food is spread over the surface of a heated drum. The drum rotates, with the food being applied to the drum at one part of the cycle. The food remains on the drum surface for the greater part of the rotation, during which time the drying takes place, and is then scraped off. Drum drying may be regarded as conduction drying. They are classified into two types viz. single and double drum dryers. Double drum dryers consist of two drums rotating in opposite directions, with a narrow, adjustable gap between the two. Applicator rolls are used for spreading viscous liquids, purees and pastes on the drum surface. Single drum dryers are extensively used with applicator rolls to produce instant mashed potato.

Limitation (s):

- The food products are always charged as a thin layer or film on the hot metal surface of drum. So, it is bound to prepare the feed which has a tendency to form a film layer like structure.
- Only the conduction mode of heat transfer is possible to dry the materials.

1.7.4 Fluidized Bed Dryers

In a fluidized bed dryer, the food material (size 0.05-10 mm) is maintained suspended against gravity in an upward-flowing air stream. There may also be a horizontal air flow helping to convey the food through the dryer. Heat is transferred from the air to the food material, mostly by convection. Due to the efficient heat and mass transfer, the product is dried rapidly.

Limitation (s):

- The particles selected for drying have a property to overcome the gravitational force, so small to medium particles are to be selected for drying operations.
- The initial moisture content of selected material must be very low.
- Only the convective heat transfer is possible during drying operations.

1.7.5 Spray Dryers

In a spray dryer, liquid or fine solid material in a slurry is sprayed in the form of a fine droplet dispersion into a current of heated air. Air and solids may move in parallel or counterflow. Drying occurs very rapidly, so that this process is very useful for materials that are damaged by exposure to heat for any appreciable length of time. The dryer body is large so that the particles can settle, as they dry, without touching the walls on which they might otherwise stick. Commercial dryers can be very large of the order of 10 m diameter and 20 m high.

Limitation (s):

- Only the liquid food items are applicable for this dryer.
- Only the convective mode of heat transfer is possible for drying.
- The control of feed rate, hot air flow rate and product quality is not an easy task to handle at a time.

1.7.6 Pneumatic Dryers

In this type of dryer, the solid food particles are conveyed rapidly in an air stream, the velocity and turbulence of the stream maintaining the particles in suspension. Heated air accomplishes the drying and often some form of classifying device is included in the equipment. In the classifier, the dried material is separated, the dry material passes out as product and the moist remainder is re-circulated for further drying.

Limitation(s):

- Use only for large solid particles.
- Initial moisture content of feed must be low.
- Only the radiation mode of heat transfer is possible.
- Operating temperature is high, so it is not to be used for heat sensitive food items.

1.7.7 Rotary Dryers

Though rotary dryers are mainly used in the chemical and mineral industry but in the area of food they are applied for dehydrating waste materials (citrus peels, vegetable trimmings) and animal feedstuffs (alfalfa). The foodstuff is contained in a horizontal inclined cylinder through which it travels, being heated either by air flow through the cylinder, or by conduction of heat from the cylinder walls. In some cases, the cylinder rotates and in others the cylinder is stationary and a paddle or screw rotates within the cylinder conveying the material through. When it reaches a position where its angle of repose has been exceeded, the material falls back to the bottom of the cylinder. Most of the drying takes place while the material falls through the air blast. Using very hot air or combustion gases, rotary dryers can also function as roasters for nuts, sesame seeds and cocoa beans.

Limitation (s):

- Products dried-up by the convection mode of heat transfer only.
- Drying temperature is high, so limitation for heat sensitive food.
- The residence time is non-uniform.

1.7.8 Trough Dryers

This is a special type of dryer, designed for the initial drying of vegetables in small pieces or grains etc. The materials (wet) to be dried are contained in a trough-shaped conveyor belt, made from mesh, and air is blown through the bed of material. The movement of the conveyor continually turns over the material, exposing fresh surfaces to the hot air. The movement of the bed causes gentle and continuous mixing of the bed, while a vertical blast of hot air circulates through the material. The blast is sufficiently strong to expand the bed slightly without fluidizing it. Evaporation is very fast and most of the water in the wet material is removed, typically less than one hour. The trough assembly is tilted towards one end, causing the material to move slowly down the slope towards the discharge.

Limitation (s):

- More energy is required for this dryer.
- Always used for the higher moisture content and heat resistant food items.

1.7.9 Bin Dryers

Tray, belt and trough dryers are quite efficient in removing most of the water of high moisture materials in the initial stage of drying. In the last stages of the falling rate period, however, removal of residual moisture takes a long time and external turbulence and mixing cannot accelerate the process. In the case of vegetables, using the more expensive types of dryers for reducing the moisture content below 15-20% would be uneconomical. Bin dryer provide the probable solution for finishing the process to the desired final moisture content of 3-6%. In bin dryers, the foodstuff is contained in a bin with a perforated bottom through which warm air is blown vertically upwards, passing through the material and so drying it.

Limitation (s):

- Only used for the large solid particles.
- Initial moisture content of feed must be low.
- Rate of drying is low.

1.7.10 Belt Dryers

The food is spread as a thin layer on a horizontal mesh or solid belt and air passes through or over the material. In most cases the belt is moving, though in some designs the belt is stationary and the material is transported by scrapers. The wet product placed in a bed of different thickness, is carried through a tunnel on perforated (mesh, slotted or louvered) conveyors. The heated air is either directed up or down through the conveyor and the layer of the product or perpendicular to the surface or through flow. It can also be directed parallel to the material surface or cross flow for products spread in thin layers on a non-perforated band. Air circulation is usually a combination of cross flow and through flow. Some dryers consist of two or more

conveyors in a series. Multiple conveyors (up to five), one above the other, can be used.

Limitation (s):

- Sufficient heat energy is required.
- Suitable only for large solid and heat resistant particles.

1.7.11 Freeze-Dryers

The primary condition of freeze-drying is the freezing water to ice and then sublime this frozen water to water vapour at very low pressure (below the pressure of triple point of water). The frozen material is held on shelves or belts in a chamber or cabinet that is under high vacuum. Heat (sublimed) is transferred to the food by conduction or radiation and the vapour is removed by vacuum pump through the condenser or moisture trap. The condensed vapour is transformed directly to ice and ice is deposited on the inside surface of the wall. Since during freeze-drying sublimation happens so the end freeze-dried product is very porous and light in physical nature. The final moisture of the product is mostly found 1-5%.

Limitation (s):

- Used only for costly items (like food, pharmaceuticals etc.) and high initial moisture content with good texture.
- Highest level of drying time (due to heat of sublimation) and hence energy required more.
- Extensively used for selective items or heat sensitive products.

1.7.12 Vacuum-Dryers

To intensify moisture removal and lower the drying temperature in order to protect heat-sensitive food components, vacuum-drying technologies are used. All vacuum-drying systems have four essential parts viz. a vacuum chamber, a heat supply, a device for producing and maintaining the vacuum (vacuum pump), and components to

collect water vapours evaporated from the food. Batch vacuum dryers are substantially the same as tray dryers, except that they operate under a vacuum, and heat transfer is largely by conduction or by radiation. The trays are enclosed in a large cabinet, which is evacuated. The water vapour produced is generally condensed, so that the vacuum pumps have only to deal with non-condensable gases. Another type consists of an evacuated chamber containing a roller dryer. Vacuum drying is similar to freeze-drying except that the product is not frozen and the vacuum is not as high.

Limitation (s):

- Initial moisture content of drying substances must be low.
- Only conduction mode of heat transfer is possible
- Since the unit operates at low pressure, so only the heat sensitive items are chosen.

Table 1.1: Applications for selected Dryers.

Sl. No.	Dryer type	Food products
1.	Tray or cabinet	Vegetables, fruits, meats, fishes, confectionary
2.	Tunnel	Vegetables, fruits
3.	Roller or Drum	Milk, soups, flakes, baby cereals, juices, potatoes
4.	Fluidized bed	Vegetables, granules, grains, peas
5.	Spray	Milk, cream, coffee, tea, juices, eggs, extracts
6.	Pneumatic	Starch, pulps, crops, granules, powders (flours)
7.	Rotary	Seeds, grains, starch, sugar crystals
8.	Trough	Pulps, grains, crops, vegetables
9.	Bin dryer	Vegetables, grains
10.	Belt dryer	Grains, vegetables, fruits, cereals, nuts
11.	Freeze-dryer	Very heat sensitive foods (fruits, vegetables, herbs, spices, fish, meat etc.)
12.	Vacuum-dryer	Heat sensitive foods (fruits, herbs, spices, selected fish, meat etc.)

Source / Reference : Ramaswamy & Marcotte (2006).

1.8 FREEZE-DRYING

Freeze-drying (lyophilization) is the latest development of food preservation for high nutritional foods. It is another method of water removal based on the sublimation of water (ice) from a frozen material under high vacuum. In this process, the food is first frozen in freezer and then subjected to high vacuum, whereby the water-ice

sublimates (i.e. evaporates directly, without melting). The water vapour released is usually trapped on the surface of a condenser at very low temperature (-40ºC). The heat of sublimation is supplied to the foods by various methods. It is based upon the dehydration of the frozen product through sublimation. Because this process does not require liquid water, and the product is at a low temperature, most of the microbiological reactions and deterioration are stopped, obtaining a final product of excellent quality. The water, being present in the freeze-drying process as a solid entity, it protects the primary structure and the shape of the products with minimum decrease volume. Despite the advantages, freeze-drying has been recognized as being the most expensive method to make a dehydrated product. These processes can affect (partially or totally) the quality of a product. Ended, a lot of changes can occur in the physical, chemical and/or biological characteristics of the products during the process, storage and distribution.

1.8.1 Historical Review

Freeze drying methods have been used for *over 100 years* for various technical purposes. The ancient Incas of Peru used mountain peaks along the Andes as natural food preservers. The extremely cold temperature and low pressure at those high altitudes prevented food from spoiling in the same basic way as a modern freeze drying machine. During *World War-II*, equipment and techniques were developed to supply blood plasma and penicillin to the armed forces. In the *late 70's* freeze drying was commonly used for taxidermy, food preservation, museum conservation and pharmaceutical production. Over 400 different types of freeze dried foods have been commercially produced *since the1960s*. Two bad candidates for freeze drying are lettuce and watermelon because they have too high water content and freeze dried poorly.

It was *Benedict* and *Manning* (*1905*), first evolved laboratory instrument for freeze drying. They actually displaced the air environment by ethyl ether, which is highly volatile in nature. After that, *Shackell* was taken the basic design of Benedict and Manning and used an electrically driven vacuum pump (to produce necessary vacuum) instead of the displacement of air with ethyl ether. It was *Shackell* who first realized that the material had to be frozen before commencing the drying process; hence *freeze drying*. In Figure 1.2, laboratory unit of freeze-drying is shown using

acetone and dry ice as a freezing-mixture (refrigerant) for both freezing of sample and condensing of sublimed vapour (from frozen sample) during drying under vacuum conditions. Both the sample (frozen) flask and condenser flask are under vacuum (using vacuum pump), and connected through a tube.

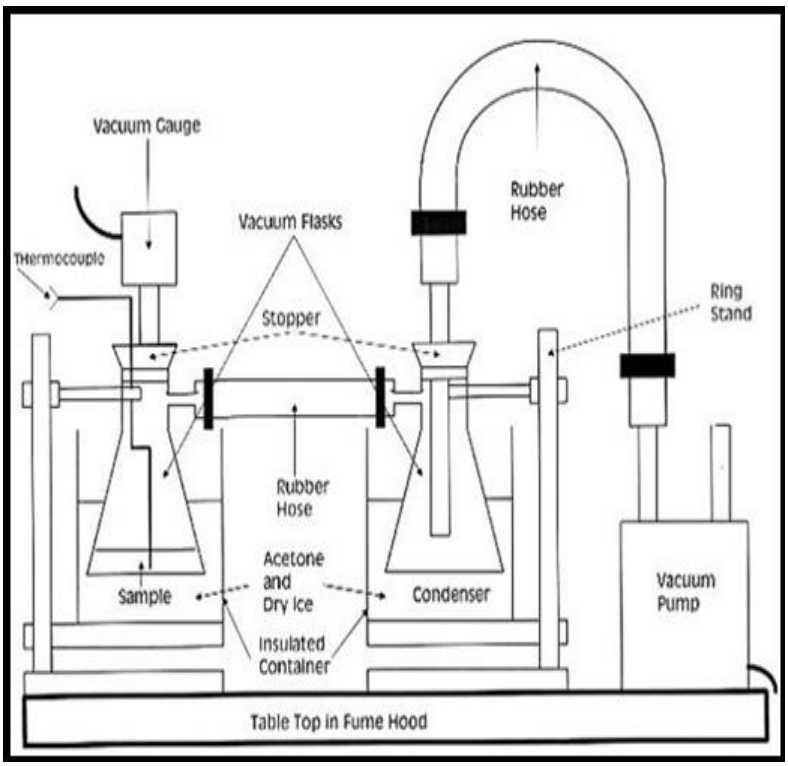


Figure 1.2: Laboratory unit of Freeze-drying *(Thomas A. Jennings)*.

Freeze-dried coffee was first produced *in 1938*, and lead to the development of powdered food products. "NESTLE" company invented the freeze-dried coffee, after being asked by Brazil to help find a solution to their coffee surplus. Nestle's own freeze dried coffee product was called *Nescafe* and was first introduced in Switzerland. Tasters choice coffee, another very famous freeze-dried manufactured product, derived from patent issued to *James Mercer*.

From the beginning of recorded history, man has struggled to find methods of preservation (since there were more surplus of crops, fruits & vegetables etc.), suitable for the long term storage of foods. Modern human has employed a variety of techniques on preservation including chemicals, mechanical refrigeration, cryogenics, sterilization, irradiation, fermentation and dehydration etc. for all the practices of long

term preservation. Lyophilization (freeze-drying) is the most natural and produces a result that favours extended storage and preserves the biological specimens.

More than *20 years ago*, astronaut *John Glenn* became the first American to orbit the Earth. Among the many tasks Glenn had to perform while in orbit were the first American space experiments in eating food in the weightless conditions of Earth orbit. Later, his valuable experience had helped to design 'space food' systems. Selective freeze-dried food items may serve the purposes of the astronauts. Besides, special freeze-dried foods need to be prepared for the requirements of soldiers posted at remote locations. Hence, extensive research is of utmost importance to develop the food items as per their demand.

Over past few years, freeze-drying had been acclaimed as a modern technology of drying. However, very few of us used this technology to preserve the *selective* food items. The term *selective* in the sense that there are certain foods which are very sensitive to temperature; the nutrition (micro-molecules) are lost or destroyed during a particular temperature profile. As these micronutrients are very much essential to our human body, thus, freeze-dried product(s) are very much significant. By this technique, all essential ingredients, color, aroma and texture are retained as that the original food item.

1.8.2 Phase for Freeze-Drying

Any food item contains water which can assist in decreasing the shelf life of particular food items. Since, microbes are hydrophilic in nature; they can grow randomly in presence of water. In freeze-drying, the liquid water molecules present in food item (so called solution) are frozen to convert into water-ice crystal. By sublimation (next step), this crystal is converted to water vapour. In Figure 1.3, transformation of phases from solid (ice) to water vapour is to be performed under sublimation process (Rao, 2010). Sublimation of water-ice can occur only if the vapour pressure and temperature are well below the atmospheric pressure [below those of the triple point of water i.e. below 612 Pa (4.58 mm Hg)] and 0.01°C respectively. Therefore, the vacuum inside the dryer must be created and operated at very low vacuum pressure, so that the transformation of solid to vapour phase will be occurred very nicely. Freeze-drying occurs at very low total pressure (vacuum). At this condition, water vapour has a large

specific volume (v, m^3 / kg). Since, large volume of vapour created (which is gaseous state), the vacuum pump must have unrealistically large displacement capacity. To overcome this problem, the vapours are condensed as ice crystals on the surface of condensers, kept at extremely low temperature (- 40°C or less).

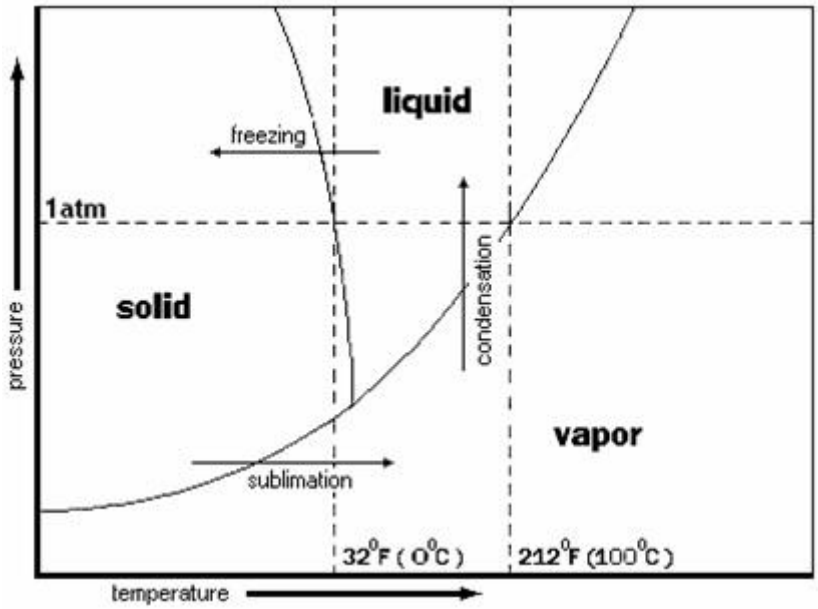


Figure 1.3: Transformation of phases for Freeze-drying.

1.8.3 Reasons of Freeze-Drying Process

Since freeze-drying (lyophilization) is a widely used method for dehydrating a vast range of materials, including foodstuffs, pharmaceuticals, biotechnology products, vaccines, diagnostics and biological materials therefore it may be carried out on a range of scales, from bench top through pilot-scale to a full-scale manufacturing process and offers a number of advantages over conventional drying and many other processing methods. Despite its wide use, however, it is still apparent that many regard freeze-drying as somewhat of an art. This is perhaps not surprising, given the lack of available texts devoted to the process itself, with many published articles tending to describe specific applications of the process. Specialized training courses are now arguably the most effective means of learning about the various aspects of freeze-drying technology. This article seeks to provide a brief overview of the process:

The foods conservation engineering - important part in program of human nutrition.

- Interdisciplinary research, for:
  - to improve human nutrition
  - Promote good health through new and traditional foods.
- For all the people - the choices - the nutritional status of the World.
- One of the best methods for foods conservation engineering is the lyophilization, (freeze-drying). Though it is a complex process that requires a careful balancing of product, equipment, and processing techniques.
- Freeze-drying (lyophilization) is a widely used method for dehydrating a vast range of materials, including foodstuffs, pharmaceuticals, biotechnology products, vaccines, diagnostics and biological materials.
- The lyophilization process, also known as freeze-drying or sublimation, has many advantages over other processing methods. Since freeze-drying is achieved at lower pressures and temperatures than other methods, it is an inherently gentle process to retain maximum nutritional quality. Lyophilization, achieved by freezing the wet substance and causing the ice to sublime directly to vapour by exposing low partial pressure of water vapour.
- In practice the substance may not be completely frozen, especially if no aqueous solutions are present, and most lyophilization processes are completed by a period of desorption drying.

  A very simplified freeze-dryer unit is represented in Figure 1.4, where the frozen sample is kept in freeze-dryer chamber. Both this chamber and condenser are under the vacuum conditions. The vacuum is created by vacuum pump. The dryer chamber and condenser are separated to each other, but they are joined by pipe (vacuum) through pressure control valve which is also used to disconnect between dryer chamber and condenser unit plus vacuum pump.

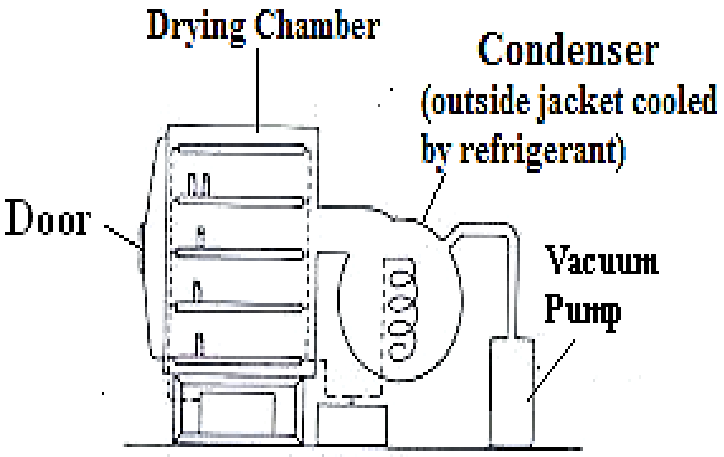


Figure 1.4: A general view of a Freeze-dryer Unit.

1.8.4 Working Stages of Freeze-Drying

According to Oregon (world's leading processors of freeze-dried products, USA) freeze-drying, the purpose of freeze-drying is to remove a solvent (usually water) from dissolved or dispersed solids. Freeze-drying is method for preserving materials, which are unstable in solution. In addition, freeze-drying can be used to separate and recover volatile substances, and to purify materials. The fundamental process steps are:

Freezing: The product is frozen. This provides a necessary condition for low temperature drying.

Vacuum: After freezing, the product is placed under vacuum. This enables the frozen solvent in the product to vaporize without passing through the liquid phase, a process known as sublimation.

Heat: Heat is applied to the frozen product to accelerate sublimation.

Condensation: Low-temperature condenser plates remove the vaporized solvent from the vacuum chamber by converting it back to a solid. This completes the separation process.

- The freeze-drying process

There are four stages in the complete drying process: pre-treatment, freezing, primary drying, and secondary drying.

- **Pre-treatment**

Pre-treatment includes any method of treating the product prior to freezing. This may include concentrating the product, formulation revision (i.e., addition of components to increase stability and/or improve processing), decreasing a high vapour pressure solvent or increasing the surface area. In many instances the decision to pre-treat a product is based on theoretical knowledge of freeze-drying and its requirements or is demanded by cycle time or product quality considerations. Methods of pre-treatment include: Freeze concentration, Solution phase concentration, Formulation to preserve product appearance, Formulation to stabilize reactive products, Formulation to increase the surface area and decreasing high vapour pressure solvents.

- **Freezing**

In a lab, this is often done by placing the material in a freeze-drying flask and rotating the flask in a bath, called a shell-freezer, which is cooled by mechanical refrigeration, dry ice and methanol, or liquid nitrogen. On a larger scale, freezing is usually done using a freeze-drying machine. In this step, it is important to cool the material below its triple point, the lowest temperature at which the solid and liquid phases of the material can coexist. This ensures that sublimation rather than melting will occur in the following steps. Larger crystals are easier to freeze-dry. To produce larger crystals, the product should be frozen slowly or can be cycled up and down in temperature. This cycling process is called annealing. Amorphous materials do not have a eutectic point, but they do have a critical point, below which the product must be maintained to prevent melt-back or collapse during primary and secondary drying.

- **Primary drying**

During the primary drying phase, the pressure is lowered (to the range of a few milibars) and enough heat is supplied to the material for the water to sublime. The amount of heat necessary can be calculated using the sublimating molecules latent heat of sublimation. In this initial drying phase, about 95% of the water in the material is sublimated. This phase may be slow (can be several days in the industry), because, if too much heat is added, the material structure could be altered (Liu, 2006).

- **Secondary drying**

The secondary drying phase aims to remove unfrozen water molecules, since the ice was removed in the primary drying phase. This part of the freeze-drying process is governed by the materials adsorption isotherms. In this phase, the temperature is raised higher than in the primary drying phase and can even be 50-55°C, to break any physico-chemical interactions that have formed between the water molecules and the frozen material. Usually the pressure is also lowered in this stage to encourage desorption (typically in the range of microbars, or fractions of a Pascal). However, there are products that benefit from increased pressure as well (Liu, 2006).

After the freeze-drying process is complete, the vacuum is usually broken with an inert gas, such as nitrogen, before the material is sealed.

As a general rule, freeze-drying occurs in three stages:

FREEZING STAGE: Here the food material (s) is / are frozen below its / their freezing point.

PRIMARY DRYING STAGE (SUBLIMATION): In which sublimation of the frozen water occurs i.e. transformation of ice crystals directly to water vapour.

SECONDARY DRYING STAGE (DESORPTION): During which most of the water adsorbed on the solid matrix is removed through vaporization.

Typically, freeze drying is carried up to a final moisture content of 1-3%.

1.8.5 Applications of Freeze-Drying

- **Food industry**

Figure 1.5: Freeze-dried coffee.

Freeze-drying is used to preserve food, the resulting product being very lightweight. The process has been popularized in the forms of freeze-dried ice cream, an example of astronaut food. It is also widely used to produce essences or flavours to add to food. Because of its light weight per volume of reconstituted food, freeze-dried product is also popular and convenient for consumers. More dried food can be carried per the same weight of wet food and has the benefit of "long life" compared to wet food that tends to spoil quickly. The consumers then reconstitute the food with water available at point of use. Instant coffee is sometimes freeze-dried (Figure 1.5) despite the high costs of the freeze-driers used. The coffee is often dried by vaporization in a hot air flow or by projection onto hot metallic plates. Freeze-dried fruits are used in some breakfast cereal or sold as a snack and are an especially popular snack choice among toddlers, dieters, as well as being used by some pet owners as a treat for pet birds. In Figure 1.6, some freeze-dried fruits such as strawberry, apples, bananas etc. are shown for representation as freeze-dried products. Culinary herbs are also freeze-dried, although air-dried herbs are far more common and less expensive. Freeze- dried tofu is a popular foodstuff in Japan ("Koya-dofu" or "shimi-dofu" in Japanese).

Figure 1.6: Freeze-dried fruits.

In chemical synthesis, products are often freeze-dried to make them more stable or easier to dissolve in water for subsequent use.

In bio-separations, freeze-drying can be used also as a late-stage purification procedure, because it can effectively remove solvents. Furthermore, it is capable of concentrating substances with low molecular weights that are too small to be removed by a filtration membrane.

Freeze-drying is a relatively expensive process. The equipment is about three times as expensive as the equipment used for other separation processes, and the high energy demands lead to high energy costs. Furthermore, freeze-drying also has a long process time; because the addition of too much heat to the material can cause melting or structural deformations. Therefore, freeze-drying is often reserved for materials that are heat-sensitive, such as proteins, enzymes, microorganisms, and blood plasma. The low operating temperature of the process leads to minimal damage of these heat-sensitive products.

- **Pharmaceutical and biotechnology**

Pharmaceutical companies often use freeze-drying to increase the shelf life of the products, such as vaccines and other injectable. By removing the water from the material and sealing the material in a vial, the material can be easily stored, shipped, and later reconstituted to its original form for injection. Another example from the pharmaceutical industry is the use of freeze drying to produce tablets or wafers, the advantage of which is less excipient as well as a rapidly absorbed and easily administered dosage form.

- **Other uses**

Organizations such as the Document Conservation Laboratory at the United States National Archives and Records Administration (NARA) have done studies on freeze-drying as a recovery method of water-damaged books and documents. While recovery is possible, restoration quality depends on the material of the documents. If a document is made of a variety of materials, which have different absorption properties, expansion will occur at a non-uniform rate, which could lead to deformations. Water can also cause mold to grow or make inks bleed. In these cases, freeze-drying may not be an effective restoration method.

In bacteriology freeze-drying is used to conserve special strains.

- In high-altitude environments, the low temperatures and pressures can sometimes produce natural mummies by a process of freeze-drying.
- Advanced ceramics processes sometimes use freeze-drying to create a formable powder from a sprayed slurry mist. Freeze-drying creates softer particles with a more homogeneous chemical composition than traditional hot spray drying, but it is also more expensive.
- Freeze-drying is also used for floral preservation. Wedding bouquet preservation has become very popular with brides who want to preserve their wedding day flowers
- A new form of burial which previously freeze-dries the body with liquid nitrogen has been developed by the Swedish company Promessa Organic AB, which puts it forward as an environmentally friendly alternative to traditional casket and cremation burials.

The Atlas freeze-drying technology also means:

- Negligible product loss
- Low energy costs
- Reliable plants

1.8.6 Different Parts of Freeze-Drying Unit

There are different parts in a freeze-drying unites (Charm, 1981). The parts are as follows:

i) Drying Chamber, ii) Vacuum pump, iii) Moisture trap or Condenser, iv) Refrigeration unit, v) Heater for sublimation, vi) Weight Balance, vii) Probe(s), viii) Controlling unit, ix) Evaporator.

i. DRYING CHAMBER:

Drying Chamber is used for the drying of frozen food stuffs with restricted or fixed area. The vacuum is created indirectly inside this chamber. All the accessories like shelves, temperature probes, the control (temperature and pressure) units, weighing balance and others which may require at the time of drying are also to be placed inside this drying chamber. The inside of the chamber is made of stainless steel and door of this chamber is well air tight locking system made of transparent and hard

fibre with good gasket or sealing system to avoid leakage of any vacuum during drying of the foods.

ii. VACUUM PUMP:

A vacuum pump is a device that removes gas / air molecules from a sealed volume in order to leave behind a partial vacuum. The first vacuum pump was invented in 1650 by Otto von Guericke.

The new Cole-Parmer Rotary Vacuum Pump (shown in Figure 1.7) addresses the problems associated with difficult freeze dryer applications. These pumps combine the low base pressure and high pumping speeds of a rotary vane pump with the high chemical resistance of a PTFE diaphragm pump. The diaphragm pump holds a vacuum on the head space of the rotary vane pump. As the corrosive vapours are brought into the rotary vane pump, the diaphragm pump pulls them on through before they can condense in the oil. A pair of condensers on the outlet of the pump act to catch much of the solvents that have been evaporated, resulting in cleaner, longer lasting oil in the rotary vane pump.

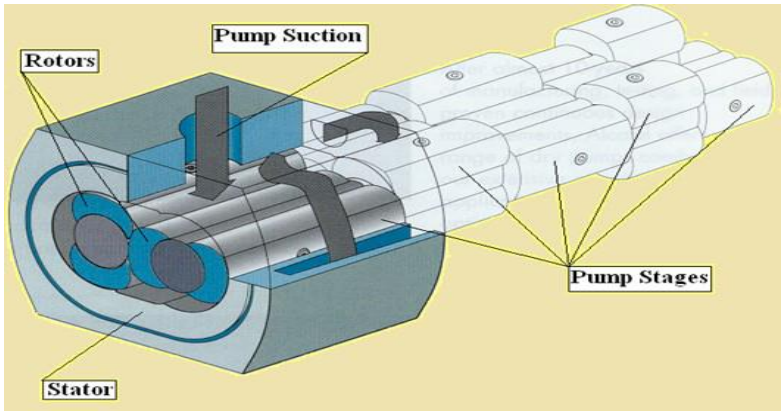


Figure 1.7: Different parts in Vacuum Pump.

iii. CONDENSER / MOISTURE TRAP:

In evaporators that are working under reduced pressure, a condenser, to remove the bulk of the volume of the vapours by condensing them to a liquid, often precedes the vacuum pump. Condensers for the vapour may be either surface or jet condensers. Surface condensers provide sufficient heat transfer surface, pipes for example,

through which the condensing vapour transfers latent heat of vaporization to cooling water circulating through the pipes. In a jet condenser, the vapours are mixed with a stream of condenser water sufficient in quantity to transfer latent heat from the vapours. The condenser captures the vapour that is removed from the product during the freeze drying process. In addition to capturing the vapour, the condenser should prevent vapour from reaching vacuum pump, where it will cause damage. In Figure 1.8, the compressor, condenser and throttling or expansion valve is shown below:

Figure 1.8: Compressor, Condenser and Throttling (valve) units.

iv. REFREGERATION UNIT:

Large, modern refrigeration units use the evaporation principle to produce the low temperatures necessary to do the job required. The process consists of a cycle of compression, cooling and condensation, then the expansion of the liquid, evaporation and re-compression of the vapour. Refrigeration is the Science of the Production of "Coldness". Refrigeration is a cooling process used to remove heat energy from a substance by the evaporation principle, to produce a temperature below that of its surroundings. As the water evaporates, the heat required for the evaporation process is removed from the interior of the tent thereby causing cooling.

The desirable properties of a refrigerant (s) are listed below:

- A Low Boiling Point: below the temperature it is expected to maintain.

- A Low Freezing Point: below the minimum temperature the system can reach - to prevent solidification of the refrigerant.
- A High Latent Heat of evaporation: Will require more latent heat of vaporization which will remove more sensible heat from the substance or space being cooled.
- A High Critical Temperature and Pressure: Can be easily condensed. A Low Critical Pressure will condense at relatively low pressure, thereby needing low energy to produce the condensation pressure.
- Non-Corrosive, Non-Toxic, Non-Flammable.

Because of the importance of the refrigeration system, a freeze-dryer must be equipped with a condenser designed and constructed with the ability to:

- Condense all vapours from the product.
- Provide a vapour route of minimum distance to avoid hindering vapour flow.
- Permit easy defrosting after the run.
- Prevent vapours from contaminating the oil in the vacuum pump.
- Provide a simple cleaning operation.
- Provide the necessary output under load to condense vapour at a maximum rate without disturbing the product's selected primary sublimation temperature.
- Insure the necessary low temperature (saturated suction) during the secondary drying to deliver the lower vacuum levels needed for this phase.

v. HEATER FOR SUBLIMATION:

An apparatus that heats or provides heat such as a hot air blower, radiator, convector etc.

- ADVANTAGES:

1. Exhaust without any smell, good environment need.
2. Normal temperature for exhaust, no dangerous for hot air out.

3. High efficiency cold system to make sure the exhaust temperature less than 25°C.

vi. WEIGHT BALANCE:

A weight balancing machine is a measuring tool used for weight of the materials. The weight balance unit have two types: (1) Rough balance: to measure the weight of the material without the decimal place (s), and (2) Accurate balance: Measure the weight of the materials accurately (upto two to four decimals). For research work, the second type is mostly used, since in research successive data are required for more accuracy or least error of the experiments.

vii. PROBE (S):

A thermometer is a device that measures temperature or temperature gradient using a variety of different principles. A thermometer has two important elements: the temperature sensor (e.g. the bulb on a mercury-in-glass thermometer) in which some physical change occurs with temperature, plus some means of converting this physical change into a numerical value. Here in freeze-drying unit many thermometer probes are used.

viii. CONTROLLING UNIT

A controlling unit (shown in Figure 1.9.) is a component of a processing device / unit that directs the operation of the processor. It directs the operation of the other units by providing timing and control signals. There are four systems of control unit by which the magnitude of the correction signal is directly proportional to the error. They are: (1) Proportional (P) control, Integral (I) control, Proportional-integral (PI) control and Proportional-integral-differential (PID) control. PID control is mostly used in industrial process control. In the PID system, the *offset* has been eliminated completely and hence the system is more stable during processing.

Figure 1.9: Temperature and Vacuum gauge control panel.

These panels are directly placed on the dryer where the drying takes place.

If four probes are used to measure four temperatures such as the sample surface temperature, sample center temperature, sublimation heater temperature and ambient (inside dryer) temperature then the temperature panel can show all the temperatures with definite / fixed time interval (s).

In Figure 1.10, the condenser and its control panel are shown. The set temperature of condenser is fixed before freeze-drying operation and the temperature of condenser is checked at a particular time by this control panel.

Figure 1.10: Condenser set and its control panel.

ix) EVAPORATOR:

A refrigerant in liquid form will absorb heat when it evaporates and it is this conditional change that produces cooling in a refrigerating process. If a refrigerant at the same temperature as ambient is allowed to expand through a hose with an outlet to atmospheric pressure, heat will be taken up from the surrounding air and evaporation will occur at a temperature corresponding to atmospheric pressure. If in a certain situation pressure on the outlet side (atmospheric pressure) is changed, a different temperature will be obtained since this is analogous to the original temperature - it is pressure dependent. The component where this occurs is the evaporator, whose job it is to remove heat from the surroundings, i.e. to produce refrigeration.

1.8.7. Different Types of Freeze-Dryer

- Pilot freeze-dryer
- Industrial freeze-dryer: it is sub-divided to
- Tray and Pharmaceutical freeze-dryer
- Multi-batch freeze-dryer
- Continuous freeze-dryer
- Tunnel freeze-dryer
- Vacuum spray freeze-dryer

Pilot freeze-dryer

Pilot freeze-dryers are appropriate for food and pharmaceutical products for the laboratory basis. These dryers are suitable for the experimental or research purpose (batch size 0.025 kg to 1 kg). The unit consists of drying chamber, heating element, condenser, refrigeration unit and vacuum system.

Tray and pharmaceutical freeze-dryer

Large number of industrial freeze-dryers in operation is of the vacuum batch type with freeze-drying of the product in trays. There are two main types depending on the type of condenser used. In the first type, the condenser plates are alongside the tray heater assembly and in the same chamber. In the second type, the condenser is in a

separate chamber joined through the first by a pipeline via butter fly valve. The second type is generally used both for food and pharmaceutical industries.

Multi-batch freeze-dryer

The freeze-drying process in a batch plant is normally program controlled to minimize the drying time and to maximize the production capacity of the plant. With a single batch plant the load on the various systems will be variable throughout the drying cycle. The optimal utilization of resources will not be possible in a single cabinet batch plant. This disadvantage can be eliminated when an industrial freeze-drying plant is built with a number of batch cabinets. This makes possible the simultaneous production of different products, which increase the operational flexibility of the plant. A large number of industrial freeze-drying plants operate today in this way as multi-cabinet batch plants.

Continuous freeze-dryers:

It is found in recent years that a growing interest on freeze-drying plants operating with a continuous flow of material through the process. Particularly the industries handled the single standardized product and the preparation of product by a continuous process; such plants are very much profitable. They give continuity on processing throughout and constant operating conditions those are easily controlled and they require less manual operation and supervision. Continuous freeze-dryers are used for freeze-drying of product in trays and for freeze-drying of large bulk products.

Tunnel freeze-dryers

In the tunnel type of freeze-dryers, the process takes place in a large vacuum cabinet into which the tray carrying trolleys are loaded at the time intervals through a large vacuum lock at one end of the tunnel and discharged similarly at other end of tunnel. Large commercial plants for processing cottage cheese and coffee have been produced by this way. The tunnel freeze-dryers have the same advantages of plant capacity utilization that can be achieved as multi-batch plants, but the flexibility of simultaneous production of different products or in switching from one product to another product is lacking.

Vacuum spray freeze-dryers

This dryer has been developed for coffee extract, tea infusion or milk. The product is sprayed from a single jet upward or downward in a cylindrical tower of 3.7m diameter by 5.5m high. The liquids solidify into small particles similar like spray dryer by evaporative freezing. The whole plant (chamber) operates under a vacuum of about 67 Pa. Frozen particles obtained by spraying into vacuum are about 150 mm diameter and lost about 15% moisture in the initial evaporation. The great advantage of this freeze- dryer is almost no nutritional loss of the small particles without sticking of them.

There are essentially **three** categories of freeze-dryers: the manifold freeze-dryer, the rotary freeze-dryer and the tray style freeze-dryer. Two components are common to all types of freeze-dryers: a vacuum pump to reduce the ambient gas pressure in a vessel containing the substance to be dried and a condenser to remove the moisture by condensation on a surface cooled to −40 to −80 °C. The manifold, rotary and tray type freeze-dryers differ in the method by which the dried substance is interfaced with a condenser. In manifold freeze-dryers a short usually circular tube is used to connect multiple containers with the dried product to a condenser. The rotary and tray freeze-dryers have a single large reservoir for the dried substance.

Rotary freeze-dryers are usually used for drying pellets, cubes and other pourable substances. The rotary dryers have a cylindrical reservoir that is rotated during drying to achieve a more uniform drying throughout the substance. Tray style freeze-dryers usually have rectangular reservoir with shelves on which products, such as pharmaceutical solutions and tissue extracts, can be placed in trays, vials and other containers.

Manifold freeze-dryers are usually used in a laboratory setting when drying liquid substances in small containers and when the product will be used in a short period of time. A manifold dryer will dry the product to less than 5% moisture content. Without heat, only primary drying (removal of the unbound water) can be achieved. A heater must be added for secondary drying, which will remove the bound water and will produce lower moisture content.

Tray style freeze-dryers are typically larger than the manifold dryers and are more sophisticated. Tray style freeze-dryers are used to dry a variety of materials. A tray freeze-dryer is used to produce the driest product for long-term storage. A tray freeze-dryer allows the product to be frozen in place and performs both primary (unbound water removal) and secondary (bound water removal) freeze-drying, thus producing the driest possible end-product. Tray freeze-dryers can dry products in bulk or in vials or other containers. When drying in vials, the freeze-dryer is supplied with a stoppering mechanism that allows a stopper to be pressed into place, sealing the vial before it is exposed to the atmosphere. This is used for long-term storage, such as vaccines.

Improved freeze drying techniques are being developed to extend the range of products that can be freeze dried, to improve the quality of the product, and to produce the product faster with less labour.

1.9 VACUUM-DRYING

For preservation and protection of heat-sensitive food components, *Vacuum-drying* technologies are commonly used. Some vegetables, as well as herbs and spices and citrus fruits are often dried by this technique. Typical batch (shelf) or continuous drying equipment is often used with some modifications to create and to maintain the vacuum created by one or more pumps connected through a moisture condenser. Vacuum-drying is like freeze-drying except that the product is not frozen and the vacuum is not as high as freeze-drying. Vacuum dryers have *four* essential components:

- A vacuum chamber / drying chamber (where drying takes place)
- Heater (supplies the heat)
- A vacuum pump (device for producing and maintaining the vacuum)
- Condenser / Moisture trap (components to collect water vapour evaporated from the food).

This equipment is a good example of conduction dryer. The vacuum oven consists of a jacketed vessel to withstand vacuum within the oven. There are supports for the shelves giving a larger area for heat transfer by conduction. The oven must be closed by a door to maintain the vacuum inside chamber and to prevent the heat transfer to

ambient temperature. The oven is connected through a condenser and liquid receiver to a vacuum pump. The operating pressure can be as low as 0.3-0.03 bar, at these pressures water boils at 25-35°C.

1.9.1 Advantages of vacuum-dryer

- Drying takes place at very low pressure and hence low temperature
- Maximum retention of nutrients
- There is little air present, so there is minimum risk of oxidation.

1.9.2 Limitations

- Sometimes burning onto trays in vacuum shelf dryers
- Shrinkage in food which reduces the contact between the food and heated surfaces of both type of equipment
- They have relatively high capital and operating costs and also low production rates.

In vacuum-dryers, heat is transferred to the material being dried through conduction and radiation. The drying cycle consists of (1) *the warming-up* phase (short or very short phase), alternatively called 'conditioning' of the food sample. Here opening of pores happen. This phase is usually short and not always observed in drying experiments. It is generally omitted in the calculation of drying time, (2) *the constant rate period* when the temperature of the material is essentially that of free water boiling under the particular vacuum. Truly this period may be observed when slowly drying wet sand or paper, (3) *a falling rate period*, during which the temperature of the material will approach the supplied heating temperature. Here, the rate of water transfer from the interior of the food particle to its surface decreases continuously as the product becomes drier. When the supply of water to the surface drops below the rate of evaporation, the moisture content of the surface begins to decrease rapidly and approaches quickly the equilibrium moisture content corresponding to relative humidity of the air on the sorption isotherm of the material. From that moment, internal transport and not the evaporation rate becomes the rate limiting factor and the falling rate period begins. This period is divided into first and second falling rate period. All the rates (constant rate, first falling rate and second falling rate) are shown in Figure 1.11.

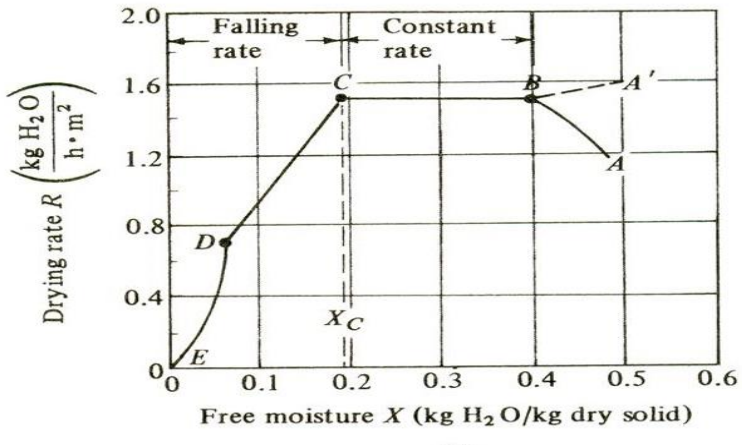


Figure 1.11: Typical drying-rate curve where phase of 'warming-up' (A or $A' \rightarrow B$), phase of 'constant rate' ($B \rightarrow C$), 'first falling rate' ($C \rightarrow D$) and 'second falling rate' ($D \rightarrow E$) are shown.

1.10 THE GENERAL PRINCIPLES OF EFFICIENT DRYING

- Large surface area for heat transfer
- Efficient heat transfer per unit area (to supply sufficient latent heat of vaporization or heat of sublimation for freeze-drying)
- Efficient mass transfer of evaporated water through any surrounding boundary layers, i.e. sufficient turbulence to minimize boundary layer thickness
- Efficient vapour removal i.e. low relative humidity air at adequate velocity
- It is convenient to categorize food dryers according to the heat transfer method used i.e. conductive, convective or radiated.

1.11 NEW DEVELOPMENT AND EMERGING DRYING TECHNOLOGY

New technologies are needed for

- Drying of new products and / or processes
- Higher capacities than current technology permits
- Better quality and quality control than currently established already
- Reduced environmental impact, use of renewable energy
- Lower energy requirement and better efficiency (for reduction of cost)

- Lower cost (operating, maintenance and capital cost)
- Shorter processing time while maintaining high product quality

1.12 FREEZE-DRYING OF KIWI FRUIT

The name of the kiwi fruit has come from a brown flightless bird *kiwi*, native of New Zealand. These mouth-watering fruit offers wide variety of health benefits as they are fully packed with health promoting nutrients such as phyto-chemicals, minerals and vitamins. It consistently ranks at the top of fruit in nutrition density models. The kiwi fruit is high in vitamin C, dietary fiber, potassium and magnesium; low in saturated fat, sodium and cholesterol. It has an oval shape, and is green on the inside with small black seeds that are edible. The kiwi has brown skin that is edible but is usually removed. The skin is relatively thin. The skin of the fruit can vary in size, shape, hairiness and colour. The flesh can also vary in colour, juiciness, texture and taste.

1.12.1 Introduction

The following scientific classifications are commonly represented for kiwi fruit:

Kingdom: Plantae

Division: Magnoliophyta

Class: Magnoliopsida

Order: Ericales

Family: Actinidiaceae

Species: Actinidia deliciosa

Binomial name: *Actinidia deliciosa*

The genus *Actinidia* contains around sixty (60) species. Though most kiwifruit are easily recognized as kiwifruit (due to basic shape) their fruit is quite variable.

Kiwifruit (genus *Actinidia*) is a fruit. The name of the kiwi fruit has come from a brown flightless bird *kiwi*, native of New Zealand. These mouth-watering fruit offers wide variety of health benefits as they are fully packed with health promoting nutrients such as phyto-chemicals, minerals and vitamins. Kiwifruit is the power

house of fruit. It consistently ranks at the top of fruit in nutrition density models. The kiwi fruit is high in vitamin C, dietary fiber, potassium and magnesium; low in saturated fat, sodium and cholesterol. It has an oval shape, and is green on the inside with small black seeds that are edible. The kiwi has brown skin that is edible but is usually removed. The skin is relatively thin. The skin of the fruit can vary in size, shape, hairiness and colour. The flesh can also vary in colour, juiciness, texture and taste. Some fruits are unpalatable while others taste considerably better than the majority of the commercial varieties.

There are different types of kiwifruit. The main types are: ------

i) Hayward
ii) Chico
iii) Saanichton 12
iv) Golden kiwifruit (A. chinensis)

1.12.2 Kiwi Fruit Plant

The most common among them is "Hayward". It is oval in shape, about the size of a large hen's egg, length 5-8 cm (2-3.1 inch), diameter 4.5 to 5.5 cm (1.8 to 2.2 inch). It has a fibrous, dull greenish-brown skin and bright green or golden flesh with rows of tiny, black, edible seeds. The fruit has a soft texture and a sweet but unique flavor, and today it is a commercial crop in several countries, such as New Zealand, Itally, Chile, Greece and France. In Figure 1.2, a full developed kiwi fruit plant is shown.

Figure 1.12: A well grown kiwi fruit plant.

The golden kiwifruit has a smooth bronze skin, with a beak shape at the stem attachment. Flesh colour varies from bright green to a clear, intense yellow. This species is sweeter and more aromatic in flavor.

Out of 100 %, over 70 % of kiwi production is in Itally, New Zealand and Chile. Itally produces roughly 10% more kiwifruit than New Zealand, and Chile produces 40 % less.

In the 1980s countries outside New Zealand began to export kiwifruit. In Italy the infrastructure and techniques required to support grape production have been adapted to the kiwifruit.

The first commercial planting of Chinese gooseberries occurred in 1937 in New Zealand by the orchardist Jim Mac Loughlin. The fruit proved popular with American serviceman in New Zealand during World War II. In year 1952 MacLoughlin partnered with the New Zealand Fruit Federation to market and export the fruit in the US market. As the local popularity of this fruit increased, New Zealanders discarded the old Chinese name for the fruit (*yang tao*) in favour of the name *Chinese Goosseberry*. It was used as a global brand for marketing the fuzzy kiwifruit, but was not registered internationally as a trademark. Kiwifruit was used as a global market as a global brand for marketing the fuzzy kiwifruit, but was not registered internationally as trademark. Kiwifruit has become a common name for all commercially grown fruit from the family Actinidia.

CLIMATE: It is classified as a subtropical plant. It will not tolerate winter temperatures much lower than 10°F (-12°C). In South-eastern North Carolina the vines can be expected to leaf in mid- to late-March and flower in mid-May. Fruit should be sufficiently mature for harvest in late October or early November. Temperature lower than 29°F (-1.67°C) between leafing and harvest can damage the leaves, blossoms and fruit. If new growth is damaged in the spring before blossoms develop, no blossoming will occur. Kiwifruit plantings can be handled much like grapes, however, they are much more susceptible to wind damage (therefore, plants are spaced 16-20 inches apart around the planting and perpendicular to the prevailing wind between every 4 to 6 kiwifruit rows) and root-knot nematodes, and require more

supplement irrigation than grapes. Many soil types are suitable, provided they are well drained. The soil pH should be adjusted to 6.0 to 6.5 and controlled before planting.

Dormant plants from a nursery can be planted in the spring after there is little chance of freezing weather. Plant to the same depth as the plants grew in the nursery. After planting, prune the plant back to a single, healthy shoot 6 to 12 inches long. The more space between rows should be allowed. On a single wire the spacing should be 10-12 ft and with the double wire, 15-16 ft.

The kiwifruit vine grows naturally at altitudes between 2,000 and 6,500 ft (600 to 2,000 m). The climate is related to heavy rainfall and an abundance of snow and ice in winter. The region where the winter mean minimum daily temperature is 40°F (4.44°C) to 42°F (5.56°C); mean maximum 57°F (13.9°C) to 60°F (15.5°C). In summer, mean minimum is 56°F (13.33°C) to 57°F (13.9°C); mean maximum is 75°F (23.9°C) to 77°F (25°C). Annual rainfall is 51 to 64 inch (130 to 163 cm) and relative humidity is 76 % to 78%. Kiwifruit vines in leaf are killed by drops in temperature below 29°F (-1.67°C).

SOIL: For good growth, the vine needs deep, fertile, moist but well-drained soil, preferably a friable, sandy loam. Heavy soils subject to water logging are completely unsuitable. The mature plants require a minimum of 150 lbs nitrogen per acre (about 150 kg / hac). In New Zealand, they are usually fertilized twice a year, once in spring and once in early summer, using a total of 500 lbs (225 kg) nitrogen, 220 lbs (100 kg) P_2O_5, 121 lbs (55 kg) K_2O per hectare. Apart from land cost, it takes a minimum of $3,500 to bring each acre in production.

HARVESTING: In California, vines generally provide their first commercial crop in the fourth season. Full production is reached within 8 to 12 years (Soyergin, 2016).

Fruit will soften off the vine if harvested after the sugar content reaches to 4%, but full flavor does not develop until the sugar content reaches 6 to 8% on the vine. Starch in the fruit is converted to sugar following harvest. When the fruit is ready to eat, it should contain 12 to 15% sugar.

Fruits are harvested by snapping the stem at the abscission layer at the base of the fruit. Commercially, kiwifruit vineyards are harvested all at one time, but in the home

garden the largest fruit can be removed first and the smaller fruit allowed to develop more size.

YIELD: In California, 4-year old vines have yielded 14,000 lbs per acre. Vines 8 years old have yielded 18,000 lbs per acre, which is nearly the maximum for mature plants (8 to 10 years old).

KEEPING QUALITY: At room temperature, 65°F to 70°F (18.3°C to -21.1°C), firm fruits can be kept for 8 weeks. Fully ripe fruits can be kept for a week or more in the home refrigerator. Fruits are harvested at the firm stage will keep for long periods at 31°F to 32°F (-0.6° to 0°C) and at least 90% relative humidity, wrapped in unsealed polyethylene in containers. Lower relative humidity (even 85%) will cause a weight loss of as much as 4.5% in 6 weeks. Fruits that are cooled to a temperature of 32°F (0°C) within 12 hours after harvesting, will keep in good condition for as long as 6 months under commercial refrigeration. Experiments have shown an atmosphere modified with 10 to 14% CO_2 will increase cold storage life by 2 months, providing the fruits enter storage within a week after harvest and are removed from the controlled atmosphere shortly in advance of marketing. The optimum storage atmosphere may be obtained with 5% CO_2 and 2% O_2, with C_2H_4 excluded and / or removed to keep it below 0.05 micro gram per liter. Kiwifruits may be stored at 28°F to 30° F (-1.8° C to -2.1 °C).

USES: Raw kiwifruit is rich in the protein dissolving enzyme actinidin (in the same family of thiol proteases as papain), which is commercially useful as a meat tenderizer. Actinidin also makes raw kiwifruit unsuitable for use in desserts containing milk or any other dairy products which are not going to be served within hours, because the enzyme soon begins to digest milk proteins. This applies to gelatin based desserts as well, as the actinidin will dissolve the collagen proteins in gelatin very quickly, either liquefying the dessert, or preventing it from solidifying.

However, U.S. Department of Agriculture suggests cooking the fruit for a few minutes before adding it to the gelatin to overcome this effect. Sliced kiwifruit has long been regularly used as a garnish atop whipped cream on New Zealand's national dessert, the *Pavlova*. It can also be used in a variety of other savory and sweet dishes.

For consumption fresh or from processing, kiwifruits are customarily kept refrigerated for at least 2 weeks to induce softening and then allowed to further soften at room temperature to improve flavor. The fruits will ripen too rapidly and loose quality if stored with other fruits, such as apples, pears, peaches, plums etc., because these fruits emit ethylene. Slightly under ripe fruits, which are high in pectin, must be chosen for making jelly, jam and chutney. The Kiwifruit Wine Company Ltd of New Zealand & Gibson wine company in California is making kiwifruit wine with 11 to 11.5 % alcohol content.

In the home kitchen, meat can be tenderized by placing slices of kiwifruit over it or by rubbing the meat with flesh. After 10 minutes, the fruit must be lifted or scraped off; otherwise the enzymatic action will be excessive. The meat should be cooked immediately.

The kiwi fruit (Figure 1.3) has a good medicinal use. The branches and leaves are boiled in water and the liquid used for treating mange (a skin disease) in dogs. In China, the fruits and juices of the stalk are esteemed for expelling "gravel". The scraped stems of the vine are used as rope in China, and paper has been made from the leaves and bark. If the bark at the base of the vine, close to the roots, is removed in one piece and placed in hot ashes, it will roll into a firm tube which can be used as a pencil.

1.12.3 Health Benefits of Kiwi Fruit

- PROTECT FROM AGE-RELATED MACULAR DEGENERATION (ARMD)

  An anti-oxidant fruit, kiwi fruit has most typical anti-oxidant vitamins C, A and E in high amount. Few studies published by the Archives of Ophthalmology proved that eating 3 to 5 serving of fruit in a day can decrease the risk of developing an eye-related disease named ARMD up to 36%. In comparison to people who only eat 1.5 servings of fruit per day, ARMD is the prime cause of vision impairment in adults.

- HELPS IN WEIGHT LOSS

  For low content fat and high content fiber it helps to lose weight loss.

- ACTS AS AN ANTI-AGING

The vitamins A, C and E content of the kiwi fruit helps to fight against the damage caused by the free radicals in our body. Free radicals have demolishing effect on the cells and are responsible for early aging signs as well as various illness: such as cardiovascular disease and cancer.

Figure 1.13: Kiwi fruits.

- LOWERS CHOLESTEROL

Dietary fibers of the kiwi fruit have been proved to reduce the cholesterol level in the body. By lowering the cholesterol levels, it also helps to reduce the risk of heart attack. Adequate sodium to potassium ratio is required for healthy heart, which is excellent in kiwifruit.

- AIDS IN DIABETICS

Kiwi fruits are rich source of fibers. The fibers have good digestive property and also help to improve diabetes. Kiwi also contains inositol, a natural occurring sugar alcohol that may have a positive effect to regulate diabetes. Inositol also helps in nerve conduction velocity, a symptom of diabetic neuropathy.

- RESPIRATORY HEALTH

Like all other vitamin C rich foods, kiwi also promotes the respiratory tract health. Some studies have shown that eating of vitamin C rich foods like kiwifruit, even in small amount, may decrease respiratory related health problems including shortness of breath, high coughing etc.

1.12.4 Other Health Benefits of Kiwi Fruit

Kiwi fruit has about the same levels of potassium as bananas but only half the calories: making kiwifruit an excellent low-sodium, high potassium fruit which can be beneficial in the maintenance of blood pressure and for heart health.

- Consumption of kiwi fruits make metabolism stronger as well as improves the nerve function.
- Prevents wheezing and coughing, particularly in children
- Helps to prevent colon cancer
- Boosts the immune system of the body so helps to fight cold and flu
- Provides sufficient amount of vitamins and antioxidants
- Prevents asthma
- Very rich source of minerals such as magnesium, potassium and copper
- It acts as a natural blood thinner
- Assists people who are suffering from depression
- Great for pregnant woman as it contains folate which is necessary for cell development
- Kiwifruit is a natural source of folic acid which is needed to prevent neural defects in babies.
- An anti-mutagenic component is present in kiwifruit which helps to prevent the mutations of DNA that may start the cancer process

1.12.5 Allergies

The actinidin found in kiwifruit can be an allergen for some individuals. Specifically, people allergic to latex, bananas, papayas or pineapples are likely to also be allergic to kiwifruit. The fruit also contains calcium oxalate crystals in the form of raphides. Reactions to these chemicals include sweating, tingling and sore mouth or throat, swelling of the lips, tongue and face, rash, vomiting and abdominal pain, heartburn

and in the most severe cases breathing difficulties, wheezing and collapse. The most symptoms are unpleasant itching and soreness of the mouth, with the most severe symptom being wheezing. Severe symptoms are most likely to occur in young children.

1.12.6 Storage and Ripening

The mature fruit can be stored for 4 to 6 months at -0.5 to 0°C if protected from dehydration. Storage life is substantially reduced if ethylene producing fruits such as apples or pears are present in storage. For maximum storage life, store kiwifruit alone. Fruit will ripen at room temperature when removed from cold storage. Ripening can be hastened by exposure to ethylene. This hastened ripening can be accomplished in the home by placing kiwifruit in a plastic bag with an apple.

1.13 VACUUM-DRYING OF SHRIMP AND FISHES

1.13.1 Introduction

In worldwide, fish is very important source of good quality protein and lipid contents. Fish contain most of the essential amino acids, among them lysine (found most cereals, millets, root crops less) is very significant (Sen, 2005). It contains good lipids (ω-3 decosahexaenoic acid), fat soluble vitamins (A, D and E). Fish contains good lipids such as triglycerides, phospholipids, cholesterol and cholesteryl esters. Besides, fat soluble vitamins (A, D and E) are present in either small amounts (vitamin A 22–132 µg, vitamin D 0.055–4 µg and vitamin E 0.11–1.6 mg) or large amounts (eg. cod, shark). It contains many fatty acids such as, SFA (saturated fatty acids), MUFA (monounsaturated fatty acids), PUFA (polyunsaturated fatty acids), EPA (ω-3 eicosapentaenoic acid), DHA (ω-3 decosahexaenoic acid) and ω-6 arachidonic acid.The presence of TMAO (trimethyl amine oxide), high content of NPN (non-protein nitrogen), variable lipid content with high degree of unsaturation and high amount of ω-3 fatty acids, low carbohydrate content with high pH (= 6), termed as "low-acid food" (pH≥4.5) are note-worthy. The combined total amount of ammonia, TMA (tri-methyl amine) and DMA (di-methyl amine) is called total volatile base nitrogen (TVBN).

1.13.2 Fish: Health Benefits

Fish are sources of both water and fat soluble vitamins. Among the water soluble vitamins, the B-complex vitamins namely thiamine (B_1), riboflavin (B_2), B_{12} are present in muscle of fish. Also, vitamin C is present in fish flesh. Among the fat soluble vitamins, vitamin A, vitamin D and vitamin E are present in body / flesh oil. It was found that presence of vitamin A is common for all types of fishes. Fish contains several important pigments such as astaxanthin, astacin, chlorophyll, haemoglobin, myoglobin and hemocyanin. Both astaxanthin and astacin are sufficiently present in prawn and apparent colour developed during processing of prawn is due to these two pigments. The only non-carotenoid natural pigment present in fish oil is chlorophyll. The colour of flesh of a fish is due to its myoglobin pigment, whereas haemoglobin acts as an oxygen carrier in blood stream. A copper-protein complex termed hemocyanin is found in many invertebrates (such as prawn, shrimp, crab etc.). The presence of copper has also a strong pro-oxidant effect.

Fish is store house of microelements such as Fe, Cu, Zn, Mn, Ca, Se, Co, P, I, Cr (III), F, Mo etcetera. Iron (Fe) is an essential constituent of haemoglobin and myoglobin. Copper (Cu) prevents anemia. Zinc (Zn) is to be required for the structure of carbonic anhydrase. Manganese (Mn) involved to activate several enzymes in human body. Selenium (Se) is known to be participated several important metabolic interactions with a variety of hazardous elements such as mercury (Hg), cadmium (Cd) and arsenic (As) which is very important in public health (Committee on Diet and Health, National Academy Press, Washington DC, USA, p. 375, 1989). Iodine (I) is required for the biosynthesis of thyroid hormones. Chromium (Cr) is required for maintenance of normal lipid, cholesterol, insulin and glucose metabolism (Guideline Document for Chromium in shellfish. Centre for Food Safety and Applied Nutrition, United States Food and Drug Administration, Washington DC, 20204 USA, Aug 1993). A small amount of molybdenum (Mo) is sufficient in the human body to satisfy the needs of many metallo-enzymes. As per the recent surveyed by UNICEF (United Nations International Children's Emergency Fund) in year 2015, it was found that millions of children are suffer from stunted growth, cognitive delays, weakened immunity and disease as a result of micronutrient deficiencies. For pregnant women, the lack of essential vitamins and minerals can be catastrophic, increasing the risk of low birth

weight, birth defects and even death. They found that Fe, Zn, Ca, I, vitamin A, and vitamin D are major micronutrient deficiency among the children and women. According to them, the general strategies are to be taken: Dietary diversification, Supplementation programs, Mass and home fortifications. In mass and home fortification, the micronutrients are added to the foods during food processing or food powders containing micronutrients can be sprinkled on the cooked foods just before serving. This can significantly improve the dietary quality of complementary foods for children, women and as well as older people.

Coronary heart disease (CHD) is a major killer in both developed and developing countries. CHD is more dreadful due to its sudden attack and debility. In Japan low death rate from CHD is found since the consumption of fish (rich in ω-3 PUFA) is estimated to about 100 g/day (Kromhout *et al.*, 1985). Hence, to reduce the risk of coronary disease it was recommended intake of 2 to 3 servings per week (Sen, 2005). Such characteristic features are present in fish make it distinct and separate from other animal food stuffs.

Figure 1.14 (a): Shrimp (*Metapenaeus monoceros*).

Figure 1.14 (b): Catla (*Catla catla*), CA.

Figure 1.14 (c): Chela (*Chela cachius*), CS.

The shrimp [Figure 1.14 (a)], two fishes viz. Catla [Figure 1.14 (b)] and chela [Figure 1.14 (c)] were selected on the basis of their nutrient values. Among them, Catla (*Catla catla*) [CA] has moderate iodine (18 µg/100g), sodium (74 mg/100g) and sulphur (170mg/100g) (Jessica *et al.*, 2015). But CA has a deficiency of vitamins (A, D, E and B_{12}), minerals (Fe, Zn, Ca, Se, P, Mg, Mn, Cu). It supplies less energy (267 kJ) per 100 g fish consumption. It is readily available and farming in freshwater pond. It has a huge demand in the Asian market and contributing much to the total fish production and economic growth of some Asian countries, such as Bangladesh and India. Chela (*Chela cachius*) [CS] fish has high energy value (349 kJ per 100 g). Both protein (15.2 g / 100 g) and fat (2.4 g / 100 g) content is less as compared with CA. CS has moderate iodine, Se, Mg. It has deficiency of Fe (like CA), Na, K, Cu (like CA), vitamin E (Vanitha, 2011). Shrimp (*Metapenaeus monoceros*) also contains many

important pigments (such as astaxanthin and astacin), vitamin E (1.6 mg/100g) [Jessica et al., 2015]. Hence, overall concluded that CA is readily available; vitamin, essential minerals and energy deficient, fat and protein rich food stuff, CS is energy rich, costly and rich particularly in Zn, Ca, Se, P, Mn, vitamin B_{12}, A and D. Shrimp has pigments, energy rich food stuffs, costly and rich in Fe, I, Se, Mg, Na, K, Mn, S and Cu and deficient in vitamin A, D.

1.14 FREEZE-DRYING OF BROCCOLLI

Broccoli (Figure 1.15) is a cool-season crop that, like spinach, can be grown in the spring or fall. It is an edible green plant in the cabbage family (closely related to cauliflower), whose large flower head is used as a vegetable. The word "Broccoli" came from the Italian plural *broccolo* refers to the flowering top of a cabbage. The botanical name of broccoli is *Brassica oleracia*. It can be stored in the refrigerator (after cleaning and surface drying) for up to 7 days. It is spoiled within 3-4 days if it is stored at room temperature [30°C] (Figure 1.15).

Figure 1.15: Fresh and Spoiled Broccoli.

1.14.1 Health Benefits of Broccoli

It has many health benefits so that it is a unique vegetable as compare with other vegetables. The following points are important to choose broccoli in research work for freeze-drying.

- It is rich in vitamin C and dietary fiber.
- It also contains B-vitamins (B_1, B_2, B_3, B_5, B_6, and B_9). It contains vitamin A, vitamin E and vitamin K. That is why it is act as an *antioxidant*rich vegetable.
- Broccoli contains no vitamin D. But it may help us to solve our vitamin D deficiency epidemic. When large supplemental doses of vitamin D are needed to offset deficiency, ample supplies of vitamin A and K help keep our vitamin metabolism in balance. It has an unusually strong combination of both vitamin A (in the form of β-carotene) and vitamin K. Broccoli may be an ideal food to include in our diet.
- The energy supply to our body is 144 kJ per 100 g of broccoli consumption. The sugar percentage is low (1 to 2%). Moisture content is 85 to 88%.
- It contains many minerals such as Fe, Ca, Cu, Mg, Mn, P, Se, K and Zn. It is low sodium (Na) and rich potassium (K) diet vegetable. Particularly, selenium (Se) has anticancer property.
- It is protein rich and poor fat diet vegetable. The cholesterol level is also found low.
- Broccoli has a strong, positive impact on our body's detoxification system. Researchers have recently identified one of the key benefits for detoxification. Glucoraphanin, Gluconasturtiian and Glucobrassicin are the three *glucosinolate* phyto-nutrients found in a special combination in broccoli. This dynamic trio is able to support all steps in body's detoxification process including activation, neutralization and elimination of unwanted contaminants. *Isothiocyanates* (ITCs) are the detoxification regulating molecules made from broccoli's *glucosinolates* and help to control the whole detoxification process at a genetic level.

- Broccoli is a particularly rich source of a flavonoid called *Kaempferol*. Recent research has shown the ability of *kaempferol* to less the impact of allergy related substance on our body.
- According to recent research studies, it is found that there are ***three*** points which are responsible for the development of cancer. They are: (i) Chronic inflammation, (ii) Oxidative stress and (iii) Inadequate detoxification. About 300 research studies revealed that broccoli has strong impact on anti-carcinogenic effect. Few antioxidant nutrients such as vitamin C, few flavonoids such as kaempferol, quercitin and few carotenoids such as lutein, Zea-Xanthin & β-carotene are enhancing the anti-carcinogenic effect. All three of these carotenoids function as key antioxidants.
- Considered as a group, vitamins, minerals, flavonoids and carotenoids contained in broccoli work to lower risk of oxidative stress in our body system. The ability of these nutrients to support oxygen metabolism and avoid excess formation of reactive oxygen (free radicals) to minimize the chance of chronic inflammation and hence cancer.
- In case of broccoli, the research shows that it has strong decrease risk of prostate cancer, breast cancer, bladder cancer and ovarian cancer. Recent studies provided us the limit of consumption of broccoli. It is half cup (45 g) per day or two cups (180 g) per week.
- Digestive support is also provided by broccoli since it contains fiber and *iso-thiocyanate* (ITC).
- The consumption of broccoli is to reduce the heart disease. Though the research on this topic is in the early stages, but few compounds such as *glucoraphanin* (present in broccoli) is responsible to reduce the heart disease. The lower level of cholesterol helps also to reduce the cardiovascular disease. The vitamin-B (B_6, B_{12}) is help to lower our risk of *hyper-homocysteinemia* (excessive formation of homocysteine) and hence reduce the cardiovascular diseases.
- Other health benefits are also provided by broccoli. The first point is our eye health. Few *carotenoids* (lutein and Zea-Xanthin) are playing an important role for maintaining our eye health.

- The second point is our skin support, including support of sun damaged skin. *Glucoraphanin* found in broccoli converted into sulforaphane by the body. Since skin cells can carry out the process of detoxification, it may be detoxification related benefits of sulforaphane that are important in helping to counterattack with sun damage.
- Broccoli is also an excellent source of *indole-3-carbinol*, a chemical which boosts DNA repair in cells and appears to block the growth of cancer cells.

1.14.2 Working Principle

Freeze-drying (lyophilization) is a widely used method for dehydrating a wide range of materials, including foodstuffs, pharmaceuticals, biotechnology products, vaccines, diagnostics and biological materials. Freeze-drying (FD) may be carried out on a range of scales, from bench top through pilot-scale to a full-scale manufacturing process and offers a number of advantages over conventional drying and many other processing methods, but its high energy cost (more drying time) did not gain popularity. But the sample's pre-processing, hence the reduction of moisture content with retention of nutrients and / or improvement of nutrients by fortification technique before freeze-drying may be alternate solution of high energy requirement.

Chakraborty *et al.* (2016) performed osmotic dehydration (OD) for kiwi sample to reduce the moisture from 85% to 4% with drying time 2.5 h by using silver coated steel heater during freeze-drying. Chakraborty and Mondal (2017) done OD with successive vacuum-drying of watermelon followed by far-infrared radiation-assisted vacuum drying to reduce moisture from 90% to less than 5%. So, pre-processing is to be accepted before freeze-drying of foodstuffs.

Blanching is a cooking process wherein the food substance, usually a vegetable or fruit, is plunged into boiling water, removed after a brief, timed interval and finally put into iced water or placed under cold running water (shocked) to halt the cooking process. Blanching is done because it

- Slows or retards enzymatic action, preserving flavour, colour and texture
- cleanses the surface of dirt, bacteria, moulds and other organisms
- brightens the colour of the product

- helps to minimize loss of vitamins
- Softens vegetables and makes them easier to pack and less susceptible to freezer burn.

It should be remembered that blanching time is crucial and varies with the vegetable and size. Under-blanching actually stimulates the activity of enzymes and is worse than no blanching. Over-blanching causes loss of flavour, colour, vitamins and minerals. Blanching applies to freezing and many drying of recipes, but not applies to canning.

1.15 THE ORGANIZATION OF THESIS

Drying is very ancient technology of food preservation. Freeze-drying methods have been used for *over 100 years* for various technical purposes. The ancient Incas of Peru used mountain peaks along the Andes as natural food preservers. The extremely cold temperature and low pressure at those high altitudes prevented food from spoiling in the same basic way as a modern freeze-drying machine. During *World War-II*, equipment and techniques were developed to supply blood plasma and penicillin to the armed forces. Over 400 different types of freeze-dried foods have been commercially produced *since the 1960s*. Two bad candidates for freeze-drying are lettuce and watermelon because they have too high water content and they are freeze-dried poorly.

In Chapter-2, Literature review and Objectives are included.

Aims and Objectives:

The present study focused towards **reducing drying time** to enhance process economy *by pretreatment* of raw food stuffs along with developing *effective heat transfer mechanism* for moisture removal. It is concluded that *reduction of drying time by a fraction could reduce the process cost* drastically keeping the *maximum retention of Quality* of the raw food stuffs.

- Different pretreatment steps, namely **osmotic dehydration** (OD), **fortification with blending** and **blanching** of raw food stuffs and finally qualities of finished products.
- To study the effects of silver coated steel plate (SCSP) & low wattage steel cubic heater as heating surface for conventional freeze-drying (FD) & vacuum drying (VD) and compare their performances with normal steel plate or copper plate. Drying kinetics, time, effective diffusivity and activation energy were investigated both for FD and VD.
- To compare the *Quality parameters* of the FD and VD food products with raw foodstuffs using standard testing protocol. The 9-point hedonic scale was applied for *Sensory analysis.*

In Chapter-3, Materials and Methods are included.

Materials required and Methods applied:

- **Four** experiments were performed viz. Experiment set-1: For osmotic dehydrated (optimized) & freeze-dried (called OPODFD) kiwi fruit preparation; Experiment set-2: For value added Kiwi fruit (called BFDKF) powder preparation; Experiment set-3: For Fortified Shrimp (called VDFS) powder preparation, and Experiment set-4: For Blanched & Freeze-dried Broccoli.
- Kiwi fruit, Shrimp, Catla, Chela fishes and broccoli were selected as basic raw materials due to their many health benefits. The different ingredients viz. rice flour, oat flour, corn flour, dried ginger powder, iodized salt, lona salt, ground sugar, calcium lactate and sodium benzoate (as a preservative) were added / fortified to improve the final nutritional quality, flavor, taste and shelf life increment.
- The freeze-dryer / vacuum-dryer were used for drying purpose. For sublimation heat treatment SCSH (silver coated steel heater), SCSRP (silver coated steel rounded plate) heaters were used during FD and SPMDH (silver plated multi-dimensional heater) was used for VD heat supply during drying.
- The successive osmotic dehydration (OD) and FD was done for Experiment set-1. The sublimation heat was supplied by self-designed SCSH. OD was optimized (called OPOD) by Taguchi orthogonal design matrix. The final product's proximate analysis was investigated. Both OD step and ODFD step the drying kinetics were studied by *four* different drying models (namely Page, Wang & Sing, Henderson & Pabis and Newton model). The effective diffusivity and activation energy during drying was calculated by Arrhenius equation for better drying kinetics.
- The value added kiwi fruit (BFDKF) powder was prepared for Experiment set-2. The short time addition and blending methodology was applied for fruit paste pulp preparation. The blended kiwi fruit (BKF) was freeze-dried using SCSRP and self-designed cubic heater. Final fruit powder (BFDKF) was tested by proximate and sensory analysis (hedonic scale).

- The fortified (by catla and chela fishes) shrimp was prepared in Experiment set-3 to meet the deficiency of nutrients (protein, minerals and vitamins) present in shrimp. Shrimp was further enriched by some raw materials. The blended material was vacuum-dried by using SPMDH and insulated cubic heater. The final two set of shrimp-fish powder products (called VDFS-1 & VDFS-2) were quality tested, along with final minerals enrichment and TVBN, histamine, Hg content were checked and compared with raw shrimp.
- The broccoli was cold blanched and freeze-dried in Experiment set-4. The final product was checked for its nutritional retention.

In Chapter-4, Results and Discussions are briefly included.

Results

Experiment set-1

- Successful OPOD technique for KF sample [which drastically cut down FD time, since moisture drop from 85.07% to 68.16%]
- At optimized OD conditions (40^0B, 50 ^{0} C and 3 h), Sucrose solution (WL= 60.75%, SG = 9.86%) is better osmotic solution than Fructose solution (WL= 54.54%, SG = 14.45%) [WL = water loss, SG = solute gain].
- High thermal conductivity material (SCSH) applied for conductive heating cut down further FD time [moisture drop from 68.16% to 4.27%]
- Drying time: using SCSH was 2.5 h, using Steel heater it was 3.82 h and for conventional freeze-drying (CFD) it was 5.75 h.
- Nutritional quality almost retained in final KF by OPODFD proce**ss.**

Experiment set-2

- Successful value addition technique for KF sample [which drastically cut down FD time, since moisture drop from 85.07% to 68%]
- High thermal conductivity material (SCSRP) applied for conductive heating cut down further FD time [moisture drop from 68% to 3%]
- Nutritional quality almost retained in final BFDKF powder.
- Vitamin C retention here is more as compared with OPODFD process [raw KF 89.10 mg/100g to 78.66 mg/100g]; here from 89.10 to 193.9 mg/100g.

- Protein, fat, Total ash and K portions were increased and Sugar/ carbohydrates, Sodium, Energy were decreased as compared with OPODFD.

Experiment set-3

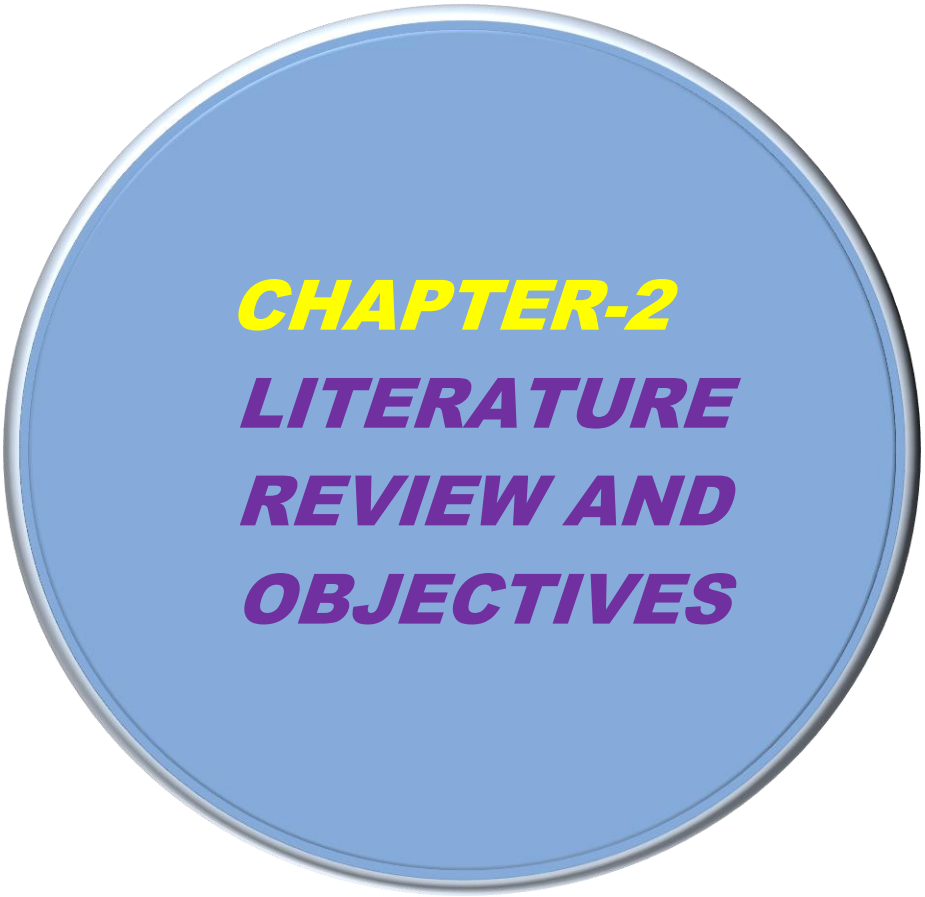
- Shrimp (*Metapenaeus monoceros*) is deficient in fat, essential minerals and vitamins. It contains high moisture (77.15 ± 0.73%), preserved by an energy-efficient process.
- Fortification protocols for enrichment of shrimp were developed employing two fishes viz. *Catla catla* (CA) and *Chela cachius* (CS).
- Pre-blended shrimps were converted to fortified shrimp (FS) by adding CF, RF (FS-1) and additionally mixing with DG (FS-2).
- Dehydrated by VD using silver/copper plated multidimensional heater to produce vacuum-dried FS (VDFS).
- Two heaters (SPMDH & CPMDH) were used for VD of FS-1 & 2.
- Drying time: SPMDH (2.55h for FS-1 and 3.25 h for FS-2). CPMDH (2.85 h for FS-1 and 3.76 h for FS-2).
- The VDFS-1 exhibited superior quality through remarkable increase in protein (188%), ω-3 fatty acids (20%), carbohydrate (35%), ash (151%) and other essential elements with acceptable water activity, rehydration ratio, TVBN, and histamine content.

Experiment set-4

- Broccoli can be spoiled within 3-4 days if it is stored at room temperature (30^0C). Therefore, it must be preserved since it is highly nutritious vegetable.
- Cold blanching technique is applied prior to FD.

The FD time was 5.5 h (using SCSH); compared with non-blanched broccoli the FD (called as CFD) time was 6.5 h.

In Chapter-5, Conclusions of work and Scope of Future work are included.


CHAPTER-2
LITERATURE
REVIEW AND
OBJECTIVES

2. 1 INTRODUCTION

2.2 KIWI PROCESSING BY OSMOTIC DEHYDRATION AND SUCCESSIVE FREEZE-DRYING

It has been already established that freeze-drying (FD) is the best drying technology for food preservation regarding flavor & nutritional parameters, reduced weight of shipping, storage, and handling and enhance shelf life of the end product(s) (Berk Z, 2009; Brennan *et al.*, 1976). FD is applied for (i) high nutritional valued foods, (ii) with initial moisture content of 70% to 80% (w/w), (iii) costly foodstuffs (Marques and Freire, 2005) such as Kiwi fruit (KF).

KF possesses health promoting nutrients such as phyto-chemicals, minerals and provides sufficient amount of vitamins and antioxidants. Besides KF is high in dietary fiber, K and Mg; low in saturated fat, Na and cholesterol (Morton, 1987; Mainland, 2006). A study found (Bali, 2010) that KF is the most enriched fruit in terms of vitamin C content compared to papaya, mango and orange. It is important for cardiovascular health. It helps in reducing weight, cholesterol level and acts as an anti-aging substance with positive effect to regulate diabetics and colon cancer.

Conventional freeze-drying (CFD) (Holdsworth, 1971) was applied for kiwifruit. The results indicated that its betterment was necessary for economization of freeze-drying. In order to intensify the freeze-drying rate, infrared radiation was applied as a source of thermal energy for prawn (Chakraborty *et al.*, 2011) and banana (Chakraborty *et al.*, 2013); however, occurrence of protein denaturation was observed. More recently, osmotic dehydration (OD) was reported to be a pre-processing before FD for reduction of FD time with improved process economy (Yadav *et al.*, 2012; Zeuthen *et al.*, 2005).

However, to the best of our knowledge no report is available on application of OD prior to FD. Besides application of fast conductive heating (Hottot *et al.*, 2009; Elia and Barresi, 1998; Boss *et al.*, 2004) using high conductivity metal (such as silver) has not be reported for reduction of FD times. Notably, silver has a high thermal conductivity and antimicrobial efficacy.

2.3 PRODUCTION OF VALUE ADDED KIWI POWDER

Kiwi fruit (*Actinidia deliciosa*) is known for its good health-benefit properties viz. low calorific value, low glycemic index (GI), more vitamins (particularly vitamin C), maximum load of potassium, high dietary fiber, presence of variety of pigments (such as chlorophylls, carotenoids, lutein and anthocyanin), few important enzymes (such as proteases), low allergic response (AR) and finally it is rich in natural antioxidants, polyphenols (Singletary, 2012). Its tiny seeds which are present in distributed form throughout the whole pulp of the fruit are also known to be very important for consumption (Singletary, 2012; Parameswaran and Murthi, 2014). So, it is better to process the fruit without separating the seeds from it.

KF is a high moisture content (>80%) fruit and classified it as highly perishable commodities (Orsat *et al.,* 2006; Chin *et al*., 2015). It is now a challenging task to keep the product fresh since it loses its freshness gradually after some days. To maintain the nutritional value of fruit intact, most preservation / storage techniques require low temperatures which are very difficult to maintain throughout the supply chain distribution system (Sagar and Suresh, 2010). Moreover, during low temperature preservation vitamin C content of the fruit decreases with time (Tavarini *et al.,* 2008). These ill effects could be overcome by using advanced drying techniques like freeze-drying (Bezyma and Kutovoy, 2006). Freeze-drying is a process at sub-zero temperature where ice sublimes to reduce water activity. It leads to decrease in the rates of all the degradative process such as non-enzymatic browning, protein deterioration and enzymatic reaction (Salunkhe *et al.,* 1991). But the cost involved in it is prohibitively high due to long drying time. To reduce the drying time and preservation cost, a new drying methodology, successive osmotic dehydration and freeze-drying (ODFD) was investigated and reported earlier (Chakraborty *et al.,* 2016).

Freeze-drying (FD) can be applied for both solid and liquid form of products. The high valued product, such as instant coffee, is a classic example of FD from liquid forms (Burmester *et al*., 2011). Modern food processing improves the quality of life and keeps humans away from diabetes, heart disease, cancers and the likes. Food additives are substances those improve the quality, nutritive value, processing parameters, shelf life and maintaining the palatability, wholesomeness and product

consistency (fssai). Therefore, addition or fortification of KF pulp with proper additives could improve cost effectiveness as well as nutritional values of the product. Few directly or indirectly food additives may be selected for KF processing. Among the major food additives, rice flour and oat flour can be chosen. Rice flour contains complex carbohydrates, no gluten (that favors non-stickiness of the final product), almost no protein and fats. It acts as a thickening agent, soft texture improver and flavor enhancer (Duncan, 2000). Whereas oat flour complement the deficiencies by providing high energy (380 kcal per 100 g), high protein (12 g per 100 g) and high dietary fiber (10 g per 100 g) values. Dietary fiber consists of cellulose, hemi-cellulose, lignin and peptic substances. They play an important role in relieving constipation by increasing water holding capacity. Its consumption is also linked to decrease incidence of cardiovascular disease, diverticulitis and colon cancer (Barrett *et al.,* 2005; Dhingra *et al*., 2012). Among the minor food additives grounded sugar, salt (blends of iodized and lona in 1:1 ratio), calcium lactate and very trace amount of sodium benzoate are used. Already sugar and salt are succeeded as preservative and taste enhancer of the final product. In fact sugar is used to prevent browning of peeled and sliced fruit. It acts in inhibiting oxidation by partially excluding air in the tissues. In KF, dry sugar powder is mixed with pulp. Since, KF is rich in ascorbic acid, citric acid, therefore addition of sugar to KF as an effective agent against loss of texture, color and flavor (Barrett *et al.,* 2005). Lona salt is used since it is low in sodium and rich in potassium (Na 23%, K 38%). The most common treatment used to improve texture retention of any fruit by addition of calcium lactate solution (maximum 1%) (Barrett *et al.,* 2012; Martin-diana *et al.,* 2007). However, calcium lactate-treated samples tend to maintain higher firmness during storage (Benitez *et al*., 2014). It serves as a firming salt for fruits and act as a gelling agent particularly for KF like fruits. Sodium benzoate is a chemical preservative, which can play a very important role in fruit processing. It is most effective in the pH range of 2.5 to 4 by addition (Barrett *et al.,* 2005). KF contains mainly citric, malic and quinic acids. During ripening, the pH value of a KF varies from 3.1 to 3.96. Therefore, it is classified as a 'high acid' food. Hence, sodium benzoate is to be added in KF. According to IOMC (Inter-Organization Programme for the Sound Management of Chemicals), 0.01% (solution of water as a solvent, w/w) sodium benzoate (EU No. 211) maximum can be added with fruit items. Though sodium benzoate is less effective against bacteria but it is more active against yeasts and molds. Since the maximum dose recommended by

IOMC is only 0.01%, so its activity will be poor above pH 4 where bacteria are the greatest problem (Barrett *et al.,* 2005).

2.4 FORTIFICATION OF SHRIMP

2.4.1 Selection of Shrimp

Worldwide shrimp (*Metapenaeus monoceros*) is considered a tasty and healthy foodstuff. Though it is costly, it is a rich source of protein, energy and elements viz. Fe, I, Se, Mg, Na, Mn, Cu, S. Shrimp is costly, protein (17.6 g / 100 g), energy rich (333 kJ/100g) and also it is rich in Fe (2.7 mg / 100g), Ca (550 mg / 100 g), I (26 µg / 100 g), Se (42 µg / 100 g), P (290 mg / 100 g), Mg (45 mg / 100 g), Na (85mg/100g), Mn (0.57 mg/100g), K (210 mg/100g), Cu (0.49 mg/100g), S (190 mg/100g), pigments (astaxanthin and astacin), vitamin E (1.6 mg/100g) (Jessica *et al.,* 2015).

2.4.2 Selection of Catla fish

For fortification of shrimp the abundantly available Catla (*Catla catla*) had been selected. Catla (CA) is a tasty fish and enriched with high nutritional value (Vanitha, 2011). It contains high amount of potassium (310 mg/100g), protein (16.2 g / 100 g) and fat (2.8 g / 100 g) / ω-3 fatty acids (Mohanty *et al.,* 2016). CA has moderate iodine (18 µg/100g), sodium (74 mg/100g) and sulphur (170mg/100g) (Jessica *et al.,* 2015). But Catla has a deficiency of vitamins (A, D, E and B_{12}), minerals (Fe, Zn, Ca, Se, P, Mg, Mn, Cu). It supplies less energy (267 kJ) per 100 g fish consumption. It is readily available and farming in freshwater pond. It has a huge demand in the Asian market and contributing much to the total fish production and economic growth of some Asian countries, such as Bangladesh and India. Data (Carbohydrate, Protein, fat, energy, minerals and vitamins) of raw edible portion flesh of the CA fish had been taken from previously published papers (Vanitha, 2011; Mohanty *et al.,* 2016; Jessica *et al.,* 2015) and therefore those were not repeatedly analyzed but the data had been used either for information or presentation of results (Table 2.1).

2.4.3 Selection of Chela fish

Chela (*Chela cachius*) [CS] fish has high energy value (349 kJ per 100 g). Both protein (15.2 g / 100 g) and fat (2.4 g / 100 g) content is less as compared with CA. CS has moderate iodine, Se, Mg. It has deficiency of Fe (like CA), Na, K, Cu (like CA), vitamin E (Vanitha, 2011). Chela fish has high energy value (349 kJ per 100 g). Both protein (15.2 g / 100 g) and fat (2.4 g / 100 g) content is less as compared with Catla. It is very much rich in Zn (4.7 mg per 100 g), Ca (1g per 100 g), P (590 mg per 100g), Mn (0.6 mg / 100 g), S (170 mg / 100 g), vitamins (A, D and B_{12}) (Jessica *et al.*, 2015). It has moderate iodine, Se, Mg. It has deficiency of Fe (like Catla), Na, K, Cu (like Catla) and vitamin E (like Catla). All the nutritional facts were shown in Table 2.1. It is costly and according to fish vendors it is available in rural markets in monsoon season only.

Table 2.1: Different elements, carbohydrate, protein, fat, ash and energy (per 100 g raw fish) present in Catla (CA), Chela (CS) and Shrimp.

Fish	**Carbo-hydrate (g)**	**Protein (g)**	**Fat (g)**	**Ash (g)**	**Fe (mg)**	**Zn (mg)**	**Ca (mg)**	**I (µg)**	**Se (µg)**	**P (mg)**	**Mg (mg)**	**Na (mg)**	**K (mg)**	**Mn (mg)**	**S (mg)**	**Cu (mg)**	**Energy (kJ)**
CA[†,‡,§]	0	16.2	**2.8**	1.1	0.83	1.1	210	18	27	260	28	74	**310**	0.07	170	0.029	267
CS[§]	0.1	15.2	2.4	**2.9**	0.84	**4.7**	**1000**	19	32	**590**	39	28	85	**0.6**	170	0.052	**349**
Shrimp[§]	0	**17.6**	1	2.2	**2.7**	1.3	550	**26**	**42**	290	**45**	**85**	210	0.57	**190**	**0.49**	333

References:

†Vanitha M. (2011)

‡ Mohanty*et al.* (2016)

§ Jessica *et al.* (2015)

2.4.4 Reasons of fortification of Shrimp with Catla and Chela fish

Shrimp has deficiency of fat (1 g / 100 g), Zn (like Catla), P (like Catla), vitamin A (trace amount) and vitamin B_{12} (same amount of Catla) and vitamin D. Nevertheless,

it has deficiency in fat, Zn, Ca, P, K, vitamins (A, B_{12} and D). So, it needs to be enriched using other foodstuffs to overcome its deficiencies.

Like chela it (selected shrimp variety) is costly and available only in monsoon season but it can be fortified with Catla which is readily available, presence of good fats (ω-3 fatty acids) and protein rich food stuff; Chela is energy rich and rich particularly in Zn, Ca, Se, P, Mn, vitamin B_{12}, A and D.

Hence, overall concluded that CA is readily available; vitamin, essential minerals and energy deficient, fat and protein rich food stuff, CS is energy rich, costly and rich particularly in Zn, Ca, Se, P, Mn, vitamin B_{12}, A and D. Shrimp has pigments, energy rich food stuffs, costly and rich in Fe, I, Se, Mg, Na, K, Mn, S and Cu and deficient in vitamin A, D.

2.4.5 Addition of additives after fortification

All the three types of fishes were step-wisely precooked (sterilized), preprocessed (fortified), vacuum-dried and stored in powder form. Two flours such as rice flour and corn flour were chosen to fortify the three sterilized fish flesh (FF). Rice flour (RF) contains complex carbohydrates, no gluten (that favors non-stickiness of the final product), almost no protein and fats. It acts as a thickening agent, soft texture improver and flavor enhancer (Duncan, 2000). Corn flour (CF) has high starch (87% w/w) content (hence good water absorption capacity), good source of lutein (a powerful antioxidant that may help age related blindness), contains less protein (0.05% w/w), less fat (0.03% w/w) and satisfactory energy (1484 kJ / 100 g). Also, CF is rich in carbohydrate, essential elements (Fe, Ca, Cu, K, P, Zn), important vitamins [thiamine, niacin, vitamin B-6] (Sabanis and Tzia, 2007). Shankar and Bandyopadhyay (2005) previously blended RF with fish powder through extrusion process. Sabanis and Tzia (2007) used CF along with RF and soy flour to improve bread characteristics. The ginger (dried) is very common spice by which three sterilized FF also fortified along with rice and corn flour to make a new set of fish powder. Ginger contains twelve anti-oxidants, carbohydrates (22% w/w), proteins (4% w/w), fat (1.5% w/w), several minerals (K, Ca, P, Fe, Mn, Mg etc.). Dried ginger powder (DG) contains high quantity of β-ionone [anticancer agent] (Liu *et al.,* 2008). It has been used as taste and flavor enhancer (Sangwan *et al.,* 2014) and also rich in

ash and Ca (Kirk and Sawyer, 1991). In Table 2.2, the nutritional constituents of RF, CF and DG are presented.

Table 2.2: Different elements (Fe, Ca, Cu), carbohydrate, protein, fat, ash and energy (per 100 g) present in Rice flour (RF), Corn flour (CF) and Dried ginger powder (DG).

Flour / Powder	**Moisture (g)**	**Carbohydrate (g)**	**Protein (g)**	**Fat (g)**	**Ash (g)**	**Fe (mg)**	**Ca (mg)**	**Cu (mg)**	**Energy (kJ)**
RF[†]	12.14	78.69	7.56	0.68	0.93	2.8	15	0.24	1490
CF[‡]	12.0	82.75	7.50	1.4	1.2	5	12	0.14	1575
DG[§,¶]	3.66	22	5.42	0.83	2.12	1.65	66.62	0.65	540

References:

† Shankar and Bandyopadhyay (2005)

‡Sabanis and Tzia (2007)

§ Kirk and Sawyer (1991)

¶ Sangwan *et al.* (2014)

2.4.6 Vacuum drying and previous works on fish powders

Brief introduction is not required for vacuum-drying (VD) process of foodstuffs. Chakraborty *et al.* (2015) reported application of VD for dehydration of alphonso mango (*Mangifera indica L.*) and alovera (*Aloe barbadensis*) blend and produced a highly nutritional value food stuffs within reasonable time. Almeida-Trasvina *et al.* (2014) optimized VD by RSM (response surface methodology) to preserve antioxidants present in apple pomace. Celeriac slices (*Apium graveolens L.*) were dried using VD by six different reduced pressures and three different temperatures (Alibas, 2012).

In recent past, fish powders were produced for the fortification of biscuits (Mohamed *et al.*, 2014), breads (Adeleke *et al.,* 2010) and ice-cream (Shaviklo *et al.*, 2011) etc. Not only that, fish bone powders were also used for removing malnutrition (Zhang *et al.*, 2016), lactose intolerance in human body (Nemati *et al.,* 2016). Sometimes, fish bones are retained in the FF to increase the nutritional load (Chattopadhyay *et al.*,

2004). Although, fish oils (Kolanowski *et al.,* 2006) contain ω-3 fatty acids and related products such as microencapsulated fish oil (Serfert *et al.,* 2010) had been widely used for preparation of different foodstuffs. However, literature review indicates that very scanty reports are available on the use of FF for the preparation of highly nutritional food stuffs containing vitamins, essential minerals and ω-3 fatty acids. Besides, production of mixed-fish stuffs through fortification of FF by different flours, spice to improve the nutritional quality of FF will be taken an utmost importance.

2.4.7 Testing of Ash, TVBN, Mercury, Histamine of final products

The ash of all foodstuffs are the measurement of inorganic residue remaining after the organic matter completely removed by burning (Kirk and Sawyer, 1991). High ash% suggests the presence of an inorganic adulterant i.e. inferior quality. Total volatile base nitrogen (TVBN) is mainly composed of ammonia, trimethyl amine (TMA) and dimethyl amine (DMA). The freshness of fish is identified by TVBN, since it is an indicator of fish detoriation (Idakwo *et al.,* 2016), and also the detection of mercury (Hg) is essential for final fish products. The main path for human exposure to mercury (Hg) is considered from the consumption of fish (EFSA, 2004). According to Food and Drug Administration (FDA, 2012) and World Health Organization (WHO, 2007) mercury (Hg) is very dangerous for both humans and ecosystems. The presence of mercury in fish is very harmful since it is highly toxic (responsible for damage the central nervous system, vision and hearing loss, delayed development, language disorders). In fish, histamine is produced from free histidine by enzymatic decarboxylation which may be due to spoiling bacteria or may be endogenous to fish itself. The presence of histamine in fish item causes many symptoms like cardiac palpitation, rash on skin, dizziness, faintness etc. Therefore, the testing of histamine is utmost importance for fish items since histamine is heat-stable (not destroyed in heat-processing).

2.5 BLANCHED AND FREEZE-DRIED BROCCOLLI

- Therefore, fresh broccoli must have to preserve by suitable technique (s) with reduction of initial moisture content and without destroying the nutrients can be alternate solution. From Table 2.3, it is found that frozen

conditioned broccoli (both cooked & uncooked) is far better vitamin C retained as compare with unfrozen conditioned broccoli (both cooked & uncooked). Therefore, after blanching always freezing is preferable. Hence, selection of FD may be beneficial for broccoli.

Table 2.3: Average Ascorbic Acid (Vitamin C) levels (mg/100g Dry Weight) in Broccoli.

Fresh / unfrozen (mg/100g)		**Frozen (mg/100g)**	
Uncooked	**Cooked**	**Non-blanched**	**Blanched**
394.91 – 1339.45	425.00 – 1000.00	706.96 – 1408.00	574.07 – 685.19

References: Favell (1998). Mahn and Reyes (2012). Martin-Belloso and Lianos-Barriobero (2001). Drake and Carmichael (1986). Wu *et al.,* (1991).

3.1 AIMS & OBJECTIVES

It is established that freeze-drying (FD) is the best drying technology for food stuffs with respect to all quality attributes of the products. But the excessive operating cost of the process prohibits this technology for its general use. It is therefore necessary to reduce the drying time in order to cut-down the energy requirement and hence to make the FD process cost effective. During FD the supply of energy for sublimation of ice and its removal as water vapours play a major role for process economy. The heat of sublimation may be supplied by either radiation heater or conduction heater. It is already established that the vacuum-drying (VD) is similar to FD except the raw material is not frozen and the vacuum is not as high as FD. Though a few works have already been done and published by using the radiation heater during either in freeze-drying or in vacuum-drying, but they were not effective or lack of directions.

The present investigations focused towards reducing drying time (for both FD and VD) to enhance process economy by pretreatment of raw food stuffs along with developing effective heat transfer mechanism for moisture removal. It is envisaged that reduction of drying time by a fraction could reduce the process cost drastically. In view of these, the main objectives of the present investigation are:

I) To study the effects on drying time of different pre-processing steps, namely osmotic dehydration (OD), fortification with blending and blanching of raw food stuffs and finally qualities of finished products.

II) To study the effects of silver coated steel plate as heating surface for conventional freeze-drying and compare its performance with normal steel plate or copper plate.

III) To develop and study the effects of silver coated steel rounded plate (SCSRP) while placing it inside a low wattage steel cubic heater with five heating surfaces (top opened) on drying time both for freeze-drying and vacuum-drying.

IV) To establish the mechanism and kinetics of drying both for freeze-drying and vacuum-drying.

V) To compare the qualitative as well as quantitative quality parameters of the freeze-dried and vacuum-dried food products using standard testing methodology.

In order to achieve objective-I,

1. A fruit (kiwi) is used as a raw material for two different experiments viz. osmotically dehydrated kiwi fruit with successive freeze-drying and blended / fortified kiwi fruit pulp by few natural ingredients with successive freeze-drying. Osmotic dehydration (OD) may be a process of reduction of freeze- drying time (Yadav *et al.,* 2012). The impregnation of solute can be performed by moist infusion or by dry infusion. Moist infusion consists in soaking the food pieces in a water-solute solution of lower a_w while dry infusion involves direct mixing of food pieces and solutes in required proportions. The infusion of not only the solute used to control a_w but also the desired quantities of antimicrobial and anti-browning agents or any solute for improving sensory and nutritional quality. This process is commonly called "osmotic dehydration" [OD] (Zeuthen, 2005) and it is used to reduce moisture load that to be remove during drying. The experiments are planned into two paths. One is conventional freeze-drying (CFD) of selected kiwi sample (s) with proper dimension (s). Another is freeze-drying (FD) after osmotic dehydration (OD), called it ODFD.

2. Food fortification is a method by which additives are to be added into the food materials either to enhancement of nutrients, to meet the deficiency of micro / macro nutrients or increase the shelf-life by reduction of moisture content. The food materials before fortification may be sterilized or not. For the selected kiwi fruit the sterilization process is not done since the fruits are highly heat sensitive. For the fish (es) the sterilization is mandatory since from a public health point, the most important microorganism in low acid food is *Clostridium botulinum*. It is a heat-resistant, spore-forming, anaerobic pathogen. It survives in processing, can potentially grow and produce the deadly botulism toxin. Since, *C. botulinum* and most spore formers do not grow or produce toxin at pH<4.5 (high and medium acid foods), so the thermal processing of low-acid foods can be done at elevated temperatures [115°C to 125°C] (Ramaswamy and Marcotte, 2006).

3. For blending, the selective additives to be added into the food materials (sterilized or not) by blending technique. For fortified kiwi powder preparation, KF pulp was converted to KF-paste by the addition (/fortification) of few natural / domestic ingredients as described on effective and corrected ratio by trial and error method to reduce the initial moisture load of the KF pulp and also to increase the quality of the product.

For the works related on Shrimp fortified with two fishes, all of them are sterilized properly with brine solution. After sterilization, sterilized and combined shrimp and two fish stuffs was prepared. The whole part of sterilized brine mix was used further in the subsequent blending step to prevent nutrient loss from the samples. From three sterilized fish stuffs, two sets of blended samples were prepared. The sterilized shrimp along with two sterilized fishes were blended using a blender to obtain pulp. Subsequently, the sterilized brine solution and pulp were further enriched by mixing with corn and rice flour in proper ratio using same blender by "step wise-short time addition and mixing methodology" with blending at moderate speed to avoid unwanted reaction(s), un-pleasant flavours and tastes and hence prepared fortified Shrimp and blended set-1. Similarly, fortified Shrimp and blended set-2 was prepared through addition of corn flour, rice flour and dried ginger powder to the sterilized brine solution and pulp in selected proportions.

4. Blanching is not the method of cooking, rather it is the preliminary / primary process followed to prepare the food item which would eventually undergo another method of preprocessing. There are many benefits of blanching are there among them it can improve the colour of foods. There are two types of blanching: (1) Hot (water) blanching and (2) Cold (water) blanching. In hot blanching the food is immersed in the heated / boiled water and cooked for the required time. In cold blanching the food is immersed in cold water and then start to heat till heated / boiled conditions are reach. At this condition, the food is blanched for required time. In this (cold) blanching the foods become more absorbent and hence it is good to them for drying. In this research, it was used cold blanching for freeze-drying of blanched broccoli. The blanching step could eliminated some portions of initial moisture content of broccoli hence cut down moisture load just before freezing and hence drying.

In order to achieve objective-II, the strategies are:

1. Selection of Heater for Sublimation during Freeze-Drying: The silver coated steel heater (SCSH) was made to supply (conductive) heat of sublimation during freeze-drying. But before the final acceptance of that heater there were some problems found during whole operation. After some investigations (by trial and error method) the design and fabrication of heaters were settled.

In order to fulfill objective-III, the following steps on calculations were done:

1. The established drying models viz. Newton, Page, Henderson & Pebis and Wang & Sing are used for osmotic dehydrated kiwi and blended freeze-dried kiwi fruit. For fortified Shrimp, along with above four models Modified Page, Linear and Modified Wang & Sing models were also investigated for drying kinetics.

2. Different statistical parameters and equations were used to calculate Root mean square error (RMSE), Mean bias error (MBE), Modeling efficiency (EF), Regression coefficient (R^2), Chi-square (χ^2), Mean relative deviation (E%) for evaluating best fitting drying model for particular food stuff under freeze-drying or vacuum-drying.

3. The effective (mass) diffusivity and activation energy were calculated by either respective equations or graphical method (Arrhenius) for all the food stuffs

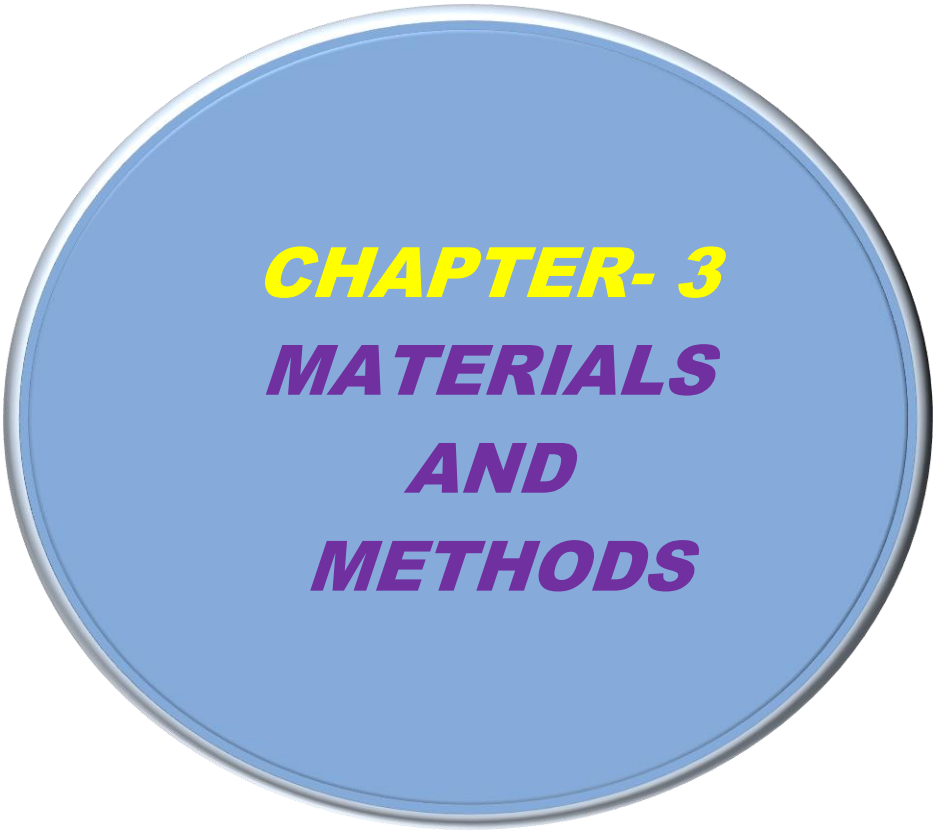
preparation and drying techniques (either freeze-drying or vacuum-drying) to identify minimum activation energy required (kJ/mole) for processing or drying.

In order to meet objective-IV, raw data for specific drying experiments will be analyzed both numerically and graphically using some known and established drying models. The best model identified for particular drying of food stuff (s) will be used to determine the rate constant, effective diffusivity and activation energy for the process.

In order to meet objective-V, the following tests were done for the establishment of good quality finished products:

1. The moisture, carbohydrate, sugar, protein, total fat, energy, ash, minerals (Na and K) and vitamins (A and K) of the raw kiwi fruit, osmotically dehydrated kiwi and freeze-dried kiwi were measured using standard methods.
2. The moisture, carbohydrate, protein, total fat, energy content, titrable acidity, crude fibre and total ash of the blended kiwi and final product blended freeze-dried kiwi fruit were tested and simultaneously sensory evaluations (by hedonic scale) were done. From the sensory evaluation, % of consumer acceptance, average score and total score were investigated by using some formulas.

3. For the fortified Shrimp preparation, there were many tests such as moisture, pH, carbohydrate, protein, total fat, energy, total ash, ω-3 fatty acids, histamine, TVBN, mercury level, rehydration ratio, water activity, microbiological assay and resistance to denaturation of protein of finished products were investigated.


CHAPTER- 3
MATERIALS
AND
METHODS

4.1 INTRODUCTION

Osmotic dehydration (OD) is a method for the partial dehydration of water rich foods. The initial moisture % of kiwi fruit is >75%. Therefore, OD technique was applied for the selected kiwi fruit for experiment set-1. By this technique the fruit sample was immersed in concentrated solutions of sugar and salt or both. At a particular temperature, time, concentration of the solution the kiwi sample was poured and hence measured the water loss (WL) and solute gain (SG). The optimize conditions were sucrose concentration (in Bx), Time (in hour) and temperature of that solution (in °C) to obtain more WL of the sample and less SG of the same sample.

The same fruit (kiwi) was used as a raw material for experiment set-2 blended / fortified kiwi fruit pulp by few natural ingredients with successive freeze-drying. For experiment set-3, shrimp and two fishes namely catla and chela were selected, where sterilized shrimp was fortified by sterilized catla, sterilized chela and few natural ingredients with successive vacuum-drying. One vegetable (broccoli) was selected for storage by blanching (cold) with successive freeze-drying. For all drying processes the sublimation heat was supplied by self-designed heater. The heating plates were selected as stainless steel, copper plate and silver coated steel plate. Their drying kinetics was studied with best possible mathematical drying models. The proximate and physico-chemical analysis were done. Sensory evaluation was done by using hedonic scale for one finished product. For fortified shrimp through analysis of macro and micronutrients were investigated with products shelf-life study; TVBN, histamine and Hg level of finished product was also detected.

4.2 EXPERIMENT SET-1: EXPERIMENTS DONE FOR OSMOTIC DEHYDRATED AND FREEZE-DRIED KIWI FRUIT

Fresh kiwi fruits (*Actinidia deliciosa*) were de-skinned and sliced in the form of slabs with a typical dimension of 35 × 33 × 5 mm (±0.1 mm). The initial weight and moisture content of the samples were determined (by moisture meter; KERN, Germany, Model MLS 50-3 HA 250, accuracy 0.01%). The OD was performed by using sucrose (99% purity) solution (SS) of three concentrations (40 Bx, 55 Bx and 70 Bx) with addition of 2 to 3% sodium chloride (W/V) solution. OD steps were applied for kiwi fruit slices as below:

4.2.1 Osmotic Dehydration Steps: Applied for Kiwi Fruits

1) The fruit was de-skinned and it was cut into three samples of same dimension [35 ± 1 × 33 ± 1 × 5 ± 1 mm; which was constant for every test].

2) Individual weights of three samples were taken and average weights of these samples were measured.

3) If average weight was W g (say), 5 times of this weight was taken and converted this value to the volume of solution i.e. $V_{solution}$ = 5W ml.

4) BRIX% (out of 40 Bx, 55 Bx and 70 Bx) was selected of sucrose solution. The weight of sucrose was selected to take according to the volume of the solution.

5) Weighed amount of sucrose was mixed to the V ml of the solvent (water). If it was not mixed to the solvent, then stirred gently until mixing was completed; The solution was heated slightly if not mixed properly.

6) The Bx was checked by refractometer (Erma RHB-0-32%, 28-62%, accuracy 0.1%); if not up to the targeted BRIX then either sucrose or water as per requirement was added.

7) After proper %BRIX (Bx), both the volume and weight of the solution were taken.

8) 2-3% salt (NaCl) (w/v) solution was added to the above solution.

9) It was mixed properly. This solution was put into glass beaker and heated by heater mantle. When the solution temperature was reached to targeted working temperature, then the three slices were poured gently to this sugar-salt solution.

10) Temperature at t°C was maintained till targeted time (in hour) was reached.

11) All the slices were removed after targeted time was reached and it was soaked in bed of tissue paper.

12) The volume and BRIX% (Bx) of the final solution was measured.

13) The moisture % of kiwi sample before and after O.D. was measured properly.

In Table 4.1, a worksheet is shown for O.D. processing of kiwi fruit slices with fixed dimensions. This sheet was filled-up for different brix, temperatures and time parameters during O.D. process.

Table 4.1: Work sheet of O.D. processing of kiwi fruit.

DATE	**Room Temp (°C)**	**40** Bx	**55** Bx	**70** Bx	**40 °C**	**45° C**	**50 °C**	**2 h**	**3 h**	**4 h**	**Initial avg. wt. (g)**	**Initial % M**	**Final avg. wt. (g)**	**Final % M**
22/02/2013	28	√			√			√			7.285	81.16	4.476	38.81
05/03/2013	33	√					√ (50)	√			7.870	81.17	3.963	35.81
06/03/2013	33	√				√		√			5.496	81.18	2.703	26.68
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Three brixes (40 Bx, 55 Bx and 70 Bx), three temperatures (40°C, 45°C and 50°C) and three time spans (2, 3 and 4 h) were chosen for OD. In Figure 4.1, the osmotic dehydration of kiwi slices with fixed dimensions is shown (already the details of OD steps are discussed in section 4.2.1). The volume of the total osmotic solutions was taken 5 times of kiwi sample weight. KF samples were placed in prepared osmotic solutions for specified times as per Taguchi orthogonal experimental design shown in next section (Table 5.4).

Figure 4.1: Osmotic dehydration (OD) of kiwi slices.

After OD, the samples were withdrawn cautiously and soaked by tissue paper and the final dimension, weight and moisture content were measured. The water loss [WL] and solute gain [SG] (Yadav *et al.*, 2012) of the kiwi samples were calculated by following equations:

$$WL = \frac{(W_0 - W_t) + (S_t - S_0)}{W_0} \times 100 \tag{4.1}$$

$$SG = \frac{(S_t - S_0)}{W_0} \times 100 \tag{4.2}$$

$$\text{Where, } S_0 = \frac{W_0(100 - M_i)}{100} \tag{4.3}$$

$$S_t = \frac{W_t(100 - M_f)}{100} \tag{4.4}$$

To evaluate the effect of fructose (99% purity) as osmotic solution OD was done at same optimal brix, temperature and time parameters as used for sucrose solution (SS). The KF sample (obtained at optimal OD using sucrose solution) was frozen through conventional refrigeration for a time span of 24 hours at temperature -16°C. Then by using SCSH and SH as thermal source, freeze-drying was performed for frozen osmotically dehydrated kiwi sample. The freeze-drying working unit is shown in Figure 4.2 and a schematic diagram of the same unit (for OPOD kiwi sample) is also shown in Figure 4.3.

Figure 4.2: Freeze-drying working chamber.

The freeze-drying (FD) chamber was subjected to fixed vacuum (5×10^{-3} mbar) and the condenser (moisture traps) temperature was fixed at - 40°C. The result of OD with fructose solution (FS) was inferior in terms of moisture reduction. Hence, FD was not done for sample obtained using fructose as osmotic solution (OS). Conventional freeze-drying (CFD) without OD was also performed at identical conditions as mentioned above and final moisture content of KF sample was recorded.

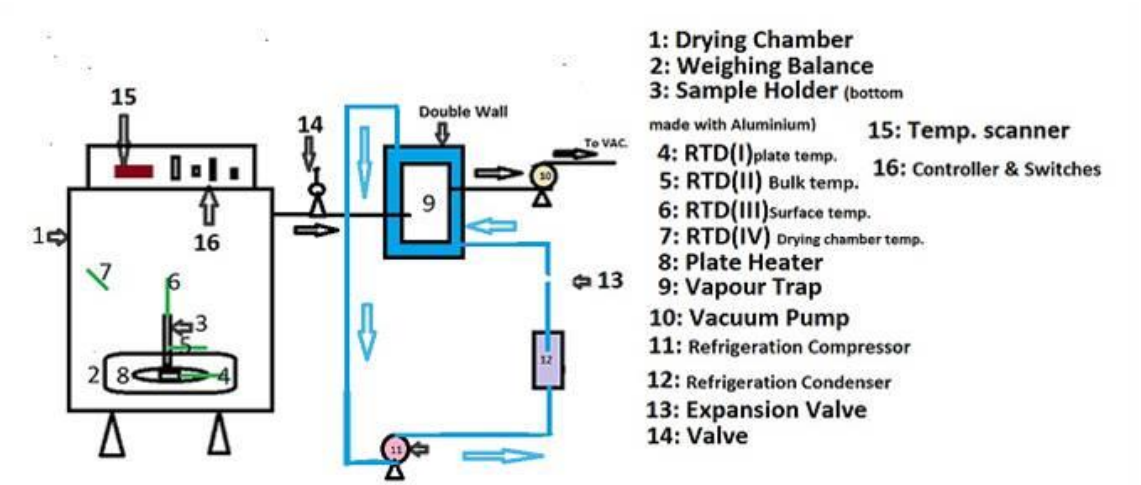


Figure 4.3: Schematic Diagram of used Freeze-dryer Unit for OPOD kiwi.

4.2.2 Experimental Methodology for Freeze-Drying

4.2.2.1 Heater design for supplying sublimation energy during freeze-drying

Heater design was mandatory for the heat required to supply sublimation energy to the frozen foods under vacuum. Before the final heater design was settled the heater was made by plywood body but it was not worked well during drying. It was replaced by the fiber body for the following possible reasons and the remedies were listed as per Table 4.2 below:

Table: 4.2: Drawback of Experiments (before fiber body chosen).

COMPONENT	PROBLEMS FOUND	REASON	REMEDY
Material (kiwi)	Cut them as per dimension encourage to create uneven both top and bottom surfaces.	The use of knife.	Use slicer.
Plywood (body of the heater made)	During freeze drying of the sample on the prepared heater, the weight of the heater (it was placed on the weighing machine) gradually decreasing.	The plywood escaped waters self during FD.	Use material for the body of the heater—which was light in weight and during heating no tendency to escape moisture.
Plate (top portion of heater)	Though plate material selection was fine but the temperature of different points of TOP plate was different. This was caused for non-uniform heating of the sample (kiwi).	Placement of heaters near the bottom plate was set up in wrong position.	Set heaters in right position of the bottom plate.
Plate (top portion of heater)	Unnecessary heat lost from the TOP plate.	TOP surface area of the plate was more.	TOP surface area of that plate to decrease i.e. dimensions (L X B) of the plate to decrease.
Plate (top portion of heater)	The kiwi sample placed on the silver plate was not got uniform heat in each point of sample (checked by RTD).	Flatness of the Plate was not proper (it was uneven surface)	To increase the thickness (t) of the silver plate.
Heater	When it was placed on the weighing m/c, the weight of the sample plus heater was nearly to the maximum weight capacity of the weighing m/c.	The total weight of the heater was excess.	To decrease the dimension of heater, plate weight (to decrease the dimension) and number of tablet heaters decrease from 3 to 2.

Heater	Temperatures of the heaters were fixed at a point. It was impossible to decrease or increase the temperature of the plate.	All heaters were connected in series with direct input lines.	Two heaters are to connect in series with series connection of two regulators.
Heater	Leakage current was found on the top plate—which might be dangerous during sample (kiwi) handling.	No earth connection.	Earth connection from silver plate to body of the freeze dryer (this is also connected to earth).
RTD	Few RTDs were not shown correct value. Errors found by those RTDs.	RTDs instrumental cause.	To change those RTDs by new ones.

It was necessary to check the moisture escaping tendency of fiber plate – which was to use for the making of HEATER (to supply sublimation heat during drying just after freezing). Earlier it was observed for *kiwi* fruit freeze drying that during heating of *frozen kiwi fruit* by using heater (which was made of silver plate top and sides were made of Bakelite sheet), the moisture was escaped from that Bakelite sheet along with sublimed vapour escaped from ice crystals. Therefore, the result of drying rate was unsatisfactory.

To remove this problem, the side walls sheet / material (fibre) were chosen very carefully, so that this sheet cannot escape moisture along with freeze- dried kiwi fruit during sublimation. A small experiment was conducted to check the escaping tendency of moisture from the selected fiber material (table 4.3).

- Weight of initial material (fiber) = 2.256 g.
- Starting time: 10:30 am; room temperature →22.4°C
- Ending time: 1:30 pm; room temperature →26.4°C
- Oven temperature →105.2 °C

Table 4.3: Recording of weight of fibre material with time inside the hot air oven.

Time (clock)	**Weight (g)**
11am	2.249
11:30 am	2.247

12 noon	2.245
12:30 pm	2.244
1 pm	2.245
1:30 pm	2.244

Weight of moisture escaped from the material = (2.256-2.244) g = 0.012g.
Therefore, the moisture was escaped from the material (in percentage) = (0.012/2.256) * 100 = 0.5319 %
The time was required to escape this amount of moisture is 3 (three) hours.

From the experience of the faults of the previous heater design, a new heater was designed (Elia, 1998). The heater was made (contained two small tablet heaters of 5 Watts, 220 Volt, 50 Hz) by own designed pattern (62 × 62 × 42 mm, thickness 4 mm) for sublimation heating purpose. Top surface (on which the frozen material was kept) was made by silver plate (62 × 62 mm, thickness 1 mm) for its higher thermal conductivity. A switch was there to regulate the number of heaters for OFF & ON purpose. Two controlling regulators were introduced to control the current and hence heat quantity produced on the top of the silver plate. Therefore, the sublimation heat was controlled to supply for primary drying stage as well as heat requirement for secondary drying stage (Hottot *et al.*, 2009). Three numbers of RTD were required for measuring the temperature of plate (Ag coated steel), temperature of frozen sample (bulk) and surface of the frozen material. The RTD of plate to control using PID temperature controller which was linked with heater input power. The plate temperature was fixed by using regulator. Once the temperature was fixed at a particular temperature then the heater was automatically controlled within ± 1°C i.e. below the temperature (1°C of set temperature) the heater was automatically ON and above the temperature (1°C of set temperature) the heater was automatically OFF.
An indigenously fabricated SCSH (circuit diagram shown in Figure 4.4) was made (fiber body, contained two small heaters (namely HEATER-1 and 2) of 2.5W each; 84×84×84 mm) for supplying conductive heat in FD for both osmotically dehydrated freeze-drying (ODFD) and Conventional freeze-drying (CFD).

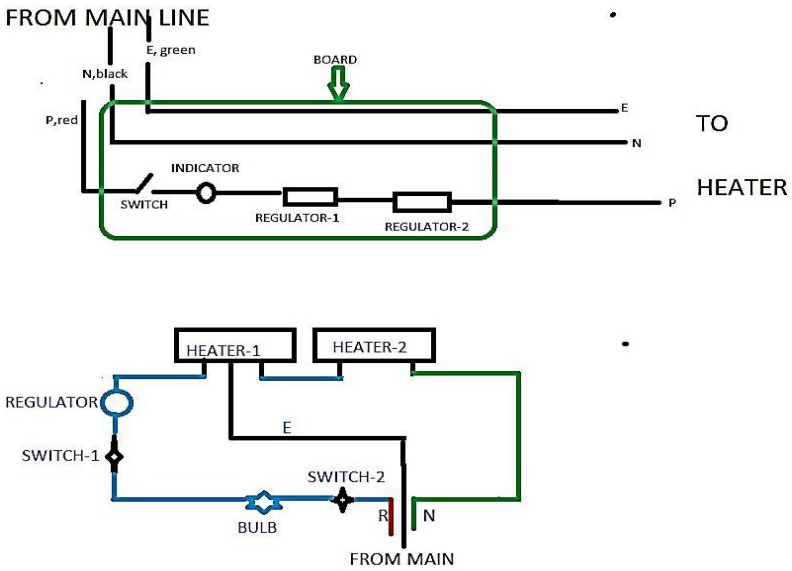


Figure 4.4: Circuit Diagram for Silver coated steel heaters (SCSH).

The sample was placed on SCSH of dimension (82 × 82 × 0.5 mm). The SCSH was placed on the weighing machine / balance by which the weight of the sample was taken in fixed time interval during drying. The SCSH and weighing machine both were placed inside the dryer chamber which is shown in Figure 4.5.

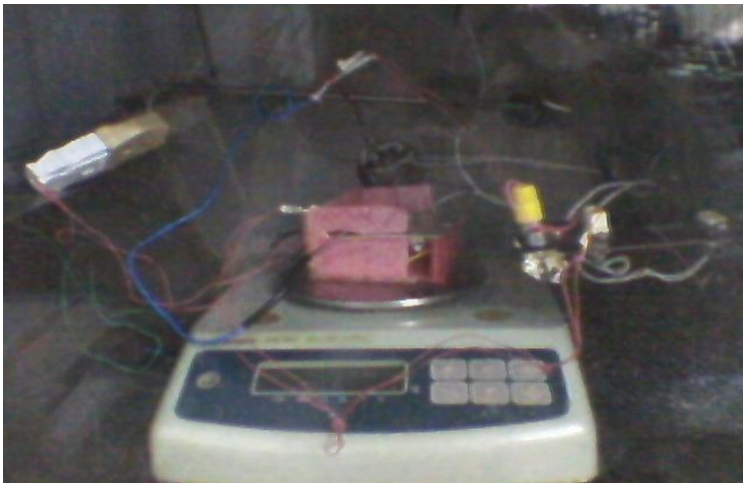


Figure 4.5: SCSH (with fiber body) placed on weighing m/c inside drying chamber.

One RTD was used for measuring the temperature of the heating plate and it was regulated with a PID controller. During FD, weight of KF sample was recorded at regular time interval. The FD time was noted when the final moisture content of the sample became ≤5% (w/w).

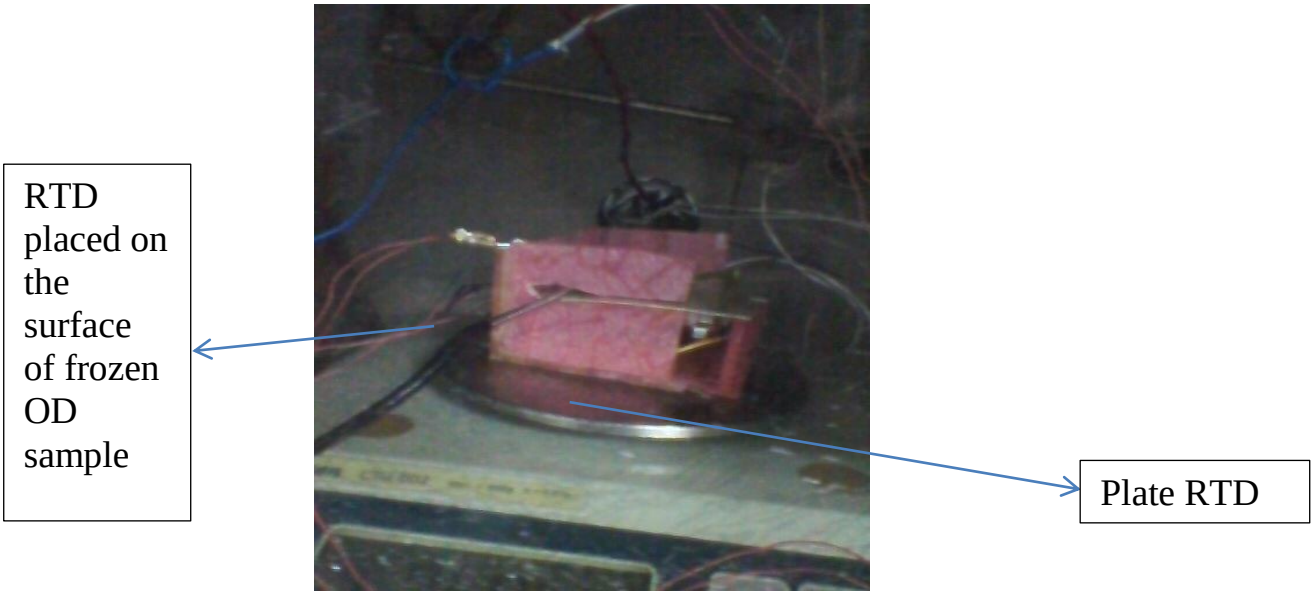


Figure 4.6: SCSH with two RTDs:- One attached with plate and another with frozen sample.

Various drying models namely Page, Wang & Sing, Henderson & Pebis and Newton models were used to compare the drying kinetics during OD and FDs. The statistical parameters such as the mean bias error (MBE), the root mean square error (RMSE) and the modeling efficiency (EF) were calculated by the following equations:

$$MBE = \frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right) \tag{4.5}$$

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)^2\right]^{0.5} \tag{4.6}$$

$$EF = \frac{\sum_{i=1}^{N}\left(MR_{\exp,i} - MR_{\exp,avge}\right)^2 - \sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)^2}{\sum_{i=1}^{N}\left(MR_{\exp,i} - MR_{\exp,avge}\right)^2} \tag{4.7}$$

The unsteady state diffusion equation for the calculation of effective diffusion coefficient (D_{eff}) is given as follow (Crank, 1975).

$$MR = \frac{8}{\pi^2} Exp\left[-\frac{\pi^2 * Deff * t}{4L^2}\right] \tag{4.8}$$

where, t : time in seconds, L: thickness or depth of sample in SCSRP.

The activation energy (E) is calculated by Arrhenius equation:

$$\ln\left(\frac{D_{eff,2}}{D_{eff,1}}\right)=\left(\frac{E}{R}\right)\left[\frac{(T_2-T_1)}{T_1*T_2}\right] \tag{4.9}$$

4.2.3 Taguchi Orthogonal Design and Optimization of Osmotic Dehydration

Water loss (WL) was selected as the response for OD operation. The signal to noise (S/N) ratios for all the OD experiments was calculated according to the following equation. For larger is better (WL) the equation is:

$$S/N=-10\log\left[1/(n\sum_{i=1}^{n}1/Y_i^2)\right] \tag{4.10}$$

Where, i and n are the number of replicates and trial experiments performed in all the combinations respectively.

4.2.4 Quality Assessment

The carbohydrate, sugar, protein, total fat, energy, ash, minerals (Na and K) and vitamins (A and K) of the raw KF, osmotically dehydrated KF and freeze-dried KF were measured using standard methods. Water activity and microbiological analysis for final dried sample was also performed by the water activity measurement device (Aqua Lab, CX-2, accuracy 0.003) and general microbiological assay (Harrigan, 1998).

4.3 EXPERIMENT SET-2: EXPERIMENTS DONE FOR VALUE ADDED KIWI FRUIT

In the previous experiment viz. optimized osmotically dehydrated freeze-drying of kiwi fruit the final quality of KF was improved in this experiment for keeping the maximum retention of vitamin C and other macro and micro nutrients enrichment by adding natural ingredients such as rice flour, oat flour etc.

4.3.1. Sample Preparation for Freeze-Drying

Fresh kiwi fruits (*Actinidia deliciosa*) were de-skinned and cut into small pieces by using sterilized stainless steel knife. The moisture content of the sample was determined by moisture meter (KERN, Germany, Model MLS 50-3 HA 250) shown in Figure 4.7.

Figure 4.7: Moisture analyzer.

A known weight of sample, about 0.1 kg, was put into a blender (PHILIPS, model no. HL 1632, 230 V, AC, 50 Hz, 500 W, maximum 1000 rpm) [shown in Figure 4.8] and was blended at moderate speed (~ 400-500 rpm) with stepwise addition of several ingredients serially. The added ingredients selected were rice flour, oat flour, ground sugar, salt, calcium lactate and a pinch of sodium benzoate as a preservative. The total blending time was 30 s and hence the average blending time for each ingredient is limited to 5 s. Excessive blending (both in time and rpm) was not done to avoid unwanted biochemical and chemical reactions which increased sourness and bitterness of the stock. This also increases shelf life, flavor, texture, nutrients and retention of vitamin C in the final powdery products.

Figure 4.8: Blender machine with bowl and lid.

The blended kiwi-paste (shown in Figure 4.9) or alternatively called Blended kiwi Fruit (BKF) was taken out from the blender and the final moisture content was measured. Finally, the blended fruit pulp is weighed (approx. 0.03 kg of batch) and poured into the SCSRP (diameter 82 mm, thickness 0.5 mm, depth 6 mm), spread uniformly (depth of about 5 mm) into the inside area of SCSRP for freezing. The BKF sample (obtained from blending technique using several natural ingredients) was frozen slowly at -18°C.

Figure 4.9: Blended kiwi fruit (BKF) inside the blender bowl.

4.3.2 Experimental Methodology for Freeze-Drying

Freeze-dryer as described in previous work (Chakraborty *et al.*, 2016) and a newly designed six sided cubic heat chamber (dimension: 90 × 90 × 90 mm; rating: 220V, 166 mA, 33W, temperature range: above ambient to 80°C) was used (shown in Figure 4.10).

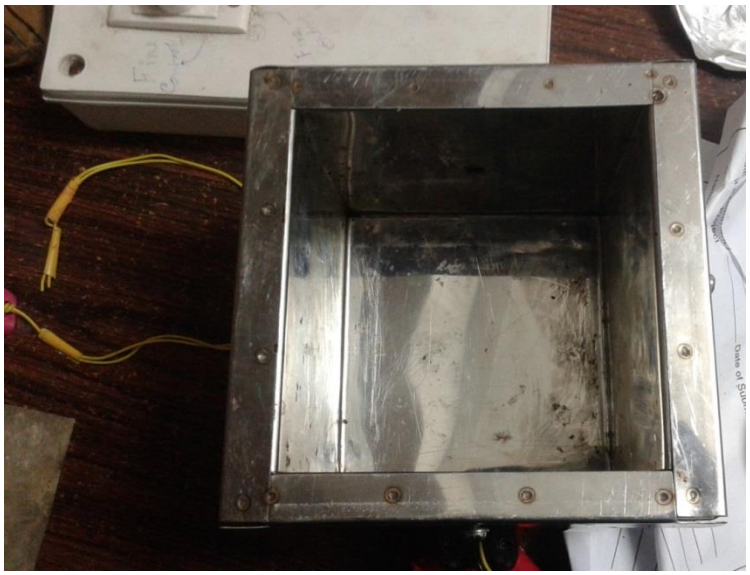


Figure 4.10: Six-sided cubic heater (SS made).

Heat was supplied from the four side-walls as well as from bottom surface. The top of this chamber was opened for allowing sublimated vapors to escape from the drying material. The FD chamber (Figure 4.11) was kept under a constant vacuum of 5×10^{-3} mbar and the condenser (moisture trap) was maintained at - 40°C. The schematic of the heating device (six sided cubic heater) is shown in Figure 4.10. The frozen material was mounted on SCSRP of cuboid heater and subsequently the entire assembly was put inside the freeze-drying chamber (Figure 4.12).

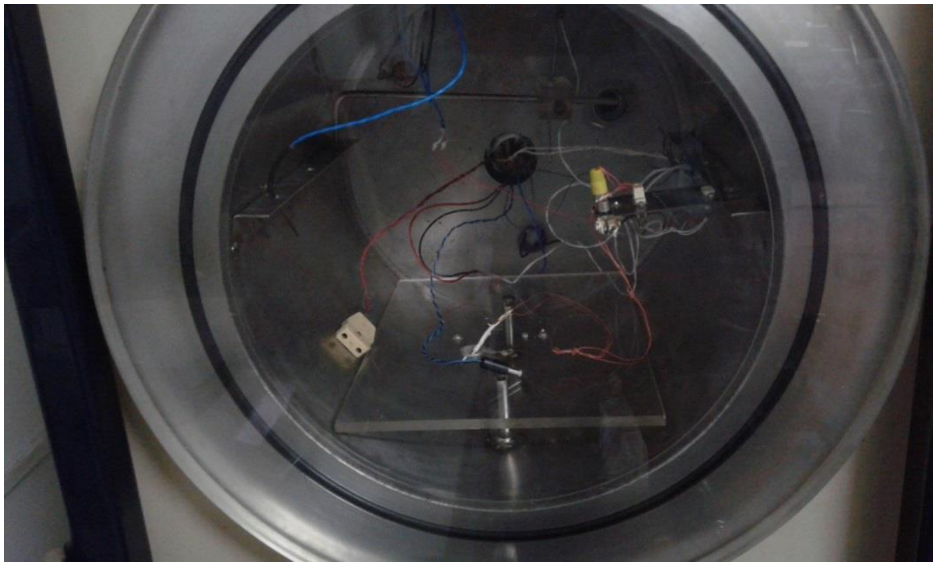


Figure 4.11: Clear view of inside drying chamber.

A PID controller with resistance temperature detector (Pt-100) was used to monitor and control the temperature of the steel surface. Simultaneously another RTD that was inserted into the drying material was used to record its temperature. During FD, instantaneous weight of KF sample was measured and recorded at fixed time interval. The FD time was also noted when the final moisture content of the sample became ≤5% (w/w).

Different drying models namely Page, Wang & Sing, Henderson & Pebis and Newton models were tested to compare the drying kinetics during FD. The statistical parameters such as the mean bias error (MBE), the root mean square error (RMSE) and the modeling efficiency (EF) were calculated by using Equations (4.5), (4.6) and (4.7).

The moisture ratio (MR) can be calculated by the following equation:

$$MR = \frac{M_C}{M_O} = \frac{\left[(W_o - W_e) - W_d\right]/W_d}{W_{i,w}/W_d} \tag{4.11}$$

The unsteady state diffusion equation for the calculation of effective diffusion coefficient (D_{eff}) was done by using Equation 4.8.

Figure 4.12: Cubic heater on weighing balance placed inside the dryer.

Figure 4.13 Freeze-dried sample (BFDKF).

Figure 4.14: Final ground and vacuum-dried kiwi (developed) product.

After freeze-drying, the sample (Figure 4.13) was ground (by the same grinder used before) with short time span. The dried powder again put into the vacuum dryer with the heater-off position for about 30 minutes and finally stored in an air-tight container (Figure 4.14) for quality assessment or other purposes.

4.3.3 Quality Assessment

The proximate analysis is a sort of yardstick for quality assessment of food products. It is basically the partitioning of the samples into six categories such as Moisture content, Ash Content, Crude protein (kjedahl protein), Crude lipids, Crude fibre and Carbohydrates (Nitrogen free extracts)) based on their chemical properties were done using standard analytical procedures. The proximate analysis and physico-chemical properties of blended freeze-dried KF (BFDKF) powder were measured given in Table 5.17 and Table 5.18 (next section) respectively.

4.3.3.1 Moisture, water activity and microbiological assay

The moisture content and water activity of the final sample was determined by moisture meter (KERN, Germany, Model MLS 50-3 HA 250, accuracy ±0.01%) and water activity measurement instrument (Aqua Lab, CX-2, accuracy 0.003)

respectively. The microbiological assay was also done (Harrigan, 1998) for the identification of microbes in final BFDKF powder sample.

4.3.3.2 Ash

5 g of BFDKF powder was taken in a porcelain bowl, ignited inside to a muffle furnace at 550 - 600°C and continued the ignition till grey ash was obtained. Cooled in a desiccator and weighed. Repeated the process of heating, cooling and weighing at 30 minutes interval till the difference in weight in two consecutive weights were <1 mg. The lowest weight for the total ash determination was noted.

4.3.3.3 Crude protein

The crude protein content was determined from the organic nitrogen content by Kjeldahl method. The various nitrogenous compounds were converted into ammonium sulfate by boiling with concentrated sulfuric acid. The ammonium sulfate formed was decomposed with sodium hydroxide solution and the ammonia liberated was absorbed in excess of standard solution of acid and then back titrated by standard alkali.

4.3.3.4 Crude lipids

Accurately 20 g BFDKF powder was weighed and transferred into extraction thimble and plugged it from the top with extracted cotton and filter paper. Put into the soxhlet apparatus, was fitted with boiling flask using petroleum ether (boiling point 40-80°C) as extraction solvent. After 5 to 6 cycles, evaporated the solvent present in flask (contained fat) on a water bath. The traces of residual solvent were removed by keeping the flask in the hot air oven (at 105°C) for about 30 minutes.

4.3.3.5 Crude fiber

The defatted BFDKF powder was transferred into boiling flask containing dilute sulfuric acid (1.25%, w/v). The flask was connected with water cooled reflux condenser. Put some glass beads to the flask to avoid unnecessary bumping of the solution during heating. Heated the solution for 30-45 minutes. After boiling, filtrated the solution through whatman filter paper and it was washed with boiling water until

the washings were free from acid. Taken the precipitation and followed the same above procedure with boiled by dilute sodium hydroxide solution (1.25%, w/v). After 30 minutes, boiling was stopped and filtered the solution till the washings were free from alkali. Dry the contents at 105±2°C in an air oven until constant weight was achieved. It was cooled, weighed and incinerated the contents in a muffle furnace until all carbonaceous matter was burnt. The ash was cooled and taken final weight of it.

4.3.3.6 Carbohydrates / Total sugars

The titration method (Lane-Eynon) was used for the determination of carbohydrates / total sugars (glucose, fructose). A burette was used to keep the carbohydrate / sugar solution being analyzed to a flask containing a known amount of Fehling solutions (Fehling A : Fehling B :: 1 :1) has reacted under boiling, any further addition of reducing sugars causes the indicator (methylene blue) from blue to red. The volume of consumed sugar solution required to reach the end point was recorded. Since the reaction is not stoichiometric, so use a standard solution (dextrose) to prepare a calibration curve by carrying out the experiment with a series of standard solutions of known carbohydrate concentration.

4.3.3.7 Titrable acidity

Titrable acidity can be expressed in grams of acid per 100 g or per 100 ml of the sample under testing. Here, titrable acidity was measured as per malic acid (1 ml of 0.1 N NaOH = 0.0067 g of malic acid). Known weight of sample diluted / mixed with distilled water (pH=7) properly. Filtered the solution and taken the filtrate as a stock solution. Transferred 2-3 ml of stock solution into about 20 ml of neutral water in a beaker. Titrated this solution with 0.1 (N) NaOH solution using phenolphthalein as an indicator till the end point was reached and noted the final titre value.

4.3.3.8 Vitamin C

Vitamin C was measured by 2,6-dichlorophenol indophenol method. The BFDKF powder (about 5 g) was mixed thoroughly with TCA (tri carboxylic acid) solution (10% w/v) in a mortar until homogeneity was achieved. Transferred this mixture into 100 ml graduated flask. The mixture was thoroughly shaken and made up the volume

to 100 ml with TCA solution. filtered the solution, taken 10 ml of the filtrate and titrated with standardized 2,6-dichlorophenol indophenol solution till the end point reached. Similar method was applied for standard ascorbic acid solution (1 mg standard, USP, ascorbic acid per 1 ml TCA solution).

4.3.3.9 Vitamin A

The BFDKF powder (about 1 g) was put into 5 ml of methanol for 2 h at room temperature (27±2°C) under dark condition in order to get a complete extraction. The β-carotene layer was separated using hexane through separating funnel. The volume was made up to 10 ml with hexane and then the layer was passed through sodium sulphonate through a funnel to remove moisture from the layer. The absorbance of the layer was measured at 436 nm using hexane as a blank (Ranganna, 1999).

4.3.4 Sensory Evaluation

4.3.4.1 Serving protocol

The final BFDKF sample was kept in air tight container and stored under refrigeration (5-8°C) condition. It was served to participants by standard American Society for Testing and Materials (ASTM) methodology (E1871-97). Each sample (BFDKF) size of 2 g was previously kept in small cleaned stainless steel bowl for all the participants. A small spoon and drinking water (kept in food grade plastic bottle) was placed nearby the bowl. The participants could use the sample as per their choice. The BFDKF powder was tested by them after rinsing of mouth with drinking water.

4.3.4.2 Evaluation process

The sensory evaluation process was done like this. All the participants had no prior information about the product and none of them were attached on this particular work. They were not given any hints about the judgment of others and no two panel members were present at the same place (whenever he/she was judging the sample). It was followed like this to avoid influence of individual judgment by others.

4.3.4.3 Sensory panel

The sensory panel consisted of 66 participants / panelists (Joshi *et al*., 2016) represented from different educated and non-educated categories in the ratio of 8:3 and their responses were recorded thrice at different span of time.

The participants were asked to indicate their preferences on a 9-point hedonic scale which has proven itself to be a simple and effective measuring device and it has a suitability for use by a certain and wide range of populations without an extensive training (Lim, 2011). The degree of liking goes: 9 = like extremely, 8 = like very much, 7 = like moderately, 6 = like slightly, 5 = neither like or dislike, 4 = dislike slightly, 3 = dislike moderately, 2 = dislike very much and 1 = dislike extremely. They were asked to rate their liking (s) of the BFDKF powder on the following organoleptic & overall tests: Sweetness, Sourness, Saltiness, Bitterless, Flavor, Texture (gummy or powdery nature) and Overall acceptability. The percentage (%) of consumer acceptance was calculated using the following equation (Jayasena; Cameron, 2008).

$$\%\ \text{of consumer acceptance} = \frac{\text{Number of panelists rated}>5}{\text{Total number of panelists}} \times 100 \qquad (4.12)$$

The Average score and Total score can be calculated by the following two equations (Singh-Ackbarali *et al*., 2014).

$$\text{Average score} = \frac{\text{Total points on individuial organoleptic test or oveall test}}{\text{Total number of panelists}} \qquad (4.13)$$

$$\text{Total score} = \frac{\text{Total points on individual organoleptic test or overall test}}{(\text{Total number of panelists})(\text{Maximum Hedonic points})} \times 100 \quad (4.14)$$

4.3.5 Stochastic Model

To model the intrinsic drift error of the sensory evaluation by Hedonic scale, the data were analyzed the Gauss-Markov Process (GM) stochastic technique.

The first-order Gauss-Markov (GM1) process is the most common model used in Kalman filtering of an integrated system because of its simplicity. The GM process is defined by the exponential Auto Correlation Function (ACF) in Equation (4.15) where variance is σ^2, the correlation time is given by $e^{-\beta(\tau)}$, and τ is the average time interval between sample taste.

$$R_x(\tau) = \sigma^2 e^{-\beta(\tau)} \quad (4.15)$$

A representation of Equation 4.15 was shown in Figure 4.15. It can be seen from this figure that its apex is at zero, and it has symmetrical descending slopes at both sides of graph going towards zero. The gradient of the slopes gets abrupt as the value of β increase. Also, it can be observed that the figure at τ equal zero is discontinuous.

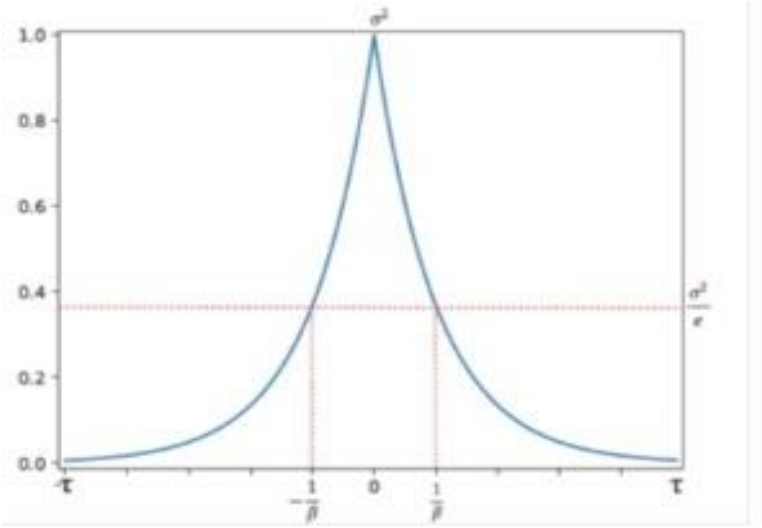


Figure 4.15: The representation of Stochastic Models equation (Eq 4.15).

4.4 EXPERIMENT SET-3: EXPERIMENTS DONE FOE FORTIFIED SHRIMP

In this experiment shrimp was fortified by catla and chela fishes for overcome its nutritional deficiency and improvement of macro and micro nutrients by adding rice flour, corn flour and with or without the addition of dried ginger powder.

4.4.1 Sampling Protocol

Shrimp, CA and CS were collected from local market with maintaining proper hygienic conditions and they were cleaned properly. After scraping off the scales and gills, CA and CS were gutted properly using sterilized knife. Filleted the flesh of CA to remove bones to yield FF. From Shrimps, only meat portions were collected. All the fishes were washed / rinsed by drinking water (0.03 to 0.04 mg/g), packaged (double covering) in polyethylene bags (51μm) and stored in refrigerator for next work.

4.4.2 Sample Preparation before Blending

Shrimp and two fishes were taken by the weight ratio Shrimp : CA : CS :: 2 : 1 : 1 in a small stainless steel (SS) container (with lid) where previously 1-2% brine solution were kept and finally lid of that container was closed. The moisture [Test Method: IS 1158: 1973 (RA 2010)] and pH (determined by Calibrated pH meter: SYSTRONICS, Ahmedabad, India, serial no.7743) of raw fishes were measured. Generally, most of the fishes are "low-acid food" ($pH \geq 4.5$). The sterilized brine solution (SBS) used in blending step (next) since the nutrient lost from all fishes (fish juices) were retained in this solution. From a public health point, the most important microorganism in low acid food is *Clostridium botulinum*. It is a heat-resistant, spore-forming, anaerobic pathogen. It survives in processing, can potentially grow and produce the deadly botulism toxin. Since, *C. botulinum* and most spore formers do not grow or produce toxin at $pH<4.5$ (high and medium acid foods), so the thermal processing of low-acid foods can be done at elevated temperatures (115°C to 125°C). Therefore, the small stainless steel (SS) container (with lid) contained the Shrimp and two selected fish samples with brine solution were sterilized at 121.1°C (15 psig) for 15 minutes. The prepared Sterilized and combined (i.e. taken Shrimp : CA: CS :: 2 : 1 : 1) three Fish

Stuffs (STFS) now ready for blending process. The moisture and pH were checked for STFS and three sterilized fishes (individually).

4.4.3 Sample Preparation for Blending

For two blended samples preparation (from STFS) the RF, CF, SBS and DG were selected and weighed (on the basis of STFS by trial-error method). For blended and fortified Shrimp set-1 (FS-1) preparation the RF, CF and SBS were added 10% (w/w), 2% (w/w) and 14% (v/w) respectively. For blended and fortified Shrimp set-2 (FS-2) preparation the RF, CF, SBS and DG were added 10% (w/w), 2% (w/w), 14% (v/w) and 1% (w/w) respectively.

The addition of RF, CF, SBS and DG (only in FS-2) to the STFS was done by "step wise-short time (30 s) addition and mixing methodology" with blending at moderate speed (~ 400-500 rpm) by blender (PHILIPS, model no. HL 1632, 230 V, AC, 50 Hz, 500 W, max. 1000 rpm) to avoid unwanted reaction(s), un-pleasant flavours and tastes.The final moisture and pH of FS-1 and FS-2 [Figure 4.16 (a) and 4.16 (b)] were measured.

(a) **(b)**

Figure 4.16: Paste of blended sample of (a) FS-1, ((b) FS-2.

Figure 4.17: Experimental set-up for vacuum-drying.

4.4.4 Sample Preparation for Vacuum-Drying

The prepared FS-1 and FS-2 were weighed (approx. 25 ± 0.01 g of batch), poured and spread uniformly into the inside area of SPMDH or CPMDH (depth or thickness of sample 0.002 m) for VD. An experimental set-up for VD is shown in Figure 4.17. The four samples (two sets multiplied by two different plates) were now ready for final stage of VD.

4.4.5 Experimental Methodology for Vacuum-Drying

VD was performed (0.5 Pa vacuum pressure, condenser temperature - 40°C) for blended four samples. Self-designed and fabricated six sides cubic (120 × 120 × 120 mm) heater (220 V, 166 mA, 36 W, temperature range: above ambient to 80°C) was made (depth 90 mm, thickness 10 mm) in which the heat was supplied from four side walls and bottom; top of the heater was opened for evaporation of water-vapor from blended material. One resistance temperature device (RTD) was used for measuring the temperature of the inside wall (steel) and it was connected with the PID controller (Figure 4.18).

The inside wall temperature was set at 55°C (accuracy ± 1°C). Another RTD was inserted into the blended material for the observation of VD sample's temperature. Weight of the drying sample was recorded at fixed time interval. The VD time was also recorded till the final moisture content of the sample became ≈ 3% (w/w). The final VD products (two) were marked as VDFS-1 and VDFS-2.

Figure 4.18: Cubic heater (insulated) connected with PID controller: heater rested on weighing balance and placed inside the VD chamber.

4.4.6 Drying Kinetics

Seven drying models viz. Newton, Page, Henderson & Pebis, Modified Page, Linear, Wang & Singh, Modified Wang & Singh were implemented to compare the drying kinetics during VD (Dongbang *et al.,* 2015; Fernando *et al.,* 2016). The selected mathematical models and their equations are shown in Table 4.4.

Table 4.4: Mathematical models selected (for SPMDH and CPMDH).

Serial no.	Model	Equation
1.	Newton	MR = exp (-Kt)
2.	Page	$MR = \exp(-Kt^n)$
3.	Henderson and Pabis	MR = A exp(-Kt)
4.	Modified Page	$MR = \exp[-(Kt)^n]$
5.	Linear	MR = A + Bt

6.	Wang and Singh	$MR = 1 + At + Bt^2$
7.	Modified Wang and Singh	$MR = (1 + At + Bt^2) / (1 + Ct)$

Note: MR is moisture ratio; A, B, C, K and n are model constants and t is the drying time in seconds.

The best models for describing the drying characteristics to produce VDFS-1 and VDFS-2 by using SPMDH and CPMDH were selected on the basis of lowest values of Chi-square (χ^2), root mean square error (RMSE) and highest coefficient of determination or regression coefficient (R^2). They were used to compare the counts of categorical responses between the experimental moisture ratios [$MR_{exp,i}$] and the predicted moisture ratios [$MR_{pre,i}$] (Dongbang *et al.*, 2015). What is more, for quality fit, R^2 (calculated from Microsoft excel 2010) should be close to 1 while the values of the RMSE and χ^2 should be close to zero (Goyal *et al.*, 2006).

$$\chi^2 = \frac{\sum_{i=1}^{N} (MR_{\exp,i} - MR_{pre,i})^2}{N - p} \tag{4.16}$$

4.4.7 Mean Relative Deviation (E %)

It is an absolute value that was used in this study to examine the goodness of the fit. It enhances the clear idea of the mean divergence of the predicted data from the experimental data. Value of *E%* < 5 indicates that extremely good fit, 5 to 10% reasonably good fit, > 10% show poor fit (Lomauro *et al.*, 1985; Gencturk *et al.*, 1986).

The formula by which the mean relative deviation has to be calculated by

$$E\% = \frac{100}{N} \sum_{i=1}^{N} \left| \frac{(MR_{\exp,i} - MR_{pre,i})}{MR_{\exp,i}} \right| \tag{4.17}$$

4.4.8 Rehydration Ratio (RR)

Rehydration ratio (RR) was measured (Chakraborty *et al.*, 2011) for both VDFS-1 and VDFS-2.

All the two sets of VD products were properly stored in air tight polythene containers and bag. After that, they were tested for rehydration ratio (RR). 5 g sample of stored VD product was taken and dispersed in 100 ml distilled water at 60°C, hold for 30 minutes. Filtered the solution of fish powder (product) and water by filter paper through 0.4 µm Whatman paper. Discarded the filtrate and precipitate was taken. Soaked the precipitate by tissue paper to remove the surface water and measured the weight of rehydrated sample of two sets (VDFS-1 and VDFS-2). The RR is expressed by the following equations:

$$RR = \left(\frac{W_{reVDFS}}{W_{VDFS}} \right) \tag{4.18}$$

W_{VDFS} = VDFS sample weight taken for testing, W_{reVDFS} = VDFS sample weight after rehydration.

4.4.9 Water activity (a_w) and Microbiological Assay

Water activity (a_w) is an important parameter to indicate the shelf life, predicting the growth of bacteria, yeast and mold. It is an indicator of the available free water (Ramaswamy and Marcotte, 2006). Food can be made safe to store by lowering the water activity to a point that will not allow pathogens such as *Clostridium botulinum* and *Staphylococcus aureus* to grow in it. It was checked by water activity measurement instrument (Aqua Lab, CX-2, accuracy 0.003). The microbiological assay was also performed for identification / presence of bacteria, yeast and mold etc. (Harrigan, 1998) in final dried products.

4.4.10 Differential Scanning Calorimetry (DSC)

Figure 4.19: Differential scanning Calorimeter.

Calorimetry is the basic technique by which the thermal properties and a relation drawn between the temperature and specific physical properties of the considered substances. The formation of protein molecules and their complexes are formed by the thermodynamically driven reactions. To achieve the thermodynamic investigations it has been required supersensitive calorimetric techniques such as DSC (Figure 4.19) to be required by which one can determine the thermal stability of proteins. DSC (Pyris Diamond DSC, Perkin Elmer Co., USA) technique was performed for two samples (VDFS-1 and VDFS-2) of final powders.

4.4.11 Quality Assessment

The following test methods were applied for the testing of Moisture (by moisture analyzer, shown in Figure 4.20) , Total Carbohydrate, Protein, Total fat, Energy, Ash, Omega-3 (ω-3) fatty acid, TVBN, Histamine and mercury (Hg) content of the VDFS-1 (Figure 4.21) and VDFS-2 (Figure 4.22) were measured properly.

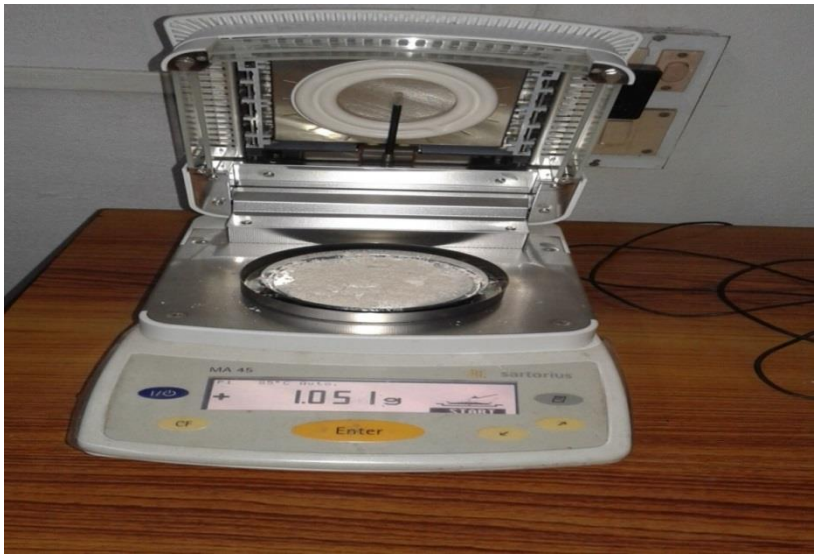


Figure 4.20: Moisture analyzer for final VDFS-1 & VDFS-2 samples.

Figure 4.21: Vacuum-dried sample (VDFS-1).

(1) Moisture (%) [Test Method: IS 1158: 1973 (RA 2010)], (2) Total Carbohydrate [Test Method: AOAC Official Methods. 19th Ed., 2012; Vol- II, 986.25], (3) Protein [Test Method: IS 7219: 1973 Rffm 2010], (4) Total fat [Test Method: AOAC Official Methods. 19thEdtn, 2012; Vol- II, 963.15], (5) Energy [Ref.: Composition & Analysis of Food by Pearson], (6) Ash [Test Method: IS: 1158: 1973 (RA-2010)], (7) Omega-3 (ω-3) fatty acid [Test Method: AOAC Official Method 19-th Edition Vol- II, 996.06], (8) Total volatile base nitrogen (TVBN) [Test Method:], (9) Histamine [Test

Method: FSSAI Lab Manual 6, 2015] and (10) Hg content [AOAC Official Methods. 19th Edition, 2012; Vol- I, 971.21].

Figure 4.22: Vacuum-dried sample (VDFS-2).

4.5 EXPERIMENT SET-4: FOR BLANCHED AND FREEZE-DRIED BROCCOLI

The raw and fresh broccoli was cold blanched, soaked and freeze-dried for improvement of its shelf-life.

4.5.1 Sample Preparation for Blanching

Fresh broccoli was purchased from local (Haldia) market of length 6 cm and breadth 2.5 cm. They were used to produce samples. Broccoli and its florets were cut into small pieces of length 4 cm. It was dark green color and the sample weight was 5.66 gm. The samples were divided into two sets. For set-1, pretreatment (cold blanching) was done (Figure 4.23) before freeze-drying of the sample (s). Half of the samples were blanched (by 2-3% brine solution) at 55° to 60°C for 10 minutes by using heater mantle. The weights of both the blanched and non-blanched (set-2) florets were measured properly. The moisture of raw broccoli was measured by moisture meter (KERN, Germany, Model MLS 50-3 HA 250).

Figure 4.23: Blanching of Broccoli.

4.5.2 Sample Preparation for Freeze-Drying and Storage

Removed the sample (s) gently from the solution after heat treatment / blanching and kept them in tray for cooling to drain the hot solution from the sample (s) as well as to prevent overcooking of the sample (s). The time was 10-15 minutes. They were now soaked by tissue paper to remove the surface water (Figure 4.24). After that, the blanched broccoli packed in Ziploc transparent bag.

Figure 4.24: Blanched and Soaked Broccoli.

The samples (both blanched and non-blanched termed as set-1 and set-2 respectively) were frozen in frozen chamber below their freezing point [at (-) 18°C]. After 24 hours of freezing, the samples were frozen completely and ready for drying.

4.5.3 Freeze-Drying of Broccoli

The sample was now dried in freeze-drying unit (Figure 4.25) under vacuum (5×10^{-3} mbar).

Figure 4.25: Freeze-dryer unit (heater placed inside) for broccoli.

By a newly designed heater [82 × 82 × 10 mm, 10 W] (Figure 4.26) sublimation heat was supplied to the frozen sample constantly. The temperature of the plate (SS made) maintained constantly (almost) by using own designed temperature controller manually. Three probes were required to set for measuring the temperature of three points. First one was plate temperature, second one was surface temperature of frozen sample and third one was ambient temperature of inside freeze-dryer unit (Figure 4.26).

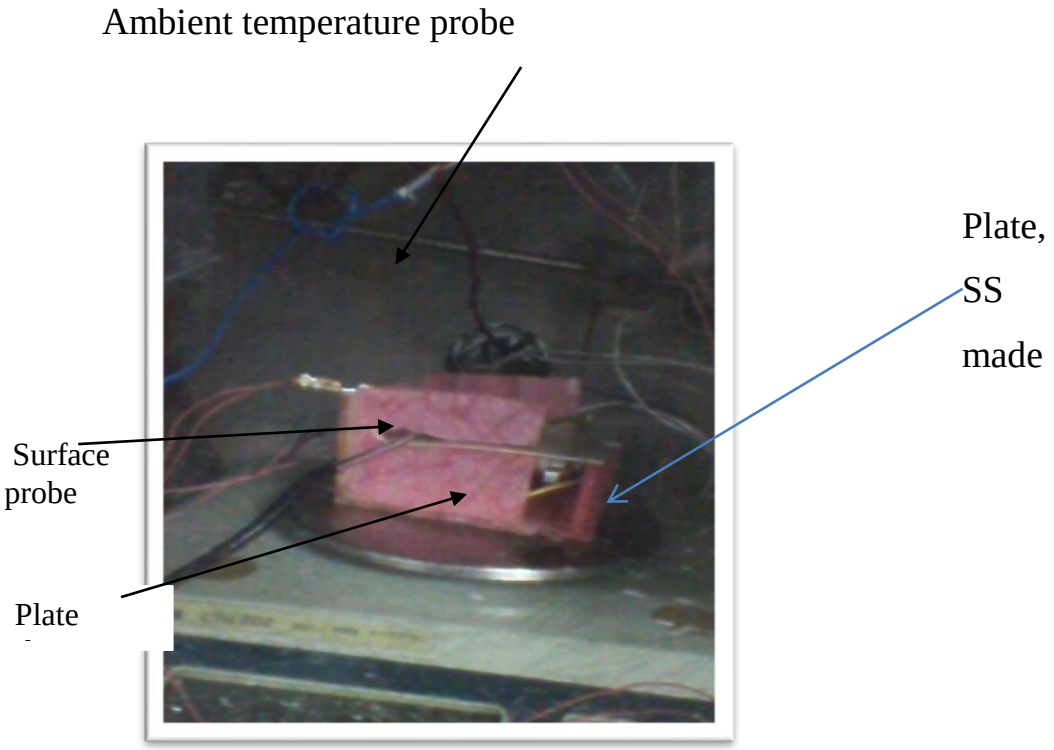


Figure 4.26: Heater with three temperature probes (RTD).

The sample weights was noted with time by weighing balance (PHOENIX, Model no. SMART 20, range 2.5 g - 2 kg, accuracy ±0.05 g) and it was run until two successive weights were nearly same (±1 mg). After complete drying, the sample (Figure 4.27) was now kept in air tight container and the final weight and moisture was measured.

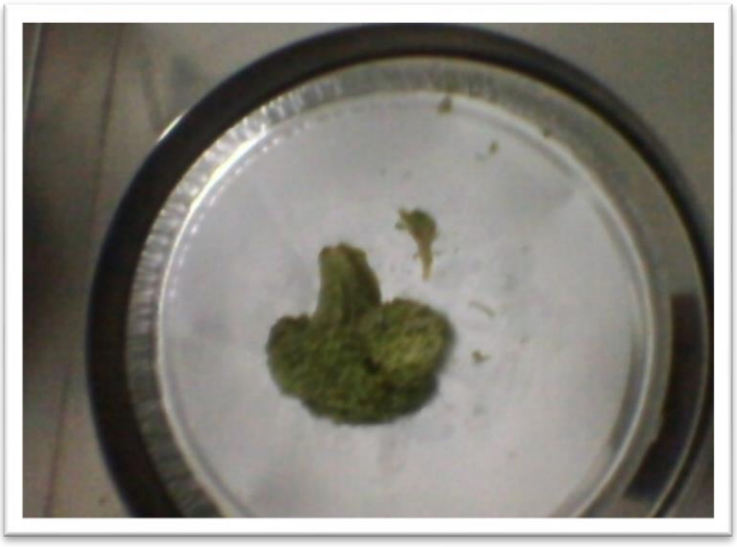


Figure 4.27: Freeze-dried Broccoli.

4.5.4 Quality Assessment

The moisture, protein, ash and vitamin C were measured for the raw, blanched and freeze-dried broccoli by using the standard protocols.

CHAPTER- 4
RESULTS AND DISCUSSIONS

5.1 EXPERIMENT SET-1: OSMOTIC DEHYDRATED AND FREEZE DRIED KIWI FRUIT

The KF was osmotically dehydrated prior to FD. This OD process was optimized (called OPOD) by Taguchi orthogonal experiment design. This OPOD sample was freeze-dried and named the sample as OPODFD. The moisture content for tested KF, OPOD sample and OPODFD sample were 85.07%, 68.16% and 4.27% respectively.

5.1.1 Water Loss and Solute Gain Calculations of Optimized Osmotic Dehydrated Samples

Each three sets of Brix (40°, 55° and 70° Bx), temperature (40°, 45° and 50°C) and time (2, 3 and 4 h) were chosen (see section 4.2.1); all of them (total $3 \times 3 \times 3 = 27$ sets) were osmotically dehydrated by same OD process discussed in section 4.2.1. The results were shown in Table 5.1.

5.1.2 Optimal Process Conditions

Before the calculations of optimal process conditions all the process variables were indicated by coded and uncoded factors (Table 5.2) and also a design matrix (related on Taguchi orthogonal design) would be arranged (Table 5.3). Finally, the analysis of S/N ratio obtained from Taguchi orthogonal design with the optimization criteria i.e. “larger is better” for WL in OD of KF were presented in Table 5.4. The maximum WL was obtained for the combination of 40° Bx, 50°C temperature and 3 h OD time.

Table 5.1: Results of water loss (WL) and solute gain (SG) for osmotic dehydration.

Exp. no.	Sucrose Bx	Sucrose Solution Temp. (°C)	O.D. time (h)	Initial Moist. % (M_i) ; before O.D.	Final Moist. % (M_f); after O.D.	Initial wt. (g) of kiwi slices, W_0	Weight (g) of the kiwi slices after O.D. W_t	S_0 (g)	S_t (g)	Water Loss, (WL) %	Solute gain, (SG) %
1(i)	40	40	2	82.526	38.81	7.285	4.473	1.273	2.737	58.697	20.097
1(ii)	40	45	2	81.18	26.68	5.496	2.703	1.034	1.982	68.058	17.240
1(iii)	40	50	2	68.83	19.11	7.897	3.469	2.461	2.806	60.435	4.363
1(iv)	40	40	3	68.83	41.09	9.653	5.34	3.009	3.146	46.099	1.419
1(v)	40	45	3	68.04	23.23	8.798	1.096	2.812	0.841	65.146	-22.396
1(vi)	40	50	3	68.83	16.55	5.922	2.89	1.846	2.412	60.753	9.555
1(vii)	40	40	4	68.83	12.22	5.875	2.69	1.831	2.361	63.235	9.022
1(viii)	**40**	**45**	**4**	**82.65**	**45.59**	**6.62**	**4.248**	1.149	2.311	**53.395**	**17.564**
1(ix)	40	50	4	68.04	21.73	7.433	2.413	2.376	1.889	60.986	-6.551
1(ix),Re	40	50	4	68.83	19.11	7.987	3.469	2.490	2.806	60.530	3.963
1(v),Re	**40**	**45**	**3**	**79.66**	**44.42**	**7.514**	**5.185**	**1.528**	**2.882**	**49.008**	**18.013**
2(i)	55	40	2	63.365	34.68	6.044	3.305	2.214	2.159	44.401	-0.917
2(ii)	55	45	2	63.365	23.52	6.836	2.604	2.504	1.992	54.406	-7.502
2(iii)	55	50	2	74.26	25.32	7.944	3.717	2.045	2.776	62.413	9.203
2(i),Re	55	40	2	76.04	41.94	8.7	4.32	2.085	2.508	55.215	4.870
2(iv)	55	40	3	71.05	14.94	7.8	3.012	2.258	2.562	65.281	3.896
2(v)	**55**	**45**	**3**	**72.92**	**47.39**	**8.344**	**5.74**	**2.260**	**3.020**	**40.319**	**9.111**
2(vi)	55	50	3	74.26	6.78	6.985	3.319	1.798	3.094	71.038	18.555
2(iii),Re	55	50	2	68.04	35.95	6.784	3.595	2.168	2.303	48.989	1.982
2(vii)	**55**	**40**	**4**	**70.24**	**20.68**	**7.948**	**5.09**	**2.365**	**4.037**	**56.996**	**21.038**
2(viii)	**55**	**45**	**4**	**71.88**	**36.24**	**8.08**	**4.754**	**2.272**	**3.031**	**50.558**	**9.394**
2(ix)	55	50	4	63.365	14.22	7.947	3.651	2.911	3.132	56.832	2.774

2(ii),Re	**55**	**45**	**2**	**72.49**	**45.46**	**5.664**	**3.255**	**1.558**	**1.775**	**46.365**	**3.833**
3(i)	70	40	2	75.37	41.1	8.57	4.496	2.111	2.648	53.808	6.270
3(ii)	70	45	2	**71.58**	**31.84**	**9.084**	**5.65**	**2.582**	**3.851**	**51.776**	**13.974**
3(iii)	**70**	**50**	**2**	**70.54**	**34.35**	**6.435**	**3.826**	**1.896**	**2.512**	**50.117**	**9.573**
3(iv)	70	40	3	71.05	2.3	6.4	3.89	1.853	3.801	69.652	30.433
3(v)	**70**	**45**	**3**	**80**	**51.85**	**5.544**	**3.408**	**1.109**	**1.641**	**48.127**	**9.599**
3(vi)	70	50	3	75.37	20.14	10	4.91	2.463	3.921	65.481	14.581
3(vii)	**70**	**40**	**4**	**71.31**	**29.77**	**9.005**	**5.766**	**2.584**	**4.049**	**52.248**	**16.279**
3(viii)	**70**	**45**	**4**	**74.14**	**39.43**	**9.297**	**5.159**	**2.404**	**3.125**	**52.260**	**7.751**
3(ix)	70	50	4	71.05	3.2	8.2	3.89	2.374	3.766	69.532	16.971
3(ii),Re	**70**	**45**	**2**	**71.55**	**40.64**	**8.163**	**5.74**	**2.322**	**3.407**	**42.973**	**13.290**

Table 5.2: Experimental ranges and levels of the factors (process variables).

Uncoded Factors	**Units**	**Uncoded values**	**Coded Factors**	**Units**	**Coded values**
Bx	% (W/V)	40	X_1	% (W/V)	-1
		55			0
		70			1
T_s	°C	40	X_2	°C	-1
		45			0
		50			1
T	hr	2	X_3	hr	-1
		3			0
		4			1

Table 5.3: Experimental design matrix.

	UNCODED FACTORS & VALUES / Operating or Process Variables			***CODED FACTORS & VALUES***			***Response / Dependent Variables***		
Sl. No.	**Brix [% (W/V)],** Bx	**Temp. (°C), T_s**	**Time (h), t**	**X_1, [% (W/V)]**	**X_2, (°C)**	**X_3, (h)**	**M_f (%)**	**WL (%)**	**SG (%)**
1	40	40	2	-1	-1	-1	38.81	58.697	20.097
2	40	45	2	-1	0	-1	26.68	68.058	17.240
3	40	50	2	-1	1	-1	19.11	60.435	4.363
4	40	40	3	-1	-1	0	41.09	46.099	1.419
5	40	50	3	-1	1	0	16.55	60.753	9.555
6	40	40	4	-1	-1	1	12.22	63.235	9.022
7	**40**	**45**	**4**	-1	0	1	**45.59**	53.395	17.564
8	40	50	4	-1	1	1	19.11	60.530	3.963
9	**40**	**45**	**3**	-1	0	0	**44.42**	49.008	18.013
10	*55*	*50*	*2*	*0*	*1*	*-1*	*25.32*	*62.413*	*9.203*
11	55	40	2	0	-1	-1	41.94	55.215	4.870
12	55	40	3	0	-1	0	14.94	65.281	3.896
13	**55**	**45**	**3**	0	0	0	**47.39**	40.319	9.111
14	55	50	3	0	1	0	6.78	71.038	18.555
15	*55*	*50*	*2*	*0*	*1*	*-1*	*35.95*	*48.989*	*1.982*
16	**55**	**40**	**4**	0	-1	1	**20.68**	56.996	21.038
17	**55**	**45**	**4**	0	0	1	**36.24**	50.558	9.394
18	55	50	4	0	1	1	14.22	56.832	2.774
19	**55**	**45**	**2**	0	0	-1	**45.46**	46.365	3.833
20	70	40	2	1	-1	-1	41.1	53.808	6.270
21	*70*	*45*	*2*	*1*	*0*	*-1*	***31.84***	*51.776*	*13.974*
22	**70**	**50**	**2**	1	1	-1	**34.35**	50.117	9.573
23	70	40	3	1	-1	0	2.3	69.652	30.433
24	**70**	**45**	**3**	1	0	0	**51.85**	48.127	9.599
25	70	50	3	1	1	0	20.14	65.481	14.581
26	**70**	**40**	**4**	1	-1	1	**29.77**	52.248	16.279
27	**70**	**45**	**4**	1	0	1	**39.43**	52.260	7.751
28	70	50	4	1	1	1	3.2	69.532	16.971
29	***70***	***45***	***2***	*1*	*0*	*-1*	***40.64***	*42.973*	*13.290*

Table 5.4: Taguchi orthogonal experimental design for OD of kiwi fruit.

Sl. No.	Brix (Bx)	Temperature (°C)	Time (h)	WL %	S/N ratio
1	40	40	2	57.229±0.2	35.15232
2	40	45	3	50.079±0.3	33.99311
3	40	50	4	61.861±0.2	35.82834
4	55	40	3	63.91±0.5	36.11138
5	55	45	4	51.397±0.5	34.21876
6	55	50	2	49.667±0.5	33.92136
7	70	40	4	52.901±0.4	34.46928
8	70	45	2	43.49±0.3	32.76779
9	70	50	3	64.376±0.1	36.17448

The sample weight, ambient (inside freeze-dryer unit during drying) and plate temperatures during OPODFD process using SCSH and SH were tabulated in Table 5.5 and Table 5.6 respectively. The same parameters were shown during CFD process using SCSH in Table 5.7. It was concluded that, for OPODFD process by using SCSH the drying time (2.5 h) required less than using SH (3.83 h). Again, by using same SCSH the drying time required more (5.75 h) for CFD process. These were ultimately concluded that OPODFD process was beneficial than CFD process and SCSH was more advantageous heater than SH for the same process followed. From all the tables it was found that the ambient temperature gradually increased as plate temperature increased i.e. heat is radiated from the plate to the ambient but since the ΔT of ambient is less than ΔT of plate at the same time interval so more heat is utilized from plate for the drying of foodstuffs than the radiated heat to the ambient.

Table 5.5: Sample weight, ambient and plate temperature during OPODFD process using SCSH.

Time (s)	Sample wt. (g)	Ambient Temp. (°C)	Plate Temp. (°C)
0	3.015	32.1	50
300	2.855	32.7	40.8
600	2.795	33	39.8
900	2.695	33.2	35.9
1200	2.605	33.3	34.1
1500	2.525	33.3	34.9
1800	2.485	33.4	32.3
2100	2.405	33.4	32
2400	2.345	33.4	32.2
2700	2.235	33.5	35.3
3000	2.165	33.4	38.8
3300	2.075	33.5	42.9
3600	1.935	33.8	46.8
3900	1.785	33.8	49.4
4200	1.685	33.7	47.7
4500	1.645	33.7	49.7
4800	1.545	33.7	51.9
5100	1.475	33.8	47.7
5400	1.395	33.8	46.1
5700	1.335	33.8	51.8
6000	1.275	33.6	50.2
6300	1.235	33.6	50.5
6600	1.205	33.9	52.8
6900	1.185	33.5	49.2
7200	1.165	33.5	49.3
7500	1.145	34	52.2
7800	1.125	34	49
8100	1.105	34	45.9
8400	1.095	34	44
8700	1.085	33.9	48.5
9000	1.085	33.8	48.3

Table 5.6: Sample weight, ambient and plate temperature during OPODFD process using SH.

Time(s)	Sample wt. (g)	Ambient Temp. (°C)	Plate Temp. (°C)
0	7.66	31	50
300	6.59	31.2	41
600	6.5	33.1	39.8
900	6.4	33.7	38.4
1200	6.22	34.1	37.5
1500	6.05	34.4	36.8
1800	5.89	34.6	35.7
2100	5.68	34.5	37.6
2400	5.45	35.1	39.5
2700	5.24	35.2	41.5
3000	5.13	35.5	42.5
3300	4.96	35.7	43.7
3600	4.76	35.6	44.5
3900	4.61	36.1	46.1
4200	4.42	36.1	47.1
4500	4.29	36.2	47.9
4800	4.12	36.4	49
5100	3.85	36.6	48.5
5400	3.79	36.6	49.7
5700	3.71	36.6	50.5
6000	3.34	36.5	49
6300	3.32	37.1	51.5
6600	3.21	37.2	49.6
6900	2.91	37.3	48.2
7200	2.87	37.3	49
7500	3.01	37.4	50.6
8100	2.11	37.7	52.1
9000	2.83	38	50
9900	2.78	38.2	48.9
10800	2.72	38.3	47.2
11700	3.14	38.6	56.2
12600	2.96	38.7	46.7
13500	2.87	38.7	43.1
13800	2.86	38.8	46.2

Table 5.7: Sample weight, ambient and plate temperature during CFD process using SCSH.

Time (sec.)	Sample wt. (g)	Ambient Temp. (°C)	Plate Temp. (°C)
0	22	32	50
300	20.94	32.8	47.4
600	20.33	33.2	44.4
900	20.06	33.7	42.2
1200	19.74	34.3	40.1
1500	19.25	34.4	38.4
1800	18.84	34.7	37.6
2100	18.54	34.5	37.1
2700	17.58	35.4	38.2
3300	16.84	35.2	40.1
3900	16.14	35.5	41.9
4500	15.4	35.7	43.2
5100	14.89	35.6	43.9
5700	14.11	36.3	45.3
6600	13.32	36.1	46.4
7500	12.44	36.2	47.6
8400	11.64	36.5	48.6
9300	10.98	36.6	49.5
10200	10.35	36.6	50.2
11100	9.7	36.7	50.9
12000	9.14	36.5	51.4
12900	8.53	37.2	52
13800	7.94	37.2	52.5
14700	7.55	37.3	53
15600	7.14	37.4	53.4
16500	6.57	37.4	53.9
17400	6.21	37.8	54.6
18300	5.85	38.1	54.3
19200	5.57	38.2	54.2
20100	5.16	38.3	55.3
20700	4.77	38.5	55.1

The change of weight versus time during OPODFD process using SCSH and SH were shown in Figure 5.1 and Figure 5.2 respectively. The same graphical representation was shown during CFD process using SCSH in Figure 5.3. For both OPODFD and CFD process using SCSH the gradual decrease of the weight of the dried sample were smoothly but by using SH the decrease was some ups and downs- it means the SH had not transferred the heat to the food surface smoothly and uniformly.

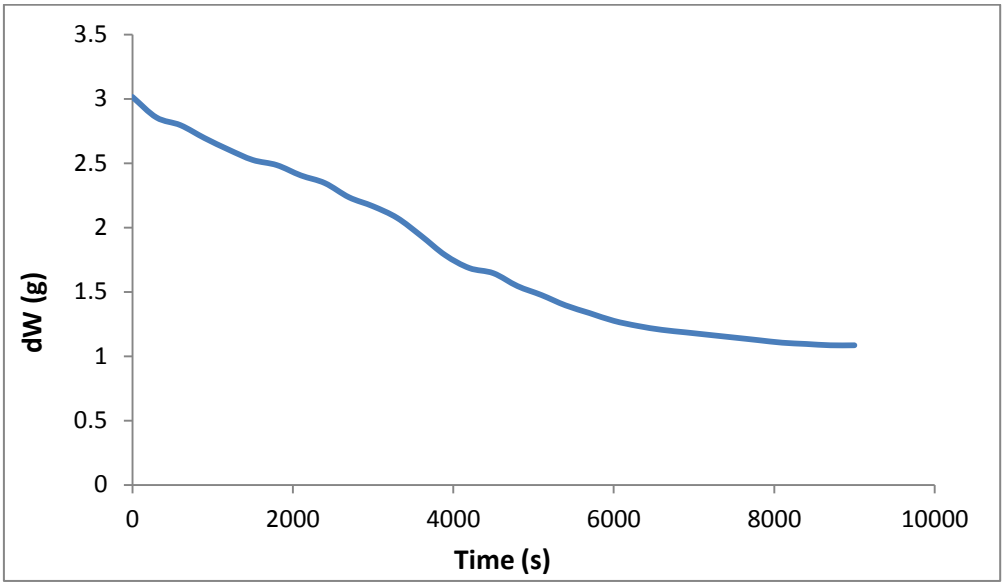


Figure 5.1: Change of weight vs. time (OPODFD using SCSH) for Experiment set-1.

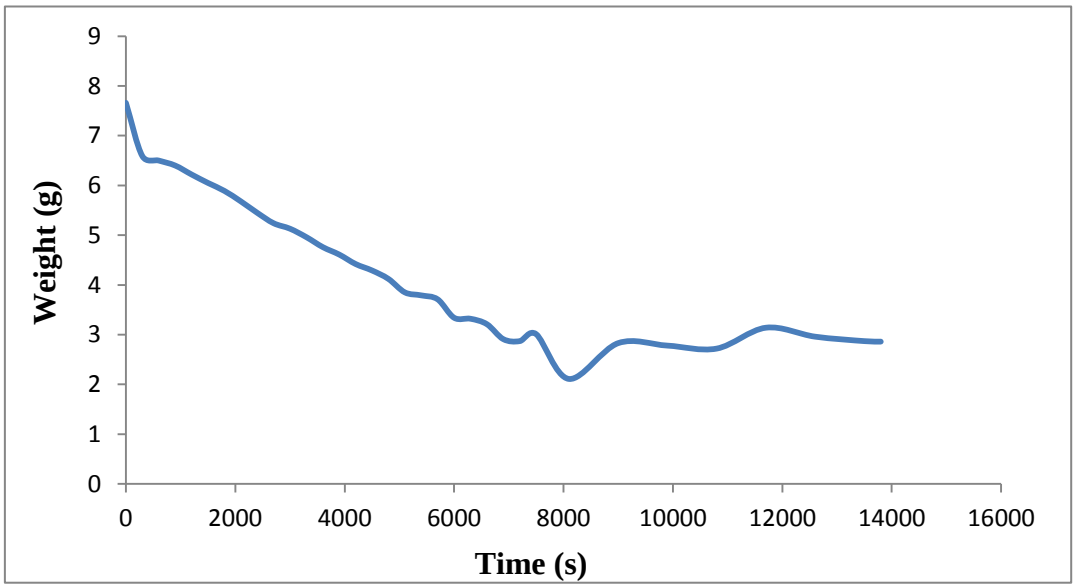


Figure 5.2: Change of weight vs. time (OPODFD using SH) for Experiment Set-1.

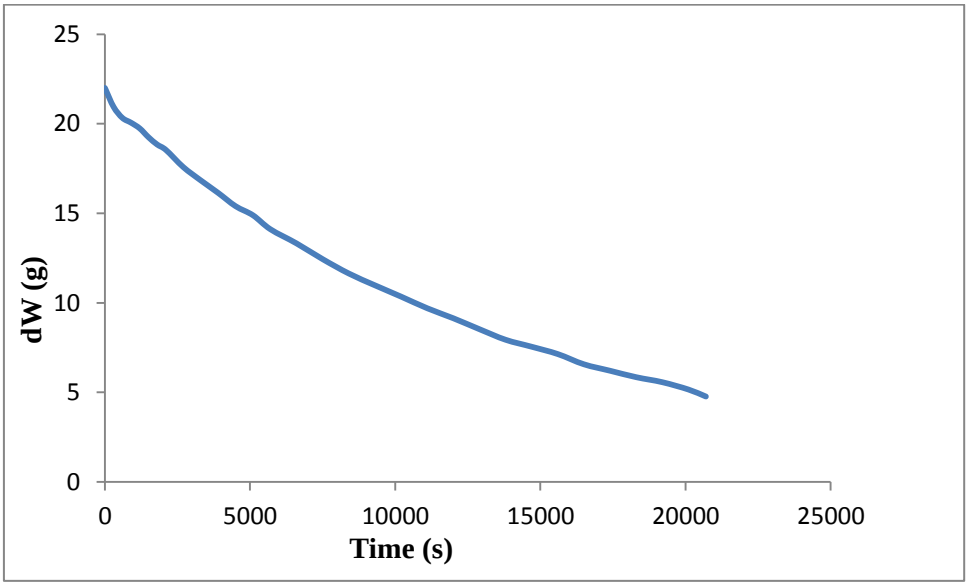


Figure 5.3: Change of weight vs. time (CFD using SCSH) for Experiment set-1.

In Figure 5.4, 5.5 and 5.6, the plate temperature and ambient temperature (inside freeze-dryer during drying) versus time of drying were shown using SCSH for OPODFD process, SH for OPODFD process and SCSH for CFD process respectively. For all the cases, the plate temperature initially decreased since the frozen product (contacted with plate surface) lowered down the plate surface temperature. In all the graphs the ambient temperature gradient (ΔT) increased very less during drying process. But the plate temperatures for OPODFD process using either SCSH or SH were fluctuating- it means the heat penetrates through the pores of foodstuffs have had some resistance but for CFD process (without OD) the heat penetrates with very minimum resistance i.e. OD have a significant effect on heat transfer during drying and this unsteady state heat transfer may leads to decrease the drying time of osmotic dehydrated kiwi fruits. By using SCSH for OPODFD process the plate temperature reached equilibrium with ambient temperature after 1200 seconds and by using SH for OPODFD process the plate temperature reached equilibrium with ambient temperature after 1800 seconds; it means SCSH had good significant role for heating than SH.

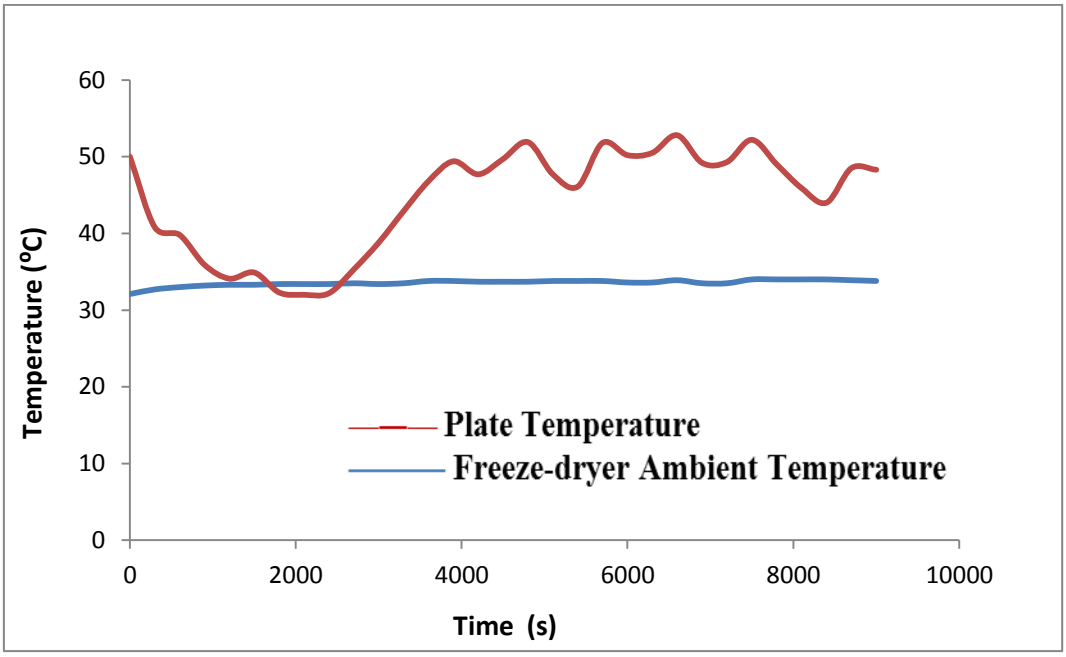


Figure 5.4: Plate temperature and Freeze-dryer (inside) ambient temperature vs. time (using SCSH for OPODFD process).

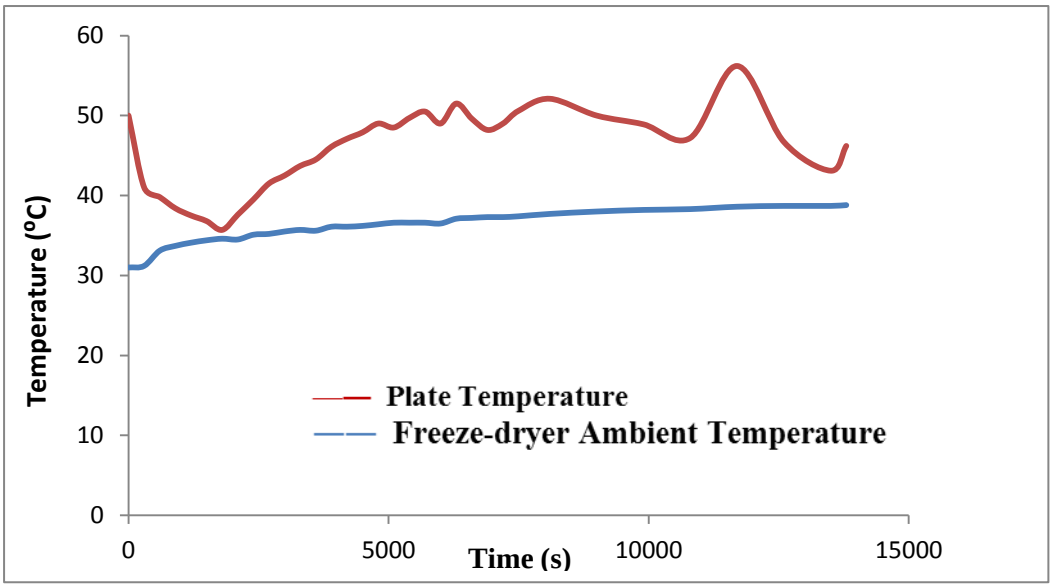


Figure 5.5: Plate temperature and Freeze-dryer (inside) ambient temperature vs. time (using SH for OPODFD process).

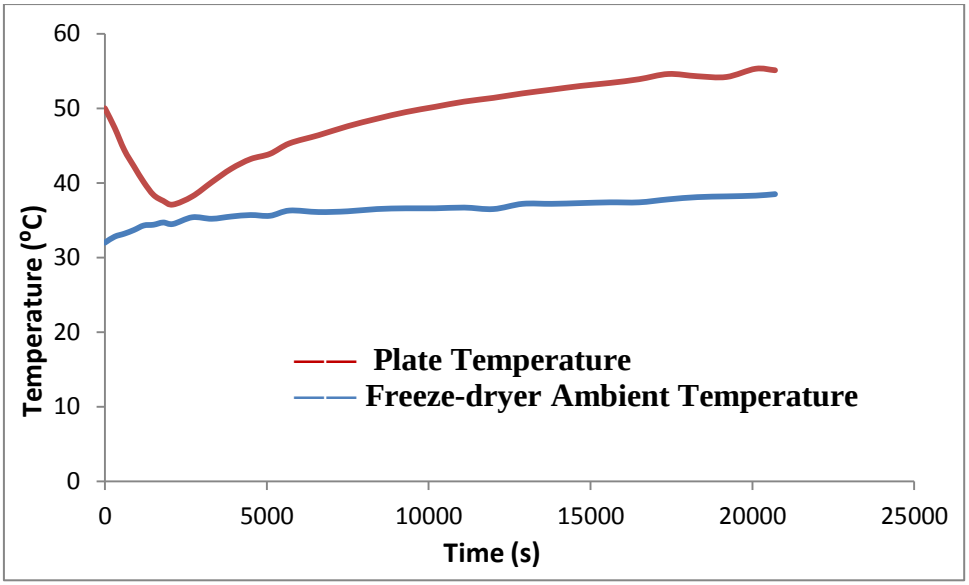


Figure 5.6: Plate temperature and Freeze-dryer (inside) ambient temperature vs. time (using SCSH for CFD process).

5.1.3 Comparison between Sucrose Solution (SS) and Fructose Solution (FS) used as an Osmotic Solution

The KF sample was also dehydrated through fructose solution (used as an osmotic solution, OS) at derived optimized conditions (40 Bx, 50°C and 3 h). Under the optimized conditions in OD the WL was 54.54% and SG was 14.45% which were much inferior to OD with SS (WL: 60.75% and SG was 9.86%). Thus, nearly10% and 32% better performance on the basis of WL (%) and SG (%) for SS as compared to FS. Therefore, it may be concluded that at optimized osmotically dehydrated (OPOD) condition, the sucrose solution (SS) was far better osmotic solution (OS) than fructose solution (FS).

5.1.4 Effects of Individual Factors on Water Loss (WL)

It was evident from Figure 5.7 that an increase in brix can cause decrease in WL% which was unfavorable; while increase in temperature from 45^0C to 50^0C could result an increase in WL. An interesting observation pertaining to the effect of OD time on WL revealed that an increased in OD time from 2 to 3 h could render a significance in WL; however further increase in OD time had contrasting effect on WL. This may be ascribed to the reverse osmosis during extended period of OD. It was found from the

results of ANOVA (Table 5.8) that all the factors (brix, time and temperature) were significant but temperature was the most important (rank 1) among all with maximum delta value (1.66).

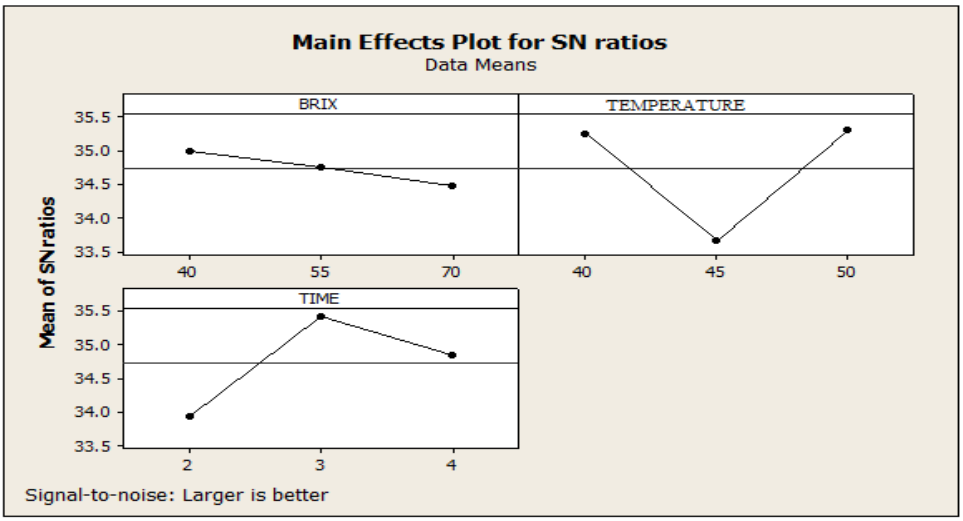


Figure 5.7: Effect of individual process factors on signal-to-noise (S/N) ratio for water loss (WL).

Table 5.8: Effects of individual factor on OD interpreted through ANOVA (analysis of variance) [for WL, larger is better].

Level	**Brix (^{0}B)**	**Temperature (^{0}C)**	**Time (h)**
1	35.01	35.25	33.95
2	34.78	33.67	35.46
3	34.47	35.33	34.85
Delta	0.54	1.66	1.50
Rank	3	1	2

5.1.5 Interaction among Process Factors in Governing WL

It is evident from Figure 5.8, that an increase in temperature from 40°C to 45°C resulted a decrease in WL% for all brix values of osmotic solution (OS). However, increase in temperature from 45°C to 50°C resulted increment in WL% for 40° Bx and 70° Bx. Again at 50°C, the WL% was more as compared with other two temperatures (40° and 45°C). Also at 3 hour time the WL% reached maximum as compared with

other two time parameters (2 h and 4 h). Therefore, to get WL% maximum the optimized process parameters were 40 Bx, 50°C and 3 h.

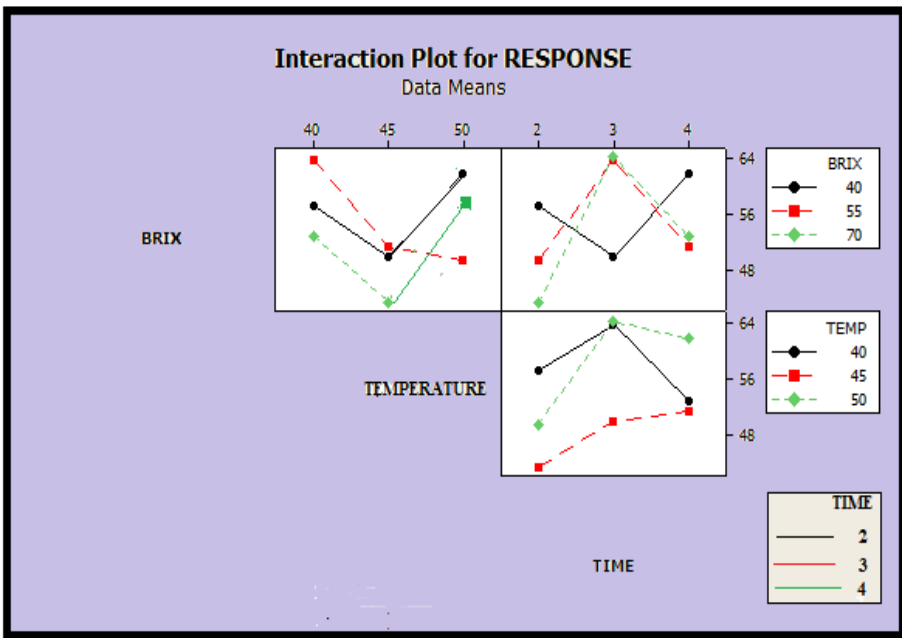


Figure 5.8: Interaction plot among process factors governing in water loss (mean) in osmotic dehydration of kiwi fruit.

5.1.6 Kinetics of Osmotic Dehydration of Kiwi Fruit Sample

The OD kinetics at optimal OD (OPOD) was compared with four models namely Page, Wang & Sing, Henderson & Pabis and Newton model (Table 5.9). Different constant values (k, a, b, n), regression coefficient (R^2) values, modeling efficiency (EF), mean bias error (MBE) and root mean square error (RMSE) values are required (Table 5.10) to evaluate the best representative model. The Newton model was accepted as the best drying models for OPOD. The moisture ratio (MR) values as a function of OD time were compared for actual (experimental) and predicted values for all the four models in Figure 5.9.

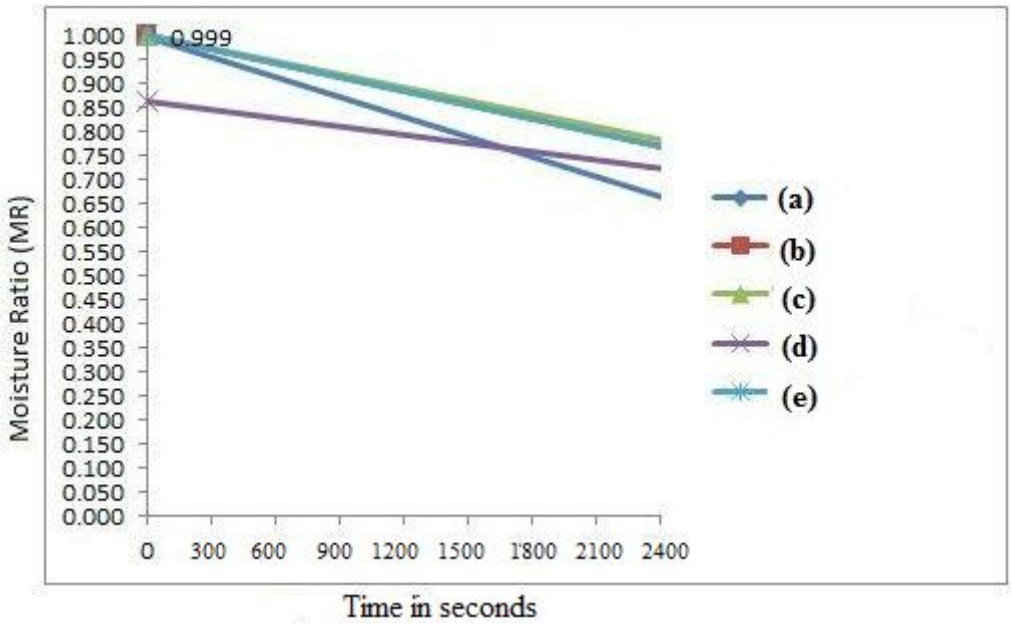


Figure 5.9: Osmotic dehydration curve at optimal conditions of OPOD. (a) Experimental value, (b) Page model, (c) Wang and Sing model, (d) Henderson and Pabis model, (e) Newton model.

Table 5.9: Mathematical models selected for Experiment -1 and Experiment-2.

Serial no.	**Model name**	**Equation used**
1.	Page[#]	$MR = \exp(-kt^n)$
2.	Wang & Sing[#]	$MR = 1 + at + bt^2$
3.	Henderson & Pabis[#]	$MR = a \exp(-kt)$
4.	Newton[#]	$MR = \exp(-kt)$

Reference:

[#]Chakraborty *et al.* (2011).

Table 5.10: Models for osmotic dehydration at optimal conditions (OPOD).

Model	**K**	**a**	**b**	**N**	**R^2**	**EF**	**MBE**	**RMSE**
Page	0.017	___	___	0.783	0.913	0.88934	0.06774	0.00769
Wang & Sing	___	-0.006	9.8×10^{-6}	___	0.965	0.82705	-0.00300	0.10965
Henderson & Pabis	0.00461	0.865	___	___	0.9745	0.83888	0.04168	0.10860
Newton	0.007	___	___	___	0.9444	0.90335	0.01708	0.08412

5.1.7 Freeze-Drying of Kiwi Fruit after OPOD

Partially dehydrated kiwi samples (30.12% on wet basis) developed through optimized OD operation (OPOD) were subsequently subjected to freeze-drying (OPODFD) using silver coated steel heater (SCSH) till the final moisture content became ≤ 5.0 % (wet basis). The actual (experimental) OPODFD kinetics obtained by plotting (Figure 5.10) moisture ratio (MR) as a function of time was compared with four standard drying models viz. Page, Wang & Sing, Henderson & Pabis and Newton model (Figure 5.10) to determine the best representative model for OPODFD dynamics. Regression coefficient (R^2), MBE, RMSE and EF values and constants (namely k, a, b, n) are presented in Table 5.11.

Table 5.11: Freeze-drying models for osmotically dehydrated kiwi samples at optimal conditions (OPODFD).

Model	**k**	**a**	**b**	**N**	**R^2**	**EF**	**MBE**	**RMSE**
Page	0.035	—	—	1.1	0.947	0.96577	0.03093	0.05773
Wang & Sing	—	-0.042	0.00048	—	0.946	0.98062	-0.00185	0.04343
Henderson & Pabis	0.0501	1.015	—	—	0.928	0.96203	0.02170	0.06080
Newton	0.0452	—	—	—	0.940	0.72485	0.04359	0.07370

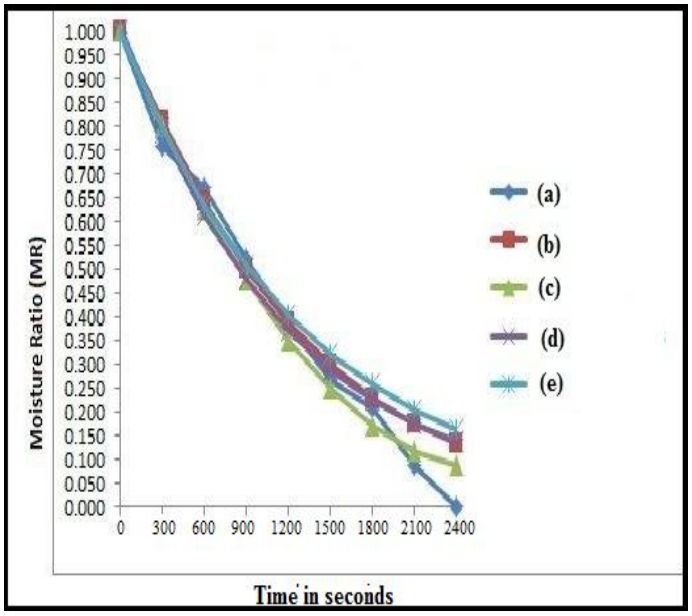


Figure 5.10: Freeze-drying curves of OPODFD. (a) Experimental value, (b) Page model, (c) Wang and Sing model, (d) Henderson and Pabis model, (e) Newton model.

From the Table 5.11 and Figure 5.10 it can be observed that Wang & Sing model was best fitted as compared with other three models i.e. it was the best representative drying model for freeze-drying kinetics of kiwi fruit sample.

5.1.8 Comparison of Freeze-Drying Performances between SCSH and SH

From Eq. (4.8), the effective moisture diffusivity (D_{eff}) for OPOD was found as 3.405×10^{-9} m^2/s (Table 5.12). Similarly, the effective moisture diffusivity in OPODFD (using SCSH) of KF was found 6.04×10^{-7} m^2/s (Table 5.12). On the other hand, the effective diffusivity for OPODFD (using conventional steel heater) was found as 2.22×10^{-7} m^2/s (Table 5.12) Therefore, SCSH was much more efficient than SH for accelerating OPODFD of kiwi fruit.

Table 5.12: Effective diffusivity for optimal osmotic dehydration (OPOD), optimal freeze-drying of osmotically dehydrated (OPODFD) kiwi samples using steel heater (SH) and silver coated steel heater (SCSH).

Temperature	**D_{eff} for OPOD (m^2 / s)**	**D_{eff} for OPODFD (m^2 / s)**	
		Using SH	**Using SCSH**
50^0C	3.405×10^{-9}	2.22×10^{-7}	6.04×10^{-7}
45 ^{0}C	1.886×10^{-9}	0.57×10^{-7}	1.50×10^{-7}
40 ^{0}C	1.045×10^{-9}	0.157×10^{-7}	0.37×10^{-7}

To attain a final moisture content (4.27%) of kiwi fruit sample the drying time was 2.5 h and 3.82 h for SCSH and SH respectively. Therefore, the drying time for SCSH was less (about 35%) than SH.

Chakraborty and Samanta (2015) reported about the effective diffusivity values (D_{eff}) for osmotic drying (OD) and vacuum drying (VD) in the range of 4.053×10^{-9} to 7.56×10^{-9} m^2/s. In Table 5.12, the D_{eff} values for OPOD are found 1.045×10^{-9} to 3.405×10^{-9} m^2/s. But using either Steel heater (SH) or Silver coated steel heater (SCSH), the D_{eff} values are found greater in the range of 1.57×10^{-8} to 6.04×10^{-7} m^2/s. The values were found greater due to the introduction of novel heater for supplying sublimation heat to the optimized osmotically dehydrated frozen materials. So, grater moisture diffusivity (D_{eff}) was found during drying.

The activation energy (E) by using Arrhenius Equation (4.9) was calculated. The activation energy required during OPODFD was computed as 23.96 kJ/mol by using SCSH; on the contrary, the activation energy required during OPODFD was 53.55 kJ/mol when using SH plate. Therefore, less activation energy is required for OPODFD by SCSH as compared with SH; indicating SCSH as an energy-efficient heating device.

For conventional freeze-drying (CFD) without OD, the drying time was required 5.75 h for KF sample to reach final moisture content 4.27%.

Table 5.13: Qualities of raw and dehydrated kiwi samples.

Quality Parameters	**Standard # values for fresh KF***	**Tested fresh KF**	**KF samples for OPOD**	**KF samples for OPODFD**	**Test method**	**Reference(s)**
Moisture (g/100g)	70-85	85.07	68.16	4.27	DGHS LAB MANUAL 5.0	Krokida et al., 1998
Carbohydrate (g/100g)	14.66	10.89	27.31	87.52	IS:1656-2007	fssai, act 2006, GOI; Maskan, 2001
Sugar (g/100g)	8.99	8.30	12.83	56.78	IS:6287-1985 (Reaffirmed-2005)	_____
Protein (g/100g)	1.14	1.31	1.34	2.82	IS:7219-1973 (R.A.-2005)	_____
Total Fat (g/100g)	_____	0.52	0.55	0.65	DGHS LAB MANUAL 5.0	_____
Energy Content (kJ/100g)	260	259.8	512.76	1548.64	IS:9487-1980 (R.A.-2005)	fssai, act 2006, GOI
Sodium (mg/100g)	3	2.81	432.48	1072.04	QA.16.5.2	fssai, act 2006, GOI
Potassium (mg/100g)	312	312.53	152.51	252.02	QA.16.5.2	fssai, act 2006, GOI; Deman, 2004
Ash (g/100g)	0.88-1.02	1.12	1.86	3.92	DGHS LAB MANUAL 5.0	fssai, act 2006, GOI; Fourie and Hansmann, 1992
Vitamin A (µg/100g)	Trace	20.0	20.0	20.0	QA.16.5.3	fssai, act 2006, GOI; Mudambi and Rajagopal, 1990
Vitamin C (mg/100g)	90	89.10	80.55	78.66	AOAC 18-th edition, 967.21	fssai, act 2006, GOI

#FSSAI *****Information on nutritional value of Fresh Kiwi

5.1.9 Quality Assessment of Raw and Dehydrated Kiwi Fruit

According to nutrition information panel (fssai) every food processing operations require to provide information on certain nutrients. Eleven quality tests were performed for three types of kiwi samples namely 'Raw', OPOD and OPODFD (final dried), shown in Table 5.13, where the reference of methods are also mentioned. The quality of all the three types with respect to the five parameters viz. moisture, carbohydrate, protein, total fat and ash content (in g per 100 g sample) were evaluated which indicated satisfactory quality.

Table 5.13 shows that carbohydrate increased by 150.78% and sugar increased by 54.58% for OPOD sample. The reason was water expulsion and sugar uptake for KF samples. Increment behavior of carbohydrates and sugars were observed for final dried samples obtained through OPODFD owing to additional moisture removal. All the dehydrated samples contained more protein than raw samples per unit mass of sample (Table 5.13). Previously, no report of fat content was made for KF. However, 0.52 g, 0.55 g and 0.65 g fat (per 100 g tested sample) was found for raw (fresh) KF, OPOD and OPODFD samples respectively. However, the temperature was kept within 50^0C therefore no loss of fats occurred due to processing. The energy content were found higher than raw sample for OPOD (512.76 kJ per 100 g) and OPODFD (1548.65 kJ per 100 g) samples. Hence, it could be concluded that the final dried kiwi sample (100g) was an energy 'store-house'. Both sodium and potassium present in kiwi were lost whenever thermally processed (fssai). Plant and their products have a higher content of potassium than of sodium (Karpagavalli *et al.*, 2014). Here, during OPOD kiwi was heated at 50^0C, accordingly sodium and potassium might be lost. To overcome this situation iodized common salt (5%, w/v) was added. Sodium content increased during OPOD, while potassium content decreased during OPOD, since it was 'salting out' quickly to the sucrose solution. The increase in potassium content from OPOD sample to OPODFD sample could be ascribed due to additional moisture reduction leading to final moisture content of 4.27%. By the same reason, the quantity of sodium for freeze-dried product also increased. Although, the dried KF sample contained less vitamin C (nearly 12 %) as compared to raw (fresh) tested KF. None the less, loss in vitamin C reported in present study was much less compared to available reports. The raw kiwi contains ash 0.88 to 1.02 g per 100 g sample (Deman,

2004). So, initial load of ash in tested KF sample was more as compared with standard value; as expected, the ash content both in OPOD and OPODFD sample increased per 100 g of dried product. Structural properties of foods are strongly affected by material moisture content (Fourie and Hansmann, 1992), therefore high moisture level (>80%) suggests that kiwi fruit was very much suited for FD preservation. It was found that, all the carbohydrate and sugar level would be back to original level (raw kiwi) when rehydration step would be performed (Maskan, 2001). Ripen kiwi is a good source of β-carotene. The daily requirement of an adult for vitamin A is of the order of 750 µg of retinol or 3000 µg of β-carotene per day derived from either animal or plant origin (Krokida *et al.,* 1998) . Table 5.13 shows that in processed kiwi vitamin A found 20 µg/100g of sample though raw kiwi contains trace amount of vitamin A as per fssai.

The water activity (a_w) found for the final OPODFD sample was 0.58 (<0.61). This value suggested that OPODFD sample has good shelf life and storability. It is confirmed by microbial test that less microbial load (total plate count was less than 10^3 log cfu cm^{-3}) was found in OPODFD sample.

5.2 EXPERIMENT SET-2: VALUE ADDED KIWI FRUIT POWDER

Here, the KF was value added by few major and minor additives and the results of moisture content at three stages viz. fresh KF, BKF and BFDKF powder were determined to be 82%, 68% and 3% (<5%) respectively on wet basis. The drying kinetics of freeze-dried BKF sample, effective diffusivity and required activation energy during BKF drying were investigated. The quality and sensory analysis of final dried product (BFDKF) was analyzed and reported under this section.

5.2.1 Kinetics of Blended Freeze-Dried Kiwi Fruit (BFDKF) Powder Sample

The kinetics of freeze-dried BKF sample (BFDKF) with four drying models were investigated (Table 5.9). Different model constant values (k, a, b, n), regression coefficient (R^2) values (using Microsoft Excel), modeling efficiency (EF), mean bias

error (MBE) and root mean square error (RMSE) values were calculated (Table 5.14) to evaluate the best representative model.

Table 5.14: Freeze-drying model constants for Blended kiwi Fruit (BKF) samples.

Model	**K**	**a**	**b**	**n**	**R^2**	**EF**	**MBE**	**RMSE**
Page	0.02	—	—	0.95	0.963	0.84243	0.00453	0.12605
Wang & Sing	—	-0.044	0.00028	—	0.926	-4.6046	-0.69806	0.75175
Henderson & Pabis	0.0112	1.002	—	—	0.985	0.66665	0.11047	0.18334
Newton	0.0155	—	—	—	0.973	0.84525	0.02348	**0.12492**

Regression coefficient is a statistical measure of the average functional relationship between two or more variables. The predicted regression coefficient (R^2_{pred}) values for all the four models are compared with the experimental value (R^2_{exp}= 0.9902) shown in Table 5.15.

Table 5.15: Comparison of Regression coefficients (R^2) for all four models with the experimental Regression coefficient (R^2_{exp}).

Model	**Predicted R^2** (R^2_{pred})	**Experimental Regression coefficient** (R^2_{exp})	**Difference** (R^2_{exp}- R^2_{pred})	**Deviation** (%)
Page	0.9634	0.9902	0.0268	2.7065
Wang & Sing	0.9261	0.9902	0.0641	6.4734
Henderson & Pabis	0.9854	0.9902	0.0048	0.4847
Newton	0.9726	0.9902	0.0176	1.7774

The R^2_{pred} values were not much deviated from R^2_{exp} values except Wang & Sing model. That means the experimental error was less. The highest R^2 and EF values are better for accepted drying model. Simultaneously, the lowest MBE and RMSE values are better for accepted drying model. What is more, the RMSE is more appropriate to

use than MBE when model errors follow a normal distribution (Chai et al., 2014). From Table 5.14, it was shown that RMSE value was least (0.12492) for the Newton model as closely compared with Page model (RMSE = 0.12605). Kept in mind all the considerations, the Newtonmodel was accepted as the best drying model as compared with other models for FD of BKF. Compared regression coefficient (R^2) for experimental value (R^2_{exp}= 0.9902) and Newton model (which is R^2_{pred} = 0.9726 ≈ 0.973), shown in Figure 5.11. In Figure 5.12, the moisture ratio (MR) values were compared for actual (experimental) and predicted values for all the four models. It was found that Wang & Sing model was far away from the experimental curve, but other models namely Page, Henderson & Pabis and Newton model were nearly closed to experimental curve (Figure 5.12).

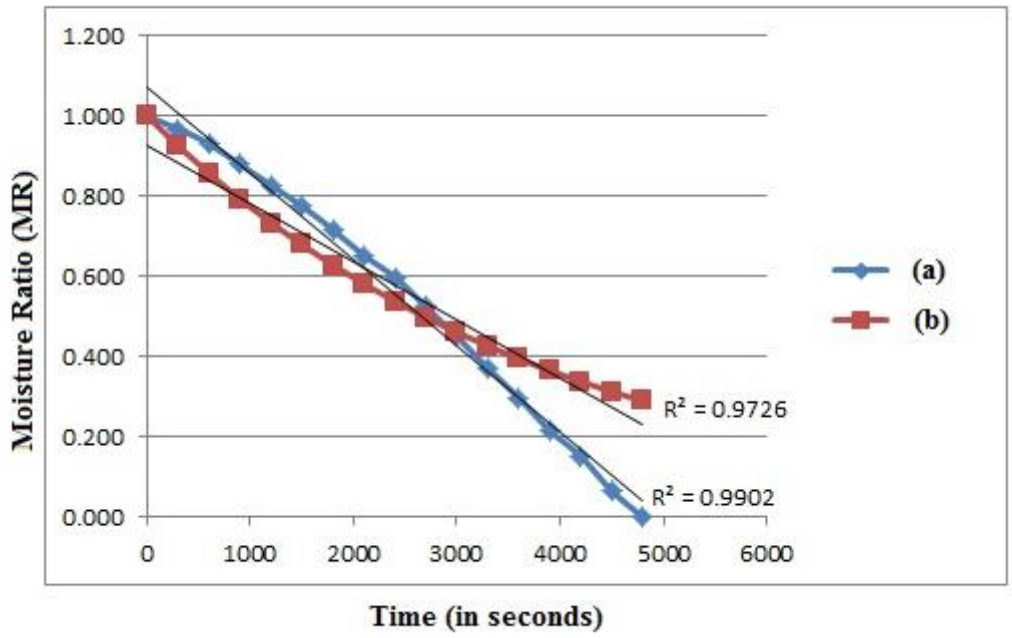


Figure 5.11: Graph of Moisture ratio (MR) vs. time to compare the regression coefficient (R^2) for (a) Experimental value (R^2_{exp}= 0.9902) and (b) Newton model (R^2_{pred} = 0.9726).

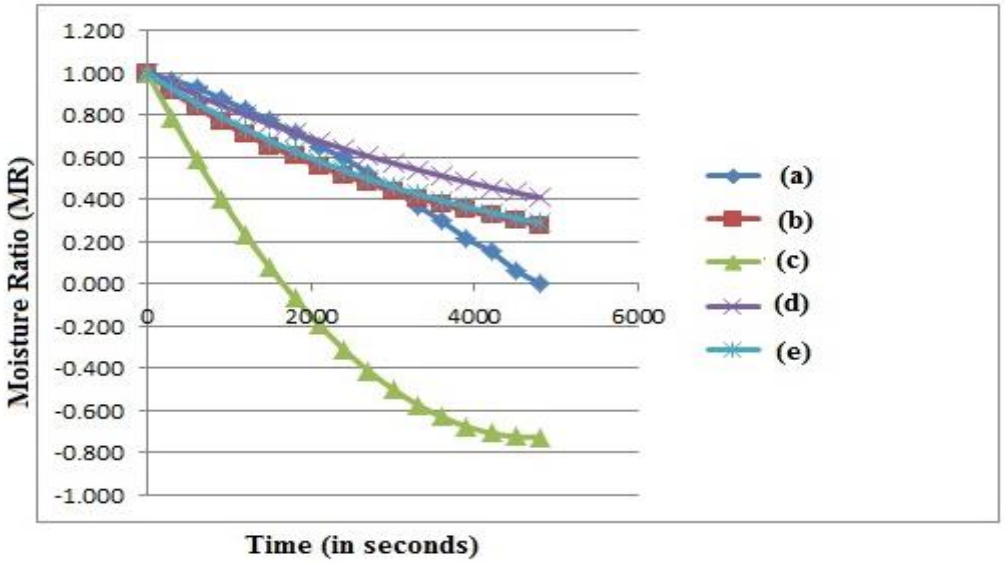


Figure 5.12: Freeze-drying curves of BFDKF sample for (a) Experimental value. (b) Page model. (c) Wang & Sing model. (d) Henderson &Pebis model. (e) Newton model.

5.2.2 Freeze-Drying Performance by Using Cubic Steel Heater

From Equation (4.8), the effective moisture diffusivity (D_{eff}) for freeze-dried blended kiwi Fruit (BKF) sample was found. The values of D_{eff} were calculated in different temperatures shown in Table 5.16.

Table 5.16: Effective diffusivity and activation energy for freeze-drying of Blended kiwi Fruit (BKF) samples using cubic steel heater (CSH).

Temperature (^{0}C)	**D_{eff} for FD of BKF**(m^2 / s)	**Activation Energy (E) required for FD of BKF** (kJ / mol)
40	1.892×10^{-7}	28.35
45	5.366×10^{-7}	
50	1.532×10^{-6}	

At 50^{0}C (material temperature), the value was found highest of 1.532×10^{-6} m^2 / s. To attain final moisture content (3%) of BFDKF sample the drying time was 4.5 h. In Table 5.16, for drying of BKF sample the D_{eff} values were found greater in the range

of 1.892 x 10^{-7} to 1.532 x 10^{-6} m^2/s. Since, multidimensional cubic steel heater is used for sublimation purpose so greater moisture diffusivity was found during drying.

By using Arrhenius Equation (4.9), the activation energy (E) was calculated. The graph of ln (D_{eff}) vs. 1/T found the activation energy (E) shown in Figure 5.13. The R^2 value of the line is 0.9565, which is satisfactory value for fitting a line. Hence, the activation energy required during FD for BFDKF sample was found 28.35 kJ/mol by using the equation developed (y = - 235.73 x + 59.513) on the graph (in Figure 5.13).

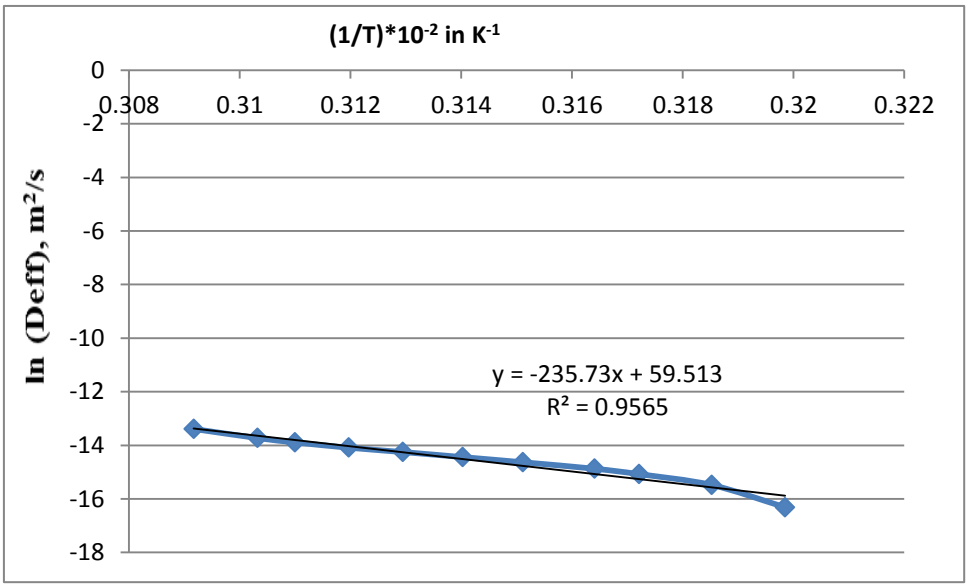


Figure 5.13: The graph of ln (D_{eff}) vs. (1/T) for Freeze-drying of BFDKF sample to find activation energy (E).

By using same cubic heater, for conventional freeze-drying (CFD) without blending (i.e. without added ingredients to KF) the drying time was found 7 h to reach same final moisture content 3%.

5.2.3 Quality Assessment of Blended KF (BKF) and Blended Freeze-Dried KF (BFDKF)

Table 5.17: Proximate analysis of raw and kiwi fruit powder sample.

Quality Parameters	**Standard # values for fresh KF***	**Tested fresh KF**	**KF samples for OPODFD**	**KF samples for BFDKF powder**	**Test method**	**Reference(s)**
Moisture (g/100g)	`70-85	85.07	4.27	3.0	DGHS LAB MANUAL 5.0	Krokida et al., 1998
Carbohydrate (g/100g)	14.66	10.89	87.52	72.17	IS:1656-2007	fssai, act 2006, GOI; Maskan, 2001
Total Sugar [Glucose & Fructose] (g/100g)	8.99	8.30	56.78	41.48	IS:6287-1985 (Reaffirmed-2005)	_____
Protein (g/100g)	1.14	1.31	2.82	5.96	IS:7219-1973 (R.A.-2005)	_____
Total Fat (g/100g)	_____	0.52	0.65	2.53	fssai LAB MANUAL 5.0	_____
Energy Content (kJ/100g)	260	259.8	1548.64	1428.22	IS:9487-1980 (R.A.-2005)	fssai, act 2006, GOI
Sodium (mg/100g)	3	2.81	1072.04	969.69	QA.16.5.2 / AOAC 19-th Edition	fssai, act 2006, GOI
Potassium (mg/100g)	312	312.53	252.02	953.72	QA.16.5.2 / AOAC 19-th Edition	fssai, act 2006, GOI; Deman, 2004
Vitamin A (µg/100g)	Trace	20.0	20.0	_____	QA.16.5.3	fssai, act 2006, GOI; Mudambi and Rajagopal, 1990
Vitamin C (mg/100g)	90	89.10	78.66	193.90	IS: 5838-1970 (RA-2005)	fssai, act 2006, GOI

fssai ***** information on nutritional value of fresh kiwi fruit

The previous work (Experiment Set-1) related on optimization of osmotically dehydrated freeze-dried (OPODFD) kiwi sample and present work (KF samples for BFDKF powder) was compared neck-to-neck. The carbohydrate and sugar content both were reduced for BFDKF powder. This is due to the no solute gain (for osmotic

dehydration) was happened. Only 1% (w/w) sugar was added during blending operation directly to the sliced KF. Since oat flour (contain 12% protein, w/w) was fortified to the sliced KF, so final BFDKF powder contain two times more protein than OPODFD process. For the same reason (oat flour contain 8% w/w total fat viz. saturated fatty acids 1.5%, MUFA 4% and PUFA 2.5% w/w), the final BFDKF powder contains six times total fat than OPODFD process. Already mentioned that the salt added to the sliced KF in the form of iodized and lona (1: 1 ratio), so final product contain low sodium and high potassium as compared with OPODFD process, which is acceptable for human health. The final powdered product is low energy high nutritional valued product suggested by low energy value (1428.22 kJ per 100 g) as compared with OPODFD process (1548.64 kJ per 100g). One great foundation on the basis of maximum vitamin C (193.9 mg per 100 g) retained on this process. Here, final product contains more vitamin C as compared with OPODFD process (78.66 mg per 100 g). The reason is in OPODFD process the optimized osmotic dehydration was performed for 3 hours at 50°C. Naturally more vitamins would be lost during this. Here, no heat treatment was done before freezing of blended sample. So, as compared the conventional FD technique in this present work more vitamin was retained. The testing of vitamin A was not performed since raw KF contain trace amount of it. Few physico-chemical properties such as titrable acidity, TA (5.92 g per 100 g sample as per malic acid), crude fiber (5.99 g per 100 g) and ash (7.88 g per 100 g) were also tested as shown in Table 5.18. Since, sugar-acid ratio plays an important part or role in the flavor of final product (Deman, 2004), so TA is a major issue for vitamin C rich fruit. The raw KF have a TA of 0.78 milli-eqivalent (as per malic acid) per 100 g KF sample and pH value 3.6 (Fattahi *et al.*, 2010). So, raw KF has TA value of 11.64 g (as per malic acid) per 100 g KF sample. Therefore, sourness of finished BFDKF powder was less as compare with raw KF. Naturally, sugar-acid ratio (calculated based on total sugar % and milli-equivalent factor of malic acid = 0.067) is increased from 10.64 to 104.57 (almost ten times) since total sugar % (as per glucose and fructose) increased (from 8.3 to 41.48% in g per 100 g). Hence, the flavor quality of final product was increased (expected) by ten times as compared with raw KF. As compared with raw KF, BFDKF powder had two times more fiber. One of the beneficial effects of fiber is its bulking capacity and the water holding capacity of the finished product (Deman, 2004). So, during consumption of the powder it may add water either directly or indirectly. On that time it will more beneficial for water

addition since the BFDKF powder contains almost 6% fiber. The ash content of final product is more as compared with OPODFD since it is fortified with many ingredients which are very rich in minerals such as magnesium, calcium, sodium, potassium etc. Structure of foods are strongly affected by material moisture content (Krokida *et al.*, 1998), so that kiwi fruit was very much applicable for FD preservation since it contains high moisture content (>80%). It was found that, all the carbohydrate and sugar level would be back to original level (raw KF) when rehydration step would be performed (Maskan *et al.*, 2001).

The water activity (a_w) found for the final BFDKF sample was 0.52 (<0.61). This value suggested that BFDKF powder has good shelf life and stability. It is confirmed by microbial test that less microbial load (total plate count was less than 10^3 log cfu cm^{-3}, coliforms < 10 cfu cm^{-3}, and *E. coli* was 0 cfu cm^{-3} and *Salmonella* spp. was 0 cfu 25 cm^{-3}) was found in BFDKF powder.

Table 5.18: Physico-chemical properties of freeze-dried kiwi fruit (BFDKF) powder.

Sample type	**Standard # values for fresh KF***	**BFDKF powder**	**Test method**	**Reference(s)**
Titrable acidity (g / 100 g) (as per malic acid)	11.64	5.92	fssai Lab Manual	Deman JM., 2004.
Crude fiber (g / 100 g)	2.9	5.99	IS: 10226 (Part I)-1982 (RA-2005)	Deman JM., 2004.
Total ash (g / 100 g)	0.88-1.02	7.88	fssai Lab Manual	fssai, act 2006, GOI; Fourie and Hansmann, 1992

fssai ***** information on nutritional value of fresh kiwi fruit

5.2.4 Sensory Evaluation

On the basis of seven indicators viz. Sweetness, Sourness, Saltiness, Bitterless, Flavor, Texture, Overall acceptability total 66 participants / panelists were given their

comments and hence given the points (1 to 9) for the BFDKF powder. The % of consumer acceptance, average score and total score were calculated for individual indicator by using Equations (4.12), (4.13) and (4.14) respectively and they were shown in Table 5.19. From Table 5.19, it was found that % of consumer acceptance was 100% for saltiness and bitterless i.e. these two indicators were well acceptable by the consumers but sweetness (68.18%) and texture (77.27%) were less acceptable. Again, sourness (90.91%) and flavor (95.45%) were almost satisfactory by the consumer acceptance. Total 54 panelists out of 66 panelists were given >5 grade points for overall acceptability. Therefore, percentage of consumer acceptance for overall acceptability of BFDKF powder became 81.82% shown in Table 5.19. As compared with previous food products (fruit juices, lemon juices etc.) survey (Singh-Ackbarali and Kitsawad, 2014; Krasaekoopt *et al.*, 2010), where the acceptance was 83.3%, the BFDKF powder acceptance value (%) was not far away from the surveyed results. On the other hand both average score (8.82) and total score (97.98) were highest for bitterless shown in Table 5.19.

Table 5.19: Judgement of different organoleptic tests & overall test by 66 panelists with 9-Hedonic points [1 (= dislike extremely) to 9 (= like extremely)].

	Sweetness	**Sourness**	**Saltiness**	**Bitterless**	**Flavor**	**Texture**	**Overall acceptability**
% of consumer acceptance	68.18	90.91	100	100	95.45	77.27	**81.82**
Average score	5.32	6.11	6.95	8.82	7.32	7.11	6.45
Total score	59.09	67.93	77.27	97.98	80.30	79.04	71.72

From Table 5.19, the average and total score for sweetness and sourness were least (almost half of maximum level). The same scores for saltiness, texture and overall

acceptability were found less as compared with bitterless, whereas these scores for flavor were found satisfactory shown in Table 5.19. So, both sweetness and sourness must have to improve in future work for betterment of BFDKF powder. For further betterment of saltiness (average score and total score), flavor and texture also have to improve (towards like extremely point) for net improvement of consumers overall acceptability.

5.3 EXPERIMENT SET-3: FORTIFIED SHRIMP WITH TWO FISHES

Under this section the pH and moisture were analyzed before and after vacuum drying of two sets. The drying kinetics, effective diffusivity and the required activation energy were studied. Different quality parameters, rehydration ratio, ω-3 fatty acid content, TVBN and Hg level, stable shelf-life of finished products (two sets) were investigated.

5.3.1 pH and Moisture Analysis of Fish Samples before Drying

From Table 5.20 it was found that, after sterilization all fishes pH were increased. Increment of pH (by sterilization) is benefitted for no microbial growth. The pH of SBS was found 5.60 ± 0.03, which was found more acidic than three sterilized fishes. It suggests, the basic ions (here, Na^+ ions mostly from brine solution) were transferred (solute gain) from solution to all sterilized fishes.

Table 5.20: pH and moisture of fish samples at different stage of processing before and after vacuum-drying (VD).

pH and Moisture	**CA**		**CS**		**Shrimp**		**STSP**[†]	**SBM**[‡]	**FS**[§]**-1**	**FS-2**	**VDFS**[¶]**-1**	**VDFS-2**
	Raw	Sterilized	Raw	Sterilized	Raw	Sterilized						
pH ± SD[*]	5.88 ± 0.02	6.47 ± 0.03	5.80 ± 0.02	6.30 ± 0.03	6.40 ± 0.02	6.66 ± 0.02	6.32 ± 0.03	5.60 ±0.03	5.90 ±0.02	6.20 ± 0.03	6.63 ± 0.02	6.33 ± 0.02
Moisture (%) ±	78.11 ± 0.75	68.87 ± 0.66	74.81 ± 0.71	67.35 ± 0.64	77.15 ± 0.73	68.76 ± 0.65	69.12 ± 0.68	—	64.97 ± 0.62	64.34 ± 0.64	3.06 ± 0.25	3.06 ± 0.25

SD												

*SD: Standard deviation.

† Sterilized Shrimp-CA-CS pulp.

‡ Sterilized brine mix.

§ Fortified Shrimp and blended set.

¶ Vacuum-dried FS product.

The moisture % of the raw fishes found almost same and moderate moisture level which was good to handle for VD process. After sterilization, three cooked fishes moisture % were decreased, that meant water decrement and soluble matters were leached along with water from fishes to brine solution (SBS) which was used (added 14%, v/w in both sets) for blending processes. After blending, two sets of moisture level were notably dropped down from STFS which suggests the addition of CF, RF and DG helps to gain nutrition and to reduce moisture level of STFS.

5.3.2 Vacuum-Drying Kinetics, Models and Drying Time

Different drying model constants (K, A, B, C, n), different statistical criteria viz. R^2, RMSE, χ^2 and E% values of VD model fittings were computed using experimental results (shown in Table 5.21 and Table 5.22 for SPMDH and CPMDH respectively) to determine the best accepted model. It was found that, for FS-1 Linear (using SPMDH) and Modified Page (using CPMDH); also for FS-2 Modified Page (using SPMDH) and Newton (using CPMDH) models were accepted as the best VD models. The experimental MR varies with time of drying had been shown in Figure 5.14 and 5.15 for FS-1, FS-2 respectively using both SPMDH and CPMDH.

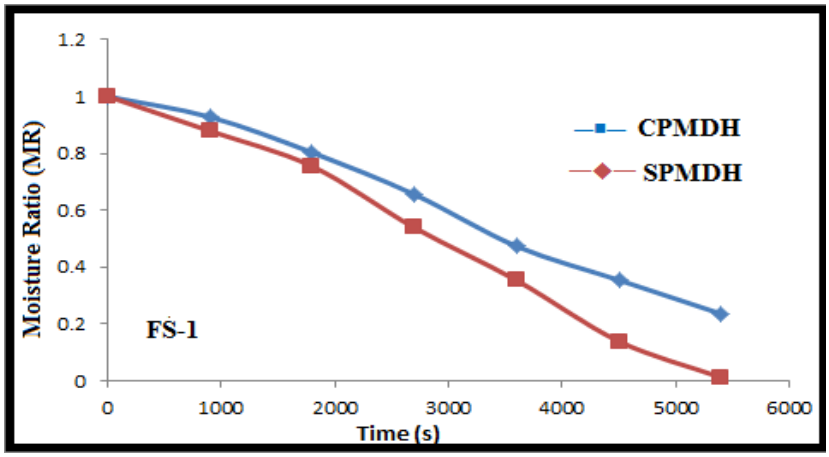


Figure 5.14: Moisture ratio (MR) vs. Time for the vacuum-drying of FS-1 sample using SPMDH and CPMDH.

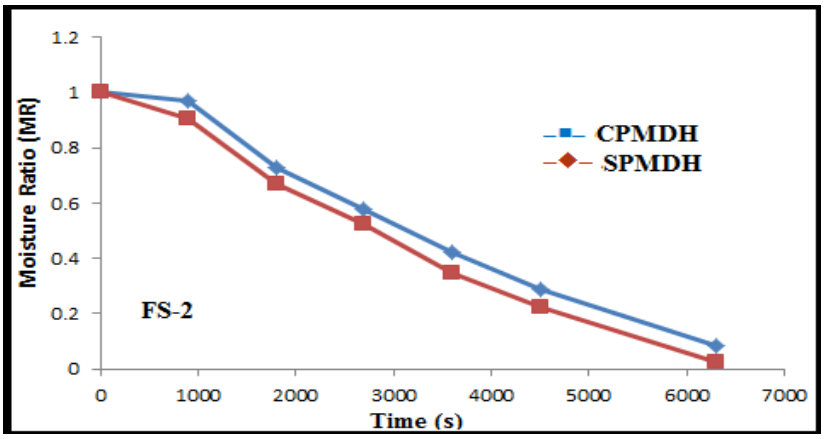


Figure 5.15: Moisture ratio (MR) vs. Time for the vacuum-drying of FS-2 sample using SPMDH and CPMDH.

TABLE 5.21: Vacuum-drying (VD) models for two sets of fish samples (FS-1 and FS-2) using SPMDH.

Model	**SET**	**K**	**A**	**B**	**C**	**N**	**R^2**	**RMSE**	**χ^2**	**E (%)**
	FS-1	0.0175					0.938	0.10886	0.0135	8.21

Newton	FS-2	0.02649	—	—	—	—	0.9005	0.19876	0.0460	14.49
Page	FS-1	0.0023	—		—	1.4919	0.9777	0.05195	0.0036	5.96
	FS-2	0.0006				1.8047	0.9888	0.05213	0.0038	2.30
Henderson & Pabis	FS-1	0.0204	1.5666	—	—	—	0.9179	0.22503	0.0675	37.81
	FS-2	0.0281	1.9044				0.8904	0.96561	0.1865	35.16
Modified Page	FS-1	0.0140	—	—	—	1.3188	0.9863	0.07441	0.0074	26.21
	FS-2	0.0171				1.8042	0.9887	0.05148	0.0037	2.29
Linear	**FS-1**	—	0.9930	-0.00942	—	—	1.0000	0.04436	0.0026	7.65
	FS-2		1.0939	-0.01234			1.0000	0.04884	0.0033	16.17
Wang & Singh	FS-1	—	-0.0087	0.000015	—	—	0.9959	0.12796	0.0218	23.49
	FS-2		-0.0087	-0.000035			0.9942	0.05499	0.0042	4.28
Modified Wang & Singh	FS-1	—	0.0648	-0.00078	0.0612	—	0.9898	0.06846	0.0075	21.88
	FS-2		-0.0246	0.00015	-0.0138		0.9497	0.28123	0.0198	9.28

Table 5.22: Vacuum-drying (VD) models for two sets of fish samples (FS-1 and FS-2) using CPMDH.

Model	**SET**	**K**	**A**	**B**	**C**	**n**	**R^2**	**RMSE**	**χ^2**	**E (%)**
Newton	FS-1	0.01771	—	—	—	—	0.9279	0.09101	0.009662	8.76
	FS-2	0.01503					0.9745	0.05195	0.003148	18.74
Page	FS-1	0.00261	—	—	—	1.6128	0.8929	0.15918	0.03547	13.79
	FS-2	0.00137				1.5986	0.9955	0.06949	0.00676	35.23
Henderson &Pabis	FS-1	0.02616	1.1433	—	—	—	0.8628	0.13761	0.02651	23.34

	FS-2	0.02441	1.7339				0.9389	1.06935	0.22870	29.48
Modified Page	FS-1	0.01596	—	—	—	1.6129	0.9814	0.032705	0.001497	25.14
	FS-2	0.01620				1.5986	0.8542	2.17199	6.60457	25.58
Linear	FS-1	—	0.99786	-0.01033	—	—	1.0000	0.06938	0.00674	22.31
	FS-2		0.97462	-0.01003			1.0000	0.04869	0.00332	23.09
Wang & Sing	FS-1	—	-0.01045	0.0000016	—	—	1.0000	0.06692	0.00627	27.51
	FS-2		-0.01157	0.000025			0.9966	0.04612	0.001298	36.03
Modified Wang & Sing	FS-1	—	0.0593	-0.0006783	0.0702	—	0.9999	0.05531	0.005354	26.41
	FS-2		-0.03036	0.0002024	-0.0205		0.9986	0.13573	0.00460	41.67

The VD time of FS-1 and FS-2 were shown in Table 5.23. SPMDH required 0.3 h and 0.51 h less VD time than CPMDH for FS-1 and FS-2 respectively. For FS-1, the VD time was less for both SPMDH (2.55 h) and CPMDH (2.85 h) than FS-2 (VD time 3.25 h and 3.76 h by using SPMDH and CPMDH respectively). The probable reason may be the internal resistance occurred during VD was more for VDFS-2 than VDFS-1 preparations.

Table 5.23: Drying time, Effective diffusivity (D_{eff}) and required activation energy (E) during VD for two blended sets (FS-1 and FS-2) of fish samples by using SPMDH and CPMDH.

SET	**PLATE**	**Drying time (h)**	**Temperature (°C)**	**D_{eff} for FD samples (m^2 / s)**	**Activation Energy, average (kJ / mol)**
FS-1	SPMDH	2.55	40	5.324×10^{-10}	28.42
			45	6.316×10^{-10}	
			50	7.464×10^{-10}	
	CPMDH	2.85	40	3.226×10^{-10}	43.09
			45	4.184×10^{-10}	
			50	5.384×10^{-10}	
FS-2	SPMDH	3.25	40	4.082×10^{-10}	37.38
			45	5.128×10^{-10}	
			50	6.366×10^{-10}	

	CPMDH	3.76	40	2.611×10^{-10}	55.59
			45	3.659×10^{-10}	
			50	5.056×10^{-10}	

What is more, though CF and RF were added same amount in both sets but DG (1%, w/w) was added in FS-2 but not in FS-1 preparation. That means DG has a significant effect on more VD time.

5.3.3 Effective Diffusivity (D_{eff}) and Activation Energy (E)

D_{eff} is generally used to know the mechanism during the transport of moisture to the surface to be evaporated (Panchariya *et al.*, 2002). D_{eff} for FS-1 and FS-2 by using SPMDH and CPMDH at considered temperatures 40°, 45° and 50°C were shown in Table 5.23. At 50°C, by using SPMDH for FS-1 the D_{eff} value found highest (7.464×10^{-10} m^2/s) than FS-2 ($D_{eff} = 4.082 \times 10^{-10}$ m^2/s). Comparing with CPMDH (at 50°C) for both FS-1 and FS-2, the D_{eff} values was found lower. From Table 5.23, the activation energy (E) required for drying for FS-1 and FS-2 (using SPMDH) were 28.42 and 37.38 kJ / mol respectively; E required for FS-1 and FS-2 (using CPMDH), 43.09 and 55.59 kJ / mol respectively. For all the sets using SPMDH and CPMDH, at 50°C the D_{eff} values were notably increased as compared with other two temperatures (40°, 45°C). All the diffusivity values were suggested that the VD time for two samples were increased from FS-1 to FS-2 (i.e. less diffusivity more VD time). Therefore, on the basis of energy effectiveness, FS-1 sample preparation was more cost effective than FS-2 sample using SPMDH and CPMDH. What is more, VDFS-1 sample preparation was more energy intensive by using SPMDH since it required least activation energy (28.42 kJ / mol).

5.3.4 Rehydration Ratio (RR)

The results of RR were found 2.68 and 2.50 for VDFS-1 and VDFS-2 respectively. It suggests that, VDFS-1 had more RR than VDFS-2.

5.3.5 Water activity (a_w) and Microbiological assay

Water activity (a_w) found 0.228 and 0.196 for VDFS-1 and VDFS-2 respectively. Very minimum a_w values for both the final products suggested that they have better shelf life and stability. But it may predict that VDFS-2 was better product than VDFS-1 to prevent the less growth of bacteria, yeast and mold. It is confirmed by microbial tests that almost no microbial load found in VDFS-2 as compared with VDFS-1.

5.3.6 Quality Assessment

Proximate analysis of two sets of fish powder sample was shown in Table 5.24. SPMDH could render ≈ 3% moisture content of the final dried VDFS-1 and VDFS-2 products within shorter VD time compared to CCHP. Silver has antimicrobial efficacy therefore the final quality of VD products (VDFS-1 and VDFS-2) would be analyzed by using SPMDH only.

Table 5.24: Proximate analysis of VDFS-1 and VDFS-2 (using SPMDH).

Quality Parameters	**Shrimp (raw)**	**VDFS-1**	**VDFS-2**	**Test method**	**Reference(s)**
Total Carbohydrate (g/100g)± SD*	0	34.99± 0.36	37.50± 0.34	AOAC Official Methods. 19th Edition, 2012; Vol- II, 986.25	fssai, act 2006, GOI.
Protein (g/100g)± SD	17.6	50.66± 0.31	48.78± 0.28	IS:7219-1973 (Rffm -2010)	Chakraborty et al. (2015)
Total Fat (g/100g)± SD	1	4.71± 0.05	4.73± 0.05	AOAC Official Methods. 19thEdtn, 2012; Vol- II, 963.15	Chakraborty et al. (2015)
Total ash (g / 100 g)± SD	2.2	5.52± 0.44	5.19± 0.42	IS: 1158: 1973 (RA-2010)	fssai, act 2006, GOI
Energy (kJ/100g)± SD	333	1609.26± 1.95	1642.28± 1.92	Composition & Analysis of Food by Pearson	fssai, act 2006, GOI
ω-3 fatty acids(g / 100 g)± SD	2.0	2.4 ± 0.03	2.2± 0.03	AOAC Official Method19-th Edition Vol- II, 996.06	KromhoutDaan et. al (1985).

Histamine (mg %)± SD	──	5± 0.01	5± 0.01	Fssai Lab Manual 6, 2015	Advances in Fish Processing Technology, D. P. Sen [ISBN 81-7764-655-9].
TVBN (mg / 100 g)	──	Below Detection Limit (5.0)	Below Detection Limit (5.0)	Fssai Lab Manual 6: 2015, Cl.no. 1.3	Idakwo et. al (2016)
Mercury (Hg) (mg / kg)	──	Below Detection Limit (0.01)	Below Detection Limit (0.01)	AOAC Official Methods. 19th Edition, 2012; Vol- I, 971.21	Torres-Escribano et.al (2010)

*SD: Standard deviation.

5.3.6.1 Moisture (%)

Both moisture level for FS-1 (64.97± 0.62%) and FS-2 (64.34± 0.64%) finally reached to 3.06 ± 0.25% (drying time: 2.55 h for FS-1 and 3.25 h for FS-2). Comparing with raw shrimp moisture (77.15± 0.73%) the final products (VDFS-1 and VDFS-2) moisture reduced to ≈ 3%.

5.3.6.2 pH

From the pH values of FS-1 (5.90±0.02), FS-2 (6.20±0.03), VDFS-1 (6.63±0.02) and VDFS-2 (6.33±0.02) it was observed that the alkalinity of fortified and blended samples increased during VD and hence the VD products were finally made into "alkaline food". According to Sarkar and Tirumkudulu (2009), evaporation during drying occurs and hence the close packing of structure increases and the structure becomes more stable and hence pH of product increases due to more ion diffusivity takes place. For VDFS-2 making, less ion diffusivity observed since pH increment was only 0.13. This was due to addition of dry ginger powder when VDFS-2 was made. For VDFS-1 making, pH increment was found 0.73. Comparing with raw shrimp pH (6.40±0.02), the pH of product VDBS-2 slight decreased and pH of VDBS-1 product increased.

5.3.6.3 Total Carbohydrate (g/100g)

Both VDFS-1 and VDFS-2 had more or less same total carbohydrate due to the same quantity addition of CF (10% w/w) and RF (2% w/w). Increment of data (about 2.51) of VDBS-2 over VDBS-1 due to the fact that, dried ginger powder (contain ≈ 22% w/w carbohydrate) was added (1% w/w) in VDBS-2 but not in VDBS-1. Since raw shrimp does not contain any carbohydrate, therefore VDFS-1 (increased 35% carbohydrate) and VDFS-2 are rich in carbohydrate by fortification with CF and RF.

5.3.6.4 Protein (g/100g)

The protein content was 50.66 ± 0.31 g and 48.78 ± 0.28 g per 100 g VD sample for VDFS-1 and VDFS-2 respectively. VDFS-1 had more protein (about 1.88g/100g) than VDFS-2. VDFS-1 has protein increment of 187% as compared with raw shrimp (17.6 g / 100g).

5.3.6.5 Total Fat (g/100g)

The total fat for all the two products were found 4.71 ± 0.05 g (VDFS-1) and 4.73 ± 0.05 g (VDFS-2) per 100 g. Therefore, both of them the fat quantity found same since they were made almost same compositions except dried ginger powder was used for VDBS-2. The dried products (VDBS-1 and VDBS-2) fat content increases almost 5 times as compared with raw shrimp (1 g / 100 g). Abraha and co-workers (2017) found 3.74 g fat per 100 g dried anchovy fish, which was well below the fat content of two VDFS products.

5.3.6.6 Energy (kJ/100g)

The energy content found 1609.26 ± 1.95 kJ and 1642.28 ± 1.92 kJ per 100 g for VDFS-1 and VDFS-2 respectively. All of the two sets were made same portions of Shrimp, CA, CS (2:1:1) and their initial (raw fish) energy content was 333 kJ, 267 kJ and 350 kJ per 100g fish for Shrimp, CA and CS respectively. So, after VD all two sets energy notably increased from their initial values. Slight increment of energy (about 33 kJ per 100g) of VDBS-2 was due to the addition of dried ginger powder in FS-2. Comparing with energy content of raw shrimp (333 kJ / 100 g), the final dried products energy content increased almost five times. Abbey and co-workers (2017) found less energy content (1333 kJ / 100 g) in dried tuna fish powder compared to the two VDFS products.

5.3.6.7 Total Ash (g/100g)

It was found from Table 5.24 that the total ash content for final VDFS-1 and VDFS-2 was 5.52 ± 0.44 g and 5.19 ± 0.42 g per 100 g dried products respectively. For both the products, the total ash increased about the range of 2 to 2.5 times as compared with raw shrimp (2.2 g per 100 g). The increment of 151% ash for VDFS-1 as compared with raw shrimp. Shaviklo (2015) reoported 1.60 g ash per 100 g freeze-dried tilapia fish and 2.08 g ash per 100 g spray-dried tuna fish, those were far lower as compared to the ash content of the VDFS products.

5.3.6.8 Histamine (mg%)

Both VDFS-1and VDFS-2 the histamine was found 5 ± 0.01 mg%. The regulatory limit for histamine by USA is 20 mg% (value is least as compare with other countries). Therefore, the tested two samples histamine content was found well below the limit. Hwang *et al*. (2012) estimated histamine in dried milkfish (*Chanos chanos*) in the range of 117.3-382.1 mg / 100 g dried fish, which was well above the histamine value of the present study.

5.3.6.9 ω-3 Fatty acids (g/100g)

The ω-3 fatty acids were found around 2.4 ± 0.03 for VDFS-1 and 2.2 ± 0.03 g/100 g for VDFS-2, which were greater than (0.55 g / 100 g) that of dried anchovy (*Stelophorus heterolobus*) fish (Abraha *et al*, 2017). According to Kris-Etherton *et al.*(2002), the intake of ω-3 PUFA is preferable from dietary (food based) approach; hence the consumption of 1 g per day ω-3 fatty acids have more beneficial to reduce cardiovascular disease, stroke (cerebral infarctions) and there are no risk of side effects (gastrointestinal disturbances and nausea) from ingestion of it. As compared with raw shrimp (ω-3: 2.0 g per 100 g), 20% increment of ω-3 fatty acids observed in VDFS-1 product.

5.3.6.10 Total Volatile Base Nitrogen [TVBN] (g/100g)

For fresh fish sample TVBN is less than 20 mg N per 100 g fish. The acceptable limit of fish sample is 20 to 30 mg N per 100g. When TVBN value reaches ≥ 40 mg N per 100 g sample then then fish is unfit for consumption. In all the two samples TVBN found below the detection limit (detection limit = 5 mg / 100 g). Koning-de (2002) reported TVBN level (for several fish meals) over the range from 85 to 170 mg / 100g

dried fish meal which was considerably higher than the present data. Abraha and co-workers (2017) also found greater TVBN content (20.12 ± 0.20 mg / 100 g) in dried anchovy (*Stelophorus heterolobus*) compared to both VDFS-1 and VDFS-2.

5.3.6.11 Mercury (mg / kg)

The joint Food and Agriculture Organization (FAO) and WHO recommended 5µg/kg body weight per week (daily intake 0.71 µg Hg/kg body weight) maximum level of Hg consumption (Torres-Escribano *et al.,* 2010). In all the two samples mercury (as Hg) was found below the detection limit (detection limit = 0.01 mg / kg of sample). Therefore, it is safe to consume all the two products. Panichev and Panicheva (2016) reported Hg content for 14 different processed fishes that ranged from 135 ng /g (0.135mg/kg) to 666 ng / g (0.666 mg/kg); evidently, these were much more than the present work.

5.3.6.12 Essential Elements

For end products, eight essential elements viz. Fe, Zn, Ca, P, K, Cu, Mg and S were measured and compared with those of raw shrimp (shown in Table 5.25). It was found that, increments of those elements were greater for VDFS-1than VDFS-2 except Ca. For VDFS-1, the increments were 119%, 260%, 129%, 198%, 152%, 71%, 108% and 141% for Fe, Zn, Ca, P, K, Cu, Mg and S respectively (Table 5.25) as compared to those elements in raw shrimp. Abbey *et al*. (2017) measured the Cu, Zn and Ca content as 0.25 mg, 1.88 mg and 1066.5 mg respectively per 100 g dried tuna fish powder. Thus, the developed VDFS products contained higher concentration of elements (viz. Cu, Zn and Ca) than dried tuna fish powder.

Table 5.25: Different essential elements analysis of VDFS-1 and VDFS-2.

Material	**Fe (mg) ± SD***	**Zn (mg) ± SD**	**Ca (mg) ± SD**	**P (mg) ± SD**	**K (mg) ± SD**	**Cu (mg) ± SD**	**Mg (mg) ± SD**	**S (mg) ± SD**	**Reference / Testing method**
Shrimp (raw)	2.70	1.30	550	290	210	0.49	45	190	Jessica *et al* (2015)
VDFS-1	5.92± 0.88	4.68± 0.51	1259.26± 0.11	864.22± 0.92	528.69± 0.78	0.8385± 0.42	93.77± 0.02	458.22± 0.64	AOAC (1999) [Yanar *et al.*

									(2004)]
VDFS-2	5.80± 0.86	4.56± 0.52	1272.97± 0.10	830.75± 0.91	504.44± 0.81	0.6345± 0.52	91.22± 0.02	445.66± 0.62	AOAC (1999) [Yanar*et al.* (2004)]

*SD: Standard deviation.

5.3.7 DSC

The denaturation of protein for VDFS-1 and VDFS-2 was studied by DSC. The range of temperature was chosen from 10°C to 100°C. From the endothermic heat flow (in mW) and the temperature (in °C) plot if multiple transition peaks are found then that denotes the denaturation of protein occurs. In Figure 5.16 (for VDFS-1 sample), no protein denaturation is found since the graph was found very smooth line up to 100°C. In Figure 5.17 (for VDFS-2 sample), many transition peaks were observed over the temperature range from 10°C to 100°C. One such transition peak is observed nearly at 65°C. Skipnes and co-workers (2004) found several denaturation peaks for cod (*Gadus morhua*) fish. By using salt and sucrose concentrate (40% w/w) on tilapia fish the denaturation found at the temperature range 50°C to 70°C (Vivanco*et al.,* 2004). No such DSC thermogram reporting was found for fish powder or fortified fish powder. Hence, the DSC thermogram in this research applied for two different powders from three different fishes to know the status of protein denaturation is new one with best of knowledge. Among two powders, VDFS-1 was found almost no denaturation during processing. In Figure 5.16 (VDFS-1), no protein denaturation was found since the profile was found very smooth up to 100°C. On the contrary, in Figure 5.17 (VDFS-2), many transition peaks were observed (around 50° and 65°C) over the temperature range from 10°C to 100°C indicating thermal degradation. For VDFS-2, the rapid increasing endothermic heat flow was possibly due to presence of DG having higher specific heat (1.92 kJ $kg^{-1}K^{-1}$). Hence, VDFS-1 indicates higher thermal stability compared to VDFS-2. Besides, higher glass transition temperature of VDFS-1(T_g = 54.57°C) over VDFS-2 (T_g = 22.02°C) implies its superior thermal stability. Moreover, relative to unfortified dried shrimp where protein denaturation

observed at 40°C (Schubring, 2009); therefore the superior thermal stability was achieved for both VDFS-1 and VDFS-2.

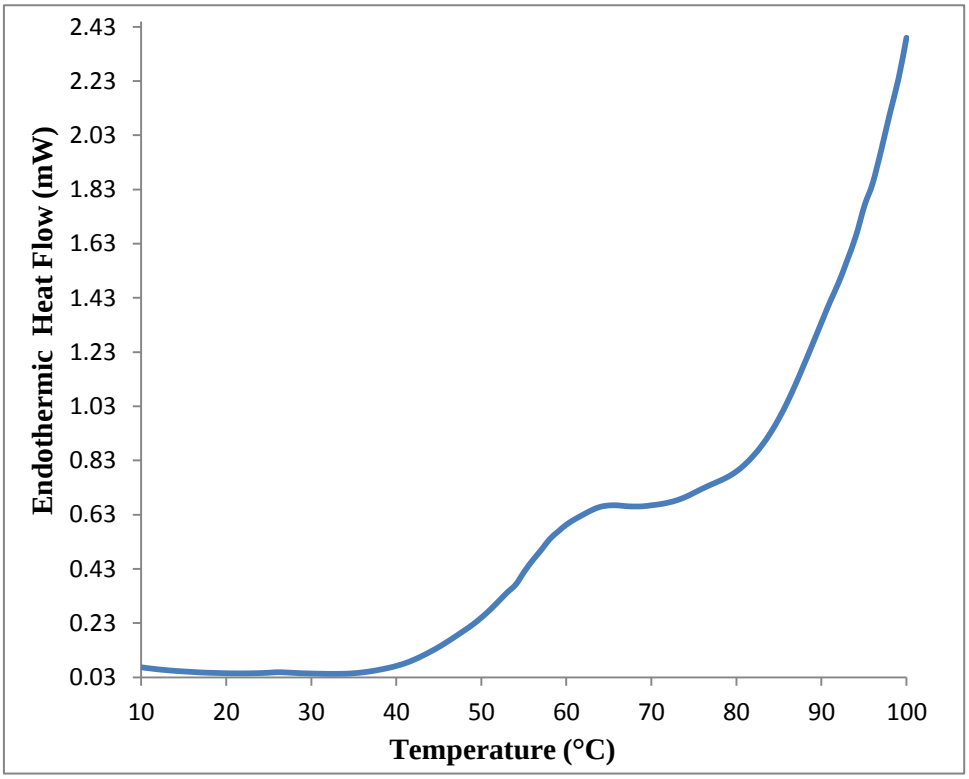


Figure 5.16: DSC thermogram of VDFS-1.

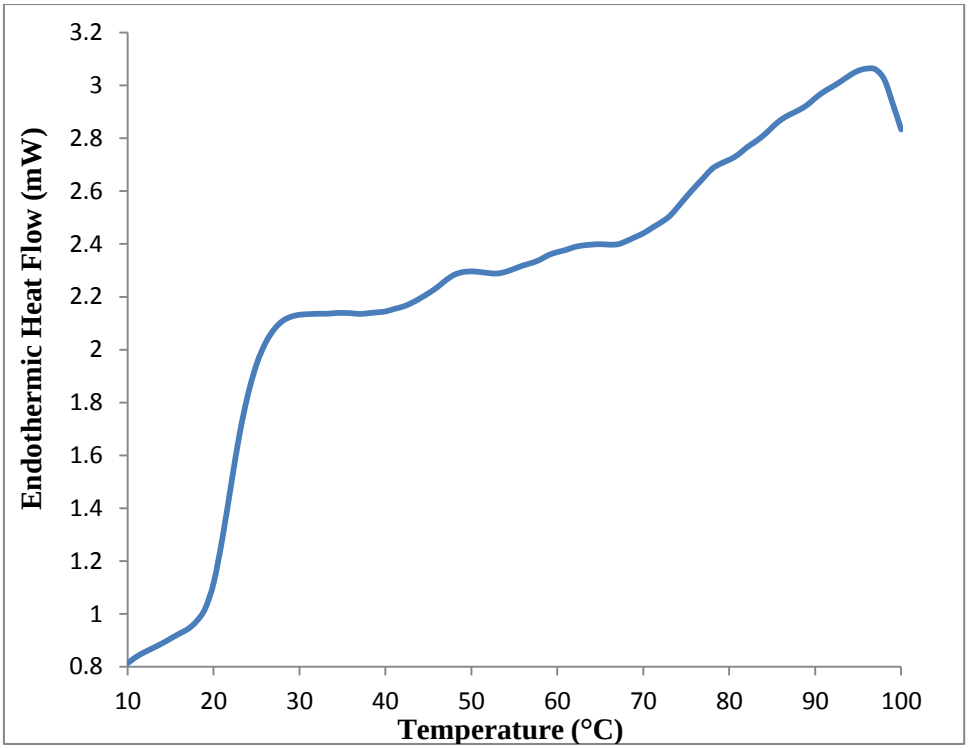


Figure 5.17: DSC thermogram of VDFS-2.

5.4 EXPERIMENT SET- 4: BLANCHED AND FREEZE-DRIED BROCCOLI

In this section the results were analyzed for the moisture, protein, ash and vitamin C content of raw, cold blanched and freeze-dried broccoli. The moisture content of raw, cold blanched and freeze-dried broccoli was found 88%, 78% and 5% respectively.

5.4.1 Analysis of Results on Drying Time, Moisture, Protein, Ash and Vitamin- C Content

The FD time for set-1 product required was 5.5 h and the same for set-2 was 6.5 h. So, the blanched broccoli drying time was less than non-blanched broccoli. So, blanching had a significant effect on lowering the drying time and hence it will be cost effective for drying. The moisture and few nutritional testing were done for the set-1 (shown in Table 5.26).

Table 5.26: Moisture , Protein, Ash, Vitamin C for raw, blanched and freeze-dried Broccoli (set-1).

Set	FD time	Moisture			Protein (g/100g)			Ash (g/100g)			Vitamin C (mg/100g)			Reference
	(hour)	R†	B‡	FD§	R	B	FD	R	B	FD	R	B	FD	
1	5.5	88%	78%	5%	5.4	5.2	11.0	0.92	0.89	3.58	84.2	78.78	74.1	fssai, act 2006, GOI

† Raw broccoli.

‡ Blanched broccoli.

§ Freeze-dried broccoli.

From Table 5.26, for set-1 the moisture % gradually decreased from raw to blanched and freeze-dried broccoli. So, both blanching and freeze-drying process have a significant effect on the reduction of moisture and hence increase the shelf-life of foodstuff. The protein content though reduced some amount in blanching but it was increased double in final product since its moisture was only 5%. The ash content of product increased almost four times as compared to raw broccoli. From raw broccoli, the vitamin C lost 6.5% and 12% for blanched and freeze-dried broccoli respectively. Brewer *et al.* (1995) reported 9.5% vitamin C lost for hot blanching of broccoli. So, cold blanching made beneficial compared to hot blanching.

A graph of rate of drying (dw/dt) versus time (t) of the frozen sample during drying was shown in Figure 5.18. The difference in successive weight of the sample was significant; therefore it was concluded that primary drying happened up to 110 minutes i.e. nearly two hours. After that the plate (heater) temperature was increased by temperature controlling unit. To start and continue the secondary drying, the sublimation heat was required more to remove bound water. Nearly from 125 minutes it was observed that the graph was constant in progress up to the end process. After 300 minutes the graph line became to absolutely constant and hence the drying process was stopped. Total drying time was 5.5 h which was better result as compared to non-blanched (set-2) freeze-drying time (6.5h).

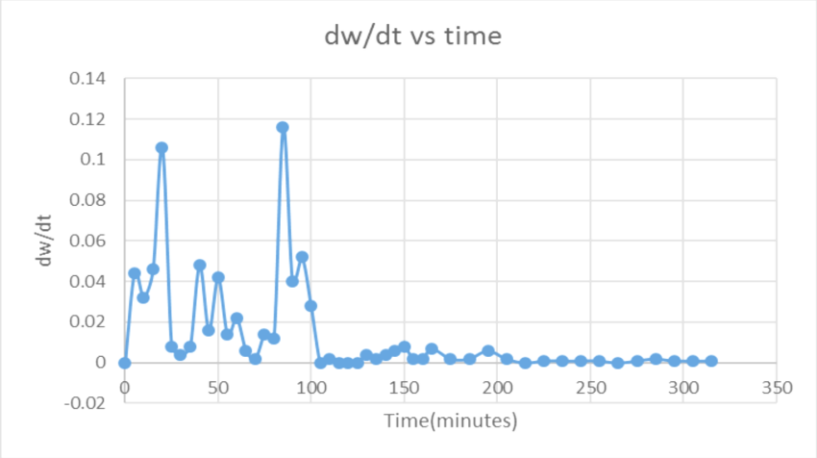


Figure 5.18: Rate of drying vs. time during freeze-drying.

Three probes were used during freeze-drying. First one for ambient temperature (inside the freeze-dryer unit), second one for surface temperature (frozen and dried material) and third one for plate temperature (by which the sublimation heat transferred). The ambient temperature during running was from 36.4°C to 39.7°C—which was steady one. Figure 5.19 shown the graph of Plate temperature (°C) versus time (in minutes). Since SS made plate have higher thermal conductivity, therefore, frozen material was heated up quickly. Initially (only for 10 minutes) the increased temperature (from 61°C to 63°C) of plate was done due to material was completely frozen and hence to decrease the time required for drying and also to avoid thawing of the frozen material quickly. After that the temperature range of the plate was 42°C to 47°C. During secondary drying stage the temperature was increased and it was in between 50°C to 55°C. Few ups and downs were shown in the graph due to temperature controlled unit was operated manually.

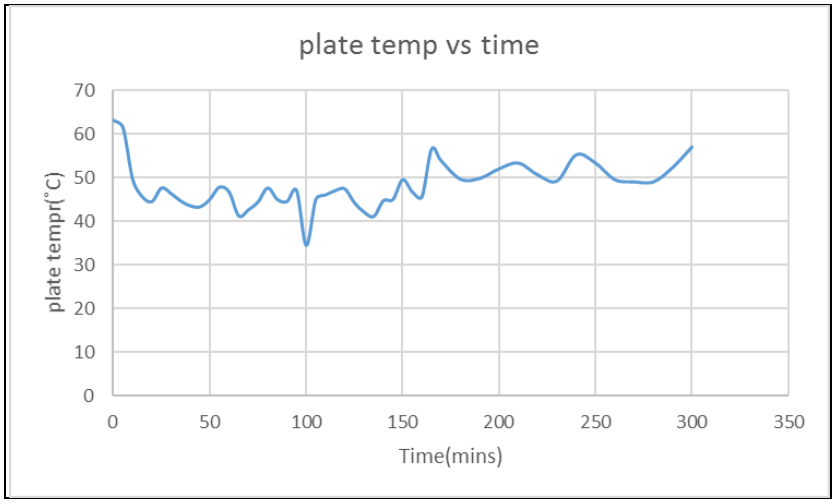


Figure 5.19: Plate (SS made) Temperature vs. Time.

From Figure 5.20, a graph of frozen surface temperature versus time, it was found that initially the frozen surface temperature was (-) 3°C. Then it was increased to 0°C and hence gradually increased to 40.5°C (at the end of drying). By drawing a trend line (black colored) on the original graph it was clearly observed that there was a tendency of increasing the surface temperature as drying carried forward. Hence, freeze-drying was proper.

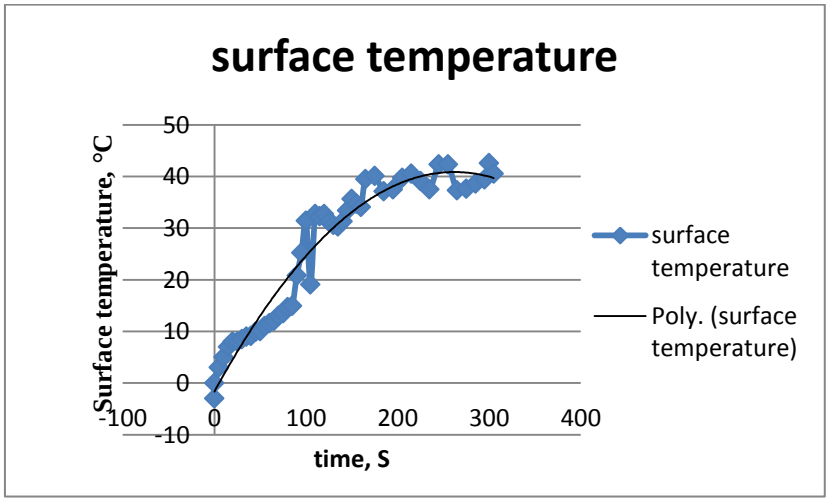


Figure 5.20: Surface (frozen) Temperature vs. Time.

CHAPTER-5
CONCLUSIONS AND SCOPE FOR FUTURE WORK

6.0 CONCLUSIONS

I. The present work highlighted the dehydration of kiwi fruits through a consecutive osmotic dehydration and freeze-drying protocol. Application of Taguchi orthogonal design could help in finding optimal conditions for osmotic dehydration. Notably, sucrose had been found to be a better osmotic solution when compared with fructose under identical OD conditions.

The silver coated steel heater had been observed much more energy efficient than conventional steel heater in consecutive freeze-drying to obtain the final dried kiwi fruit. The osmotically dehydrated kiwi fruit at optimal conditions subsequently dehydrated employing energy-efficient silver coated heater resulted in a final dried kiwi fruit containing acceptable quality parameters conforming to market status. It is expected that the developed economically-sustainable dehydration protocol may be applied to similar foodstuffs.

The higher effective moisture diffusivity and lower activation energy in freeze-drying are indicative of a faster and energy saving dehydration protocol through application of novel silver (coated steel) heater making the kiwi dehydration process economically sustainable.

II. A value added kiwifruit powder had been developed at reduced cost. It was tested and found to be well accepted by all for its good taste and quality. The conversion of kiwi fruits to its powder through a consecutive value addition of blended kiwi fruit and freeze-drying protocol reduced the drying time as well as processing cost. The cubic steel heater designed for this purpose and silver coated steel plate were found to be very efficient for drying of frozen blended kiwi fruit.

The higher effective moisture diffusivity and lower activation energy in freeze-drying are indicative of a faster and energy saving dehydration protocol through application of novel cubic heater making the kiwi dehydration process economically sustainable.

III. Fortification of shrimp with two fishes (Catla and Chela) was performed to improve preservation and nutritional quality of shrimp. The whole processs was done by successive sterilization, blending, fortification (by CF, RF, with or without DG) and VD techniques to reach best quality protein, fats (and fatty acids), richness of carbohydrate, vitamins, essential micronutrients. VD was performed by an indigenously fabricated six sides cubic heater for blended four samples to reach the

final moisture content ≈ 3%. Seven drying models were selected to compare the VD kinetics. By using SPMDH for FS-1 the VD time, D_{eff} and E were found 2.55 h, 7.464 x 10^{-10} m^2 / s and 28.42 kJ / mol respectively which to be considered to maintain best drying protocol.

All the two sets the histamine, TVBN and Hg was found below the detection limit. Though, VDFS-2 ha little advantages over VDFS-1 regarding less microbial count detection, more carbohydrate, ash and energy content but VDBS-1 was better on cost effective (less drying time protocol), tends to alkaline product, protein rich and more ω-3 fatty acids content as compare with VDBS-2.

IV. Food hunger and food starvation is now a major problem in developing countries. The unstoppable population growth, gap of both a surplus and a shortage of food, lack of application of technology in food production sector and lack of awareness for utilization of waste food will be life-threating or food crisis after 30 years. So, preservation of food with less energy utilization can be a path of solution to overcome this global problem. Few drying, freezing or the combination of freezing and drying (Freeze-drying) may be pioneer of preservation.

V. In freeze-drying, the nutrition retention is more but it is not cost effective. Pre-processing without loss of essential nutrients before freeze-drying for fruits and vegetables may be alternate solution to reduce the energy. The nutritional rich broccoli has so many health benefits for human beings, particularly presence of few natural antioxidants make it as an anti-carcinogenic. Cold blanching technique was done as pre-processing of broccoli and hence drying time was less (5.5 h) as compared with non-blanching (6.5 h). The final freeze-dried product protein and ash incremented by 2 and 4 times respectively, vitamin C lost 12% to reach final moisture level 5% from 88% of raw broccoli. Therefore, pre-processing may be cost effective step for freeze-drying of highly nutritious foodstuffs.

VI. The freeze-drying (FD) time of **2.5 h** (using SCSH), **3.82 h** (using SH) required for OPODFD process and **5.75 h** time required for CFD (without OD for KF sample) process. For making of value added KF powder the FD time (using SCSRP) of **4.5 h** (with additives) and **7 h** (without additives) were required. For the preservation of shrimp (by fortification with two fishes and flours employing fast vacuum-drying), for FS-1, VD time was **2.55 h** (using SPMDH) and **2.85 h** (using CPMDH); for FS-2, VD time was **3.25 h** (using SPMDH) and **3.76 h** (using CPMDH). For the preparation of blanched and freeze-dried broccoli the FD time was **5.5 h** (using SCSH) and for non-

blanched broccoli the FD (CFD) time was **6.5 h**. From these drying time data it is clearly found that pre-processing prior to FD / VD process is advantageous and energy efficient as compared with no pre-processing (called conventional drying).

VII. The nutritional quality of all the final products (osmotically dehydrated kiwi fruit, value added kiwi fruit powder, fortified shrimp and cold blanched broccoli) were checked and compared with starting raw materials; found up-gradation of the final products.

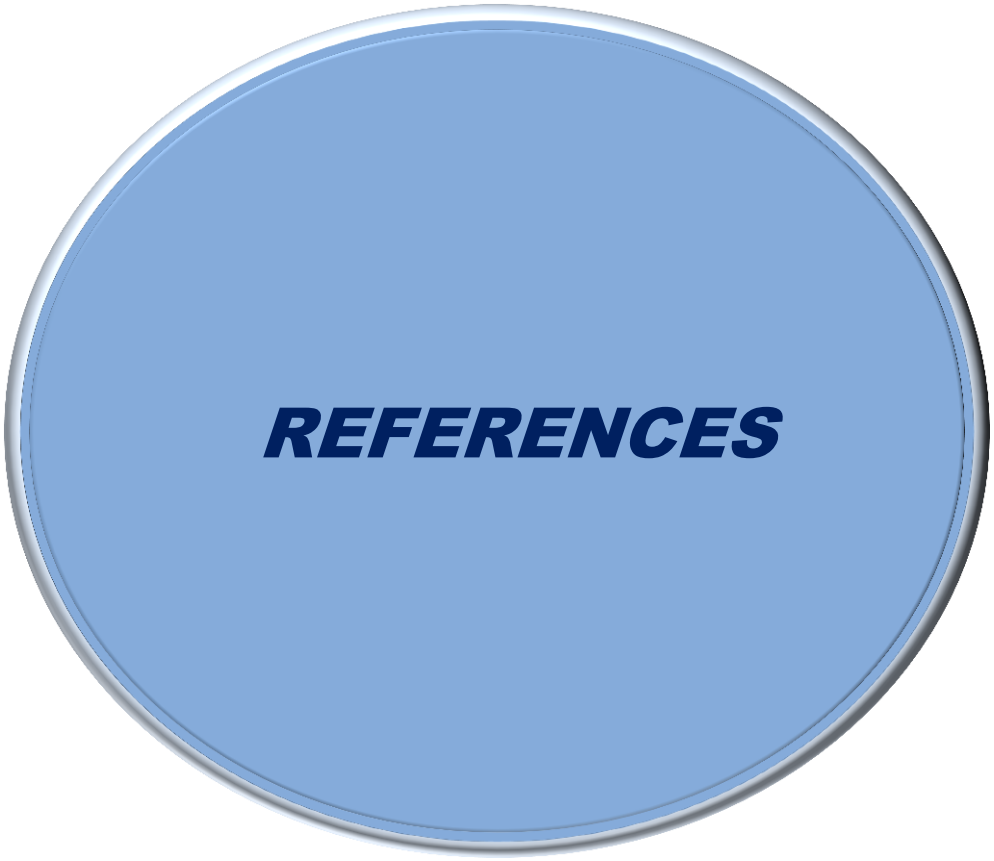
7.0 SCOPE OF FUTURE WORK

I) In future, there will scope on different pre-processing steps to choose by which the reduction of principle processing energy and hence product (s) selling cost may be reduced. This will benefit many costly processing like freeze-drying such as spray drying, Ohmic heating, separation by membrane technology so that costly food items may reach to the financial backward people.

II) In future, further studies may be required for further development of kiwi fruit related fruit powder and its analysis for final powder on the basis of flowability, particle size, degree of caking, porosity, cohesiveness and bioactive components measurements.

III) In future there will be a scope of working for a particular nutrient deficient fish may be fortified by other nutrient rich fish with the no addition of chemical preservatives and addition of other flours from different cereals and hence may produce value added fish powder. Several drying methodology, different raw materials for value addition, different fish (es) selection may apply as a permutation and combination process to get a less costly and good nutritional rich fish products which can solve so many societal and human health problems.

IV) Another set of heater (s) to be developed with good temperature controlling factors for the supply of heat according to the need of primary and / or secondary drying.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ANNEXURE-1

CALCULATIONS

Table 5.1:

Experiment no. (i)

$$WL = \frac{(W_0 - W_t) + (S_t - S_0)}{W_0} \times 100 \quad (4.1)$$

$$SG = \frac{(S_t - S_0)}{W_0} \times 100 \quad (4.2)$$

$$\text{Where, } S_0 = \frac{W_0(100 - M_i)}{100} \quad (4.3)$$

$$S_t = \frac{W_t(100 - M_f)}{100} \quad (4.4)$$

Initial Moist. (M_i); before O.D. = 82.526%.

Final Moist. (M_f); after O.D. = 38.81%.

Initial wt. of kiwi slices, W_0 = 7.285 g.

Weight (g) of the kiwi slices after O.D. W_t = 4.473 g.

S_0 = 1.273 g [using equation (4.3)]; S_t (g) = 2.737 g [using equation (4.4)].

Therefore, **Water Loss, (WL)** = 58.697 % [using equation (4.1)].

Solute gain, (SG) = 20.097% [using equation (4.2)].

Table 5.9: Mathematical models selected for Experiment -1 and Experiment-2.

Serial no.	Model name	Equation used
1.	Page	$MR = \exp(-kt^n)$
2.	Wang & Sing	$MR = 1 + at + bt^2$
3.	Henderson & Pabis	$MR = a \exp(-kt)$
4.	Newton	$MR = \exp(-kt)$

Where, $MR = \frac{M_C}{M_O} = \frac{\left[(W_o - W_e) - W_d\right]/W_d}{W_{i,w}/W_d}$ (4.11)

Where, M_c = moisture content at particular time with respect to dry solids, M_0 = initial moisture content with respect to dry solids, W_o = initial weight of sample (g), W_e = amount of evaporated water (g), W_d = dry matter content of sample, $W_{i,w}$ = initial weight of water in sample (g).

At, t = 0 sec: W_o = 4.49 g; W_e = 0 g; W_d (M%=14.94%) = 3.82 g (constant throughout the FD); $W_{i,w}$ = 0.671 g.

Therefore, MR = 0.999 [using Eq. 4.11].

Similarly, At, t = 2100 sec: W_o = 3.88 g; W_e = 0.61 g; W_d (M%=14.94%) = 3.82 g (constant throughout the FD); $W_{i,w}$ = 0.671 g.

Therefore, MR = 0.089 [using Eq. 4.11]. So, at different time intervals got different MR values (taken these values are MR_{exp}).

For Page Model equation used $MR = \exp(-kt^n)$

By putting MR and t values, solved for k= 0.035, n = 1.1.

For Wang and Sing Model Equation used: $MR = 1 + at + bt^2$

By putting MR and t values, solved for a = - 0.042, b = 0.00048.

For Henderson & pabis Model Equation used: $MR = a \exp(-kt)$.

By putting MR and t values, solved for a = 1.015, k = 0.0501.

For Newton Model Equation used: $MR = \exp(-kt)$.

By putting MR and t values, solved for k = 0.0452.

MR_{pre} values were calculated graphically to match with the (MR_{exp} versus time) graph. The model constant values (got from the graph) for different Models were put into respective Model equations and hence found MR_{pre} values.

Regression coefficient (R^2) was calculated by-

Plotted MR_{exp}vs t in excel sheet and from this chart R^2 value displayed on chart.

EF value calculated from the equation.

$$EF = \frac{\sum_{i=1}^{N}\left(MR_{\exp,i} - MR_{\exp,avge}\right)^2 - \sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)^2}{\sum_{i=1}^{N}\left(MR_{\exp,i} - MR_{\exp,avge}\right)^2}$$

Calculated: $MR_{exp,avge}$ value. From MR_{exp}, $MR_{exp,avge}$ and MR_{pre} values, the EF values were found from all models.

Similarly, MBE and RMSE values are calculated by using their respective equations for all selected models.

$$MBE = \frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)$$

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)^2\right]^{0.5}$$

All values are shown in the following table (5.11).

NOTE: For all the experiments (SET-2 and SET-3) the similar type of calculations were done.

A sample calculation sheet (done in Excel) is produced for this purpose:

Page model

MRpre = EXP(-k.t^n)	MRexp	MRpre – Mrexp	SQ. of(Mpre - Mexp)	Mexp, avg(= 3.905/ 9)	(Mexp - Mexp,avg)	Sq. of (Mexp-Mexp,avg)	MBE=(1/9)∑ (MRpre-MRexp)	RMSE=Sq.root[(1/9)∑(MRpre-MRexp)^2]	EF=(N13-K13)/ N13	R^2 value(from chart / graph)
1.00000	***1.000***	0.00149	0.000002	***0.434***	0.565	0.31867	**0.03093**	**0.05773**	**0.96577**	**0.947**
0.81419	***0.968***	0.05413	0.002930	***0.434***	0.326	0.10631				
0.64363	***0.931***	-0.02701	0.000729	***0.434***	0.237	0.05600				
0.50244	***0.879***	-0.01917	0.000368	***0.434***	0.088	0.00768				
0.38887	***0.827***	0.00139	0.000002	***0.434***	-0.047	0.00216				
0.29901	***0.775***	0.03076	0.000946	***0.434***	-0.166	0.02747				
0.22869	***0.716***	0.02005	0.000402	***0.434***	-0.225	0.05079				
0.17412	***0.652***	0.08470	0.007174	***0.434***	-0.345	0.11874				
0.13205	***0.598***	0.13205	0.017437	***0.434***	-0.434	0.18836				
	SUM	0.2784	0.0299	3.906		0.8762				

Similar, calculations of R^2, MBE, RMSE and EF were done for all Models used in this thesis.

The best Model was selected on the following basis:

	R^2			MBE			RMSE			EF			TOTAL SCORE
Model	Value	RANK	SCORE	Value	RANK	SCORE	Value	RANK	SCORE	Value	RANK	SCORE	
PAGE	**0.947**	I	20	0.03093	II	10	0.05773	III	15	0.96577	II	15	60
Wang & Sing	**0.946**	II	15	-0.00185	IV	20	0.04343	IV	20	0.98062	I	20	**75**
Handerson & Pebis	0.928	IV	5	0.0217	III	15	0.0608	II	10	0.96203	III	10	40
Newton	0.94	III	10	0.04359	I	5	0.0737	I	5	0.72485	IV	5	25

As per Table, here **Wang & Sing model** was best selected / fitted for OPODFD.

Table 5.11: Freeze-drying models for osmotically dehydrated kiwi samples at optimal conditions (OPODFD).

Model	K	a	B	N	R^2	EF	MBE	RMSE
Page	0.035	—	—	1.1	0.947	0.96577	0.03093	0.05773
Wang & Sing	—	-0.042	0.00048	—	0.946	0.98062	-0.00185	0.04343
Henderson & Pabis	0.0501	1.015	—	—	0.928	0.96203	0.02170	0.06080
Newton	0.0452	—	—	—	0.940	0.72485	0.04359	0.07370

The effective diffusivity (D_{eff}) and activation energy are calculated by the following two equations:

$$MR = \frac{8}{\pi^2} Exp\left[-\frac{\pi^2 * Deff * t}{4L^2} \right]$$

where, t : time in seconds, L: thickness or depth of sample in SCSRP.

The activation energy (E) is calculated by Arrhenius equation:

$$\ln\left(\frac{D_{eff,2}}{D_{eff,1}} \right) = \left(\frac{E}{R} \right)\left[\frac{(T_2 - T_1)}{T_1 * T_2} \right]$$

A sample calculation done in excel sheet shown (for OPODFD for SCSH) the Table below:

t (sec)	MRexp	ln (MR)	[-ln(MR)-0.2089]	2.4649 t	L (meter)	L^{-2}	Deff = [-ln(MR)-0.2089]/(2.4649*t*L^{-2})	Temp.,°C	T, Kelvin
0	***1.000***	0	-0.2089	0	0.036	0.001296	#DIV/0!	38	311.15
300	***0.795***	-0.22941316	0.020513164	739.47	0.036	0.001296	**3.69515E-08**	**40**	313.15
600	***0.745***	-0.29437106	0.085471061	1478.94	0.036	0.001296	7.48986E-08	**41.6**	314.75
900	***0.686***	-0.37687765	0.167977651	2218.41	0.036	0.001296	9.81329E-08	**43.4**	316.55
1200	***0.576***	-0.55164762	0.342747618	2957.88	0.036	0.001296	**1.50175E-07**	**45**	318.15
1500	***0.392***	-0.93649344	0.727593439	3697.35	0.036	0.001296	2.55037E-07	**46.6**	319.75
1800	***0.198***	-1.61948825	1.410588248	4436.82	0.036	0.001296	4.12034E-07	48.2	321.35
2100	***0.073***	-2.61729584	2.408395838	5176.29	0.036	0.001296	**6.03996E-07**	**50**	323.15
2400	***0.000***	#NUM!	#NUM!	5915.76	0.036	0.001296	#NUM!	52.5	325.65

From, the two effective diffusivity values (D_{eff}) corresponding to two temperatures (in Kelvin), the activation energy (E) was calculated by the equation (just above Table).

Table 5.12: Effective diffusivity for optimal osmotic dehydration (OPOD), optimal freeze-drying of osmotically dehydrated (OPODFD) kiwi samples using steel heater (SH) and silver coated steel heater (SCSH).

Temperature	D_{eff} for OPOD (m^2 / s)	D_{eff} for OPODFD (m^2 / s)	
		Using SH	Using SCSH
50^0C	3.405×10^{-9}	2.22×10^{-7}	**6.04×10^{-7}**
45^0C	1.886×10^{-9}	0.57×10^{-7}	**1.50×10^{-7}**
40^0C	1.045×10^{-9}	0.157×10^{-7}	**0.37×10^{-7}**

SENSORY EVALUATION & SCORES

$$\%\ \text{of consumer acceptance} = \frac{\text{Number of panelists rated}>5}{\text{Total number of panelists}} \times 100 \quad (7)$$

$$\text{Average score} = \frac{\text{Total points on individuial organoleptic test or oveall test}}{\text{Total number of panelists}} \quad (8)$$

$$\text{Total score} = \frac{\text{Total points on individual organoleptic test or overall test}}{(\text{Total number of panelists})(\text{Maximum Hedonic points})} \times 100 \quad (9)$$

FIRST LOT OF PANELISTS (22 persons)

NO. of Panelists or Testers	SWEETNESS	SOURNESS	SALTINESS	BITTERNESS	FLAVOUR	TEXTURE	OVERALL ACCEPTABILITY	AVERAGE (Total grades / 7)
1	**5.5**	**6**	**7**	**9**	**6**	**6**	**6**	6.50
2	**5.5**	**7**	**6**	**9**	**8**	**7**	**8**	7.21
3	7	**6**	**8**	**9**	**7**	**8**	**7**	7.43
4	**6**	**5.5**	**7**	**9**	**8**	**6**	**5**	6.64
5	6	6	7	**9**	7	8	4	6.71
6	6	7	8	**8**	**7**	**7**	**6**	7.00
7	**5.5**	7	7	**9**	**7**	**8**	**7**	7.21
8	**5.5**	6	7	**9**	8	**6**	**8**	7.07
9	6	7	7	**9**	**6**	**7**	5	6.71
10	5	6	8	**9**	**7**	**8**	**8**	7.29
11	5	**5.5**	**6**	**8**	**9**	**7**	**6**	6.64
12	**5.5**	6	7	**9**	**8**	**6**	**8**	7.07
13	6	6	7	**9**	**7**	7	**7**	7.00
14	6	6	6	**9**	**8**	**8**	**6**	7.00
15	5	7	7	**9**	**8**	6	**7**	7.00
16	5	7	7	**9**	9	7	5	7.00
17	6	5	7	**8**	**8**	8	**8**	7.14
18	6	**5.5**	**8**	**9**	**9**	**7**	**7**	7.36
19	3	6	7	**8**	**7**	**7**	**7**	6.43
20	**5.5**	5	7	**9**	**6**	**6.5**	**6**	6.43
21	3	6	6	**9**	6	**8**	**5.5**	6.21
22	3	6	6	**9**	5	**8**	**5.5**	6.07

TOTAL or SUM→→	117	134.5	153	**194**	161	**156.5**	**142**	
AVERAGE SCORE(Total Score / No.of testers)→→	5.318181818	6.1136364	6.9545455	8.8181818	7.318182	7.11364	**6.454545455**	
TOTAL SCORE:↓								
[SUM/(22*9)]*100	59.09090909	67.929293	77.272727	97.979798	81.31313	79.0404	**71.71717172**	
CONSUMER ACCEPTANCE→	(15/22)*100 =68.18%	(20/22)*100 =90.91%	(22/22)*100 =100%	(22/22)*100 =100%	(21/22)*100 =95.45%	(17/22)*100 =77.27%	(18/22)*100 = **81.82%**	

Table 5.19: Judgement of different organoleptic tests & overall test by 66 panelists with 9-Hedonic points [1 (= dislike extremely) to 9 (= like extremely)].

	Sweetness	**Sourness**	**Saltiness**	**Bitterless**	**Flavor**	**Texture**	**Overall acceptability**
% of consumer acceptance	68.18	90.91	100	100	95.45	77.27	**81.82**
Average score	5.32	6.11	6.95	8.82	7.32	7.11	6.45
Total score	59.09	67.93	77.27	97.98	80.30	79.04	71.72

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{\exp,i} - MR_{pre,i})^2}{N - p} \qquad (4.16)$$

$$E\% = \frac{100}{N}\sum_{i=1}^{N}\left|\frac{(MR_{\exp,i} - MR_{pre,i})}{MR_{\exp,i}}\right| \qquad (4.17)$$

Both $\chi 2$ and E% were calculated by using equations (4.16 and 4.17) in Excel sheet. Some sample calculations are shown below:

FS-1 (SPMDH): *[Sample calculation for Newton model].*

exp[(-)kt]	**MRpre - MRexp**	**SQ. of(MRpre - MRexp)**	**RMSE=Sq.root[(1/8)∑(MRpre-MRexp)^2]**	N-p(no. of constants)	**Chai sq.=(1/7)∑(MRpre-MRexp)^2]**	**MRexp - MRpre**	MOD. Of[(MRexp - MRpre)/MRexp]	E%=(100/N)*∑
					0.013543969			
1	0	0	**0.108862172**	8-1 = 7		0	0	**8.206162848**
0.769588	-0.198252	0.039303864				0.198252021	0.198252021	
0.5922657	-0.1344143	0.018067216				0.134414343	0.134414343	
0.4558005	-0.1240395	0.01538579				0.12403947	0.12403947	
0.3507786	-0.0717814	0.005152568				0.071781392	0.071781392	
0.269955	-0.016825	0.000283081				0.016825	0.016825	
0.2077541	0.0608841	0.003706876				-0.060884123	0.085985877	
0.1598851	0.1136151	0.012908385				-0.113615075	0.025194925	
		0.09480778				∑=	**0.656493028**	

FS-2 (SPMDH): *[Sample calculation for Newton model].*

MRpre							MOD. Of column L	
exp[(-)kt]	MRpre - MRexp	SQ. of(MRpre - MRexp)	RMSE=Sq.root[(1/7)∑(MRpre-MRexp)^2]	N-p(no. of constants)	Chai sq.=(1/6)∑(MRpre-MRexp)^2]	MRexp – Mrpre	MOD. Of[(MRexp - MRpre)/MRexp]	E%=(100/N)*∑
1	0	0	**0.198759919**	7-1 = 6	**0.046089756**	0	0	**14.49078004**
0.6721996	-0.286340428	0.081990841				0.286340428	0.286340428	
0.4518523	-0.332707735	0.110694437				0.332707735	0.332707735	
0.3037349	-0.234915101	0.055185105				0.234915101	0.234915101	
0.2041705	-0.149409531	0.022323208				0.149409531	0.149409531	
0.1372433	-0.002196698	4.82548E-06				0.002196698	0.002196698	
0.0922549	0.079624889	0.006340123				-0.079624889	0.008785111	
		0.276538538				∑=	1.014354603	

TABLE 5.21: Vacuum-drying (VD) models for two sets of fish samples (FS-1 and FS-2) using SPMDH. *[Sample result for Newton model].*

Model	SET	K	A	B	C	N	R^2	RMSE	χ^2	E (%)
Newton	FS-1	0.0175	—	—	—	—	0.938	**0.10886**	**0.0135**	**8.21**
	FS-2	0.02649					0.9005	**0.19876**	**0.0460**	**14.49**

ANNEXURE-2

All the testings were done by two renowned food testing houses such as Mitra S.K. Private Limited, Kolkata and Edward Food Research & Analysis centre Limited (efrac), Kolkata. Few selective tests (such as water activity, microbiological analysis) were done in IIT, Kharagpur (Central Facility Lab).

All the standard methods have been followed for the execution of the test parameters (Moisture, Total Carbohydrate, Total Sugar, Protein, Total fat, Total ash, Crude fiber, Energy, vitamin A, vitamin C, Titrable acidity, fatty acids, histamine, TVBN, mercury, different essential elements such as Na, K, Fe, Zn, Ca, P, K, Cu, Mg, S). The procedures and hence instruments used for the detection of those parameters are well calibrated according to their calibration protocol.

The **certificate of accreditation of NABL** (National Accreditation Board for Testing and Calibration Laboratories) in favour of **Mitra S. K. Private Limited** is attached, which signify that the instruments used for the testing are well calibrated.

A sample **test report done by efrac** of a fortified and blended kiwi fruit powder sample (dated: 31/12/2016) is attached herewith.

ANNEXURE-3

COST ANALYSIS

EXPERIMENT SET-1

MATERIALS COST

Name of the material	Required amount (in g)	Unit cost (in Rs.)	Total cost (in Rs.)
Kiwi fruit	5.922 g (Weight of kiwi slice before OD at 40 Bx, 50°C and 3 h).	Rs. 40 / 90 g (average wt. of one kiwi)	Rs. 2.63
Sugar	32 g	Rs. 40 / kg	Rs. 1.28
Table or common salt	2 g	Rs.30 / kg	Rs. 0.06
TOTAL:	39.922 g		**Rs. 3.97**

After OPOD, the weight of kiwi slice = 2.89 g.

ENERGY UTILIZATION

[A] For Osmotic Dehydration:

Heater power = 500 W = 0.5 kW.

Total time (optimization of OD) = 3 h.

Total energy = 0.5 × 3 = **1.5 kWh**.

[B] For Sublimation purpose:

Two heaters (5 W each) = 10 W = 0.01 kW.

Freeze-drying time (using SCSH) = 2.5 h.

Total energy = 0.01 × 2.5 = **0.025 kWh**.

[C] For Freeze-drying (under vacuum): 1.5 kW / kg of material.

For 2.89 g (OD) material energy required = 0.005 kW

Total energy utilization for FD = 0.005 kW × 2.5 h = **0.0125 kWh**.

[D] Total energy = (1.5 + 0.025 + 0.0125) kW = **1.5375 kWh**.

Cost of 1 kWh = Rs.6/-

Therefore, cost on energy = Rs. 6 × 1.5375 kWh = **Rs. 9.22/-**.

[E] Cost of Raw materials + energy = Rs.(3.97 + 9.22) = **Rs. 13.19/-**.

[F] Labour + others cost = 10% of [Cost of Raw materials + energy] (Timmerhaus *et al.*, 2011) = 10% of [E] i.e. 10% of Rs.32.60 = **Rs. 1.32/-**.

[G] Therefore, TOTAL COST = [E] + [F] = Rs. (13.19 + 1.32) = **Rs.14.51/-**.

Final product weight = 2.52 g (M= 4.38%).

Therefore, Product cost = **Rs. 575.80/- per 100 g.**

EXPERIMENT SET-2

MATERIALS COST

Name of the material	**Required amount (in g)**	**Unit cost (in Rs.)**	**Total cost (in Rs.)**
Kiwi fruit	100 g	Rs. 40 / 90 g (average)	Rs. 44.44
Ground Sugar	0.5 g	Rs. 50 / kg	Rs. 0.025
Lona salt	0.5 g	Rs. 40 / 100 g	Rs. 0.2
Rice flour	1 g	Rs. 40 / kg	Rs. 0.04
Oat flour	1 g	Rs. 100 / 500 g	Rs. 0.2
Calcium lactate	1 g	Rs. 28 / 25 g	Rs. 1.12
Sodium benzoate	0.02 g	Rs. 20 / 20 g	Rs. 0.02
TOTAL:	104.02 g		Rs. 46.045

Out of 104.02 g (cost Rs. 46.045), only **30 g** is used (BKF) for FD.

Therefore, the cost of 30 g blended product = **Rs.13.28**.

ENERGY

[A] For Blending:

Motor power = 500 W = 0.5 kW.

Total time (blending) = 30 s (maximum) = 0.0083 h.

Total energy = 0.5 × 0.0083 = **0.0042 kWh**.

[B] For sublimation purpose:

Cubic heater = 33 W = 0.033 kW.

Freeze-drying time (using SCSRP) = 4.5 h.

Total energy = 0.033 × 4.5 = **0.1485 kWh**.

[C] For Freeze-drying (under vacuum): 1.5 kW / kg of material.

For 30 g (BKF) material energy required = 0.045 kW

Total energy utilization for FD = 0.045 kW × 4.5 h = **0.2025 kWh**.

[D] Total energy = (0.0042 + 0.1485 + 0.2025) kWh = **0.3552 kWh**.

Cost of 1 kWh = Rs.6/-

Therefore, cost on energy = Rs. 6 × 0.3552 kWh = **Rs. 2.14**.

Cost of Raw materials + energy = Rs.(13.28 + 2.14) = Rs. 15.42/-

[E] Labour + others cost = 10% of [D] i.e. 10% of Rs.15.42 = Rs. 1.54/-

[F] Therefore, TOTAL COST = Rs. (15.42 + 1.54) = Rs.16.96/-

Final product weight = 9.9 g (M= 3.0%).

Therefore, Product cost = **Rs. 171.33/- per 100 g.**

EXPERIMENT SET-3

MATERIALS COST

Name of the material	**Required amount (in g)**	**Unit cost (in Rs.)**	**Total cost (in Rs.)**
Shrimp	50 g	Rs. 400 / kg	Rs. 20
Catla fish	25 g	Rs. 200 / kg	Rs. 5
Chela fish	25 g	Rs.300 / kg	Rs. 7.50
Table or common salt (for making of 1.5% brine solution, volume 50 ml)	0.75 g	Rs.30 / kg	Rs. 0.02
Rice flour	10 g	Rs. 40 / kg	Rs. 0.40
Corn flour	2 g	Rs. 27 / 100 g	Rs. 0.54
Dried ginger powder	1 g	Rs. 60 /100 g	Rs.0.60
TOTAL:	113.75 g		Rs. 33.46 (FS-1) Rs. 34.06 (FS-2)

Out of 113.75 g [cost Rs. 33.46 (FS-1)], for VD only **25 g** is used.

Therefore, the cost of 25 g fortified and blended product [FS-1] = **Rs.7.35**.

Out of 113.75 g [cost Rs. 34.06 (FS-2)], for VD only **25 g** is used.

Therefore, the cost of 25 g fortified and blended product [FS-2] = **Rs.7.48**.

ENERGY

[A] For Sterilization:

Heater power = 500 W × 2 = 1 kW.

Total time (come-up + sterilization) = (10 + 15) minutes = 25 minutes = 0.42 h.

Total energy = 1 × 0.42 = **0.42 kWh**.

[B] For Blending:

Motor power = 500 W = 0.5 kW.

Total time (blending) = 30 s (maximum) + 30 s (maximum) = 0.017 h.

Total energy = 0.5 × 0.017 = **0.0085 kWh**.

[C] For sublimation purpose:

Cubic heater = 36 W = 0.036 kW.

Vacuum-drying time (using SPMDH for FS-1) = 2.55 h.

Vacuum-drying time (using SPMDH for FS-2) = 3.25 h.

Total energy (for FS-1) for VD = 0.036 × 2.55 = **0.0918 kWh**.

Total energy (for FS-2) for VD = 0.036 × 3.25 = **0.117 kWh**.

[D] For vacuum-drying: 1.5 kW / kg of material.

For 25 g (FS-1 and FS-2) material energy required = **0.0375 kW**.

Total energy utilization for FD [FS-1] = 0.0375 kW × 2.55 h = **0.0956 kWh**.

Total energy utilization for FD [FS-2] = 0.0375 kW × 3.25 h = **0.1219 kWh**.

[E] Total energy [FS-1] = (0.42 + 0.0085 + 0.0918 + 0.0956) kW = **0.6159 KWh.**

Total energy [FS-2] = (0.42 + 0.0085 + 0.117 + 0.1219) kW = **0.6674 kWh.**

Cost of 1 kWh = Rs.6/-

Therefore, cost on energy [FS-1] = Rs. 6 × 0.6159 kWh = **Rs. 3.70**.

Therefore, cost on energy [FS-2] = Rs. 6 × 0.6674 kWh = **Rs. 4.00**.

[F] Cost of Raw materials + energy [FS-1] = Rs.(7.35 + 3.70) = Rs. 11.05/-

Labour + others cost = 10% of [F] = 10% of Rs.11.05 = Rs. 1.10/-

[G] Therefore, TOTAL COST [FS-1] = Rs. (11.05 + 1.10) = Rs.12.15/-

Final product weight [FS-1] = 9.05 g (M= 3.06%).

Therefore, **Product cost [VDFS-1] = Rs. 134.25/- per 100 g**.

[H] Cost of Raw materials + energy [FS-2] = Rs.(7.48 + 4.00) = Rs. 11.48/-

Labour + others cost = 10% of Rs.11.48 = Rs. 1.15/-

[I] Therefore, TOTAL COST [FS-2] = Rs. (11.48 + 1.15) = Rs.12.63/-

Final product weight [FS-2] = 9.20 g (M= 3.06%).

Therefore, Product cost [VDFS-2] = **Rs.137.28/- per 100 g**.

EXPERIMENT SET-4

MATERIALS COST

Name of the material	**Required amount (in g)**	**Unit cost (in Rs.)**	**Total cost (in Rs.)**
Broccolli	5.66 g (before blanching)	Rs. 220 / kg	Rs. 1.25
Table or common salt	2 g	Rs.30 / kg	Rs. 0.06
TOTAL:			Rs. 1.31

After cold blanching, the weight of broccoli = **3.10 g**.

ENERGY

[A] For Cold blanching:

Heater power = 500 W = 0.5 kW.

Total time (blanching) = 10 minutes = 0.17 h.

Total energy = 0.5 × 0.17 = **0.085 kWh**.

[B] For sublimation purpose:

Two heaters (5 W each) = 10 W = 0.01 kW.

Freeze-drying time (using SCSH) = 5.5 h.

Total energy = 0.01 × 5.5 = **0.055 kWh**.

[C] For Freeze-drying (under vacuum): 1.5 kW / kg of material.

For 3.10 g (blanched) material energy required = 0.005 kW

Total energy utilization for FD = 0.005 kW × 5.5 h = **0.0275 kWh**.

[D] Total energy = (0.085 + 0.055 + 0.0275) kWh = **0.1675 kWh**.

Cost of 1 kWh = Rs.6/-

Therefore, cost on energy = Rs. 6 × 0.1675 kWh = Rs. 1.00/-.

[E] Cost of Raw materials + energy = Rs.(1.31 + 1.00) = Rs. 2.31/-

[F] Labour + others cost = 10% of [E] = 10% of Rs.2.31 = Rs. 0.24/-

[G] Therefore, TOTAL COST = Rs. (2.31 + 0.24) = Rs.2.55/-

Final product weight = 1.4 g (M= 5%).

Therefore, Product cost = **Rs. 182.14/- per 100 g**.

SUMMARY:

Expt. Set no.	Product	Total Cost of product per 100 g
1	Osmotic dehydrated & freeze-dried kiwi (OPODFD)	Rs. 575.80/-
2	Blended & fortified freeze-dried kiwi powder (BFDKF powder)	Rs. 171.33/-
3	Sterilized Shrimp with two fishes fortified with flours and vacuum-dried fish powder (VDFS-1 and VDFS-2)	Rs. 134.25/- (VDFS-1)
		Rs. 137.28/- (VDFS-2)
4	Cold blanched and freeze-dried broccoli	Rs. 182.14/-

The profit is 30% of total cost of the product (Satyanarayana *et. al*., 2007), so the product selling price in the market as per Table below:

Expt. Set no.	Product	Market price of product per 100 g
1	Osmotic dehydrated & freeze-dried kiwi (OPODFD)	Rs. 748.54/-
2	Blended & fortified freeze-dried kiwi powder (BFDKF powder)	Rs. 222.73/-
3	Sterilized Shrimp with two fishes fortified with flours and vacuum-dried fish powder (VDFS-1 and VDFS-2)	Rs. 174.52/- (VDFS-1)
		Rs. 178.46/- (VDFS-2)
4	Cold blanched and freeze-dried broccoli	Rs. 236.78/-

Expt. Set-1] However, if OPODFD slice (weight 2.52 g, moisture 4.38%) is rehydrated to initial raw kiwi slice (moisture 82.5%), then the weight of final slice will be 13.77 g [by the total mass and water component balance]. The cost of this 13.77 g rehydrated sample = cost of 2.52 g slice i.e. Rs. 14.51. Again, cost of 90 g raw kiwi = Rs. 40. Therefore, the cost of 13.77 g raw kiwi = Rs. 6.12/-

Alternatively, 100 g raw kiwi cost = Rs. 44.44/-

100 g rehydrated kiwi slice cost = Rs. 105.37/-

Expt. Set-2] However, if BFDKF powder (weight 9.90 g, moisture 3%) is rehydrated to BKF (moisture 68%), then the weight of final BKF will be 30.01 g [by the total mass and water component balance]. The cost of this 30.01 g rehydrated sample = cost of 9.9 g BFDKF powder i.e. Rs. 16.96.

Again, cost of 104.02 g BKF = Rs. 46.045. Therefore, the cost of 30.01 g BKF = Rs. 13.28/-

Alternatively, 100 g BKF cost = Rs. 44.26/-

100 g rehydrated BKF cost = Rs. 56.51/- [NOTE: 100 g raw kiwi cost = Rs. 44.44/-]

Expt. Set-3] However, if VDFS-1 (weight 9.05 g, moisture 3.06%) is rehydrated to FS-1 (moisture 64.9%), then the weight of final FS-1 will be 25.04 g [by the total mass and water component balance]. The cost of this 25.04 g rehydrated sample = cost of 9.05 g VDFS-1 i.e. Rs. 12.15/-.

Again, cost of 113.75 g FS-1 = Rs. 33.46/-. Therefore, the cost of 25.04 g FS-1 = Rs. 7.36/-

Alternatively, 100 g FS-1 cost = Rs.29.39/-

100 g rehydrated VDFS-1 cost = Rs. 48.52/-

However, if VDFS-2 (weight 9.20 g, moisture 3.06%) is rehydrated to FS-2 (moisture 64.34%), then the weight of final FS-2 will be 25.01 g [by the total mass and water component balance]. The cost of this 25.01 g rehydrated sample = cost of 9.20 g VDFS-2 i.e. Rs. 12.63/-.

Again, cost of 113.75 g FS-2 = Rs. 34.06/-. Therefore, the cost of 25.01 g FS-2 = Rs. 7.49/-

Alternatively, 100 g FS-2 cost = Rs.29.94/-

100 g rehydrated VDFS-2 cost = Rs. 50.50/-

Expt. Set-4] However, if freeze-dried broccoli (weight 1.40 g, moisture 5%) is rehydrated to initial raw broccoli (moisture 88%), then the weight of final broccoli will be 11.08 g [by the total mass and water component balance]. The cost of this 11.08 g rehydrated sample = cost of 1.4 g FD broccoli i.e. Rs. 2.55/-.

Again, cost of 1000 g raw broccoli = Rs. 220/-. Therefore, the cost of 11.08 g raw broccoli = Rs. 2.44/-

Alternatively, 100 g raw broccoli = Rs.22/-

100 g rehydrated freeze-dried broccoli cost = Rs. 23.01/-

SUMMARY

Expt. Set no.	Product	Cost of raw material per 100 g	Cost of rehydrated product	Increment Price
1	Osmotic dehydrated & freeze-dried kiwi (OPODFD)	Rs. 44.44/-	Rs. 105.37/-	137.11%
2	Blended & fortified freeze-dried kiwi powder (BFDKF powder)	Rs. 44.26/- (BKF)	Rs. 56.51/-	27.68% (w.r.t. BKF)
				27.16% (w.r.t. raw KF)
3	Sterilized Shrimp with two fishes fortified with flours and vacuum-dried fish powder (VDFS-1 and VDFS-2)	Rs. 29.39/- (FS-1) Rs. 32.50/- (Shrimp, Catla, Chela; 2:1:1)	Rs.48.52/-	65.09% (w.r.t. FS-1)
				49.29% (w.r.t. shrimp, catla & chela)
		Rs. 29.94/- (FS-2)	Rs.50.50/-	68.67% (w.r.t. FS-2)
				55.38% (w.r.t. shrimp, catla & chela)
4	Cold blanched and freeze-dried broccoli	Rs. 22/-	Rs. 23.01/-	4.6%

ANNEXURE-4

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Dehydration of Kiwi Fruit (*Actinidia deliciosa*) by Consecutive Osmotic Dehydration and Freeze-Drying

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Abstract

Objectives: In this study, a highly nutrient food rich in vitamins and minerals, kiwi fruit (*Actinidia deliciosa*), is dehydrated effectively at a faster rate keeping almost all its original properties. **Methods/Analysis:** A new methodology, consecutive Osmotic Dehydration (OD) and Freeze Drying (FD), was developed and applied. This technique was found to be very effective with respect to drying time, cost and quality. The OD was performed using sucrose solution at three concentrations (40°B, 55°B, and 70° B), three temperatures (40°C, 45°C, 50°C) and three time spans (2, 3 and 4 h). OD parameters were optimized by "Taguchi orthogonal design" methodology. **Findings:** Optimal time for OD was found to be 3 h, at 40°B, 50°C corresponding to 60.75% water loss and 9.86% solute gain. Notably, at optimal OD conditions, application of fructose solution resulted unfavorable result. In FD of osmotically dehydrated kiwi fruit, the thermal energy was supplied by an innovative Silver Coated Steel Heater (SCSH), which was found to be much more efficient (required only 2.5 h drying time) compared to an equal power conventional steel heater (SH) (required 3.82 h drying time) to achieve a final kiwi fruit moisture content ≤ 5% (w/w). At both optimal OD and FD, the drying kinetics was assessed using four models. The values of effective diffusivity (D_{eff}) and activation energy indicated higher energy-efficiency for SCSH compared to SH. The proximate nutritional analyses for fresh and dehydrated kiwi samples indicated very good quality of the processed product. **Novelty/Improvement:** The uniqueness and novelty of this research work is less drying time and almost no nutritional loss as compared with other conventional techniques.

Keywords: Consecutive Osmotic Dehydration and Freeze Drying, Kiwi Fruit, Silver Coated Steel Heater

1. Introduction

Freeze-Drying (FD) has been considered as the best drying technology for food preservation regarding flavor and nutritional parameters, reduced weight of shipping, storage, and handling and enhance shelf life of the end product(s)[1,2]. FD is applied for 1. High nutritional valued foods, 2. With initial moisture content of 70% to 80% (w/w), 3. Costly foodstuffs[3] such as Kiwi Fruit (KF).

KF possesses health promoting nutrients such as phyto-chemicals, minerals and provides sufficient amount of vitamins and antioxidants. Besides KF is high in dietary fiber, K and Mg; low in saturated fat, Na and cholesterol[4,5]. A study found[6] that KF is the most enriched fruit in terms of vitamin C content compared to papaya, mango and orange. It is important for cardiovascular health. It helps in reducing weight, cholesterol level and acts as an anti-aging substance with positive effect to regulate diabetics and colon cancer.

Conventional Freeze-Drying (CFD)[7] was applied for kiwifruit. The results indicated that its betterment was necessary for economization of freeze-drying. In order

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to intensify the freeze-drying rate, infrared radiation was applied as a source of thermal energy for prawn[8] and banana[9]; however, occurrence of protein denaturation was observed. More recently, Osmotic Dehydration (OD) was reported to be a pre-processing before FD for reduction of FD time with improved process economy[10,11]. However, to the best of our knowledge no report is available on application of OD prior to FD. Besides application of fast conductive heating[12–14] using high conductivity metal (such as silver) has not be reported for reduction of FD times. Notably, silver has a high thermal conductivity and antimicrobial properties.

In the present study, the OD of KF using sucrose solution has been optimized applying Taguchi orthogonal method. At the derived optimal condition, effect of fructose as osmotic solution on OD of KF has been evaluated. Subsequent FD of osmotically dehydrated KF was conducted using Silver Coated Steel Heater (SCSH) and its performance in FD was compared with conventional Steel Heater (SH). Kinetic parameters in OD and FD after OD (ODFD) were also compared. The final dehydrated KF was evaluated for quality assessment viz. Carbohydrate, sugar, protein, total fat, ash, vitamins etc.

2. Materials and Methods

2.1 Sample Preparation for Osmotic Dehydration (OD)

Fresh kiwi fruits (*Actinidia deliciosa*) were de-skinned and sliced in the form of slabs with a typical dimension of 35×33×5 mm (±0.1 mm). The initial weight and moisture content of the samples were determined (by moisture meter; KERN, Germany, Model MLS 50-3 HA 250). The OD was performed by using sucrose (99% purity) solution (SS) of three concentrations (40°B, 55°B, and 70° B) with addition of 2 to 3% sodium chloride (W/V) solution. Three temperatures (40°C, 45°C, 50°C) and three time spans (2, 3 and 4 h) were chosen for OD. The volume of the total osmotic solutions was taken 5 times of kiwi sample weight. KF samples were placed in prepared osmotic solutions for specified times as per Taguchi orthogonal experimental design shown in Table 1. After OD, the samples were withdrawn cautiously and soaked by tissue paper and the final dimension, weight and moisture content were measured. The Water Loss (WL) and Solute Gain (SG)[10] of the kiwi samples were calculated by following equations:

$$WL = \frac{(W_0 - W_t) + (S_t - S_0)}{W_0} \times 100 \quad (1)$$

$$SG = \frac{(S_t - S_0)}{W_0} \times 100 \quad (2)$$

$$\text{Where, } S_0 = \frac{W_0(100 - M_i)}{100} \quad (3)$$

$$S_t = \frac{W_t(100 - M_f)}{100} \quad (4)$$

To evaluate the effect of fructose (99% purity) as osmotic solution OD was done at same optimal brix, temperature and time parameters as used for Sucrose Solution (SS). The KF sample (obtained at optimal OD using sucrose solution) was frozen through conventional refrigeration for a time span of 24 hours at temperature -16°C. Then by using SCSH and SH as thermal source, freeze-drying was performed for frozen osmotically dehydrated kiwi sample. The Freeze-Drying (FD) chamber was subjected to fixed vacuum (5×10^{-3} mbar) and the condenser (moisture traps) temperature was fixed at – 40°C. The result of OD with Fructose Solution (FS) was inferior in terms of moisture reduction. Hence, FD was not conducted for sample obtained using fructose as Osmotic Solution (OS).

Conventional Freeze-Drying (CFD) without OD was performed at identical conditions as mentioned above and final moisture content of KF sample was recorded.

2.2 Experimental Methodology for Freeze-Drying (FD)

An indigenously fabricated SCSH was made (fiber body, contained two small heaters of 2.5W each; 84×84×84 mm) for supplying conductive heat in FD for both Osmotically Dehydrated Freeze-Drying (ODFD) and Conventional Freeze-Drying (CFD). The sample was placed on silver coated steel plate of Silver Coated Steel Heater (SCSH) of dimension (82×82× 0.5 mm). One RTD was used for measuring the temperature of the heating plate and it was regulated with a PID controller. During FD, weight of KF sample was recorded at regular time interval. The FD time was noted when the final moisture content of the sample became ≤ 5% (w/w).

Various drying models namely Page, Wang and Sing, Henderson and Pebis and Newton models were used to compare the drying kinetics during OD and FDs. The statistical parameters such as the Mean Bias Error (MBE), the Root Mean Square Error (RMSE) and the modeling efficiency (EF)[15] are calculated by the following equations:

$$MBE = \frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right) \quad (5)$$

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)^2\right]^{0.5} \quad (6)$$

$$EF = \frac{\sum_{i=1}^{N}\left(MR_{\exp,i} - MR_{\exp,avge}\right)^2 - \sum_{i=1}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)^2}{\sum_{i=1}^{N}\left(MR_{\exp,i} - MR_{\exp,avge}\right)^2} \quad (7)$$

The unsteady state diffusion equation for the calculation of effective diffusion coefficient (D_{eff}) is given as follow Crank[23].

$$MR = \frac{8}{\pi^2}Exp\left[-\frac{\pi^2 * Deff * t}{4L^2}\right] \quad (8)$$

The activation energy (E) is calculated by Arrhenius equation:

$$\ln\left(\frac{D_{eff,2}}{D_{eff,1}}\right) = \left(\frac{E}{R}\right)\left[\frac{(T_2 - T_1)}{T_1 * T_2}\right] \quad (9)$$

2.3 Taguchi Orthogonal Design and Optimization of OD

Water Loss (WL) was selected as the response for OD operation. The signal to noise (S/N) ratios for all the OD experiments was calculated according to the following equation. For larger is better (WL) the equation is:

$$S/N = -10\log\left[1/(n\sum_{i=1}^{n}1/Y_i^2)\right] \quad (10)$$

Where i and n are the number of replicates and trial experiments performed in all the combinations respectively.

2.4 Quality Assessment

The carbohydrate, sugar, protein, total fat, energy, ash, minerals (Na and K) and vitamins (A and K) of the raw KF, osmotically dehydrated KF and freeze-dried KF were measured using standard methods as given in Table 2.

3. Results and Discussion

Moisture content of fresh KF was determined to be 75.75% (average) on wet basis.

3.1 Optimal Process Conditions

The analysis of S/N ratio obtained from Taguchi orthogonal design with the optimization criteria i.e., "larger is better" for WL in OD of KF are presented in Table 1. The maximum WL was obtained for the combination of 40° brix, 50°C temperature and 3 h OD time.

Table 1. Taguchi orthogonal experimental design for OD of kiwi fruit

Sl. No.	Brix (°B)	Temperature (°C)	Time (h)	WL %	S/N ratio
1	40	40	2	57.229±0.2	35.15232
2	40	45	3	50.079±0.3	33.99311
3	40	50	4	61.861±0.2	35.82834
4	55	40	3	63.91±0.5	36.11138
5	55	45	4	51.397±0.5	34.21876
6	55	50	2	49.667±0.5	33.92136
7	70	40	4	52.901±0.4	34.46928
8	70	45	2	43.49±0.3	32.76779
9	70	50	3	64.376±0.1	36.17448

3.2 Comparison between Sucrose Solution (SS) and Fructose Solution (FS) used as an Osmotic Solution

The KF sample was also dehydrated through fructose solution (used as an osmotic solution, OS) at derived optimized conditions (40°B, 50°C and 3 h). Under the optimized conditions in OD the WL was 54.54% and SG was 14.45% which were much inferior to OD with SS (WL: 60.75% and SG was 9.86%). Thus, nearly10% and 32% better performance on the basis of WL (%) and SG (%) for SS as compared to FS. Therefore, it may be concluded that at optimized osmotically dehydrated (OPOD) condition, the Sucrose Solution (SS) was far better Osmotic Solution (OS) than Fructose Solution (FS).

3.3 Effects of Individual Factors on WL

It is evident from Figure 1 that an increase in brix can cause decrease in WL% which is unfavorable; while increase in temperature from 45°C to 50°C could result an increase in WL. An interesting observation pertaining to the effect of OD time on WL revealed that an increased in OD time from 2 to 3 h could render a significance in WL; however further increase in OD time had contrasting effect on WL. This may be ascribed to the reverse osmosis during extended period of OD. It was found from the results of ANOVA (Table 3.) that all the factors (brix, time and temperature) were significant but temperature was the most important (rank 1) among all with maximum delta value (1.66).

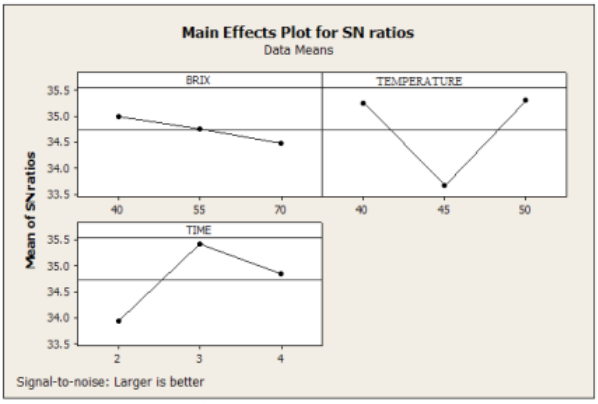

Figure 1. Effects of individual process factors on signal-to-noise (S/N) ratio for water loss (WL).

Table 3. Effects of individual factor on OD interpreted through ANOVA (analysis of variance) [for WL, larger is better]

Level	Brix (°B)	Temperature (°C)	Time (h)
1	35.01	35.25	33.95
2	34.78	33.67	35.46
3	34.47	35.33	34.85
Delta	0.54	1.66	1.50
Rank	3	1	2

3.4 Interaction among Process Factors in Governing WL

It is evident from Figure 2, that an increase in temperature from 40°C to 45°C resulted a decrease in WL% for all brix values of Osmotic Solution (OS). However, increase in temperature from 45°C to 50°C resulted increment in WL% for 40°B and 70°B. Again at 50°C, the WL% was more as compared with other two temperatures (40°C and 45°C). Also at 3 hour time the WL% reached maximum as compared with other two time parameters (2 h and 4 h). Therefore, to get WL% maximum the optimized process parameters were 40°B, 50°C and 3 h.

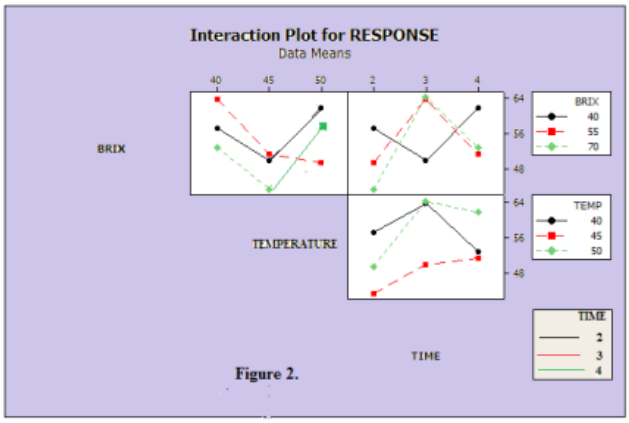

Figure 2. Interaction plot among process factors governing in water loss (mean) in osmotic dehydration of kiwi fruit.

3.5 Kinetics of Osmotic Dehydration of Kiwi Fruit Sample

The OD kinetics at optimal OD (OPOD) was compared with four models namely Page, Wang and Sing, Henderson and Pabis and Newton model (Table 4). Different constant values (K, a, b, n), regression coefficient (R^2) values, modeling efficiency (EF), Mean Bias Error (MBE) and Root Mean Square Error (RMSE) values are required (Table 5) to evaluate the best representative model. The Newton model was accepted as the best drying models for OPOD. The Moisture Ratio (MR) values as a function of OD time were compared for actual (experimental) and predicted values for all the four models in Figure 3.

Table 4. Mathematical models selected

Serial no.	Model name	Equation used
1.	Page	$MR = \exp(-kt^n)$
2.	Wang & Sing	$MR = 1 + at + bt^2$
3.	Henderson & Pabis	$MR = a \exp(-kt)$
4.	Newton	$MR = \exp(-kt)$

Table 5. Models for osmotic dehydration at optimal conditions (OPOD)

Model	K	a	b	n	R^2	EF	MBE	RMSE
Page	0.017	___	___	0.783	0.913	0.88934	0.06774	0.00769
Wang & Sing	___	- 0.006	9.8 x10^{-6}	___	0.965	0.82705	-0.00300	0.10965
Henderson & Pabis	0.00461	0.865	___	___	0.9745	0.83888	0.04168	0.10860
Newton	0.007	___	___	___	0.9444	0.90335	0.01708	0.08412

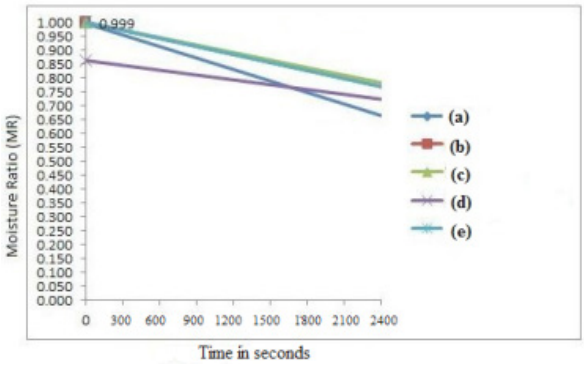

Figure 3. Osmotic dehydration curves at optimal condition of OPOD.(a) Experimental value.(b) Page model.(c) Wang and Sing model.(d) Henderson and Pabis model. (e) Newton model.

3.6 Freeze-Drying of KF after OPOD

Partially dehydrated kiwi samples (30.12% on wet basis) developed through optimized OD operation (OPOD) were subsequently subjected to freeze-drying (OPODFD) using Silver Coated Steel Heater (SCSH) till the final moisture content became ≤ 5.0 % (wet basis). The actual (experimental) OPODFD kinetics obtained by plotting (Figure 4.) Moisture Ratio (MR) as a function of time was compared with four standard drying models viz. Page, Wang and Sing, Henderson and Pabis and Newton model (Figure 4.) to determine the best representative model for OPODFD dynamics. Regression coefficient (R^2), MBE, RMSE and EF values and constants (namely k, a, b, n) are presented in Table 6.

From the Table 6 and Figure 4 it can be observed that Wang and Sing model was best fitted as compared with other three models i.e., it was the best representative drying model for freeze-drying kinetics of kiwi fruit sample.

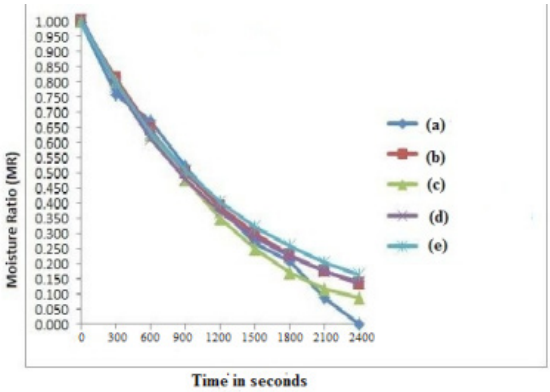

Figure 4. Freeze-drying curves of OPODFD for.(a) Experimental value.(b) Page model.(c) Wang and Sing model.(d) Henderson and Pabis model.(e) Newton model.

3.7 Comparison of Freeze-Drying (FD) Performances between SCSH and SH

From Equation (8), the effective moisture Diffusivity (D_{eff}) for OPOD was found as 3.405×10^{-9} m^2/s (Table 7). Similarly, the effective moisture diffusivity in OPODFD (using SCSH) of KF was found 6.04×10^{-7} m^2/s (Table 7). On the other hand, the effective diffusivity for OPODFD (using conventional steel heater) was found as 2.22×10^{-7} m^2/s. Therefore, SCSH was much more efficient than SH for accelerating OPODFD of kiwi fruit.

Table 7. Effective diffusivity for optimal osmotic dehydration (OPOD), optimal freeze-drying of osmotically dehydrated (OPODFD) kiwi samples using steel heater (SH) and silver coated steel heater (SCSH)

Temperature	D_{eff} for OPOD (m^2 / s)	D_{eff} for OPODFD (m^2 / s)	
		Using SH	Using SCSH
50°C	3.405×10^{-9}	2.22×10^{-7}	6.04×10^{-7}
45 °C	1.886×10^{-9}	0.57×10^{-7}	1.50×10^{-7}
40 °C	1.045×10^{-9}	0.157×10^{-7}	0.37×10^{-7}

Table 6. Freeze-drying models for osmotically dehydrated kiwi samples at optimal conditions (OPODFD)

Model	K	a	b	n	R^2	EF	MBE	RMSE
Page	0.035	—	—	1.1	0.947	0.96577	0.03093	0.05773
Wang and Sing	—	-0.042	0.00048	—	0.946	0.98062	-0.00185	0.04343
Henderson and Pabis	0.0501	1.015	—	—	0.928	0.96203	0.02170	0.06080
Newton	0.0452	—	—	—	0.940	0.72485	0.04359	0.07370

To attain a final moisture content (4.27%) of kiwi fruit sample the drying time was 2.5 h and 3.82 h for SCSH and SH respectively. Therefore, the drying time for SCSH was less (about 35%) than SH.

The activation energy (E) by using Arrhenius Equation (9) was calculated. The activation energy required during OPODFD was computed as 23.96 kJ/mol by using SCSH; on the contrary, the activation energy required during OPODFD was 53.55 kJ/mol when using SH plate. Therefore, less activation energy is required for OPODFD by SCSH as compared with SH; indicating SCSH as an energy-efficient heating device.

For Conventional Freeze-Drying (CFD) without OD, the drying time was required 5.75 h for KF sample to reach final moisture content 4.27%.

3.8 Quality Assessment of Raw and Dehydrated Kiwi Fruit

According to nutrition information panel[16] every food processing operations require to provide information on certain nutrients. Eleven quality tests were performed for three types of kiwi samples namely 'Raw', OPOD and OPODFD (final dried), shown in Table 2, where the reference of methods are also mentioned. The quality of all the three types with respect to the five parameters viz. moisture, carbohydrate[17], protein, total fat and ash content (in g per 100 g sample) were evaluated which indicated satisfactory quality.

Table 2 shows that carbohydrate increased by 150.78% and sugar increased by 54.58% for OPOD sample. The

Table 2. Qualities of raw and dehydrated kiwi samples

Quality Parameters	Standard # values for fresh KF*	Tested fresh KF	KF samples for OPOD	KF samples for OPODFD	Test method	Reference(s)
Moisture (g/100g)	70-85	85.07	68.16	4.27	DGHS LAB MANUAL 5.0	Krokida et al., 1998
Carbohydrate(g/100g)	14.66	10.89	27.31	87.52	IS:1656-2007	fssai, act 2006, GOI; Maskan, 2001
Sugar(g/100g)	8.99	8.30	12.83	56.78	IS:6287-1985 (Reaffirmed-2005)	_____
Protein (g/100g)	1.14	1.31	1.34	2.82	IS:7219-1973 (R.A.-2005)	_____
Total Fat (g/100g)	_____	0.52	0.55	0.65	DGHS LAB MANUAL 5.0	_____
Energy Content (kJ/100g)	260	259.8	512.76	1548.64	IS:9487-1980 (R.A.-2005)	fssai, act 2006, GOI
Sodium (mg/100g)	3	2.81	432.48	1072.04	QA.16.5.2	fssai, act 2006, GOI
Potassium (mg/100g)	312	312.53	152.51	252.02	QA.16.5.2	fssai, act 2006, GOI; Deman, 2004
Ash (g/100g)	0.88-1.02	1.12	1.86	3.92	DGHS LAB MANUAL 5.0	fssai, act 2006, GOI; Fourie and Hansmann, 1992
Vitamin A (µg/100g)	Trace	20.0	20.0	20.0	QA.16.5.3	fssai, act 2006, GOI; Mudambi and Rajagopal, 1990
Vitamin C (mg/100g)	90	89.10	80.55	78.66	AOAC 18-th edition, 967.21	fssai, act 2006, GOI

#FSSAI*Information on nutritional value of Fresh Kiwi

reason was water expulsion and sugar uptake for KF samples. Increment behavior of carbohydrates and sugars were observed for final dried samples obtained through OPODFD owing to additional moisture removal. All the dehydrated samples contained more protein than raw samples per unit mass of sample (Table 2). Previously, no report of fat content was made for KF. However, 0.52 g, 0.55 g and 0.65 g fat (per 100 g tested sample) was found for raw (fresh) KF, OPOD and OPODFD samples respectively. However, the temperature was kept within 50°C therefore no loss of fats occurred due to processing. The energy content were found higher than raw sample for OPOD (512.76 kJ per 100 g) and OPODFD (1548.65 kJ per 100 g) samples. Hence, it could be concluded that the final dried kiwi sample (100g) was an energy 'store-house'. Both sodium and potassium present in kiwi were lost whenever thermally processed[16]. Plant and their products have a higher content of potassium than of sodium[18]. Here, during OPOD kiwi was heated at 50°C, accordingly sodium and potassium might be lost. To overcome this situation iodized common salt (5%, w/v) was added. Sodium content increased during OPOD, while potassium content decreased during OPOD, since it was 'salting out' quickly to the sucrose solution. The increase in potassium content from OPOD sample to OPODFD sample could be ascribed due to additional moisture reduction leading to final moisture content of 4.27%. By the same reason, the quantity of sodium for freeze-dried product also increased. Although, the dried KF sample contained less vitamin C (nearly 12%) as compared to raw (fresh) tested KF. None the less, loss in vitamin C reported in present study was much less compared to available reports. The raw kiwi contains ash 0.88 to 1.02 g per 100 g sample[19]. So, initial load of ash in tested KF sample was more as compared with standard value; as expected, the ash content both in OPOD and OPODFD sample increased per 100 g of dried product. Structural properties of foods are strongly affected by material moisture content[20], therefore high moisture level (>80%) suggests that kiwi fruit was very much suited for FD preservation. It was found that, all the carbohydrate and sugar level would be back to original level (raw kiwi) when rehydration step would be performed[21]. Ripen kiwi is a good source of β-carotene. The daily requirement of an adult for vitamin A is of the order of 750 μg of retinol or 3000 μg of β-carotene per day derived from either animal or plant origin[22]. Table 2 shows that in processed kiwi, vitamin A found 20 μg/100g of sample though raw kiwi contains trace amount of vitamin A as per fssai[16].

4. Conclusion

The present work highlighted the dehydration of kiwi fruits through a consecutive osmotic dehydration and freeze-drying protocol. Application of Taguchi orthogonal design could help in finding optimal conditions for osmotic dehydration. Notably, sucrose has been found to be a better osmotic solution when compared with fructose under identical OD conditions.

The silver coated steel heater has been observed much more energy efficient than conventional steel heater in consecutive freeze-drying to obtain the final dried kiwi fruit. The osmotically dehydrated kiwi fruit at optimal conditions subsequently dehydrated employing energy-efficient silver coated heater resulted in a final dried kiwi fruit containing acceptable quality parameters conforming to market status. It is expected that the developed economically-sustainable dehydration protocol may be applied to similar foodstuffs.

The higher effective moisture diffusivity and lower activation energy in freeze-drying are indicative of a faster and energy saving dehydration protocol through application of novel silver (coated steel) heater making the kiwi dehydration process economically sustainable.

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Quality enrichment and preservation of shrimp by fortification with fishes and flours employing fast vacuum-drying

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Abstract

Shrimp (*Metapenaeus monoceros*) is low in fat, essential minerals and vitamins. It contains high moisture (77.15 ± 0.73%), so it needs to be preserved by an energy-efficient process. Therefore, fortification protocols for the enrichment of shrimp were developed employing two fishes namely *Catla catla* (CA) and *Chela cachius* (CS). Pre-sterilised shrimps were blended with sterilised CA and CS at 2:1:1 weight ratio. Pre-blended shrimps were converted to fortified shrimp (FS) by adding corn, rice flour (FS-1) and additionally mixing with dried-ginger (FS-2) and subsequently dehydrated by vacuum-drying (VD) using silver/copper plated multidimensional heater to produce vacuum-dried FS. The VD kinetics indicated faster dehydration of FS-1 as compared to FS-2 in both heaters. Higher effective diffusivity (7.464×10^{-10} m^2/s) and lower activation energy (28.42 kJ/mol) were computed for FS-1 in silver plated heater. The vacuum-dried FS-1 exhibited superior quality through remarkable augmentation in protein (188%), ω-3 fatty acids (20%), carbohydrate (35%), ash (151%) and other essential elements with acceptable water activity, rehydration ratio, TVBN and histamine content.

Abbreviation list

CA: catla fish (*Catla catla*); CS: chela fish (*Chela cachius*); CPMDH: copper-plated multidimensional heater; CF: corn flour; CHD: coronary heart disease; DSC: differential scanning calorimetry; DG: dried ginger; FS-1: fortified shrimp and blended set-1; FS-2: fortified shrimp and blended set-2; MR: moisture ratio; RR: rehydration ratio; RTD: resistance temperature detector; RF: rice flour; SPMDH: silver-plated multidimensional heater; STSP: shrimp-CA-CS pulp; SBM: sterilised brine mix; TVBN: total volatile base nitrogen; VD: vacuum-drying; VDFS-1: vacuum-dried fortified shrimp and blended set-1; VDFS-2: vacuum-dried fortified shrimp and blended set-1.

Introduction

Shrimp (*Metapenaeus monoceros*) is considered a tasty and healthy foodstuff worldwide. Though it is costly, it is a rich source of protein, energy, minerals (Fe, I, Se, Mg, Na, Mn, Cu, S), pigments (astaxanthin and astacin), and vitamin E (Bogard *et al*., 2015). However, it is low in fat, Zn, Ca, P, K, and vitamins A, B_{12} and D (Bogard *et al*., 2015). So, it needs to be enriched using other foodstuffs to overcome these deficiencies. The presence of high moisture (77.15 ± 0.73%) in shrimp also enhances its deterioration, thereby reducing its shelf-life. Shrimp preservation is thus needed to extend its shelf-life. Therefore, the enrichment of nutrients through fortification and successive reduction of moisture content through energy-efficient drying for preservation of fortified shrimp are very important.

Coronary heart disease (CHD) is a major killer in both developed and developing countries. CHD is also dangerous due to its sudden attack. In Japan, the low death rate from CHD is reported since the

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consumption of fish (rich in ω-3 PUFA) is estimated to be about 100 g/day (Kromhout *et al*., 1985). Hence, to reduce the risk of CHD, the recommended intake of fish is two to three servings per week (Sen, 2005).

The tropical fish such as catla (*Catla catla*) [CA] contains a high amount of K, protein and fat/ω-3 fatty acids (Vanitha, 2011; Bogard *et al*., 2015; Mohanty *et al*., 2016). Vanitha (2011) has developed value-added products from CA. Another tropical fish, chela (*Chela cachius*) [CS], is rich in Zn, Ca, P, Mn, S, and vitamins A, D and B_{12} (Bogard *et al*., 2015). Small indigenous fish species like CS has been used for contributing dietary vitamin A, Ca and Fe in total household fish consumption (Roos *et al*., 2003). Therefore, the deficiency in shrimp can be mitigated by fortification with CA and CS at specific proportions.

Rice flour (RF) functions as a thickening agent, and texture and flavor improver (Duncan, 2000), while corn flour (CF) is rich in carbohydrate, essential elements (Fe, Ca, Cu, K, P, Zn), and vitamins [thiamine, niacin, vitamin B_6] (Sabanis and Tzia, 2009). Jaya Shankar and Bandyopadhyay (2005) previously blended RF with fish powder through an extrusion process. Sabanis and Tzia (2009) used CF along with RF and soy flour to improve bread characteristics. Dried ginger powder (DG) contains a high quantity of β-ionone [anticancer agent] (Liu *et al*., 2008). It has been used as taste and flavor enhancer (Sangwan *et al*., 2014), and is also rich in ash and Ca (Kirk and Sawyer, 1991).

Chakraborty and Samanta (2015) reported an application of vacuum-drying (VD) for producing highly nutritious foodstuffs by enriching alphonso mango with aloe vera (*Aloe barbadensis* Miller) blend. Almeida-Trasvina *et al*. (2014) optimised VD [pressure (46.33 kPa, abs) and temperature (68.33°C)] to preserve antioxidants present in apple pomace, and Alibas (2012) investigated the drying of celeriac slices using VD by different reduced pressures (0.1, 3, 7, 10, 13 and 17 kPa) and temperatures (55, 65 and 75°C). Recently, fish powders were produced for the fortification of biscuits (Mohamed *et al*., 2014), bread (Adeleke and Odedeji, 2010) and ice-cream (Shaviklo *et al*., 2011). Fish oils (Kolanowski and Laufenberg, 2006) contain ω-3 fatty acids, and related products such as microencapsulated fish-oil (Serfert *et al*., 2010) had been used for the preparation of different foodstuffs.

From the literature, however, it is evident that enrichment of shrimp with fish (CA and CS) has not been reported till date. Accordingly, in the present work, shrimp has been fortified by blending with two different fishes (CA, CS) for improvement in nutritional qualities. Moreover, RF, CF, and DG have also been amalgamated with pre-fortified shrimp for further improvement in overall quality. To improve the shelf-life, the fortified shrimp was vacuum-dried using novel silver-plated multidimensional heater (SPMDH) or copper-plated multidimensional heater (CPMDH) to enhance the drying operation. The final qualities of dried products were assessed using standard protocols.

Materials and methods

Sampling protocol

Shrimp, CA and CS were collected from the local market (Haldia, India) and were cleaned with deionised water. After scraping off the scales and gills, CA and CS were gutted using a sterilised knife. CA and CS were then filleted. Shrimps were de-headed, de-tailed and de-veined to obtain the flesh. After cleaning with deionised water, shrimp was stored along with CA and CS in polyethylene bags (51 μm) under refrigeration (5 - 8°C).

Sample sterilisation

Shrimp, CA and CS were taken at weight ratio of shrimp:CA:CS 2:1:1, and immersed into 1.5% (w/v) brine solution kept in a 250 mL stainless steel container (with lid) for sterilisation (at 121°C, 15 psi, 15 min). The moisture [Test Method: IS 1158: 1973 (RA-2010)] and pH (determined by calibrated pH meter: SYSTRONICS, Ahmedabad, India, serial no.7743) of brine solution (1.5%), raw and sterilised shrimp (along with CA and CS) were measured. The whole part of sterilised brine mix (SBM) was used further in the subsequent blending step to prevent nutrient loss from the samples. The pH of SBM was measured by the same instrument.

Fortification of shrimp

The sterilised shrimp along with sterilised CA and CS were blended using a blender (PHILIPS, model no. HL 1632, 230 V, AC, 50 Hz, 500 W) to obtain shrimp-CA-CS pulp (STSP). Subsequently, STSP and SBM were further enriched by mixing with CF and RF at STSP:CF:RF ratio of 2:0.2:0.04 in the same blender by "step wise-short time (20 s to 30 s) addition and mixing methodology". The blending was performed at moderate speed (~400 - 500 rpm) to avoid development of unwanted reaction(s), unpleasant flavours and tastes during preparation of fortified shrimp and blended set-1 (FS-1). Similarly, fortified shrimp and blended set-2 (FS-2) was prepared through the addition of CF, RF and DG to the STSP and SBM at a STSP:CF:RF:DG ratio of 2:0.2:0.04:0.02. The final moisture content and pH

Table 1. Parts (weight basis) of shrimp, CA, CS, STSP, SBM, RF, CF and DG for preparations of FS-1 and FS-2 samples by successive sterilisation, fortification and blending steps.

Fortified shrimp and blended product	Shrimp (sterilised)	CA (sterilised)	CS (sterilised)	Shrimp-CA-CS pulp (STSP)	SBM [1.5% (w/v) brine solution]	Added part to STSP		
						RF	CF	DG
FS-1	2 parts	1 part	1 part	4 parts	whole part (50 mL)	0.4	0.08	-
FS-2	2 parts	1 part	1 part			0.4	0.08	0.04

of both FS-1 and FS-2 were determined. The details of FS-1 and FS-2 preparation are shown in Table 1.

Vacuum-drying (VD) of FS-1 and FS-2

The prepared FS-1 and FS-2 were weighed (approximately 25 ± 0.01 g of a batch) and placed on SPMDH or CPMDH (depth or thickness of sample 0.002 m) and subjected to VD (0.5 Pa vacuum pressure, condenser temperature -40°C). Self-designed and fabricated cuboid (120 × 120 × 90 mm) heater (220 V, 166 mA, 36 W) viz. SPMDH or CPMDH in which the heat was supplied from four sides metallic (stainless steel) walls and bottom; top of the heater was kept open for diffusion of water-vapour from FS-1/FS-2. One resistance temperature detector (RTD) was used for measuring the temperature of the inside wall (stainless steel) and it was connected with the PID controller. The inside wall temperature was set at 55°C (± 1°C). Another RTD was inserted into FS-1/FS-2 for measuring the VD sample's temperature over 40 to 50°C. The weight of the sample undergoing VD was recorded at fixed time interval (15 min) for evaluation of drying kinetics. The VD was continued till the final moisture content of the sample became ≈3% (w/w). The final VD products were marked as VDFS-1 and VDFS-2, corresponding to the FS-1 and FS-2, respectively.

Drying kinetics

Seven drying models namely Newton, Page, Henderson and Pebis, Modified Page, Linear, Wang and Singh, and Modified Wang and Singh were implemented to assess the best representative drying kinetics during VD (Dongbang and Pirompugd, 2015; Fernando and Amarasinghe, 2016; Chakraborty and Samanta, 2017). The selected mathematical models and their equations are shown in Table 2.

The regression coefficient [R^2], root mean square error [RMSE], Chi-square [χ^2] and mean relative deviation [E%] have been evaluated to determine the 'goodness of fit' of the selected models for both FS-1 and FS-2 employing SPMDH and CPMDH. The diffusion equation (unsteady state) for the calculation of effective diffusion coefficient (D_{eff}) is given as reported by Taheri-Garavand and Meda (2018):

$$MR = \frac{8}{\pi^2} Exp\left[-\frac{\pi^2 * D_{eff} * t}{4L^2}\right] \quad \text{(Eq. 1)}$$

where, MR = moisture ratio, t = time in seconds, L = thickness or depth of sample in SPMDH or CPMDH. R^2 was evaluated by Microsoft Excel 2010, while RMSE and χ^2 were calculated by the following equations (Chakraborty and Roychowdhury, 2013):

Table 2. Mathematical models selected and statistical data (R^2, RMSE, χ^2, E%) for two sets of fish samples (FS-1 and FS-2) using SPMDH and CPMDH.

Model	Equation	SET	SPMDH				CPMDH			
			R^2	RMSE	χ^2	E (%)	R^2	RMSE	χ^2	E (%)
Newton	MR = exp (-Kt)	FS-1	0.938	0.109	0.013	8.21	0.928	0.091	0.009	8.76
		FS-2	0.901	0.199	0.046	14.49	0.974	0.052	0.003	18.74
Page	MR = exp(-Kt^n)	FS-1	0.978	0.051	0.004	5.96	0.893	0.159	0.035	13.79
		FS-2	0.989	0.052	0.004	2.30	0.995	0.069	0.007	35.23
Henderson and Pabis	MR = A exp(-Kt)	FS-1	0.918	0.225	0.067	37.81	0.863	0.138	0.026	23.34
		FS-2	0.890	0.966	0.186	35.16	0.939	1.069	0.229	29.48
Modified Page	MR = exp[-$(Kt)^n$]	FS-1	0.986	0.074	0.007	26.21	0.981	0.033	0.001	25.14
		FS-2	0.989	0.051	0.004	2.29	0.854	2.172	6.605	25.58
Linear	MR = A + Bt	FS-1	1.000	0.044	0.003	7.65	1.000	0.069	0.007	22.31
		FS-2	1.000	0.049	0.003	16.17	1.000	0.049	0.003	23.09
Wang and Singh	MR = 1 + At + Bt^2	FS-1	0.996	0.128	0.022	23.49	1.000	0.067	0.006	27.51
		FS-2	0.994	0.055	0.004	4.28	0.997	0.046	0.001	36.03
Modified Wang and Singh	MR = (1 + At + Bt^2) / (1 + Ct)	FS-1	0.989	0.068	0.007	21.88	0.999	0.055	0.005	26.41
		FS-2	0.949	0.281	0.019	9.28	0.998	0.136	0.005	41.67

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{exp,i}\right)^2\right]^{0.5} \quad \text{(Eq. 2)}$$

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{exp,i} - MR_{pre,i})^2}{N-p} \quad \text{(Eq. 3)}$$

where, $MR_{pre,i}$ = predicted moisture ratio, $MR_{exp,i}$ = experimental moisture ratio, N = number of observations, and p = number of constants.

Mean relative deviation (E%)

Absolute value, which provides a clear idea of the mean divergence of the predicted data from the experimental data, was used in the present work to examine the 'goodness of fit'. Value of $E\% < 5$ indicates an 'extremely good fit', 5 to 10% 'reasonably good fit', and > 10% shows 'poor fit' (Lomauro *et al*., 1985; Gencturk *et al*., 1986). The formula to calculate the mean relative deviation is as follows:

$$E\% = \frac{100}{N}\sum_{i=1}^{N}\left|\frac{(MR_{exp,i} - MR_{pre,i})}{MR_{exp,i}}\right| \quad \text{(Eq. 4)}$$

Table 3. pH and moisture of fish samples at different stages of processing before and after vacuum-drying (VD).

pH and Moisture	CA		CS		Shrimp		STSP	SBM	FS-1	FS-2	VDFS-1	VDFS-2
	Raw	Sterilised	Raw	Sterilised	Raw	Sterilised						
pH	5.88 ± 0.02	6.47 ± 0.03	5.80 ± 0.02	6.30 ± 0.03	6.40 ± 0.02	6.66 ± 0.02	6.32 ± 0.03	5.60 ± 0.03	5.90 ± 0.02	6.20 ± 0.03	6.63 ± 0.02	6.33 ± 0.02
Moisture (%)	78.11 ± 0.75	68.87 ± 0.66	74.81 ± 0.71	67.35 ± 0.64	77.15 ± 0.73	68.76 ± 0.65	69.12 ± 0.68	-	64.97 ± 0.62	64.34 ± 0.64	3.06 ± 0.25	3.06 ± 0.25

Data are means ± standard deviation. STSP: sterilised shrimp-CA-CS pulp; SBM: sterilised brine mix; FS: fortified shrimp and blended set; VDFS: vacuum-dried FS product.

Table 4. Proximate analysis and different elements present in VDFS-1 and VDFS-2 (using SPMDH).

Quality Parameter	Shrimp (raw)	VDFS-1	VDFS-2	Test method (s) / [Reference(s)]
Total Carbohydrate (g/100 g) ± SD*	0	34.99 ± 0.36	37.50 ± 0.34	AOAC 986.25 [FSSAI (2010)]
Protein (g/100 g) ± SD	17.6	50.66 ± 0.31	48.78 ± 0.28	IS: 7219 - 1973 [Chakraborty and Samanta (2015)]
Total Fat (g/100 g) ± SD	1	4.71 ± 0.05	4.73 ± 0.05	AOAC 963.15 [Chakraborty and Samanta (2015)]
Total ash (g/100 g) ± SD	2.2	5.52 ± 0.44	5.19 ± 0.42	IS: 1158 - 1973 [FSSAI (2010)]
Energy (kJ/100 g) ± SD	333	1609.26 ± 1.95	1642.28 ± 1.92	Pearson's composition and analysis of foods [FSSAI (2010)]
ω-3 fatty acids (g/100 g) ± SD	2.0	2.4 ± 0.03	2.2 ± 0.03	AOAC 996.06 [Kromhout *et al*. (1985)]
Histamine (mg %) ± SD	-	5 ± 0.01	5 ± 0.01	FSSAI Lab Manual 6 [Sen (2005)]
TVBN (mg/100 g)	-	Below detection limit (5.0)	Below detection limit (5.0)	FSSAI Lab Manual 6: Cl.no. 1.3 [Idakwo *et al*. (2016)]
Mercury (Hg) (mg/kg)	-	Below detection limit (0.01)	Below detection limit (0.01)	AOAC 971.21 [Torres-Escribano *et al*. (2010)]
Fe (mg) ± SD	2.70	5.92 ± 0.88	5.80 ± 0.86	AOAC (1999) [Yanar *et al*. (2004)]
Zn (mg) ± SD	1.30	4.68 ± 0.51	4.56 ± 0.52	AOAC (1999) [Yanar *et al*. (2004)]
Ca (mg) ± SD	550	1259.26 ± 0.11	1272.97 ± 0.10	AOAC (1999) [Yanar *et al*. (2004)]
P (mg) ± SD	290	864.22 ± 0.92	830.75 ± 0.91	AOAC (1999) [Yanar *et al*. (2004)]
K (mg) ± SD	210	528.69 ± 0.78	504.44 ± 0.81	AOAC (1999) [Yanar *et al*. (2004)]
Cu (mg) ± SD	0.49	0.8385 ± 0.42	0.6345 ± 0.52	AOAC (1999) [Yanar *et al*. (2004)]
Mg (mg) ± SD	45	93.77 ± 0.02	91.22 ± 0.02	AOAC (1999) [Yanar *et al*. (2004)]
S (mg) ± SD	190	458.22 ± 0.64	445.66 ± 0.62	AOAC (1999) [Yanar *et al*. (2004)]

SD: Standard deviation.

The relation between activation energy (E) and D_{eff} were calculated by the Arrhenius equation:

$$\ln\left(\frac{D_{eff.2}}{D_{eff.1}}\right)=\left(\frac{E}{R}\right)\left[\frac{(T_2-T_1)}{T_1*T_2}\right] \quad \text{(Eq. 5)}$$

Quality assessment

The moisture (%), pH, total carbohydrate, protein, total fat, energy, ash, essential elements (Fe, Zn, Ca, P, K, Cu, Mg, and S), ω-3 fatty acid, TVBN, histamine and Hg content of the VDFS-1 and VDFS-2 were measured in triplicate, and the means and standard deviations (SD) were reported (Table 3 and Table 4).

Rehydration ratio (RR)

Rehydration ratio (RR) was also measured (Chakraborty *et al*., 2011) for both VDFS-1 and VDFS-2. All the two sets of VD products were properly stored in airtight polythene containers and bag. After that, they were tested for rehydration ratio (RR). Briefly, 5 g sample of stored VD product was taken and dispersed in 100 mL distilled water at 60°C for 30 min. The solution of fish powder (product) and water were filtered by 0.4 µm Whatman filter paper. The filtrate was discarded, and the precipitate was taken. The precipitate was blotted with tissue paper to remove excess water, and the weight of rehydrated sample of two sets (VDFS-1 and VDFS-2) was measured. The RR is expressed by the following equation:

$$RR=\left(\frac{W_{reVDFS}}{W_{VDFS}}\right) \quad \text{(Eq. 6)}$$

where, W_{VDFS} = VDFS sample weight taken for testing (initial), W_{reVDFS} = VDFS sample weight (after rehydration).

Water activity (a_w) and microbiological assay

Water activity (a_w) is an indicator of the available free water (Ramaswamy and Marcotte, 2005). Food can be made safe to store by lowering the a_w to a point that does not allow pathogens such as *Clostridium botulinum* and *Staphylococcus aureus* to grow in it. a_w was measured by a water activity measurement instrument (Aqua Lab, CX-2). The microbiological assay was also performed for the identification/ presence of bacteria and yeasts and moulds (Harrigan, 1998) in final dried products.

After six months, the refrigerated (5 - 8°C) products (VDFS-1 and VDFS-2) were assessed through a_w and microbiological assay to determine the 'shelf-life' and 'antimicrobial activity'. For both products, the a_w data were measured in triplicate, and reported as means and SD.

Differential scanning calorimetry

Calorimetry is the basic technique by which a relationship is drawn between the temperature and specific physical properties of the considered substance. The protein molecules and their complexes are formed by the thermodynamically driven reactions. The supersensitive calorimetric techniques such as differential scanning calorimetry (DSC) is used to determine the thermal stability of proteins. From the endothermic heat flow (mW) vs. temperature (°C) plot, exhibition of multiple transition peaks denotes the denaturation of protein. Accordingly, DSC (Pyris Diamond DSC, Perkin Elmer Co., USA) was performed (from 10 to 100°C) for VDFS-1 and VDFS-2 to assess their thermal stability.

Results and discussion

pH and moisture analysis of shrimp and fishes before and after vacuum-drying

From Table 3, it was found that after sterilisation, the pH levels of shrimp, CA and CS had increased. The pH of SBM decreased to 5.60 ± 0.03 from 6.90 ± 0.03 (1.5%, w/v, brine solution). It was suggested that the basic Na+ ions present in brine solution were partly transferred to all sterilised samples.

After blending with SBM, CF, RF, with or without DG, the moisture content of the two fortified samples (FS-1 and FS-2) dropped, implying that the addition of CF, RF, and DG helped gaining nutrition while reducing the moisture of STSP. The final vacuum-dried products (VDFS-1 and VDFS-2) were found to contain 3.06 ± 0.25% moisture (wet basis). The alkalinity of VDFS sample(s) increased; thus making the products "alkaline food". This happened due to more ion diffusivity into dried samples owing to reverse water removal (Sarkar and Tirumkudulu, 2009). In comparison with raw shrimp (pH = 6.40 ± 0.02), the pH of VDFS-1 was determined to be higher (6.63 ± 0.02) and VDFS-2 was lower (6.33 ± 0.02).

Vacuum-drying kinetics, models and drying time

The VD model constants (K, A, B, C, n), for seven model equations (Table 2) for both the products FS-1 and FS-2 (using the corresponding heater) were computed using experimental data. The variation of experimental MR with VD time is shown in Figure 1(A) and Figure 1(B) for FS-1, FS-2, respectively, using both SPMDH and CPMDH.

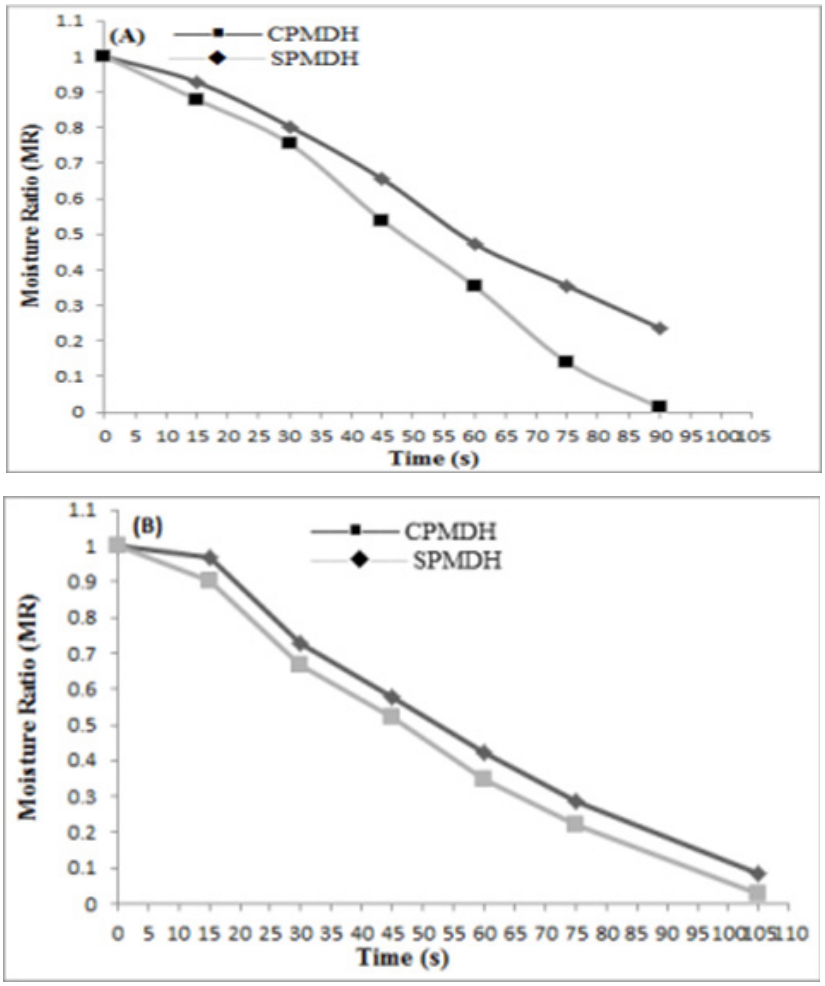

Figure 1. Moisture ratio (MR) vs. time for (A) vacuum-drying of FS-1 sample using SPMDH and CPMDH, and (B) vacuum-drying of FS-2 sample using SPMDH and CPMDH.

For FS-1, the VD time was found less for both SPMDH [2.55 h] and CPMDH [2.85 h] in comparison with FS-2 [3.25 h and 3.76 h by using SPMDH and CPMDH, respectively]. The probable reason might be the enhanced internal resistance to moisture diffusion during VD for FS-2 than FS-1, since DG (1%, w/w) was additionally added to FS-2 only. That means addition of DG had a significant adverse effect on VD time.

Statistical analysis of VD models

The numerical values of R^2 (Microsoft Excel 2010), RMSE, χ^2, and *E*% values as expressed by equations (2), (3), and (4), respectively pertaining to the vacuum-drying (VD) models for both FS-1 and FS-2 (using corresponding SPMDH and CPMDH) were calculated to know the best fitting/accepted model out of seven models, and are presented in Table 2. Highest R^2, lowest values of RMSE, as well as χ^2 and *E*% (< 5) values were computed and found in Linear (using SPMDH) and Modified Page (using CPMDH) models in VD of FS-1, while similar results were determined in Modified Page (using SPMDH) and Newton (using CPMDH) models in VD of FS-2. Accordingly, these models were considered the best representative of the VD kinetics.

Effective diffusivity (D_{eff}) and activation energy (E)

The Deff in VD of FS-1 and FS-2 under SPMDH and CPMDH over a temperature range from 40°C to 50°C were calculated using equations (1) and (5). At 50°C, by using SPMDH for FS-1, the D_{eff} value was found higher (7.464×10^{-10} m^2/s) than FS-2 (Deff = 6.366×10^{-10} m^2/s). In the case of CPMDH (at 50°C), the Deff values were found lower for both FS-1 and FS-2 as compared to SPMDH. The activation energy (E) required for VD for FS-1 and FS-2 (using SPMDH) were 28.42 and 37.38 kJ/mol, respectively which were much lower than 43.09 and 55.59 kJ/mol for FS-1 and FS-2 (using CPMDH), respectively. Thus, SPMDH was observed to be more energy efficient than CPMDH.

As SPMDH rendered higher drying rate in comparison with CPMDH; beside SPMDH

possessing higher antimicrobial efficacy; hence, both the final products (VDFS-1 and VDFS-2) were developed using SPMDH and subsequently tested for quality assessments.

Rehydration ratio (RR)

By using equation (6), the results of RR were found to be 2.68 ± 0.03 and 2.50 ± 0.03 for VDFS-1 and VDFS-2, respectively. This suggested that VDFS-1 had more RR than VDFS-2. Chakraborty *et al*. (2011) reported a RR value of 2.41 for infrared-assisted freeze-dried tiger prawn product, which was lower as compared to both VDFS products reported in the present work.

Water activity (a_w) and microbiological assay

The a_w was determined as 0.228 ± 0.005 and 0.196 ± 0.005 for VDFS-1 and VDFS-2, respectively. As per FSSAI (2010) guidelines, the a_w of any finished fish products must be < 0.78; the a_w values of both VDFS products were notably less than the FSSAI limit, thus conforming to the market status. Low a_w values for both the final products suggested that they possessed acceptable shelf-life and stability. It may further be observed that VDFS-2 had longer shelf-life and higher antimicrobial activity than VDFS-1.

Quality assessment

The results of the proximate analysis of VDFS-1 and VDFS-2 (100 g each) are shown in Table 4.

Total carbohydrate, protein, total fat, total ash

For VDFS-2, the increment in carbohydrate content was found over VDFS-1. In comparison with raw shrimp sample, VDFS-2 had 37.5 units more than raw shrimp; whereas VDFS-1 had 35 units more carbohydrate content since DG was added for FS-2 making.

The protein content was 50.66 ± 0.31 g and 48.78 g ± 0.28/100 g for VDFS-1 and VDFS-2, respectively. Thus, protein content increment in VDFS-1 and VDFS-2 were 188% and 177%, respectively as compared to raw shrimp (17.6 g/100 g).

The total fat content of the two products was found almost the same [4.71 ± 0.05 g (VDFS-1) and 4.73 ± 0.05 g (VDFS-2)/100 g] since they were made of almost similar compositions except for DG in VDFS-2. The fat content increased almost five times in comparison with that of raw shrimp (1 g/100 g). Abraha *et al*. (2017) found 3.74 g fat per 100 g dried anchovy fish, which was well below the fat content of two VDFS products.

The total ash content for VDFS-1 and VDFS-2 was 5.52 ± 0.44 g and 5.19 ± 0.42 g per 100 g dried products, respectively. For both the products, the total ash content increased about 2 to 2.5 times as compared to raw shrimp (2.2 g/100 g). Thus, 151% increment in total mineral content was achieved in VDFS-1 which was higher than the increment in VDFS-2 (136%). Shaviklo (2015) reported 1.60 g ash per 100 g freeze-dried tilapia fish, and 2.08 g ash per 100 g spray-dried tuna fish, both of which were lower as compared to the ash content of the VDFS products assessed in the present work.

Essential elements

For end products, eight essential elements namely Fe, Zn, Ca, P, K, Cu, Mg, and S were measured and compared with those of raw shrimp. Abbey *et al*. (2016) measured the Cu, Zn and Ca content as 0.25 mg, 1.88 mg and 1066.5 mg, respectively per 100 g dried tuna fish powder. Thus, the developed VDFS products contained higher concentration of elements (Cu, Zn and Ca) than dried tuna fish powder. It was found that increments of those elements were greater for VDFS-1 than VDFS-2 except fot Ca. For VDFS-1, the increments were 119%, 260%, 129%, 198%, 152%, 71%, 108% and 141% for Fe, Zn, Ca, P, K, Cu, Mg and S, respectively (Table 4) as compared to those in raw shrimp.

Energy

The energy content was almost equal at 1609.26 ± 1.95 kJ and 1642.28 ± 1.92 kJ/100 g for VDFS-1 and VDFS-2, respectively which were much higher as compared to 333 kJ, 267 kJ and 349 kJ per 100 g raw shrimp, CA and CS, respectively. Marginal difference in energy content (about 33 kJ/100 g) between VDFS-1 and VDFS-2 could be due to the addition of DG to FS-2. Abbey *et al*. (2016) found less energy content (1333 kJ/100 g) in dried tuna fish powder as compared to the two VDFS products assessed in the present work.

ω-3 fatty acids

The ω-3 fatty acids were measured around 2.4 ± 0.03 for VDFS-1 and 2.2 ± 0.03 g/100 g for VDFS-2, which were significantly greater than (0.55 g/100 g) that of dried anchovy (Stelophorus heterolobus) (Abraha *et al*., 2017). According to Kris-Etherton *et al*. (2002), the intake of ω-3 PUFA is preferable from dietary approach; since the consumption of 1 g per day ω-3 fatty acids is beneficial to reduce cardiovascular disease, stroke (cerebral infarctions) and there is no risk of side effects (gastrointestinal disturbances and nausea) through its ingestion. As compared to raw shrimp (ω-3: 2.0 g/100 g); about 20% increment in ω-3 fatty acids could be achieved.

Histamine and total volatile base nitrogen (TVBN)

For both VDFS-1 and VDFS-2, the histamine content was 5 ± 0.01 mg%. The regulatory limit for histamine by the USA is 20 mg% (lowest among all countries). Therefore, the histamine content of the prepared foodstuffs was found well below the permissible limit. Hwang *et al*. (2012) estimated histamine in dried milkfish (*Chanos chanos*) in the range of 117.3 - 382.1 mg/100 g dried fish, which was well above the histamine value found in the present work.

In both end products, TVBN was found below the detection limit (detection limit: 5 mg/100 g); thus, indicating acceptability for human consumption; since TVBN was much lower than the threshold 40 mg N per 100 g sample. Notably, de Koning (2002) reported TVBN level (for several fish meals) over the range from 85 to 170 mg/100 g dried fish meal which was considerably higher than the present data. Abraha *et al*. (2017) also found greater TVBN content (20.12 ± 0.20 mg/100 g) in dried anchovy (*Stelophorus heterolobus*) as compared to both VDFS-1 and VDFS-2 assessed in the present work.

Mercury (Hg)

The joint Food and Agriculture Organization (FAO) and World Health Organization (WHO) recommended 5 µg/kg body weight per week (daily intake 0.71 µg Hg/kg body weight) maximum level of Hg consumption (Torres-Escribano *et al*., 2010). In both product samples, Hg was found below the detection limit (0.01 mg/kg). Therefore, it was safe to consume both the fortified shrimp products. Panichev and Panicheva (2016) reported Hg content for 14 different processed fishes that ranged from 135 ng/g (0.135 mg/kg) to 666 ng/g (0.666 mg/kg); evidently, these were much higher than that found in the present work.

DSC

In Figure 2(A) (VDFS-1), no protein denaturation was found since the profile was found very smooth up to 100°C. On the contrary, in Figure 2(B) (VDFS-2), many transition peaks were observed (around 50 and 65°C) over the temperature range from 10°C to 100°C indicating thermal degradation. For VDFS-2, the rapid increment in endothermic heat flow

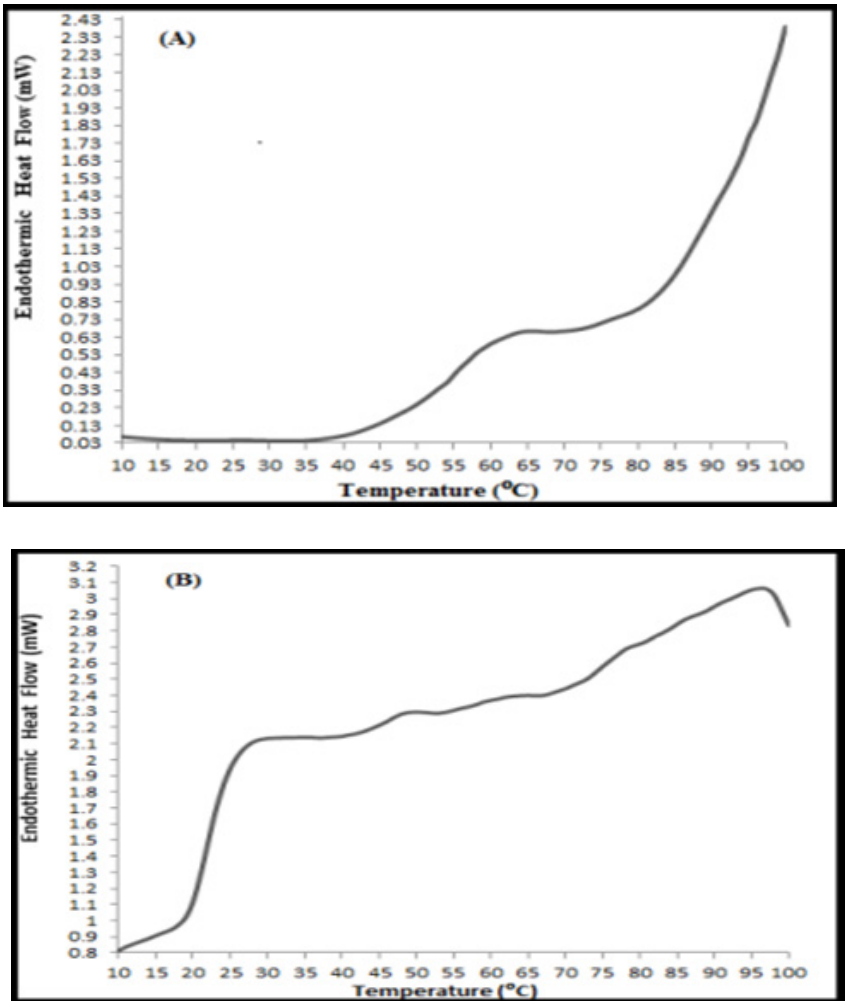

Figure 2. DSC thermogram of (A) VDFS-1, and (B) VDFS-2.

was possibly due to the presence of DG having higher specific heat (1.92 kJ/kg/ K). Hence, VDFS-1 demonstrated higher thermal stability as compared to VDFS-2. Besides, the higher glass transition temperature of VDFS-1 (T_g = 54.57°C) than VDFS-2 (T_g = 22.02°C) implied its superior thermal stability. Moreover, relative to unfortified dried shrimp where protein denaturation was observed at 40°C (Schubring, 2009), superior thermal stability was displayed by both VDFS-1 and VDFS-2.

Conclusion

In the present work, the shrimp was enriched for improvements of its nutritional attributes and energy content by fortification with two fishes (*Catla catla* and *Chela cachius*) along with the addition of corn and rice flours (with or without adding dried ginger). Though VDFS-2 possessed higher shelf-life, antimicrobial activity, carbohydrate and energy content than VDFS-1, VDFS-1 was superior in terms of alkalinity, lower drying time, rehydration ratio and thermal stability than VDFS-2. VDFS-1 exhibited better quality due to remarkable increments in protein, ω-3 fatty acids, carbohydrate, ash and other essential elements as compared to raw shrimp. Moreover, VDFS-1 possessed acceptable a_w, TVBN and histamine content, making it an attractive foodstuff. Thus, enrichment of shrimp by fortification with low-cost fishes and flours was demonstrated to be an effective protocol. Additionally, the energy-efficient fast vacuum-drying could emerge as a proficient preservation method.

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Once accepted in the peer review your manuscript will be directed for references, abstract and vernacular revisions. You will received the proofs for approval.

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Are the interpretations and conclusions justified by the results?: Yes

Are adequate and update (5 years) references made to other works in the field?: No

Is the language acceptable?: Yes

Please rate the priority for publishing this article (1 is the highest priority, 10 is the lowest priority): 3

Length of article is: Adequate

Number of tables is: Adequate

Number of figures is: Adequate

Please state any conflict(s) of interest that you have in relation to the review of this paper (state "none" if this is not applicable).: None

Rating:

Interest: 2. Good

Quality: 2. Good

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Date Sent: 12-Aug-2019

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National Accreditation Board for Testing and Calibration Laboratories

(A Constituent Board of Quality Council of India)

CERTIFICATE OF ACCREDITATION

MITRA S. K. PRIVATE LIMITED

has been assessed and accredited in accordance with the standard

ISO/IEC 17025:2005

"General Requirements for the Competence of Testing & Calibration Laboratories"

for its facilities at

Building No. P-48, Udayan Industrial Estate, 3,
Pagladanga Road, Kolkata, West Bengal

in the field of

TESTING

Certificate Number TC-6950

Issue Date 18/09/2018 **Valid Until** 17/09/2020

This certificate remains valid for the Scope of Accreditation as specified in the annexure subject to continued satisfactory compliance to the above standard & the relevant requirements of NABL.

(To see the scope of accreditation of this laboratory, you may also visit NABL website www.nabl-india.org)

Signed for and on behalf of NABL

8907697010003000113J

Anil Relia
Chief Executive Officer

TEST REPORT

ISSUED TO :
Haldia Institute of Technology-
P.O : HIT,Haldia, Purba Medinipur - 721657

KIND ATTENTION : Mr.Niladri Chakrabotry

REPORT NO.	: EFRAC/2016/FDS/10313
ISSUE DATE	: 05/01/2017
CUSTOMER REF	: TRF
REF. DATE	: 31/12/2016
PAGE NO.	: 3 of 4

SAMPLE DETAILS

SAMPLE REGISTRATION DETAILS

Sample Receipt Date : 31/12/2016
Sample Registration Date : 31/12/2016
Sample Registration No : EFRAC/2016/FDS/10313
Sample Type : Fruit Powder
Batch No. : Nil

Sample Quantity Received : 60 gm
Sample Submitted/Drawn by : Client

SAMPLE ANALYSIS DETAILS

Analysis Starting Date : 02/01/2017
Analysis Completion Date : 05/01/2017

TEST RESULT

S. No.	TEST PARAMETER	UOM	METHOD	RESULTS
Proximate Analysis				
1	Energy	Kcal/100g	IS:9487-1980(RA-2005)	335.29
2	Carbohydrates	g/100g	IS:1656-2007	72.17
3	Protein	g/100g	IS:7219-1973 (RA 2005)	5.96
4	Fat	g/100g	FSSAI LAB MANUAL	2.53

UOM : Unit of Measurement
Instruments Used : HOT AIR OVEN
Limit of Quantitation : N/A
Remarks : None

Authorized Signato
Kishan Pal Singh
Manager