Development of a Predictor Tool for the Estimation of Minimum Free Fat in Pasting for Achieving Desired Particle Size (Dv90) in Chocolate on a Given Refiner

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CERTIFICATE

This is to certify that the thesis entitled '**Development of a Predictor Tool for the Estimation of Minimum Free Fat in Pasting for Achieving Desired Particle (Dv90) Size in Chocolate on a Given Refiner'** submitted by **Avilash Roy** bearing registration no: **160272 of 2021-22** is in partial fulfillment of the requirements for the award of degree of **Master of Technology in Food Technology & Biochemical Engineering** of **Jadavpur University.**

This is an authentic work carried by him under our supervision and guidance. To the best of our knowledge, the matter embodied in the thesis has not been submitted to any other university/institute for the award of my degree.

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The foregoing thesis is hereby approved as a creditable study in **Master of Technology in Food Technology and Biochemical Engineering** and presented in a manner satisfactory to warrant its acceptance as a prerequisite to the degree for which it has been submitted. It is understood that by this approval the undersigned do not necessarily endorse or approve any statement made, opinion expressed or conclusion therein but approve this thesis only for the purpose for which it is submitted.

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I hereby declare that this thesis contains literature survey and original research work done by me as part of my **Master of Technology in Food Technology and Biochemical Engineering** studies.

All information in this document have been obtained and presented in accordance with academic rule and ethical conduct.

I also declare that, as required by these rules and conduct, I have fully cited and reference all materials and results that are not original to this work.

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Student's signature with date

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ABBREVIATIONS

AFM : Atomic Force Microscopy

AMF : Anhydrous Milk Fat

CBD : Cocoa Butter Deodorized

DOE : Design of Experiment

LMC : Liquid Milk Chocolate

PGPR : Polyglycerol Polyricinoleate

PSD : Particle Size Distribution

3RR : 3 Roll Refiner

5RR : 5 Roll Refiner

RMC : Refined Milk Chocolate

SEM : Scanning Electron Microscopy

SMP : Skimmed Milk Powder

WMP : Whole Milk Powder

YN : Yolkin

NOTATIONS

L : Coating Thickness

N : Number of Particles

$\mathbf{V_{ds}}$: Percentage Volume Distribution at Different particle Diameter

$\mathbf{V_{RMC}}$: Total Volume of RMC

$\mathbf{V_{PC}}$: Volume of a Core Particle

X : Percentage of Each Solid with respect to Total Solid

Z : Percentage Solid to Percentage CBD Ratio

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ABSTRACT

The research in this paper was aimed to develop a predictor tool for the estimation of minimum free fat in pasting in an attempt to increase the refining efficiency to achieve the targeted Dv90 on a given 600mm refiner. In this study the corresponding minimum fat coating thickness on each particle and the total number of particles present per kilogram of refined milk chocolate (RMC) was also calculated. This study also aimed to give a clear in depth understanding of particle size distribution as this project based on PSD. The effect of Dv90 on fat coating thickness and number of particles was evaluated in this paper through the performance of the refiner at different refining condition by using excel calculator.

The theory of excel calculator and the process of development of excel calculator was also described in this paper through explaining the derived mathematical equation with the help of chocolate science involved in it. Later this excel calculator was upgraded to predictor tool with the help of regression model. A data pool was created with the obtained data after taking multiple trials in the pilot plant. This data pool was further used as data bank for the predictor tool. This is a statistical tool made over excel based on the real time practical data.

Total 7 trials were conducted in this project and particle size was measured. For example for a particular recipe that means at a constant fat percentage coating thickness was varied with particle size. At Dv90 40 micron coating thickness on each particle was calculated 0.25 micron while at Dv90 48 micron coating thickness was found to be 0.27 micron when number of particles are in 10^{13}.

This is the first statistical tool based on real time practical data which is able to predict the total number of particles present per kg of mass and the fat coating thickness on each particle in micron based on the user input recipe within just a few a second along with the required minimum fat to obtain desired user input Dv90 from a given 600mm refiner.

Keywords: Refining, Dv90, Particle size distribution, regression, Predictor, Coating thickness, Number of particles, Excel calculator.

1. INTRODUCTION

As a continuous lipid matrix made up of both solid and liquid fat, mostly cocoa butter (CB), chocolate is defined as a dispersion of solid particles that are scattered [35]. As a result of the widespread production of meals that may be coated in chocolate or have chocolate as their foundation over a long period of time, it has become one of the most popular food kinds and flavours globally. In recent years, chocolate has come to be recognized as a useful food and may be considered a buddy. Contrary to popular belief, CB does not increase cholesterol levels. According to recent studies, stearic acid has neutral effects that prevent blood cholesterol levels from rising. Additionally, chocolate plays a significant role in the provision of several nutritional clusters. Nutritionally, essential fatty acids (FA) and the natural antioxidants in chocolate improve the in vitro and in vivo preservation of lipids. Additionally, because it contains a higher amount of cocoa than milk chocolate, dark chocolate retains its nutritional benefits [36]. Milk chocolate, white chocolate, and black chocolate are the three primary categories of chocolate.

Milk is added to solid chocolate in the form of milk powder, liquid milk, or condensed milk to create **milk chocolate**.

Higher cocoa content is used to make **dark chocolate**, also referred to as "plain chocolate" or "black chocolate," though there are also dark milk chocolates and various degrees of hybrids. Dark chocolate is typically produced with cocoa butter rather than milk. Dark chocolate can be consumed on its own or used in cooking; thicker baking bars are often produced using elevated cocoa concentrations between 70% and 99%. Even though the proportion of cocoa butter to solids might change, dark is a synonym for semisweet and especially dark for bittersweet.

Without the cocoa solids, **white chocolate** is formed of sugar, milk, and cocoa butter.

Couverture is a term used for chocolates rich in cocoa butter.

Technically speaking, **compound chocolate** refers to a confection that substitutes vegetable fats for cocoa butter, typically tropical fats and/or hydrogenated fats. It is often used for candy bar coatings. In many countries it may not legally be called ‘chocolate’.

As per FSSR 2011, The term "chocolate" refers to an uniform product made from a combination of one or more ingredients, specifically cocoa materials such as cocoa beans, cocoa nibs, cocoa mass (cocoa liquor/cocoa paste), cocoa press cake, and cocoa powder (cocoa fines or cocoa dust), encompassing fat reduced cocoa powder regardless of the addition of sugars, cocoa butter, and milk solids such as milk fat. After deducting the total weight of any other added edible consumables and without lowering the minimum cocoa material contents, the inclusion of vegetable fats apart from cocoa butter shouldn't go over 5% of the final item. Standard of different chocolates are given below;

Sl. No.	Characteristics	Requirements for		
		Milk Chocolate	White Chocolate	Plain Chocolate
1	Total Fat (on dry basis), percentage by weight. Not Less Than	25	25	25
2	Milk Fat (on dry basis), percentage by weight. Not Less Than	2	2	Not Contain
3	Cocoa Solid (on moisture free & fat free basis), percent by weight	2.5	Not Contain	12
4	Milk Solid (on moisture free & fat free basis), percent by weight. Minimum	10.5	10.5	Not Contain
5	AIA (on moisture, sugar & fat free basis), percent by weight. Not more Than	0.2	0.2	0.2

Table 1.1: Standards of Chocolate

1.1 OVERVIEW OF LIQUID CHCOCOLATE MAKING

There are three basic unit operations that are involved in the transition of powder ingredients to liquid chocolate. They are pasting, refining & conching.

1.1.1 Pasting:

In this stage powder ingredients (sugar, cocoa powder, SMP, WMP, lactose) are kneaded in a jacketed mixer in which water is heated (45^0C - 55^0C). Kneading action is carried out by using rotating shaft. In pilot plant jacketed planetary mixer- Hobart is used to make paste. As per requirement time, temperature and speed needs to be adjusted. Main objective of this stage is to mix the ingredient to produce a homogeneous mass and to coat dry component with fat. About 95% of the chocolate recipe is brought together into the mixer (generally some portion of the fat and emulsifier are not added in pasting). The image of Hobart mixer that was used in this project is attached below;

Fig.1.1.1: Jacketed Planetary Mixer-Hobart

1.1.2 Refining:

Process of applying pressure and shear to solid particles in chocolate paste to mill them and to reduce their particle size is called refining. Main objective of refining is to reduce the particle size. Industrially refining is done in two stages, they are pre-refining by using 2 roll refiner and

fine refining by using 5 roll refiner. But in pilot plant there are two types of refiner, one is 300mm refiner and another one is 600mm refiner and these both refiners are 3 roll refiners, only difference is in the roller length. The 3RR is operated differently to the full-scale factory equipment. The 3-roll refiner is used as both the pre-refiner (first pass) and refiner (second pass). The pre-refining with the three roll achieves a significantly lower particle size than the factory two roll pre-refiners; this is good because it makes achieving the final particle size with the three roll easier but also comes with its own difficulties. Rollers are positioned horizontally to each other and the chocolate pass through the two roll gap. Due to the decreased particle size, the surface area is increased. This requires more fat to coat the surface and so the paste seems drier, and this makes it very difficult to further refine as the paste doesn't run through the rolls well. To combat this, the pre-refined paste can be returned to the mixer and pasted with a small amount of extra fat to bring the dry flake back to a workable paste before refining. In this particular project Buhler 600mm refiner is used. A picture of this refiner is attached below;

Fig.1.1.2: Buhler 600mm refiner

For optimum refining there are some parameters that need to be controlled. These parameters are discussed below.

I. Roller gap:

Amount of mass enters into the refiner is depends upon the roller gap. Parallelism of gap is highly important to achieve consistent product fineness. Product fineness must be consistent across the total roll length. A zero gap calibration is performed when a difference between left and right fineness is noticed.

II. Roller temperature:

Roller temperature is important for achieving good roll coverage and therefore optimal refining. Cooling of roll is very important due to heat produced during refining process and as the paste is sheared a lot of frictional force is produced which heats up the rolls. If the rolls are not cooled they would get very hot and cause the paste to melt and stick to the roll surface hindering the refining process. The lower the temperatures, the more mass is pulled in. Each roll has a different set temperature that aids mass transfer from one roll to the other until the mass reaches the final roller.

III. Roller pressure:

Correct pressure across the refiner is required to achieve consistent product fineness across the entire roll. In pilot plant maximum 20 bar pressure can be applied across the refiner.

IV. Paste Plasticity:

Final particle size is influenced by the feeding passage. The more mass to enter, the more mass to leave which results in a thicker layer on roll 5 and therefore a coarser particle size. Amount of mass to enter in the intake passage is influenced by mass plasticity. If the paste is drier, there is a high feeding rate; if the paste is softer, there is a low feeding rate.

1.1.3 Conching:

It is the final step in liquid chocolate making. It takes place in a conche which is basically a large jacketed mixer with a powerful motor. During conching refiner flake is combined with fat, emulsifier, flavor and possibly further ingredients and turned into liquid chocolate. Conching has mainly two phases- active conching and liquefaction. Active conching is further divided into two stages- dry conching and paste.

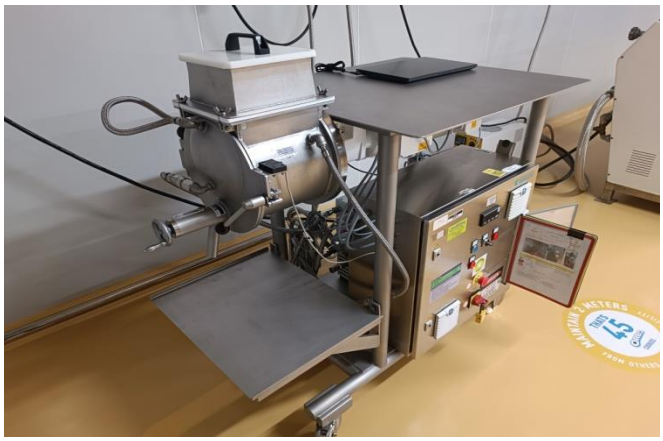

Fig.1.1.3: Elk Conche of capacity 5kg

1.1.3.1 Aims of conching

I. To change the phase of mass from powdery flake to flow-able liquid.
II. Aroma and flavor development and redistribution
III. Removal of moisture and volatiles (organic acids)

1.1.3.2 Ingredients for Conching:

Refiner flake, fats (cocoa butter, AMF, Vegetable fats), emulsifiers (Yolkin, PGPR), and additives like flavors are added in conche.

1.1.3.3 Stages of Conching:

Conching process is divided into four phases – Filling, active conching (dry conching, Paste) liquefaction and discharge. For dark chocolate active conching part is larger as more volatiles need to be removed. For crumb chocolate active conching part is often shorter as most flavors are developed during crumb making process.

I. Filling

Refiner flake is manually added into the conche according to the recipe. But industrially refiner flake is directly fed to conche via belts. During filling some of the lecithin and fat should be added as pre-charge. A maximum of the total 0.1% of the total batch weight should be added in pre charge as 2-3 steps. Temperature is kept around 55^0C for milk chocolate. If temperature is too high moisture will be released from ingredients potentially causes grits to form.

II. Dry conching

Main objective of this stage is to remove moisture and volatiles from the mass. Filling materials are slowly mixed and heated about 50-55^0C for milk chocolate. At this stage mass is dried and some of the bound fat is released from the refiner flake.

III. Paste Conching:

The mass has a more pasty texture in this phase. At this point shear is introduced by changing the mixing direction from clockwise to anti-clockwise as the mass smeared between paddles and conche walls. Breaking up of agglomerates and covering the particles with fat during his process allow them to slide past one another.

IV. Liquefaction

Remaining fat and lecithin are added in 2-3 addition with around 10-20 minutes interval in this process. Temperature is reduced to 45^0C in this stage. At the end viscosity of the mass is adjusted with PGPR addition depending on the recipe.

- 1st addition- lecithin
- 2nd addition – remaining fat
- 3rd addition - remaining lecithin
- 4th addition- additives like flavors
- 5th addition- PGPR for viscosity adjustment

V. **Discharge:**

At this stage liquid chocolate mass is discharged from the conche. Before discharging speed of the agitator is reduced and water temperature is also reduced to 40^0C.

1.1.3.4 Process involved in conching:

Conching is combination of mixing, heating and shearing.

I. Mixing

Mixing ensures that the fat and aroma compounds are evenly distributed within the mass. During dry conching it is usually turns in clockwise direction and in anti-clockwise durin the other part of active conching. Clockwise direction results good mixing, drying effect as well as kneading when the mass gets more dough like consistency. Anti-clock direction enhances shear and emulsifying effect.

II. Heating

During conching process the mass must be exposed to air either by actively blowing air or by opening the louvers at the top of the conche. This will allow unwanted volatiles flavor compound (from cocoa) and water to be evaporated. Heating helps to drive off volatiles and moisture and contribute flavor development.

III. Shearing

Shear helps in breaking up of agglomerates and covering particle surfaces with fat. This process allow them to slide past one another which lowers the mass viscosity.

1.2 TESTING PARAMETERS:

There are basic parameters that's need to be tested during chocolate making these includes particle size measurement (Dv90), viscosity, total fat and moisture content. Malvern

Mastersizer 3000 is used for PSD measurement (Dv90), Thermo Scientific HAAKE viscometer is used for viscosity measurement and NIR is used to measure total fat content. Each of this testing procedure will be discussed in Materials & Methods section.

1.3 BACKGROUND OF THE PROJECT:

Dv90 is critical to quality (CTQ) parameter for chocolate. Dv90 is not only important from the sensorial perspective but also for the flow properties of the chocolate. Particle size depends on various factors like type and composition of ingredient, type and performance of the refiner and on some process parameter like refiner roll temperature, roll gap, pressure between the roller and pasting temperature. But in pilot plant it is one of the biggest challenges to achieve targeted Dv90 in one or two shot. Achieving desired refiner flake Dv90 in the pilot plant using 300mm and / or 600mm refiner takes substantial amount of time, sometimes resulting in loss of material due to failure. So this project aimed to minimize this problem through the development of a predictor tool for the estimation of minimum free fat required in pasting in an attempt to increase the refining efficiency to achieve targeted Dv90 on a given 600mm refiner.

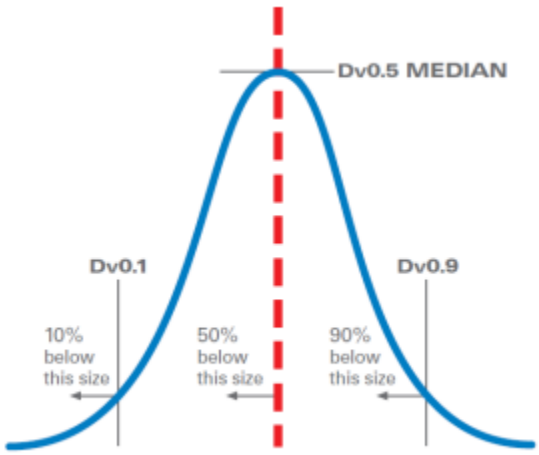

Fig.1.3.1: Normalized PSD curve

In Mondelez there is a statistical software named PSD App which is generally deconvolute the PSD curve given by Malvern into 2 log-normal distributions. Deconvolution generates a small set of parameters that much more fully describe the PSD. It is true Dv90 is CTQ parameter for chocolate but to understand the PSD better Dv99 is more significant than Dv90 because it talks about 99% of the sample. These facts triggered me to work with not only Dv90 but also with the whole volume distributions at different diameter given in the detailed Malvern report. To run this deconvolution program in PSD app, first detailed Malvern report which contains percentage volume distribution at different diameter is need to be downloaded in a excel file

format and then upload to the PSD app. These data of percentage volume distribution at different diameter are attached in Appendix 1.

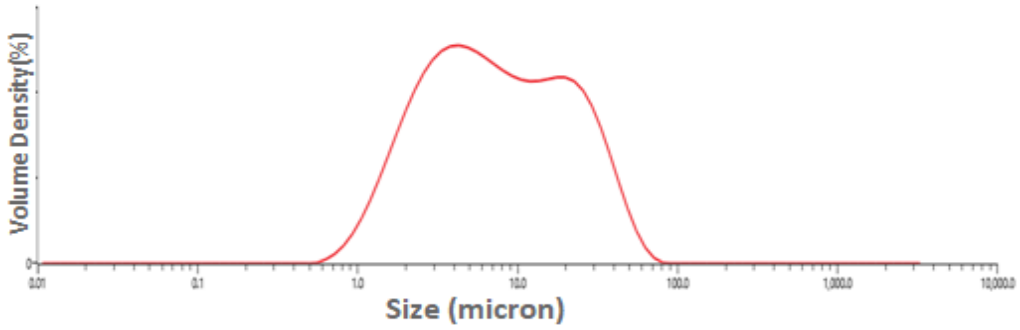

Fig.1.3.2: PSD Curve from Malvern.

Malvern shows this kind of PSD curve (Fig.1.3.2) after particle size measurement. Particle size is taken in X axis and Percentage volume density is in the Y axis.

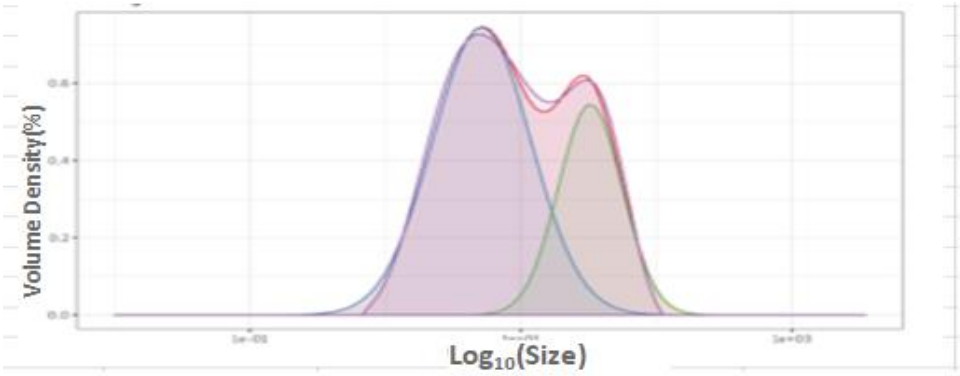

Fig.1.3.3: Deconvoluted PSD Curve from PSD App

In Fig.1.3.3 deconvoluted plot of Fig.1.3.2 is shown. Bimodal distribution shown in Fig.1.3.2 is broken into two normal distributions in Fig.1.3.3, where in X axis logarithmic particle size is taken and Y axis represents percentage Volume distribution. Red line represents the experimental line given by PSD App and violet line represents the actual line given by Malvern. Deconvolution report of the above shown plot says in between 2.5 to 6.9 micron diameter 66% volume of particles lies which represents peak 1 & in between 21.23 to 24.67 micron 34 % volume of particle lies which represents peak 2. It also gives Dv99 that is 56 micron, whereas Dv90 is 30 micron.

1.4 OBJECTIVE & AIM OF THE PROJECT:

Objective of the project is to understand the mechanism of fat distribution through transition of powder ingredients to liquid chocolate.

Aim is to develop a predictor tool for the estimation of minimum free fat required in pasting in an attempt to increase the refining efficiency to achieve targeted Dv90 on a given refiner.

1.5 PROJECT HYPOTHESIS:

If fat is considered to have a thin coating over solid in pasting and RMC with known particle size distribution of RMC then the amount of fat required for effective refining (fat discrete) and conching (fat continuous) can be predicted.

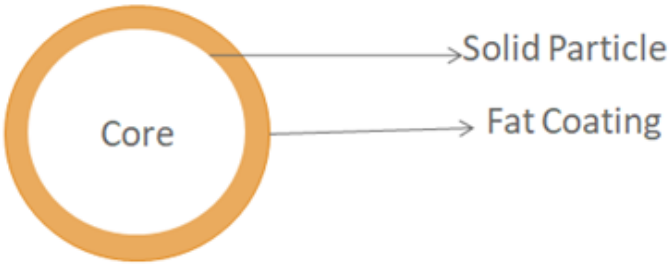

Fig.1.5.1: Schematic of fat coated particle

1.6. KEY STEPS :

i. To study the particle size distribution in depth (discussed in the section 1.3)
ii. To know the amount of fat needed to make the system fat continuous from fat discrete phase.
iii. To see the difference of fat distribution between refined milk chocolate (RMC) particle and liquid milk chocolate particles (LMC), samples needs to be observed under scanning electron microscope(SEM).

1.7 KEY CHALLENGES:

i. Particles don't have any specified shape and are porous in nature.
ii. Extent of refine ability & post refining distribution of Solid ingredients is unknown.

1.8 LITERATURE REVIEW:

A liquid chocolate is a type of suspension that contains refined sugar crystals with varying surface areas. These crystals are formed during the grinding process under water-free conditions. The high energy input from the process leads to the formation of crystalline and amorphous structures. [1].They cause phase separation in the suspension and aid in the agglomeration of solid particles [2]. By raising viscosity [3] and enabling the measurement of a yield value (defined as the shear strength at the place where slip flow crosses over into shear flow [4]), As a result, the interparticular spaces of the clumps partially immobilise the phase that is continuous. Emulsifiers have long been recognised to have macroscopic effects in lipophilic solutions [6]. These macroscopic alterations are believed to be the result of reduced concentrations of immobilised cocoa butter at the solid and emulsifier coatings of the surfaces of sucrose particles [7], [5]. Operating experiences led to the conclusion that soy lecithin is the most useful emulsifier for chocolate manufacturing. It nicely complements the hard chocolate particle's amphiphilic surface features. [8], since it is a blend of several phospholipids.

The head-tail emulsifiers that lecithins are. The molecules' hydrophilic head groups are oriented towards the suspension's hydrophilic phase, while the molecules' lipophilic tails are oriented towards the suspension's lipophilic phase. However, more information on its mode of operation is still a mystery. Lecithin is still used as an emulsifier because it works in real-world situations.

However, little is known about a different common emulsifier. Vegetable polyglycerol polyricinoleic acid with a high molecular mass between 1200 and 2000 g/mol makes up the exceedingly complex mixture of macromolecules known as polyglycerol polyricinoleate (PGPR) [9]. Without having a considerable impact on viscosity, it decreases or even eliminates yield value [10, 11, 12]. It is proven that PGPR adsorption increases the lipophilicity of sucrose particles by reducing the acidity of the surface. As a result, interactions between sucrose particles are reduced, while suspensions based on fat have more fluidity. Given the differences between lecithin and PGPR's basic properties, it has been hypothesised that the polycondensed ricinoleic acid's oxygen atoms play a key role [13]. Other hypotheses centre on how particles repel one another, get rid of excess cocoa butter from particle surfaces, or use PGPR to bind water [11].

Atomic force microscopy (AFM) can be used to learn more about the microscopic interactions between emulsifiers and sucrose surfaces. AFM provides the chance to look into the workings of several emulsifiers. Recent AFM investigations using emulsifier adsorption were conducted [14]. It was demonstrated that the decrease in particle-particle interactions supported theories from the literature and was in good accord with the findings of sedimentation tests of sugar-oil suspensions. In order to make chocolate, Lecithin and PGPR were examined for their effects on the outermost layers of crushed sugar particles in liquefied cocoa butter. Both a microscopic

and macroscopic investigation of the impact of emulsifier adsorption was conducted. AFM findings and quantitative adsorption of emulsifier quantities on surfaces of particles were connected to flow behaviour and fat immobilization. In terms of AFM, topography imaging and force spectroscopy methods were applied in particular. As a result, disparities in the use of PGPR and soy lecithin were identified, and further information about PGPR's mode of action was acquired.

Sample Preparation:

To make model suspensions, liquid cocoa butter was stirred at 55°C and 500 min^{-1} to dissolve 0.5% emulsifier linked to sucrose content. Following the addition of 45% commercially available crystalline sucrose, the crystals were pulverized in a ball mill at 45^{o} C using 10 kg of 1.5 cm steel balls. Grinding continued until 90% of the particles had a maximum particle size of 30 mm and the mass median diameter was 10–12 mm. The suspension was then conched for 4 hours using 1500 liter per hour of 22°C airflow. The kneader rotated at a rate of 130 min^{-1} while the water bath was 75^{o}C in temperature[15]. Along with the model formulations that contained emulsifiers, a couple of reference formulations without them were also made and conched. The grounding mentioned above was applied to one of the suspensions but not the other.

Atomic Force Microscopy (AFM) Approach:

Using centrifugation, sugar particles were removed from the bulk mixture for AFM observations (10 minutes, 55^{o}C, 5000 1/min), acetone washing, and 24-hour drying in a desiccator with dried nitrogen. Then, using Neuner's two-step preparation process (G3305A, Plano GmbH, Wetzlar, Germany), sucrose particles were adhered to a mica disc once again in a dry nitrogen atmosphere [15].

The MFP-3D SA (Asylum Research, Santa Barbara, CA, USA) was used for the AFM investigations, and conventional silicon cantilevers with a nominal spring constant of 2 N/m were used [15]. Topographical imaging was performed in intermittent contact mode in a nitrogen atmosphere that was dried out (with a relative humidity < 5%). After completing 60 × 60 force-versus-distance curves for each force map with a size of 2 micron × 2 micron in contact mode, adhesion forces were estimated. [15]. The precise cantilever spring constant for each tip was calculated using the thermal noise approach [16]. A contact trigger point of 1 V and a 2 mm/s scan speed were used. Each sucrose particle had two force maps, which were inspected at various surface regions and recorded as duplicates. At least 15 sucrose particles per sample were examined. The amount of occurrence of a certain adhesion force per map in percent was contrasted to each other in the histogram created from the force maps. Quantities less than 0.5% of the entire force graphs were not in calculation. Using IGOR Pro Software and Microsoft Excel 2010, measurements and analyses of images and maps were conducted.

Analysis of Interfacial Layer:

Centrifugation was used to separate the sugar particles. The acetone phase's absence of the emulsifier indicates that it was maintained on the particle surface. Acetone was then used to remove the remaining, and so immobilized, cocoa butter. To make sure that none emulsifier was rinced off the outer layer, the acetone-wash phase had its emulsifier concentration checked (information not provided).Then, with the use of chloroform or hexane, the emulsifier layer was removed, leaving just the sucrose particles [15]. The emulsifier layer containing chloroform and hexane was further examined.

Quantification of Cocoa Butter and Emulsifiers:

After the triglycerides were hydrolyzed with a methanolic potassium hydroxide solution, In the emulsifier layer, the amount of cocoa butter was calculated. [15]. Gas chromatography-mass spectrometry was used to measure the resulting fatty acid methyl esters using the DGF technique C-VI 11d (DGF = German Society for Fat Science). By contrasting the methyl ester of stearic acid from cocoa butter with the methyl ester of palmitic acid from a standard , the amount of cocoa butter present was calculated. Prior to esterification, 125 mg of glyceryl tripalmitate per 100 ml was added to every single specimens and the cacao butter standards for calibration.

According to direct extraction from the lipophilic phase, PL was produced [17]. According to [18], high-performance thin-layer chromatography (HPTLC) was utilised for quantification Prior to esterification, 125 mg of glyceryl tripalmitate per 100 ml was added to every single specimens and the cacao butter standards for calibration.. Detecting agent made up of 80.4 g of 85% H_3PO_4 and 25.8 g of $CuSO_4.5H_2O$ dissolved in 400 mL of distilled water. Ricinoleic acid concentrations in samples that included PGPR were measured using high-resolution gas chromatography. Then, its content based on 83.6% was calculated using the amount of ricinoleic acid in PGPR [15]

Adsorption of Emulsifiers on sucrose Surface:

As anticipated, the inclusion of an emulsifier decreased the quantity of fat that was immobilised (see Table I). The division of the particles by the combination of soy lecithin and PGPR prevents the association of particles and cocoa butter entrapment.. Since soy lecithin's impact was marginally stronger than that of PGPR, this indicates that soy lecithin, with its many PL components, is ideally suited for the surface of sucrose. Unexpectedly, grinding also led to a drop in the amount of immobilised fat. In theory, grinding increases particle surfaces and causes surface activation. Therefore, it was anticipated that the amount of fat would grow. However, it must be remembered that molasses residues remain on ungrounded sucrose particles and have a significant impact on surface characteristics. It has been demonstrated in

the past that smaller particles increase cocoa mass viscosity by lowering the quantity of mobile fat [19]. However, there was no equivalent change in the characteristics of the model dispersion or the sugar particles. While viscosity nearly remained unaltered, grinding increased the yield value from 0.11 to 1.82 Pa (see Table I).

Sample	Immobilized fat content [g/m^2 surface]	Equilibrium viscosity [Pa s]	Yield value [Pa]
Unground	0.68 ± 0.02	0.28 ± 0.01	0.11 ± 0.01
Ground	0.27 ± 0.01	0.32 ± 0.02	1.82 ± 0.01
Ground + PGPR	0.25 ± 0.01	0.33 ± 0.01	0.36 ± 0.01
Ground + soy lecithin	0.22 ± 0.01	0.29 ± 0.01	1.16 ± 0.02

Table 1.8.1: Viscosity, Immobilized fat and yield value of sugar particles

Fat phase that is continuous is created when the water-loving phosphatidyl groups of a Phospholipid molecule react with the hydrophilic outer layer of sugar. While the long polyricinoleic acid chains of the lipophilic components of PGPR compete with one another and goes into the cocoa butter phase, the hydrophilic portions of PGPR are anticipated to cover outer most layer of the solid particles. In this instance, the force of steric repulsion is used for segregating the particles. Alternative explanations for the system's workings is that more cocoa butter is forced into the bulk continuous phase by a PGPR covering of solid particles. The yield value reduces as cocoa butter levels are raised.

Emulsifier addition has a significant impact on the flow characteristics of the suspension of cocoa butter as well as the interaction of the sucrose particles with one another in the continuous phase [7]. Lecithin is one of the most commonly used emulsifiers in chocolate. Lecithin is frequently used in conjunction with PGPR [12]. As shown by the findings in Table I, the addition of PGPR had no discernible impact on viscosity but did increase yield value. The force range of the histogram was increased by emulsifier adsorption, going from a range of 5–40 nN for exposed particles to a limit of around 10 to >90 nN for soy lecithin-coated particle surfaces. Adsorption of various PL fractions of soy lecithin can explain wider dispersion and inhomogeneities of adhesion forces. For each chunk, a different amount of emulsifier molecule adheres to the sucrose surface and to the silicon tip of the AFM. Additionally, the variation in adhesion to the limit of higher pressures demonstrates that the lecithin fractions had extensively covered the particle surface areas, leaving just a few exposed surface areas. Adhesion forces fill the lower force range of grounded but exposed particles very well, are equally distributed there, and their distribution width is fairly tiny. As a result, the histogram's breadth and homogeneity, along with the force ranges that are occupied, serve as broad indicators of the type and quality of emulsifier interactions with sucrose surfaces.

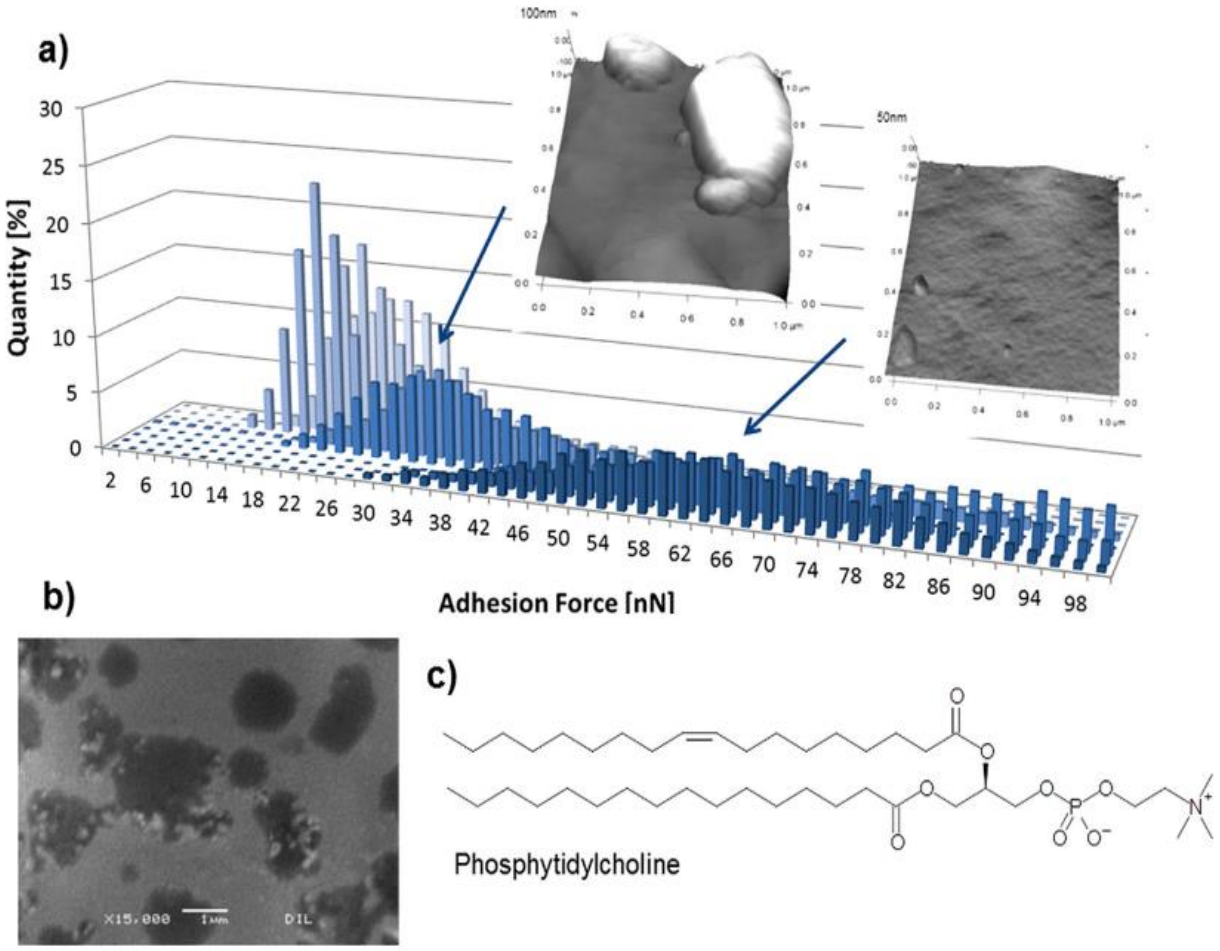

Fig. 1.8.1 –(a) Histogram of adhesion forces estimated from exemplary AFM force maps taken on soy lecithin-coated outer layer of ball-milled sugar particles; (b) SEM micrograph of soy lecithin-coated sugar outer layer; and (c) Formula of a phosphatidylcholine. Source- [15].

One may make the same observations and come to the same conclusions using the AFM information of sugar particles reacting with PGPR in Fig. I.B.a. Large numbers of PGPR molecules do appear to be however certain areas of the surface also get deposited on the surfaces of particles seem to be left unaffected. This is likely due to a more limited and uniform distribution with little to no change in adhesion forces at higher force ranges. Sugar particle contraction with PGPR molecules typically found to be a little less robust than with soy lecithin or the PL component. Figures I.A.b and I.B.b, which show SEM micrographs and AFM topography, both concur with this observation.

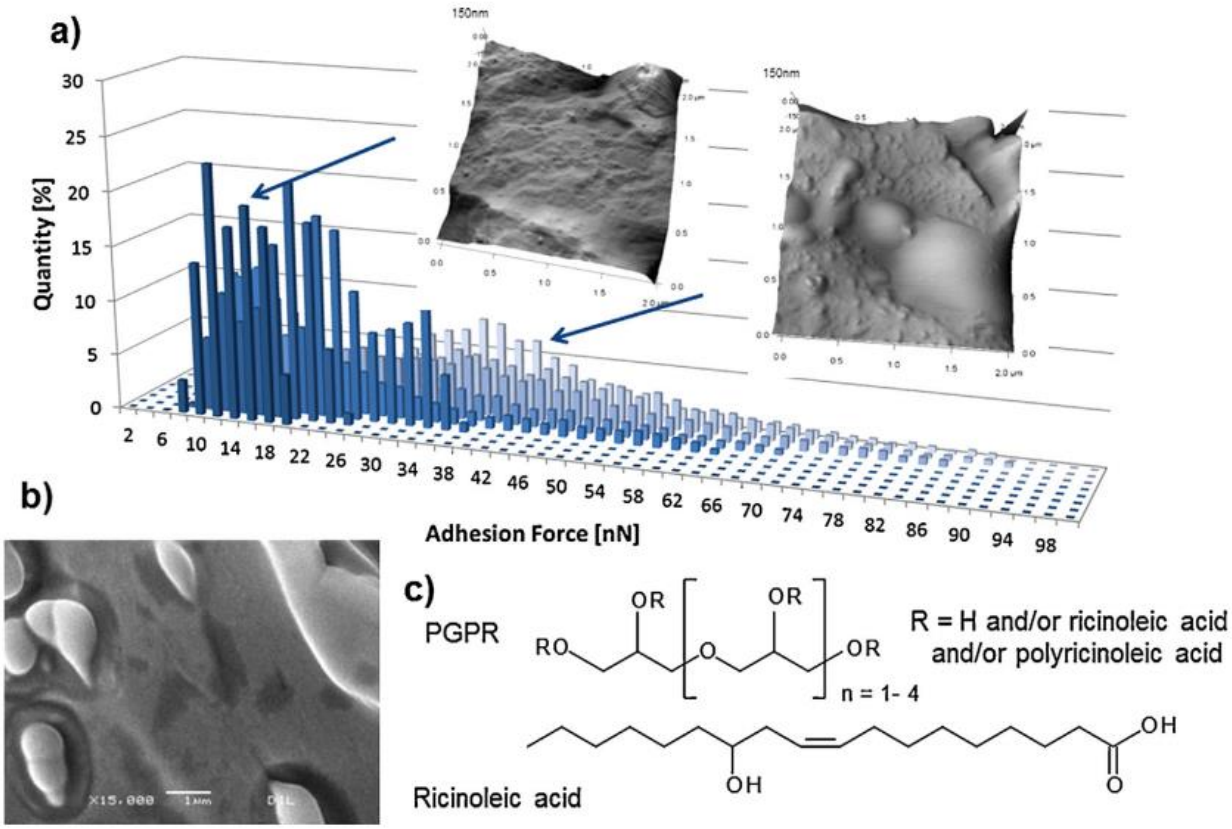

Fig. 1.8.2 – (a) Histogram analysis of estimated adhesion forces from reference AFM force maps taken on ball-milled sugar particle outer layer reacting with PGPR, 3D height images found by AFM (b) SEM micrograph of sugar outer layer reacting with PGPR; (c)structural formula of PGPR, subjected to production process R stands for hydrogen atoms, ricinoleic acid and/or poly-ricinoleic acid residues. Source-[15]

For confirmation, the amount of emulsifier that adsorbs on a square metre of sucrose surface was estimated. Only 0.18 ± 0.01 mg/m^2 of sugar outer layer was computed for PGPR adsorption, while 2.69 ± 0.05 mg/m^2 were found for soy lecithin[15]. In comparison to soy lecithin, PGPR content is therefore only around 7%. This result is strikingly different from the strong effects of PGPR inclusion on the macroscopic parameters of the dispersion (see Table I) and the microscopic properties of the surfaces of sucrose particle (see Fig. I.A): However, there is a significant variation in the yield value of the suspensions between particles coated in soy lecithin and particles interacting with PGPR. Chloroform/hexane was used to remove the coating after acetone was used to extract the particles out of the oil-loving phase. Interfacial layer thickness was estimated to be 0.33 ± 0.2 mg/m^2 of sugar surface area. This coating has a minimum cocoa butter content of 83.2 ± 1.9%, hence the maximum PGPR content is just 16.8%. It was discovered that there were 2.92 ± 0.20 mg of soy lecithin per square metre of sucrose

surface area [15]. These findings support the notion that PGPR's interaction with cocoa butter significantly affects how it functions as an emulsifier.

When particle surfaces were investigated using AFM and SEM (see Figs. I.A and I.B), it was discovered that coronas were encircling the regions of surfaces where emulsifier molecules interact. As they are creating a sort of ditch around the locations of PGPR-sugar engagement, these coronas might be thought of as regions of cocoa butter starvation, exposing the naked sugar surface. However, the quantity of immobilised cocoa butter was somewhat increased rather than decreased as would have been anticipated owing to depletion after the addition of PGPR. Therefore, quantitative examination of a layer of interfacial cocoa butter showed that these regions only have modest concentrations of PGPR but high levels of lipids. PGPR interacts the bulk phase of lipids as a result, in addition to drawing cocoa butter from the sucrose surface nearby and attracting it to it. In order to explain how PGPR affects yield value, the notion that cocoa butter is moved entering the bulk from the outermost layer of the particle must be adjusted [20].

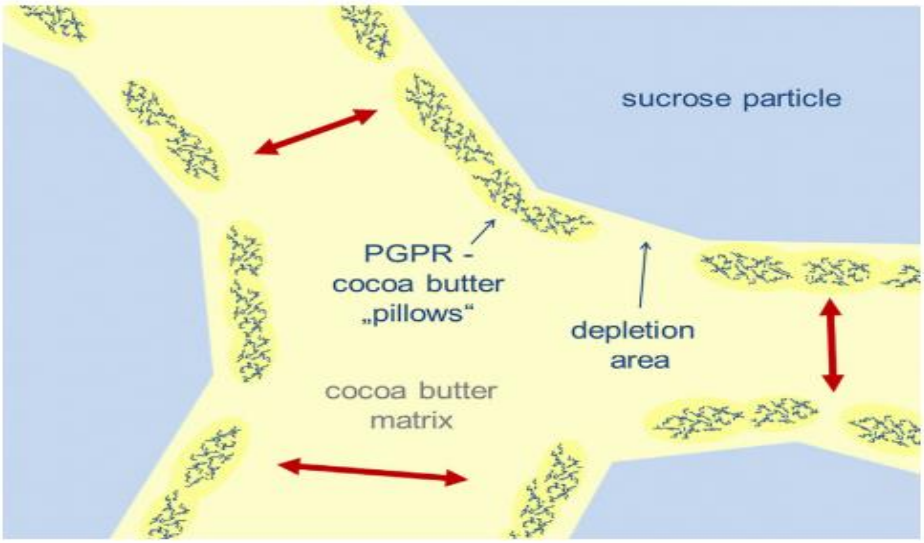

Fig 1.8.3- Pillow-like accumulation made of cocoa butter and PGPR polymers linked to pulverised sugar particles are an example. These deposits operate as unfirmly bonded buffers and gappings between the molecules of sucrose, drastically reducing the yield value. Source-[15]

Given the information above, it is quite plausible that PGPR, which reacts with the cocoa butter that was immobilised on the outermost layer of sugar to produce pillow-like accumulation The single sugar particles are kept apart from one another by steric hindrance, and these arrangements serve as separators and weakly bonded barriers between them(see Fig. I.C). By successfully reducing the contact between sucrose particles, pillow-like structures have little impact on viscosity. Particles are able to repel one another as a result of the decreased particle-

particle interactions, and less structure must be disrupted in order to produce flow. Thus, yield value drastically declines.

Small quantitative quantities of PGPR, which bound on sugar surfaces considerably lower the yield value of suspensions of cocoa butter and sugar when compared to the usage of soy lecithin. However, according to AFM and SEM measurements, the surface effect after PGPR incorporation is not so minor and is even reasonable to that of soy lecithin. Large quantities of immobilised cocoa butter and starvation zones associated with pillow-like formations on the outermost layer of sugar were also seen. Additional interfacial layer tests indicated that 83.2% of the cocoa butter is immobilised alongside the PGPR. There is a significant interaction between PGPR and cocoa butter. Depletion zones around the covered surface regions show that these deposits were made from nearby cocoa butter in order to produce pillow-like structures [15].

II. Free Fat & Bound Fat in WMP/SMP

Amorphous lactose creates a continuous matrix in spray-dried full cream milk powder where fat globules, proteins and air cells are distributed[21] .Just a small percentage of the entire fat mass may be taken out with solvents that are organic under controlled circumstances; this portion is referred to as "free fat." This liberated fat in full-fat milk powder can be found on the outermost layer of the granules as well as inside capillary pores and fissures. Another component is the fat that is contained in the powder particles and is more prevalent in homogenised milk. This fat is enclosed, making extraction difficult. When compared to milk powder that has been roller-dried Whole milk powder that has been spray-dried has fewer free fat. This is a result of spray drying's quick particle creation process This keeps some fat enclosed inside the component structure. [22,23] Milk powder, which is included in the formulation at a rate of roughly 20% w/w, is a crucial component. Other forms of chocolate may use milk fat for cocoa butter to improve the flavour and consistency of the chocolate mixture. Utilising whole milk powders containing a lot of free fat can reduce the amount of cocoa butter required to make a flowable product. The viscosity of the chocolate mass decreases with the amount of free fat in the milk powder.[24] Additionally, the amount of milk fat affects the treatment characteristics of the liquid chocolate mass, such as flowability, as well as the sensory profile of the chocolate (taste and texture). [25] Milk fat can only be used in chocolate in proportion to 30% cocoa butter due to its softening properties. [26,7] Typically, during conching, cocoa butter and surfactants (such as lecithin) are added to chocolate paste in order to get the correct final viscosity. Milk powders that have higher available fat concentrations and greater concentrations of particles change the viscosity of chocolate paste, which might lead to cheaper ingredient costs.

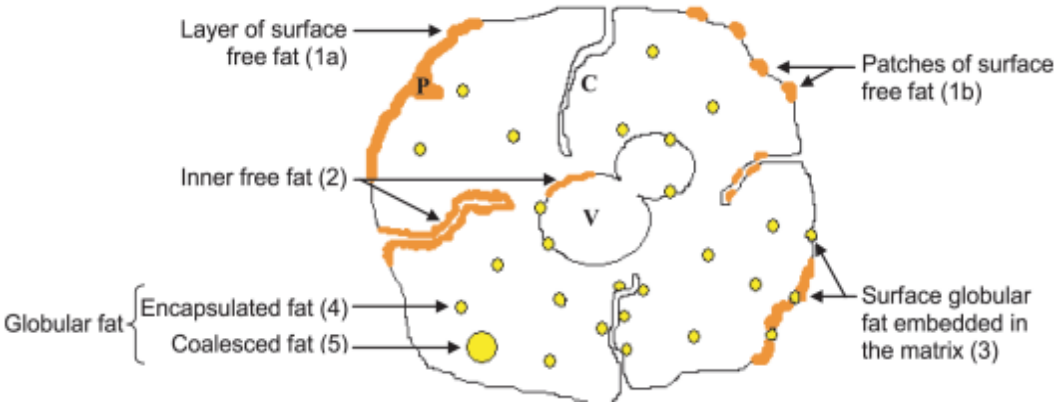

Fig.1.8.4: Schematic illustration of the different types of fat within a powder particle and their location. Porosity includes capillaries, pores and Vacuoles. Source-[27]

When lactose crystallises in milk powder and protein and lactose's molecular bonds are rearranged, fat is likely to be liberated. After storing fat-containing powders in humid conditions, Faldt and Bergenstahl [28] detected the dissolution of fat that has been sealed inside the powder using XPS. Using scanning electron microscopy (SEM), Saito [29] observed a related occurrence in whole milk powder maintained in humid circumstances. They proposed that this emission of fat was caused by the crystallisation of unstructured milk sugar. Milk Sugar can be found in crystalline or unstructured, glassy forms in spray-dried powdered whole milk [30]. The matrix of the bulk of spray-dried whole milk powders is mostly composed of unstructured lactose. Lactose crystallisation creates a phase distinction between milk sugar and other components of milk by altering the molecular structure and rupturing hydrogen bonds with other components. This reorganisation leads to the formation of a region of capillary pores across the whole particle. In this process, enclosed lipids are forced to break loose on the outermost layer of the particle, where they physically disintegrate due to the mechanical action of the rough crystal edges.

Extraction of Surface Free Fat:

When exposed for a relatively short period of time to organic solvents, the available fat on the outermost layer of a granular particle becomes soluble. Accurately weighing one gramme of the powder on filter paper No. 1 by Whatman, it was then rinsed with 4 mL of n-hexane. The resultant filtrate solution, which included the extracted fat, was transferred to a ceramic Petri plate and allowed to air out in a vacuum drier for 20 minutes before being dried by vacuum at ambient temperature for an hour. It was found that the weights of the cleaned powder and the evaporated solution were equivalent. The recovered fat content was thereafter expressed as an amount of the powder's surface-available fat unit weight. [27].

Removal of Inner Available Fat

The particle's inner available fat degrades significantly much slower than its outside unbound fat. After the surface free fat was extracted, the powder residue was added to 20 mL of n-hexane and vigorously shaken by hand for 48 hours. Filtration using filter paper No. 1 separated the solvent and powder residue. The extracted fat was added to the filtrate solution, which was then given 20 minutes in a hoover dryer to evaporate. The amount of the recovered fat was then expressed as a proportion of internal independent fat per unit quantity of powder. [31].

Removal of Total Fat

A bench vortex mixer was used to shake 1 g of the powder in 10 mL of hot water (50 °C) for 2 minutes. The solution was taken out with 20 mL of n-hexane.The mixture was mixed for five minutes, and then centrifuged at 2000 X g for a further five minutes. An Eppendorf pipette was used to capture the transparent organic phase. The organic phase, which contained the collected fat, was then placed in a Petri plate and allowed to dry out in a vacuum drier for 20 minutes.The amount of the isolated fat was subsequently presented as proportion of all the fat in a single unit of powder.

Fluidization Conditions for powder Processing:

All of the experimental setups used in this investigation are listed in Table II.A. Three main circumstances that produce distinctly different outcomes have been discussed below; however, A variety of combinations of time required for processing, relative humidity, and warmth were used. The operational rules have been categorised as increased-humidity (indicating to the relatively driest processing), elevated temperatures (in reference to the relatively warmest process), and moderate (a combination of humidity and temperature that falls among increased-humidity and elevated-temperature).

	Fluidization air temperature,°C	Fluidization air RH, %	Fluidization time, min	Comments
Cluster one	45–48	78 ± 1	15	High-Humidity
Cluster two	55	60 ± 1	15	Mild
Cluster three	65	53 ± 1	15	High-Temperature

Table 1.8.2 : Experimental conditions used to process the powders in the fluidized bed [31].

Fat content of different powders is shown below;

Powders	Surface free fat %, (g)	Inner free fat %, (g)	Encapsulated fat* %, (g)	Total fat %, (g)
Raw Powder	18, (0.008)	25, (0.011)	57, (0.025)	100, (0.044)
High-Humidity	57, (0.025)	39, (0.017)	5, (0.002)	100, (0.044)
Mild	64, (0.028)	32, (0.014)	5, (0.002)	100, (0.044)
High-Temperature	75, (0.033)	23, (0.01)	2, (0.001)	100, (0.044)

*Encapsulated fat was calculated based on the free fat (surface and inner) deducted from the total fat.

Table 1.8.3: Different powders' fat content [31].

Bound to Free Fat Development:

The entire amount of fats, unbound fats, and enclosed fats of unprocessed and treated powders were calculated using the mechanism provided. The trials employed similar fresh powders, therefore the overall fat content of each powder was comparable. It has been shown that the readily accessible spray-dried specimens (raw powders) contain the bulk of their fat as encapsulated particles. This enclosed ratio of fat differs from other research that revealed that majority fat in experimental spray-dried powdered full cream milk had gathered on the outermost layer. However, it is comparable with measurements made for some commercial milk powders. [28] Table 2 displays the fat content of several powders for comparison. Commercial WMP (raw powder) has 25% inner and 18% surface free fat. Only five percent of the entire fat was still enclosed in the treated powder from elevated humidity settings, which had 57% surface available fat and 39% interior available fat. 75% of the surface available fat in treated powder produced under elevated temperatures and 23% of the interior available fat. During elevated-temperature treatment, greater amounts of unbound fat were created on the outer most layer and lesser levels of available fat were created within. It's possible that during high-temperature processing, more liquid fat diffuses outward to the surface, resulting in a decreased amount of available inner fat. The consequences of processing in high-humidity settings will be discussed later. The fat has greater viscosity (due to its reduced temperature) and spreads to the outside more gradually after being released from the matrix that is solid. [31].

Powders	Surface free fat	Inner free fat	Encapsulated fat
High-Humidity	213%	55%	–92%
Mild	250%	27%	–92%
High-Temperature	313%	–9%	–96%

Table 1.8.4: Different processed powders' variations in free fat as compared to raw WMP [31].

Table 3 displays the variations in powder-unbound fat levels as well as the enhancements over raw powders. Surface-free fat content was enhanced by 250% after moderate treatment (55°C and 60% Relative humidity for fifteen min). Surface available fat content was improved by 313% after high-temperature processing, 213% after high-humidity processing, and 313% after high-humidity processing. Elevated temperature and moisture and mild treatment resulted in 55, 27, and 9% variations in the inner free fat concentrations relative to raw powder. More than 90% of the fat in each processed powder is released from its encapsulation to produce additional free fat. All processing settings resulted in a similar amount of total fat release; however, processing at a lower temperature kept the particles with more available fat. The emission of fat into the

outermost layer of the powders may result from the softening of the fat contained inside the particles, where liquified fat may quickly flow towards the surface. More inner unbound fat (from the framework) that is guarded by a particle layer is therefore preferred. Milk fat oxidises easily and affects shelf life. The amount of fat on the surface influences how much cholesterol is oxidised [7, 33, 34]; as a result, interior available fat may be shielded by the membrane of the particle and lower oxidation is observed. When commercialised WMP was processed with warm air, some of the basic interior liberated fat was brought to its outermost layer. Due to fat melting, It's conceivable that the liquidified fat globules in the rigid matrix will enhance the interior pressure and processing at higher temperatures is likely to result in greater liquid fat thermal expansion. More liquidified fat will probably rise to the surface as a result of the intense internal pressure. Because there is less interior fat to plug the interior fractures and holes, this process also results in increased porosity in the particles after cooling.

This section has describes how two distinct mechanisms—the melting of fat and the crystallisation of lactose—can cause fat to release and migrate to particle surfaces. The enhanced crystallinity of the powders produced by this method may also improve the texture, flavour, and overall appeal of chocolate. This study's recommended method shows the capacity to raise available fat (surface and interior) and emits all bulk fat. This section has demonstrated two unique methods by which fat can release and migrate to particle surfaces: melting of fat and lactose crystallisation. The texture, taste, and overall attractiveness of chocolate may all be enhanced by the increased crystallinity of the powders made using this technique. The suggested strategy from this study has the ability to increase both surface and internal free fat and release all bulk fat.

2. MATERIALS & METHODS:

In the 1st phase of trial under this project I made liquid chocolate in pilot plant to study the PSD in depth and to see process of chocolate making and operations of chocolate making equipments and to calculate the fat coating thickness on each particle of both RMC & LMC by using excel calculator, which is very important to understand the phase change i.e fat discrete to fat continuous. The process of development of the excel calculator is elaborated later under this section.

In the 2nd phase of this project I took 5 trials to determine the minimum free fat required in pasting to achieve desired Dv90 from a given refiner & to determine the corresponding fat coating thickness and by using these data I developed a predictor tool. The process of development of the predictor tool is elaborated later under this section.

2.1 EQIPMENTS USED IN LIQUID CHOCOLATE MAKING IN PILOT PLANT

i. Jacketed planetary mixer – Hobart (Ref. Fig.1.1.1) of capacity 15 kg
ii. 300 mm Buhler refiner
iii. 600mm Buhler refiner (ref. Fig.1.1.2)
iv. Elk conche of capacity 5 kg (ref. Fig.1.1.3)
v. Malvern Mastersizer 3000 (ref. Fig.10)

2.2 INGREDIENTS FOR LIQUID CHOCOLATE MAKING

i. Crystal sugar
ii. Lactose monohydrate
iii. Cocoa powder
iv. Skimmed milk powder
v. Whole milk powder
vi. Cocoa butter
vii. AMF
viii. YN
ix. PGPR

2.3 TRIAL PHASE 1:

The below mentioned DoE is developed to determine the number of particles per kg of RMC and LMC and the corresponding fat coating thickness in both RMC and LMC to understand the distribution of fat. Paste and conch recipe are shown below.

TRIAL PHASE 1 - PASTE RECIPE (Batch Size 10 kg)		
Ingredient	**%**	**Kg**
Crystal Sugar	54.3	5.43
Coco Powder	7	0.7
SMP	11.8	1.18
WMP	0	0
Lactose Monohydrate	7	0.7
CBD	20.2	2.02
Total	100.3	10.03

Table 2.3.1: Phase 1 paste recipe

TRIAL PHASE 1 - CONCH RECIPE (Batch Size 5 kg)		
Ingredient	**%**	**Kg**
RMC	93	4.65
CBD	3.5	0.175
AMF	2.5	0.125
YN	0.52	0.026
PGPR	0.2	0.01
Total	99.7	5.0

Table 2.3.2: Phase 1 conch recipe

At first all the solid ingredients were weighed as per the recipe shown in the table 2. Then all solid ingredients were poured into the jacketed Hobart and mixed well for 10 minutes at 50^{o}C (water temperature). After 10 minutes of mixing melted CBD addition was started gradually.CBD was added in 10 minutes of interval while Hobart was running at gear 2. Almost 50% of the total paste CBD was added into the mixture for the 1st pass of refining.

Refining was done by using 300mm Buhler refiner. For the 1st pass of refining temperature was set at 25^{o}C for each of the roller. Gap between R1 & R2 was kept 1.5 and between R2 & R3 was 1.0. 1st pass refining was done at 8 bar roller pressure.

After 1st pass of refining, flakes are transferred to the Hobart and the temperature was set to 50^{o}C. After 10 minutes of mixing rest 50% paste CBD was gradually added in 7 minutes of interval varying gear 1 to 2. After addition of all CBD mixing was continued for 15 to 20 minutes to make the paste proper and consistent for 2nd pass of refining.

In the 2nd pass of refining 43.502 micron particle size was achieved at 14 bar pressure and gap between R1 and R2 was 0 and the gap between R2 and R3 was maintained 0.5. When the pressure was increased to 16 bar from 14 bar and gap was reduced to 0.25 from 0.5 between R2 and R3, Dv90 was reduced to 30.406 micron. Temperature for 1st, 2nd and 3rd roll was kept at 40^{o}C, 42^{o}C and 30^{o}C respectively in each case.

Fig.2.3.1: Paste for 2nd pass in phase 1 trial

After 2nd Pass of refining according to the conch recipe 4.65 kg of refiner flake was transferred to the 5kg Elk conche. Conch profile was shown below;

Conch Profile					
	Duration	Speed	Direction	Water Temp	
Loading	5	750	Tip forward	55	
Dry Conching	60	750	Tip forward	55	
Pasting	60	1500	Tip backwards	50	AMF + 70% CB + YN
Mixing	20	2400	Tip forward	45	Remaining CB + PGPR
Discharge	Manual				

Table 2.3.3: Trial phase 1 conch profile

At the starting of paste conching AMF and 33% of YN were added, at 20 minutes of pasting 35% of CBD of conch CBD and 33% of YN were added and at 40 minutes of pasting rest of the YN and 35% of CBD were added. In the liquefaction stage remaining CBD and PGPR is added. Then the mass was manually discharged from the conch. Particle size was measured of this LMC and found to be 29.164 micron.

2.4 DEVELOPMENT OF EXCEL CALCULATOR:

Under this project I developed an excel calculator which is able to determine the number of particles in per kg of refined milk chocolate and liquid milk chocolate and fat coating thickness on each particle which is very important to understand the distribution of fat around the RMC and LMC particles.

2.4.1 Aims:

i. To determine the number of particle in RMC

ii. To determine the thickness of fat coating on each particle in RMC (Assuming uniform coating happens in RMC)

2.4.2 Assumptions:

As I discussed in the section 1.7 there are some key challenges in this project, so to proceed further in this project below mentioned assumption need to be taken;

i. All particles are non porous and spherical.
ii. Refine ability of each recipe subcomponent is equal.
iii. All solid particles are present in each volume distribution at recipe combination in RMC.

2.4.3 Theory:

Cocoa butter, fats in WMP/SMP and fats in cocoa particle are present in paste & RMC as total fat. In RMC percentage of fat coverage on solid particles like Sugar, Cocoa, Lactose, Protein is not uniform. But during conching with the help of mixing, heating and shearing & addition of remaining fat and emulsifier particles are got well coated with fat so that flow properties of mass get improved. Hence thickness of fat coating on particles of LMC is more uniform than that of RMC.

2.4.4 Terminologies:

i. Percentage Volume distribution at different diameter (**V_{ds}**) : Malvern gives Dv90 along with % volume distribution of core solid particles at different diameter. These data is given in appendix section.
ii. Percentage of each solid wrt total solid**(x):** (% Solid in ingredient 1/total solid). Total solid is defined as amount of solid present in the recipe excluding moisture & fat.
iii. Percentage volume distribution for each Solid **(V_{ds}*x):** this is to be calculated for each type of solid present in different diameter like for sugar, cocoa powder, lactose SMP & WMP**.**
iv. RMC volume **(V_{RMC}):** Summation of Volume of Solid (excl. fat & moisture) of each solid ingredient of the recipe. Volume of each solid will be given by (mass of each solid * true density).
v. Absolute Volume **(V_{RMC}* V_{ds}*x):** volume of each solid (in m^3) present at different diameter given by Malvern. This is calculated for each solid present in the recipe.
vi. No. of particle **(N):** It is a important parameter to determine the fat coating thickness .Lower the Dv90 greater the no. of particle and lesser the coating thickness and vice versa.
N= (V_{RMC}* V_{ds}*x)/ volume of 1 particle. This is to be calculated for each solid.
vii. Core Volume **(V_{pc}):** Volume of 1 particle without fat coating that is (0.523*d_i^3), where d_i is the core particle diameter given by Malvern.
viii. Coating thickness **(L):** Thickness of fat coating on each particle of RMC and LMC.

2.4.5 Calculation Process:

To understand the distribution of fat around the particle of RMC and LMC it is very important to determine the fat coating thickness around the particle assuming all particles are spherical. To calculate this coating thickness, number of particle in a certain mass should be a known parameter. So to find out the number of particle we need to know the total volume of mass that is to be divided by volume of one particle. Total volume of mass per kg can be calculated from the ingredient composition in the recipe and their true density as density is mass upon

volume. True densities of all ingredients are shown below from which volume of each ingredient is calculated to calculate total volume per kg of mass.

Ingredients	True Density in kg/m^3
Crystal sugar	1590
Lactose	1520
Cocoa solids	1350
Milk protein	1460
CBD / Cocoa fat	974
AMF / Milk fat	920

Table 2.4.1: Densities of Ingredients

In the report of Malvern diameters of each different particle along with their percentage volume distribution is given. From this diameter volume of one particle can be calculated. As Malvern gives only the core diameter of particles excluding fat and moisture, total volume of RMC is calculated excluding fat and moisture from each ingredient. Ingredient specification is shown below;

Ingredient	Moisture(%)	Fat(%)	SNF(%)
Crystal sugar	0.08	0	99.92
Coco powder	4	11	85
SMP	4	0.9	95.1
WMP	4	26.5	69.5

Table 2.4.2: Ingredient specification

To run this calculator recipe composition and percentage volume distribution at different diameter is to be given as input. Ingredient specification and true densities are already predefined in the excel calculator and considered within the formula as required. Coating thickness on each particle and number of particle per kg are the two outcomes from this calculator. SMP or WMP in the recipe are splitted in two fractions – milk protein and lactose with ash for accuracy and ease of the calculation. This percentage volume distribution at different diameter changes with the Dv90. These data are added in appendix section for different Dv90 associated with this project. Calculation process is shown below;

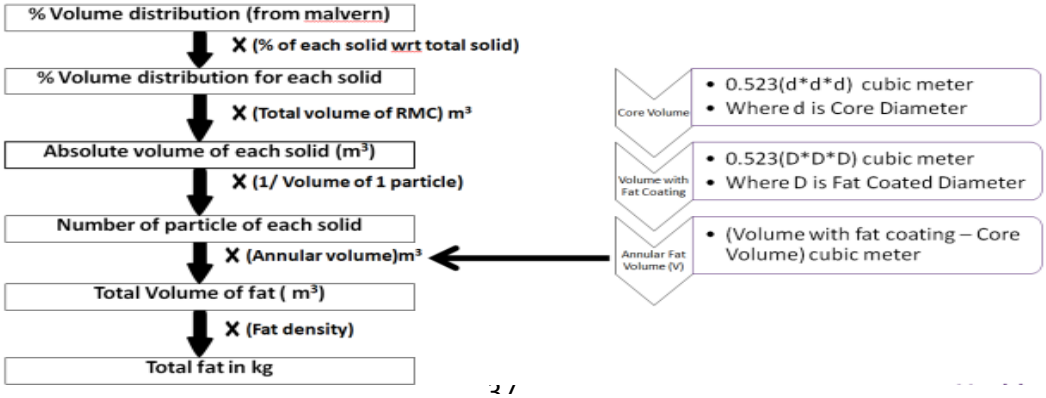

Fig.2.4.1: Mathematical calculation process

This whole calculation was done in Microsoft excel by applying some of the existing formulas in excel and also by creating simple mathematical formula as described in fig.2.4.1. Derived equation for the above mentioned calculation process is given by

$$N = V_{RMC}\left(\sum_{i=1}^{n} \frac{V_{ds_i}}{V_{pc_i}}\right)\left(\sum_{j=1}^{m} x_j\right)$$

$$V_F = \left\{0.523\left(\sum_{i=1}^{n} d_i + 2L\right)^{(3)} - \sum_{i=1}^{n} V_{pc_i}\right\} \times \sum_{j=1}^{m} \sum_{i=1}^{n} N_{ji}$$

Where,

V_{ds} = Volume distribution given by Malvern (%)

V_{pc} = Core volume of particle (m^3)

x = solid of each ingredient/total solid

V_{RMC} = Total volume of RMC (m^3)

V_F = Total volume of fat (m^3)

N = Total number of particles

d_i = Diameter of core particle (m)

L = Fat coating thickness particle(m)

$i = No.\ of\ reading\ of\ \%\ volume\ distribution\ \&\ corresponding\ diameter$

$j = No.\ of\ Solid\ ingredient\ in\ recipe$

If coating thickness is given, we can determine the amount of fat and if the amount of fat is known, then we can find out the coating thickness assuming all particles are spherical and get coated uniformly**.**

Paste recipe and conch recipe of phase 1 trial (ref. to table 2.3.1 & table 2.3.2) and their corresponding percentage volume distribution at different diameter was uploaded to input section of this calculator to get the coating thickness of both LMC and RMC that will be discussed in Result & Discussion section (3.1).

2.5 TRIAL PHASE 2:

The DoE(s) are made by using a statistical tool called minitab to determine the minimum amount of free fat in pasting to achieve desired Dv90 on a given refiner and the corresponding

fat coating thickness on each particle by using that excel calculator as discussed in the section 2.4.5. From this data I developed a predictor tool for the estimation of minimum fat to achieve desired Dv90 on a given refiner and corresponding minimum fat coating thickness on each particle.

There are total 5 DoE(s) consists of 4 prototypes (P2, P4, P8, P12) and a standard (STD). DoE(s) are shown below;

DoE							
	Paste Recipe 100%						RMC Total Fat(%)
	Crystal Sugar	Cocoa Powder	SMP	Lactose	WMP	CBD	
STD	54.3	7	11.83	7	0	19.9	**20.58**
P2	54.3	6.9	12.12	7	0	19.68	**20.35**
P4	54.3	6.9	10	0	9.44	19.36	**22.52**
P8	54.3	8.86	12.96	0	5.88	18	**20.47**
P12	54.3	6.9	10	0	11.8	17	**20.81**

Table 2.5.1: DoEs for trial phase 2

In this case all RMC were made by using 600mm Buhler refiner due to unavailability of 300mm refiner in pilot plant.

Pasting was done in two stages- paste before 1st pass of refining and paste before 2nd pass of refining. Pasting temperature was varied from 50^0C to 55^0C for each recipe and after addition of all the solid ingredients it was mixed for 10 minutes in the Hobart then CBD addition was started. In the 1st pasting around 36%-37% of CBD of total CBD for pasting was added, remaining CBD was added in 2nd pass pasting and this technique was followed for each prototype including standard. After addition of CBD in 1st pass pasting, it was mixed in Hobart for more 10 minutes. Then 1st pass refining was done. After that 1st pass refiner flakes were transferred to the Hobart and after 10 minutes of mixing remaining CBD for 2nd pass pasting that was around 64%-65% of CBD of total CBD in the paste recipe was started to add. After completion of this addition, the mass was mixed in the Hobart for more 20-25 minutes at slow speed to make paste like structure. After that 2nd pass of refining was done and particle size was measured. Refining condition is discussed below.

In 1st pass of refining roller temperature for three rollers were kept 25^0C and the gap between 1st and 2nd roller was 1.5 and the gap between 2nd and 3rd roller was 1. Roller pressure for both left and right side was 8 bar. These conditions were kept constant for each prototypes including standard for 1st pass of refining.

In 2nd pass of refining roller temperature of 1st, 2nd, and 3rd roll was 40^0C, 42^0C and 30^0C respectively. Roller pressure and gap was varied from low to extreme condition to achieve

lowest possible Dv90 according to recipe from 600mm refiner, which was shown in Result and Discussion section.

2.6 ANALYTICAL PROCEDURES:

Particle Size Measurement:

- **Equipment Used:** Malvern Mastersizer 3000
- **Objective:** To determine the particle size (Dv90) of chocolate

Fig.2.6.1: Malvern experimental setup

- **Working Principle: Laser diffraction**

The magnitude of the light that scatters is evaluated by angular fluctuation after the beam of laser traverses a sample of dispersed particulate matter. In relation to the laser beam, big particles scatter light at small angles and microscopic particles scatter light at large angles. Using the Mie theory of scattered light, the angular scattering intensity data is then examined to determine the size of the particles that produced the scattering pattern**.**

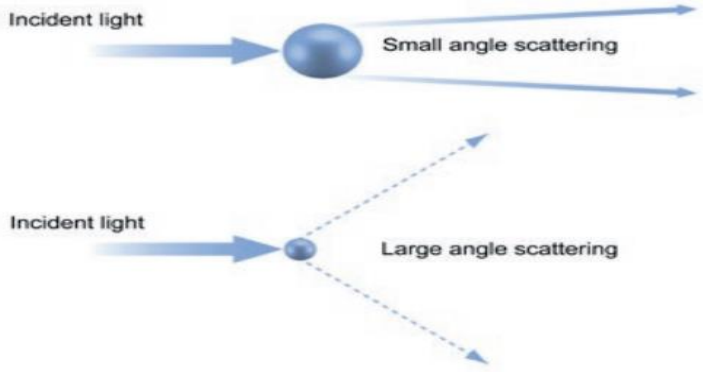

Fig.2.6.2: Laser diffraction in Malvern

- **Sample Preparation for RMC**

i. After 2nd pass of refining, refiner flakes was mixed properly by using ladle.

i. After that 2g refiner flakes was weighed in petri dish.
ii. Then 10-12 drops of MCT oil was added in it by using dropper and mixed with a glass rod to form dough like structure.
iii. From that dough 0.16g was measured in a conical container.
iv. Then 15 ml of MCT oil was poured in that container containing that sample.
v. Then the mixture was sonicated for sharply 2 minutes by using a sonicator. This dispersion would be the sample for particle size measurement.

- **Sample preparation for LMC:**
  i. 0.16 g of LMC was weighed in a conical container.
  ii. Then 15 ml of MCT oil was poured in that container containing that LMC.
  iii. Then the mixture was sonicated for sharply 2 minutes by using a sonicator. This dispersion would be the sample for particle size measurement.

- **Procedure of Measuring Sample:**
  i. Hardware was switched on.
  ii. Malvern 3000 software was open in the laptop connected to the machine.
  iii. New folder was created with date in that software.
  iv. To make the equipment ready for measurement machine background which was shown the laptop should be okay. To settle the background rpm was increased immediately to 2500 for 10 seconds and then immediately reduced it to 0. This process was repeated until all the air bubbles of the MCT oil removes from sample dispersion unit. After removal of all air bubbles background would be ready and then the tab 'Measure Background' was clicked in the monitor and message ' Add sample to measure' was shown in the display.
  v. Then from the prepared sample as discussed earlier whatever it may be LMC or RMC sample was added drop wise until the obscuration level goes up by 13. Obscuration level should be in between 13 to 17. When obscuration level showed in green then 'Measure sample' tab was clicked in the display.

- **Cleaning Procedure:**
  i. After measuring a sample, MCT oil containing sample was drained from the sample dispersion unit.
  ii. Then sample dispersion unit was refilled with fresh MCT oil by hand pumping from the MCT oil tank.
  iii. Machine RPM was increased to highest for 10 seconds and reduced it to zero. This process was repeated 2 times.

iv. Then MCT oil was drained from the sample dispersion unit.
v. ii, iii, iv was repeated once again to was the sample dispersion unit properly.
vi. Then again sample dispersion unit was refilled with fresh MCT oil and background was made ready to measure new samples.

- **Test Report:**

Malvern shows different parameters like concentration, uniformity, specific surface area, Dv10, Dv50 & Dv90 etc. Malvern also gives particle size distribution curves as shown in fig.1.3.2 and the percentage volume distribution at different diameter. In this project the concerned parameters are Dv90, PSD curve and percentage volume distribution at different diameters.

Diameter (micron)	% Vol Dis
0.675	0.13
0.767	0.25
0.872	0.42
0.991	0.64
1.125	0.91
1.279	1.24
1.453	1.6
1.651	1.98
1.875	2.37
2.131	2.75
2.421	3.09
2.75	3.38
3.125	3.61
3.55	3.77
4.034	3.86
4.583	3.88
5.207	3.85
5.916	3.77
6.722	3.65
7.637	3.51
8.677	3.34

Table2.6.1: **Snapshot of partial data of Percentage volume distribution at different diameter**.

In table 2.6.1, a partial data of RMC of P12 (ref. table.2.5.1) is shown of Dv90 55 micron. In the left column different particle diameters are shown and in the right their corresponding percentage volume distribution. For example in table 2.6.1 the first row implies that at 0.675 micron particle diameter 0.13% volume of particles lies of total volume of particle and so on. It is assumed that each volume distribution is in recipe combination.

These whole testing procedure was performed for trial taken in Phase 1 of RMC & LMC and also in Phase 2 for multiple RMC samples of STD, P2, P4, P8, & P12 to determine the Dv90. All of these results are shown and discussed in Result and Discussion section. Percentage volume

distribution data of each prototype for every Dv90 was added in Appendix section as these are huge data set.

3. RESULT & DISCUSSION:

3.1 FOR PHASE 1 TRIAL:

After measuring particle sizes of two RMC samples of trial phase 1 (discussed in the section 2.3) following the process discussed in section 2.6, I got two different Dv90, **43.502 micron** and **30.406 micron** depending on different refining condition by using 300mm Buhler refiner. I did not use the RMC of 43.502 micron in liquid chocolate making because of its higher Dv90. LMC was made by using RMC of 30.406 micron.

From the detailed Malvern report of RMC of 30.406 percentage volume distribution at different diameters (attached in appendix) along with paste recipe combination (ref. table 2.3.1) were given as input in the excel calculator. After calculation as the process discussed in section 2.4.5, it was found that the **number of particle per kg of RMC was 6.241E+13 and fat coating thickness on each particle was 0.249 micron.**

Fig.3.1.1: RMC in phase 1.

In left 43.502 micron & in right 30.406 micron

In Fig.3.1.1 it is clearly seen that the color of RMC of 43.502 micron is slightly darker than RMC of 30.406 micron. As higher size particle scatters light at lower angle and lower size particle scatters light larger angle, so RMC of 30.406 micron looks comparatively brighter in color. There may also have another reason of this color difference that is the wet ability of particles with fat. As recipe composition is same for two RMCs and normally RMC with higher Particle size contains less number of particles than the RMC with lower particle size in a certain amount of

mass so naturally wet ability or coating thickness should be more in case of RMC of higher particle size. So it looks comparatively darker in color.

Particle size (Dv90) of LMC was found **29.164 micron**. From the detailed Malvern report of LMC of 29.164 percentage volume distribution at different diameters (attached in appendix) along with conch recipe combination (ref. table 2.3.2) were given as input in the excel calculator. After calculation as the process discussed in section 2.4.5, it was found that the **number of particle per kg of LMC was 7.211E+13 and fat coating thickness on each particle was 0.305 micron.**

It is clearly observed that as the particle size of RMC is greater than that of LMC so the same amount of RMC contains lesser number of particles than that of LMC.

Although LMC contains more number of particles than RMC but still Coating thickness is higher in case of LMC because of addition of more CBD in conching and removal of some portion of bound fat from milk particles and cocoa powder due to conching. These fats are become effective to coat particles.

RMC was fat discrete phase but LMC became fat continuous phase with coating thickness difference of 0.056 micron on each particle. Though this difference is very less numerically but numbers of particles are in 10^{13} numbers per kg, so it will have a huge impact in overall mass. Besides for mixing , heating and shearing in conching and due to addition of emulsifiers immobilized fat becomes mobilized that turns the system fat continuous from fat discrete phase which gives more uniform coating thickness on each particle than that of RMC.

3.2 FOR PHASE 2 TRIAL:

In the continuation of section 2.5, 2nd pass of refining was done for all the prototypes STD, P2, P4, P8 and P12 at low to high refining condition and as a result of it different particle sizes were obtained from Malvern according to the process discussed in the section 2.6. The result is shown below in a tabular form;

Prototype	Attempt	Roller Pressure (bar)	Roller Gap B/W R1 & R2	Roller Gap B/W R3 & R4	Dv90 (micron)
STD	1st	**18**	**0.5**	**0**	**40.124**
	2nd	18	0	0	42.86
	3rd	20	0.5	0	54.096
	4th	20	0	0	48.35
P2	1st	**18**	**0.5**	**0**	**47.38**
	2nd	18	0	0	48.024
P4	1st	16	0	0	46.477
	2nd	20	0.5	0	45.351
	3rd	**18**	**0.5**	**0**	**44.66**
P8	1st	18	0.5	0	58.23
	2nd	**20**	**0.5**	**0**	**53.55**
	3rd	20	0	0	55.75
P12	1st	16	0.5	0	63.023
	2nd	18	0.5	0	60.35
	3rd	**20**	**0.5**	**0**	**55.99**
	4th	20	0	0	63.004

Table 3.2.1: **Obtained Dv90 at different refining condition**

These green highlighted rows in Table 3.2.1 are indicating that at that free fat percentage in pasting of each prototype including standard that Dv90 could be the lowest possible Dv90 that was achieved, as I explored refiner from low to high condition.

As the DoE discussed in the section 2.5, CBD percentage was varied from 17% to 19.9, while RMC total fat was varied from 20.35% to 22.52%. Pictures of 2nd pass pasting and RMC of lowest achieved Dv90 (RMC of green highlighted rows in table 3.2.1) for each prototype is shown below.

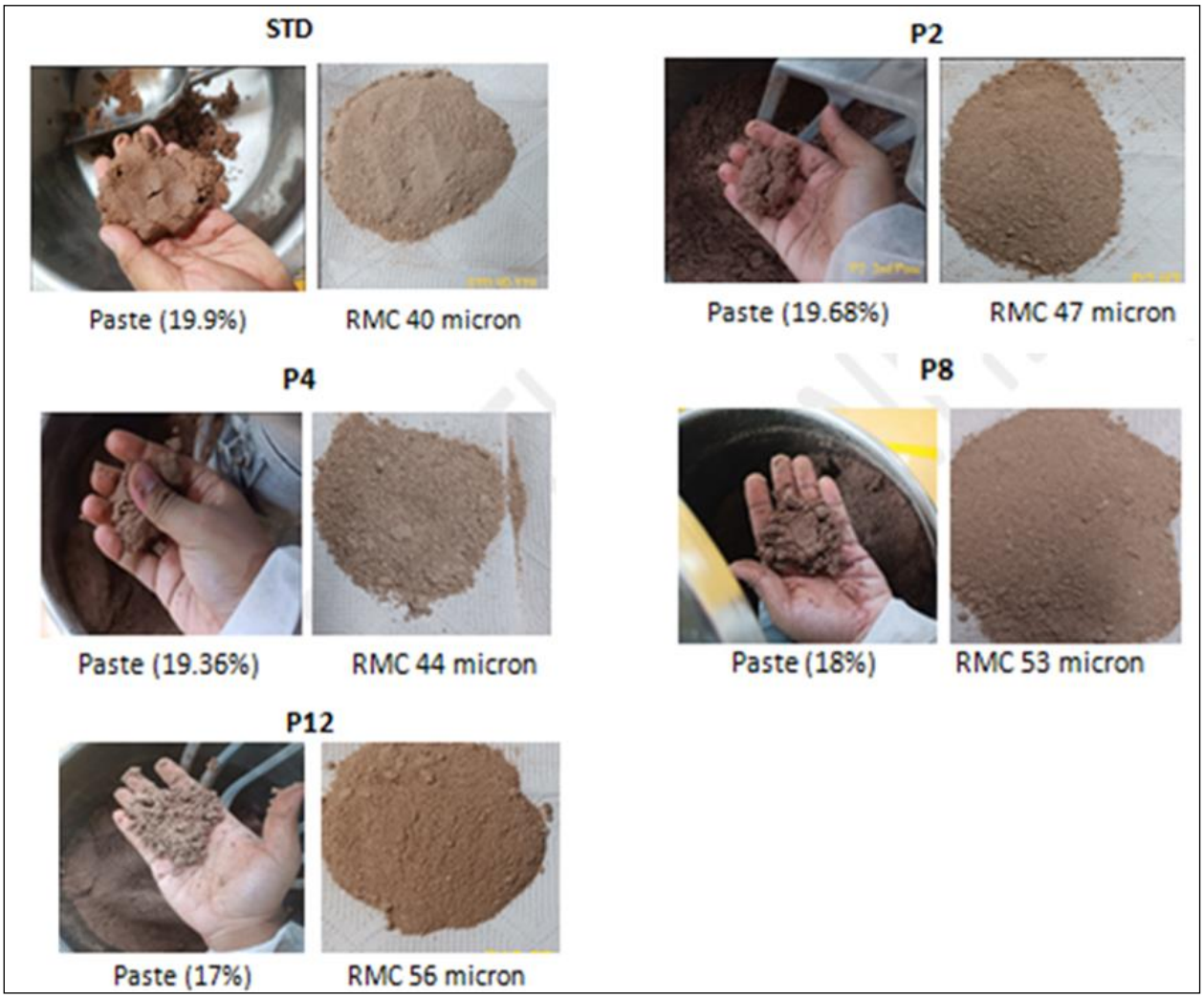

Fig.3.2.1: **Outcomes from DoE(s); comparison of paste and RMC at different fat percentage**

It is clearly observed that at 17% of CBD in Paste recipe (P12), the paste was very dry compared to STD that contains 19.9% of CBD in paste recipe and hence at this fat percentage it is not possible to achieve 40.124 micron particle size at any operating condition by using 600mm Buhler refiner that was used in our pilot plant. So there is a requirement of a minimum free fat

percentage in paste recipe to achieve desired particle size from a given refiner. Free fat percentage becomes lower gradually from STD to P12 and so paste becomes drier and hence lowest possible Dv90 becomes larger from STD to P12.

To calculate number of particles per kg (N) and the corresponding fat coating thickness (as per section 2.4.5) on each particle for each prototype, recipe composition for each prototype and percentage volume distribution corresponding to obtained Dv90 (added in the Appendix-1) was uploaded to excel calculator as a input. The results are shown below;

	From Excel Calculator		From Malvern
PROTOTYPES	No. of Particles/kg[1] (N)	Coating Thickness(L), micron	Dv90 (micron)
STD	5.55	0.269	48.35
	6.12	0.255	40.124
	5.96	0.26	42.86
	5.68	0.271	54.096
P2	6.13	0.2549	47.38
	5.76	0.26	48.02
P4	5.8	0.2597	44.66
	5.71	0.261	45.351
	5.79	0.2616	46.022
P8	6.37	0.231	53.55
	5.98	0.234	55.75
P12	5.6	0.24	55.99
[1]No. of Particles/kg are in 10^{13}numbers			

Table3.2.2: **Result from excel caculator in phase 2**

From the table 3.2.2 the following conclusion can easily be drafted;

i. Coating thickness is dependent on two factors- No. of particles (N) & % CBD. If I consider any of the prototype, it is clearly seen that coating thickness is lesser when number of particle (N) is higher and vice-versa. So number of particle and coating thickness is inversely proportional at constant CBD percentage.
ii. Coating thickness is proportional to CBD percentage when N is constant.
iii. No. of particle (N) is dependent on 2 factors they are Dv90, type and composition of the ingredient used.
iv. If I consider any one of the prototype it is clearly observed that at higher Dv90, N is lesser and vice-versa. So N and Dv90 is inversely proportional provided type and composition of ingredient should be same.
v. In the table it is clearly observed that in case of STD at 40.124 micron N is 6.12×10^{13} and on the other hand in case of P8 at 53.55 micron N is 6.37×10^{13}. From these result

dependency of number of particle on type and composition of ingredients can be studied. As per the calculation process discussed in section 2.4.5 N was calculated from the volume of solid and this volume was calculate the true density of that solid. So if any recipe contains more amount of low density ingredient than that of other recipe, obviously number of particles will be more in that low density ingredient recipe because lower density implies higher volume and hence higher the number of particles. In this case P8 contains lesser amount of lactose than STD and the density of lactose is 1520 kg/m^3 and instead of that amount of lactose P8 contains more amount of milk protein of density 1460 kg/m^3 and Cocoa solids of density 1350 kg/m^3. So P8 contains more number of particles per kg than STD.

vi. If the color of RMC is observed it is clearly seen that larger the particle size darker the color and vice-versa. Light scatters at smaller angle from the larger particle so it looks darker but in case of smaller particles light scatters at higher angle so it looks brighter. Wet ability of particles with fat may be another reason of this color difference.

With the data obtained from the excel calculator, I developed a predictor tool, which is discussed in the next section.

4. DEVELOPMENT OF PREDICTOR TOOL:

I made an excel calculator as discussed in section 2.4. I upgrade this excel calculator to a predictor tool over excel based on the DoE discussed in section 2.5 (table 2.5.1) and data obtained from the DoE (table 2.5.1) discussed in section 3.2 of table 3.3.2.

The aim of this tool is to predict the minimum percentage of free fat required in pasting to achieve desired Dv90 on 600 mm Buhler refiner used in pilot plant and the corresponding fat coating thickness on each particle of RMC.

This predictor model is based on two step regression. 1^{st} regression was between lowest achieved Dv90 & Solid to CBD ratio (Z; dry basis) and 2^{nd} one was bi variant regression that was between coating thickness (L), Number of particles per kg (N) & Solid to CBD ratio (Z).

Dv90 is not only depends on the free fat percentage but also depends on the type and composition of the solid. So to reduce the number of variable in 1^{st} regression it is better to take solid to CBD ration instead of taking %solid and %CBD as two separate variables.

As the Dv90 measured by Malvern is only of the core solid particles, so this Z is calculated on dry basis. Moisture and fat % from each of the ingredients is subtracted to calculate percentage solid in dry basis; specification given in table 2.4.2.

User should give their desired lowest Dv90 and percentage of solid ingredient present in the recipe as input and also user will get an option to choose refiner either 300mm or 600mm, though as of now this tool will only work for 600mm refiner so user should choose only 600 mm refiner for now. When user give input lowest Dv90 then from the data pool it will take the corresponding percentage volume distribution at different diameter which is required for calculation as discussed in section 2.4.5. This will show minimum CBD percentage and the corresponding fat coating thickness on each particle as output. To develop this tool three worksheets are used in which 1st was user interface sheet, 2nd and 3rd one were calculation sheet and data pool sheet only for the use of developers.

4.1 CHARACTERISTICS OF TOOL:

i. This tool is applicable for powder-based chocolate which may contain sugar, cocoa Powder SMP, WMP & lactose in its paster recipe.
ii. This whole calculation based on considering CBD only as free fat in Pasting.
iii. It will show the min. CBD% for pasting at which user targeted D90 can be achieved at any refining condition in 600mm refiner along with coating thickness on each particle.
iv. This is applicable only for 600mm refiner used in the pilot plant of Thane Technical Centre, Mondelez thus far.

4.2: REGRESSION BETWEEN LOWEST Dv90 ACHIEVED AND SOLID TO CBD RATIO (Z):

The aim of this step is to generate a prediction equation of lowest Dv90 that can be achieved at a certain minimum free fat % and the corresponding Solid to CBD ratio (Z).
In section 3.2, table 3.2.2. Green highlighted Dv90 of each prototype are the lowest possible Dv90 that was achieved at that fat percentage on a given 600 mm refiner implies, that fat percentage was the required minimum fat percentage to achieve that green highlighted Dv90. In these cases Solid to CBD ratio (Z) was maximum as the fat % was minimum for that Dv90 and as % fat was at denominator in Z.

So to predict maximum Z and hence minimum CBD% only those green highlighted data are selected which is shown below;

Prototypes	% CBD	% SOLID	Solid/CBD (Z; dry basis)	Lowest Dv90 (micron)
STD	19.9	80.1	3.941	40.124
P2	19.68	80.32	3.996	47.38
P4	19.36	80.64	3.935	44.66
P8	18	82	4.344	53.55
P12	17	83	4.578	55.99

Table 4.2.1: **Selected data for Regression between Z & lowest Dv90**

The regression equation is given by;

%Solid/%CBD (Z) = 1.816 + 0.0523 Lowest Dv90

Regression plot is shown below;

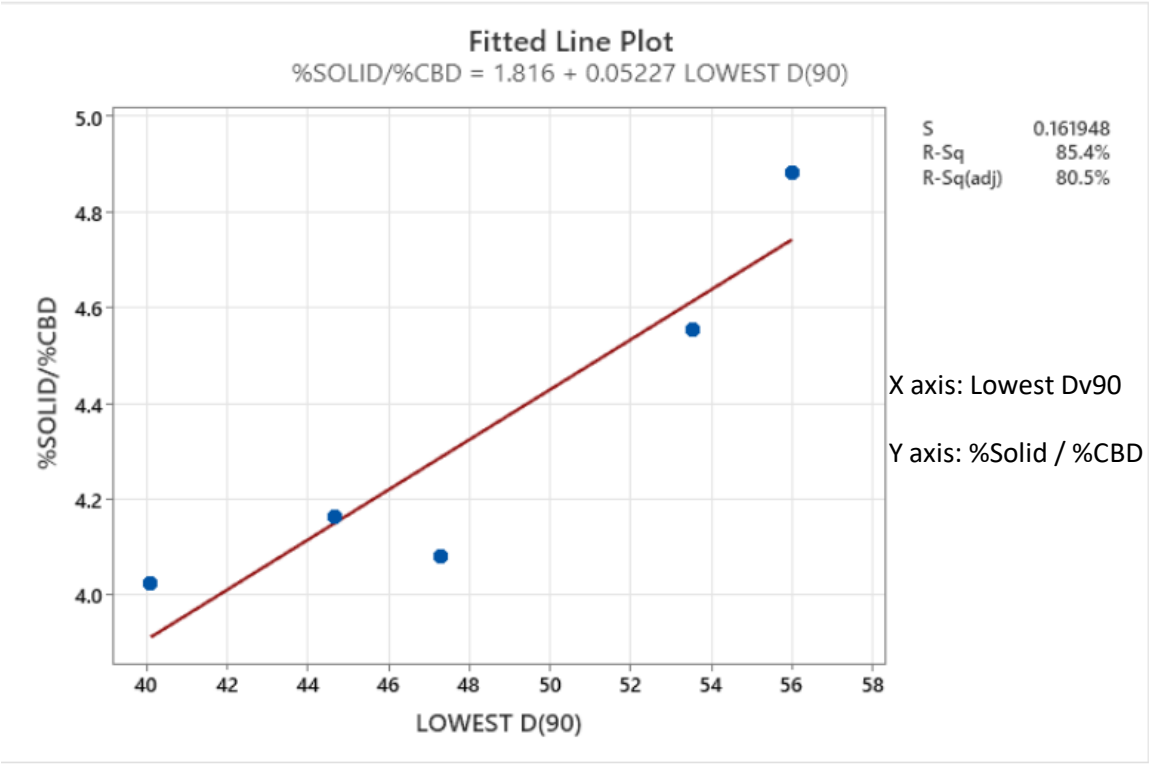

Fig.4.2.1: Regression plot between Z & Dv90

Model summary is given below;

$R^2 = 0.853$
P-Value(95% Confidence Level) = 0.024
Standard Error= 0.162

From the model summary it is clear that 85.3% of data are satisfying this model. For more better fit I need to collect more data conducting more trials. Pareto chart for the above regression is shown below;

Fig.4.2.2: Pareto chart of regression plot between Z & Dv90

From the pareto chart it is clearly observed that histogram crossed the threshold value that is 3.182 so lowest Dv90 can be a good predictor of Z from which minimum percentage of CBD is going to be calculated.

4.3 REGRESSION OF COATING THICKNESS (L) ON NUMBER OF PARTICLES (N) AND SOLID TO CBD RATIO (Z):

As I discussed in the section 3.2 coating thickness highly depends on number of particle and CBD percentage which can be proved by this regression and the corresponding pareto chart.

The aim of this stage is to predict the fat coating thickness on each particle through an equation in which coating thickness will be unknown and number of particles (N) and Solid to CBD ratio (Z) will be known variable. N was calculated in the backend of tool by using the calculation process discussed in section 2.4.5 and Z was calculated from the regression equation discussed in the section 4.1.

Regression is shown below;

From Recipe		From Excel Calculator	
PROTOTYPES	Solid/CBD (Z; dry basis)	No. of Particles/kg[1] (N)	Coating Thickness(L), micron
STD	3.941	5.55	0.269
		6.12	0.255
		5.96	0.26
		5.68	0.271
P2	3.996	6.13	0.2549
		5.76	0.26
P4	3.935	5.8	0.2597
		5.71	0.261
		5.79	0.2616
P8	4.344	6.37	0.231
		5.98	0.234
P12	4.578	5.6	0.24

[1]No. of Particles/kg are in 10^{13}numbers

Table 4.3.1: Selected data for regression between L, N & Z

Regression was done in Minitab. Obtained regression equation is shown below;

Coating thickness = 0.584 - 2.39*10^{-15} N – 0.0466 Z

From this equation coating thickness is going to be calculated.

As this is a bi variant regression, obtained regression plot is a matrix plot that is shown below;

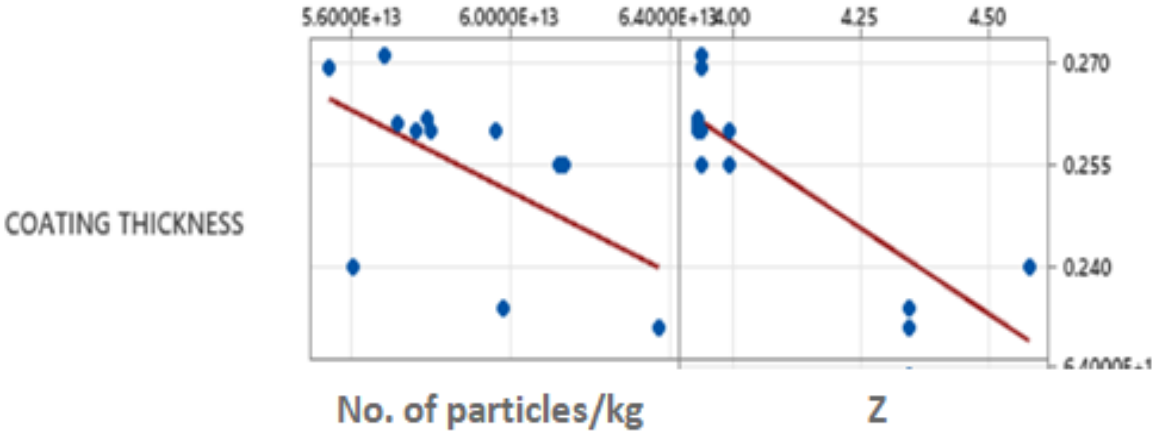

Fig.4.3.1: Regression plot between L, N & Z

Model summary is shown below;

R^2= 0.946
P-Value (95% Confidence Level)
For No. of particles/kg = 0.0056
For %solid/%CBD= 0.0022
Standard Error= 0.001

It is clearly observed hat 94.6% of data mentioned in the table 4.2.1 are satisfying this model. How coating thickness (L) is depends on the number of particles (N) and percentage sold to percentage CBD ratio that is shown below with a pareto chart.

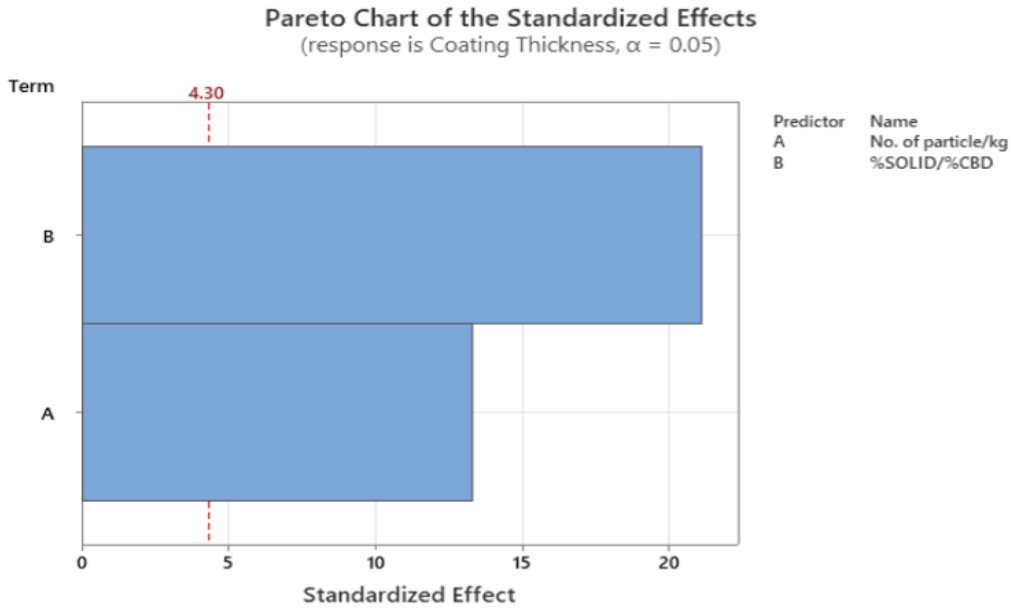

Fig.4.3.2: Pareto chart of regression between L, N & Z

From pareto chart (Fig. 4.3.2) it is clearly observed that coating thickness is highly depends on N & Z. So these two parameters can be good predictor for coating thickness. But coating thickness is more highly depends on percentage solid to percentage CBD than number of particles.

4.4 Data bank/pool:

As I mentioned earlier to calculate number of particles and coating thickness percentage, volume distribution at different diameter is one of the required data obtained from detailed report of Malvern and these data are different for different Dv90 value.

So in that tool I made a excel sheet consists of percentage volume distribution at different diameter for Dv90 ranging from 40 micron to 56 micron. I got these data from my trial by using 600mm refiner. So when user will give input desired Dv90 in user sheet it will take the percentage volume distribution data of the corresponding nearest Dv90 value of the user input desired Dv90 with the help of multiple index match formula applied in that sheet. This data set going to be transferred to the calculation sheet for further calculation.

4.5 Working process of tool:

User should give desired Dv90 and percentage solid present in recipe as input in the user sheet of that tool. Then based on the CBD (free fat) present in the user input paste recipe number of particles (N) is going to be calculated as per the process discussed in the section 2.4.5. To calculate N, the required percentage volume distribution at different diameter is taken up from data pool correspond to nearest user input Dv90 to calculation sheet of that tool.

Based on user input Dv90 and percentage solid recipe, max solid to CBD ratio (Z) is calculated from regression 1 as discussed in the section 4.2. From these Z minimum percentage of CBD is calculated to achieve that user input Dv90.

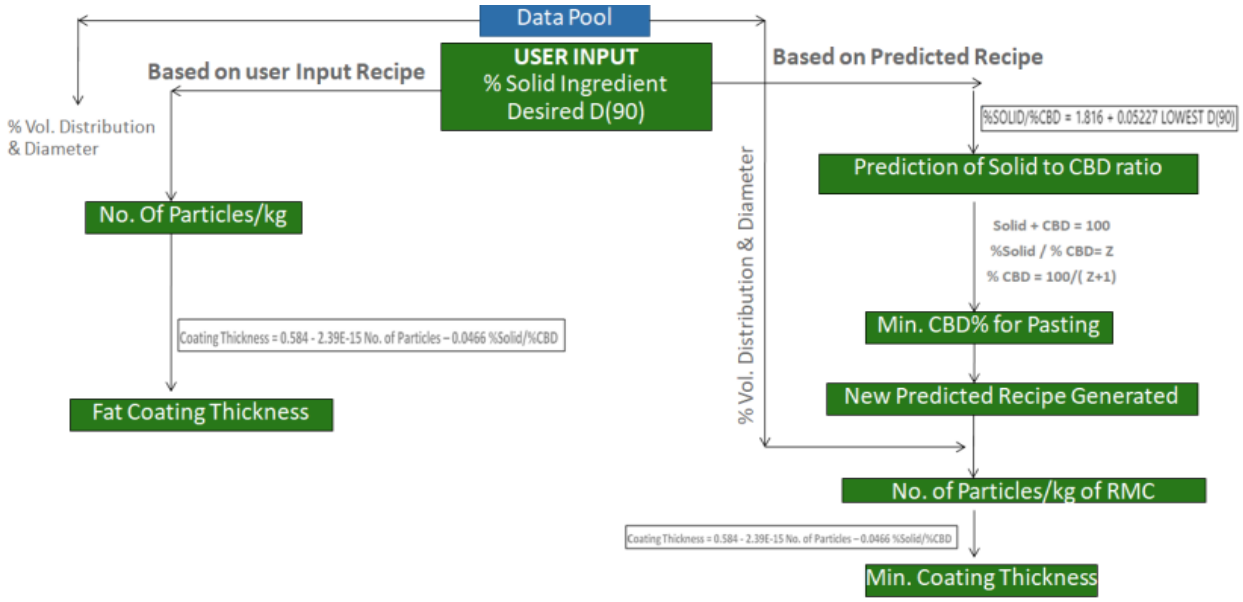

Fig.4.5.1: Working Principle of tool

On the other hand Coating thickness is calculated by the prediction equation discussed in the section 4.3 where Z is already calculated from regression 1 (discussed in section 4.2) and N will be calculated as the process discussed in section 2.4.5.

If user input recipe contain more CBD than the required minimum CBD it will show “PASS” message that user can achieve desired Dv90 value with their input recipe but that one is not the minimum fat recipe to achieve that Dv90. So this tool will also show the required minimum fat to achieve that Dv90. If user recipe contains less than the minimum required CBD to achieve user input Dv90 then it will show “FAILURE” message for the user input recipe along with the required minimum CBD percentage for achieving that Dv90. Formula was designed in excel in such a way so that in case of “FAILURE” a new predicted recipe will be generated by proportionately deceasing the solid percentage of each ingredient and increasing the same amount of CBD percentage from the user input recipe. But in case of “PASS” a new predicted recipe will be generated by proportionately increasing the solid percentage of each ingredient and decreasing the same amount of CBD percentage from the user input recipe.

On the basis of this predicted recipe, number of particles is calculated as per the process discussed in the section 2.4.5 and then coating thickness on each particle corresponding to the minimum CBD percentage is calculated with the help of 2nd prediction equation discussed in the section. Till now this tool was not validated, it is ongoing. User interface of the proposed tool is shown below;

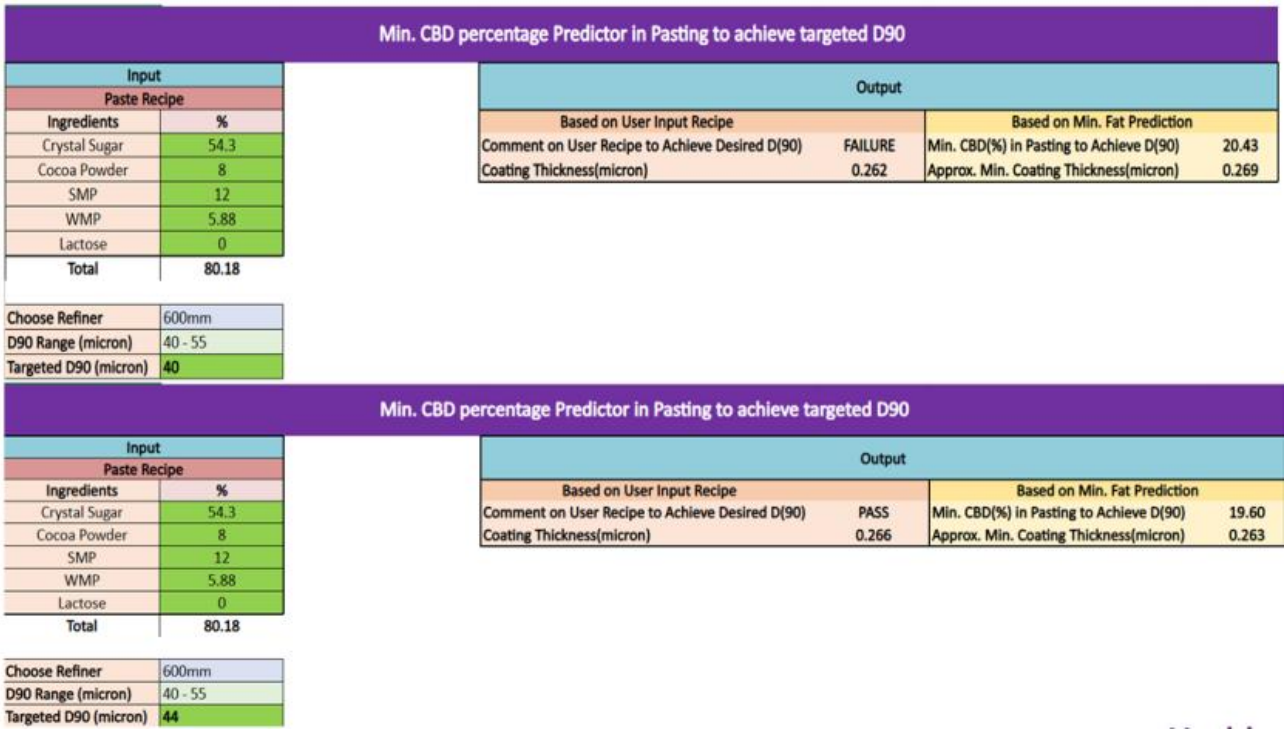

Fig 4.5.2: User interface of the proposed tool

4.6 Future recommendation:

i. To expand the working zone and to reduce the error more data of volume distribution of corresponding Dv90 obtained from 600mm refiner of the same ingredient recipe should be added in the data pool from Malvern detailed report.
ii. More DoE should be designed in this way and need to run on the same logic to generate more data for regression to get more accurate result.
iii. To validate this model multiple trial should be taken and need to give input to the tool to get the results. Simultaneously these data can be used for regression to make it more robust.
iv. RMC & LMC sample should be analyzed under scanning electron microscopy to correlate the report with the obtained coating thickness.
v. Same model can be established for 300 mm refiner.

5. CONCLUSION:

These whole study gives a very clear view of the mechanism of phase changes happens in chocolate that is from powdery (RMC) to liquid (LMC) and explains this phase change with the help of fat coating thickness at the particle level.

The base of this project was PSD and fat distribution. Deconvolution and the concept of Dv99 triggered me to work with particles and hence Number of particle per kg of mass had a huge impact in this project. The procedure of number of particle calculation, the effect of ingredient on number particle with relevance to the true density was explained. How number of particles varies with Dv90 that was also discussed.

Under this project an excel calculator was developed which is able to determine the fat coating thickness on each particle in micron and hence able to capture the phase transition in terms of changes in fat coating thickness from RMC to LMC.

Upgraded or modified version of this calculator is the predictor tool which is able to find out the minimum CBD percentage to achieve the desired Dv90 on a given 600mm refiner and it also shows the corresponding fat coating thickness on each particle. The validation and betterment process of this tool is also mentioned in this report. This is first statistical tool for the prediction of minimum fat required in pasting to achieve user desired Dv90 along with corresponding fat coating thickness on each particle.

APPENDIX 1: Percentage Volume Distribution Data

Phase 1 RMC 30.406 micron

Diameter (micron)	% Vol Dis
0.675	0.12
0.767	0.25
0.872	0.44
0.991	0.7
1.125	1.03
1.279	1.42
1.453	1.85
1.651	2.31
1.875	2.76
2.131	3.18
2.421	3.54
2.75	3.83
3.125	4.04
3.55	4.16
4.034	4.22
4.583	4.21
5.207	4.15
5.916	4.03
6.722	3.88
7.637	3.72
8.677	3.58
9.858	3.5
11.201	3.5
12.726	3.57
14.458	3.69
16.427	3.83
18.664	3.93
21.205	3.93
24.092	3.78
27.373	3.46
31.1	2.98
35.335	2.39
40.146	1.77
45.613	1.17
51.823	0.67
58.88	0.31
66.897	0.09

Phase 1 LMC 29.164 micron

Dia(micron)	% Vol
0.594	0.07
0.675	0.18
0.767	0.34
0.872	0.57
0.991	0.87
1.125	1.23
1.279	1.65
1.453	2.09
1.651	2.55
1.875	2.99
2.131	3.39
2.421	3.72
2.75	3.98
3.125	4.15
3.55	4.25
4.034	4.26
4.583	4.22
5.207	4.13
5.916	4.01
6.722	3.88
7.637	3.76
8.677	3.66
9.858	3.58
11.201	3.55
12.726	3.56
14.458	3.6
16.427	3.63
18.664	3.64
21.205	3.57
24.092	3.39
27.373	3.08
31.1	2.65
35.335	2.14
40.146	1.59
45.613	1.07
51.823	0.62
58.88	0.29
66.897	0.09

STD RMC 40.124 micron

Diameter (micron)	% Vol Dis
0.675	0.13
0.767	0.27
0.872	0.45
0.991	0.7
1.125	1
1.279	1.36
1.453	1.75
1.651	2.17
1.875	2.58
2.131	2.96
2.421	3.31
2.75	3.59
3.125	3.81
3.55	3.95
4.034	4.02
4.583	4.03
5.207	3.98
5.916	3.88
6.722	3.76
7.637	3.6
8.677	3.44
9.858	3.29
11.201	3.16
12.726	3.08
14.458	3.05
16.427	3.08
18.664	3.16
21.205	3.26
24.092	3.35
27.373	3.38
31.1	3.31
35.335	3.12
40.146	2.8
45.613	2.38
51.823	1.88
58.88	1.36
66.897	0.88
76.006	0.48
86.355	0.2

STD RMC 42.86 micron

Diameter (micron)	% Vol Dis
0.675	0.13
0.767	0.26
0.872	0.44
0.991	0.68
1.125	0.97
1.279	1.32
1.453	1.7
1.651	2.11
1.875	2.51
2.131	2.89
2.421	3.23
2.75	3.51
3.125	3.73
3.55	3.87
4.034	3.94
4.583	3.95
5.207	3.91
5.916	3.82
6.722	3.7
7.637	3.55
8.677	3.4
9.858	3.25
11.201	3.11
12.726	3.02
14.458	2.98
16.427	2.99
18.664	3.07
21.205	3.18
24.092	3.29
27.373	3.36
31.1	3.36
35.335	3.24
40.146	2.99
45.613	2.62
51.823	2.15
58.88	1.63
66.897	1.12
76.006	0.66
86.355	0.31
98.114	0.04

STD RMC 48.35 micron

Diameter (micron)	% Vol Dis
0.675	0.11
0.767	0.23
0.872	0.4
0.991	0.62
1.125	0.91
1.279	1.24
1.453	1.61
1.651	2.01
1.875	2.4
2.131	2.78
2.421	3.12
2.75	3.41
3.125	3.63
3.55	3.78
4.034	3.87
4.583	3.89
5.207	3.85
5.916	3.77
6.722	3.66
7.637	3.52
8.677	3.36
9.858	3.2
11.201	3.05
12.726	2.92
14.458	2.83
16.427	2.81
18.664	2.84
21.205	2.93
24.092	3.05
27.373	3.17
31.1	3.26
35.335	3.27
40.146	3.17
45.613	2.94
51.823	2.58
58.88	2.11
66.897	1.6
76.006	1.09
86.355	0.64
98.114	0.3
111.473	0.09

STD RMC 54.096 micron

Diameter (micron)	% Vol Dis
0.675	0.12
0.767	0.25
0.872	0.42
0.991	0.65
1.125	0.94
1.279	1.27
1.453	1.63
1.651	2.01
1.875	2.38
2.131	2.73
2.421	3.04
2.75	3.29
3.125	3.49
3.55	3.62
4.034	3.69
4.583	3.7
5.207	3.67
5.916	3.6
6.722	3.5
7.637	3.38
8.677	3.25
9.858	3.11
11.201	2.97
12.726	2.85
14.458	2.76
16.427	2.71
18.664	2.7
21.205	2.76
24.092	2.85
27.373	2.98
31.1	3.11
35.335	3.21
40.146	3.24
45.613	3.15
51.823	2.94
58.88	2.58
66.897	2.11
76.006	1.57
86.355	1.02
98.114	0.55
111.473	0.21

P2 RMC 47.38 micron

Diameter (micron)	% Vol Dis
0.675	0.13
0.767	0.26
0.872	0.45
0.991	0.7
1.125	1.01
1.279	1.37
1.453	1.77
1.651	2.19
1.875	2.6
2.131	2.98
2.421	3.31
2.75	3.58
3.125	3.77
3.55	3.88
4.034	3.93
4.583	3.91
5.207	3.85
5.916	3.76
6.722	3.63
7.637	3.49
8.677	3.33
9.858	3.16
11.201	2.99
12.726	2.85
14.458	2.75
16.427	2.7
18.664	2.71
21.205	2.79
24.092	2.9
27.373	3.03
31.1	3.14
35.335	3.17
40.146	3.09
45.613	2.87
51.823	2.52
58.88	2.06
66.897	1.54
76.006	1.02
86.355	0.57
98.114	0.24
111.473	0.04

P2 RMC 48.024 micron

Diameter (micron)	% Vol Dis
0.675	0.12
0.767	0.24
0.872	0.41
0.991	0.64
1.125	0.94
1.279	1.28
1.453	1.66
1.651	2.07
1.875	2.49
2.131	2.88
2.421	3.24
2.75	3.53
3.125	3.76
3.55	3.91
4.034	3.99
4.583	3.99
5.207	3.94
5.916	3.84
6.722	3.71
7.637	3.55
8.677	3.38
9.858	3.21
11.201	3.04
12.726	2.89
14.458	2.78
16.427	2.73
18.664	2.72
21.205	2.78
24.092	2.88
27.373	2.99
31.1	3.08
35.335	3.11
40.146	3.05
45.613	2.86
51.823	2.54
58.88	2.11
66.897	1.61
76.006	1.09
86.355	0.62
98.114	0.27
111.473	0.04

P4 RMC 44.66 micron

Diameter (micron)	% Vol Dis
0.675	0.13
0.767	0.26
0.872	0.44
0.991	0.67
1.125	0.97
1.279	1.31
1.453	1.7
1.651	2.1
1.875	2.51
2.131	2.9
2.421	3.24
2.75	3.53
3.125	3.75
3.55	3.9
4.034	3.98
4.583	3.99
5.207	3.96
5.916	3.88
6.722	3.76
7.637	3.61
8.677	3.44
9.858	3.25
11.201	3.07
12.726	2.92
14.458	2.82
16.427	2.78
18.664	2.82
21.205	2.92
24.092	3.06
27.373	3.19
31.1	3.28
35.335	3.27
40.146	3.12
45.613	2.82
51.823	2.38
58.88	1.85
66.897	1.28
76.006	0.76
86.355	0.35
98.114	0.05

P4 RMC 45.351 micron

Diameter (micron)	% Vol Dis
0.675	0.13
0.767	0.25
0.872	0.43
0.991	0.66
1.125	0.95
1.279	1.29
1.453	1.67
1.651	2.08
1.875	2.49
2.131	2.88
2.421	3.24
2.75	3.54
3.125	3.77
3.55	3.93
4.034	4.01
4.583	4.03
5.207	3.99
5.916	3.9
6.722	3.77
7.637	3.61
8.677	3.42
9.858	3.22
11.201	3.03
12.726	2.87
14.458	2.76
16.427	2.73
18.664	2.76
21.205	2.87
24.092	3.01
27.373	3.16
31.1	3.25
35.335	3.24
40.146	3.11
45.613	2.83
51.823	2.41
58.88	1.9
66.897	1.36
76.006	0.85
86.355	0.44
98.114	0.17

P4 RMC 46.022 micron

Diameter (micron)	% Vol Dis
0.675	0.13
0.767	0.26
0.872	0.44
0.991	0.68
1.125	0.98
1.279	1.32
1.453	1.69
1.651	2.09
1.875	2.48
2.131	2.85
2.421	3.18
2.75	3.45
3.125	3.66
3.55	3.81
4.034	3.89
4.583	3.91
5.207	3.88
5.916	3.81
6.722	3.71
7.637	3.58
8.677	3.42
9.858	3.24
11.201	3.07
12.726	2.92
14.458	2.81
16.427	2.77
18.664	2.79
21.205	2.89
24.092	3.03
27.373	3.18
31.1	3.3
35.335	3.33
40.146	3.23
45.613	2.96
51.823	2.54
58.88	2
66.897	1.41
76.006	0.85
86.355	0.39
98.114	0.05

P8 RMC 53.75 micron

Diameter (micron)	% Vol Dis
0.675	0.14
0.767	0.27
0.872	0.46
0.991	0.71
1.125	1.02
1.279	1.37
1.453	1.77
1.651	2.18
1.875	2.59
2.131	2.98
2.421	3.32
2.75	3.6
3.125	3.81
3.55	3.94
4.034	4
4.583	4
5.207	3.95
5.916	3.86
6.722	3.74
7.637	3.58
8.677	3.4
9.858	3.2
11.201	3
12.726	2.81
14.458	2.65
16.427	2.53
18.664	2.45
21.205	2.43
24.092	2.45
27.373	2.51
31.1	2.58
35.335	2.65
40.146	2.69
45.613	2.67
51.823	2.56
58.88	2.33
66.897	2
76.006	1.58
86.355	1.12
98.114	0.68
111.473	0.32
126.652	0.07

P8 RMC 55.75 micron

Diameter (micron)	% Vol Dis
0.675	0.12
0.767	0.24
0.872	0.41
0.991	0.65
1.125	0.94
1.279	1.29
1.453	1.68
1.651	2.11
1.875	2.55
2.131	2.97
2.421	3.36
2.75	3.69
3.125	3.94
3.55	4.11
4.034	4.18
4.583	4.17
5.207	4.09
5.916	3.95
6.722	3.77
7.637	3.56
8.677	3.34
9.858	3.11
11.201	2.89
12.726	2.7
14.458	2.53
16.427	2.42
18.664	2.34
21.205	2.32
24.092	2.34
27.373	2.4
31.1	2.48
35.335	2.57
40.146	2.64
45.613	2.66
51.823	2.59
58.88	2.41
66.897	2.12
76.006	1.72
86.355	1.26
98.114	0.81
111.473	0.42
126.652	0.15

P8 RMC 55.99 micron

Diameter (micron)	% Vol Dis
0.675	0.13
0.767	0.25
0.872	0.42
0.991	0.64
1.125	0.91
1.279	1.24
1.453	1.6
1.651	1.98
1.875	2.37
2.131	2.75
2.421	3.09
2.75	3.38
3.125	3.61
3.55	3.77
4.034	3.86
4.583	3.88
5.207	3.85
5.916	3.77
6.722	3.65
7.637	3.51
8.677	3.34
9.858	3.16
11.201	2.99
12.726	2.83
14.458	2.7
16.427	2.61
18.664	2.56
21.205	2.56
24.092	2.61
27.373	2.69
31.1	2.79
35.335	2.89
40.146	2.96
45.613	2.95
51.823	2.84
58.88	2.6
66.897	2.23
76.006	1.75
86.355	1.22
98.114	0.71
111.473	0.31
126.652	0.06

APPENDIX 2: Excel Calculator

Diameter (micron)	% Vol Dis	Volume Distribution of Each Solid (%)				Absolute Volume (m3)				No. of Particle				Fat Coated Dia (micron)	Fat Coated Vo (m3)	core volu (m3)	Annular Vol (m3)	Volume Of Fat (m3)			
		Sugar	ocoa sol	Lactose	Protein	Sugar	Cocoa solid	Lactose	Protein	Sugar	Cocoa Solic	Lactose	Protein					Sugar	ocoa soli	Lactose	Protein
0.675	0.13	0.0899	0.0099	0.0235	0.0067	4.553E-07	4.993E-08	1.192E-07	3.367E-08	2.83E+12	3.10E+11	7.41E+11	2.09E+11	1.185	8.70275E-19	1.61E-19	7.09428E-19	2.008E-06	2.202E-07	5.257E-07	1.485E-07
0.767	0.27	0.1868	0.0205	0.0489	0.0138	9.456E-07	1.037E-07	2.475E-07	6.993E-08	4.01E+12	4.39E+11	1.05E+12	2.96E+11	1.277	1.08912E-18	2.36E-19	8.5313E-19	3.419E-06	3.749E-07	8.949E-07	2.528E-07
0.872	0.45	0.3113	0.0341	0.0815	0.0230	1.576E-06	1.728E-07	4.126E-07	1.165E-07	4.54E+12	4.98E+11	1.19E+12	3.36E+11	1.382	1.38047E-18	3.47E-19	1.03369E-18	4.698E-06	5.152E-07	1.230E-06	3.474E-07
0.991	0.7	0.4843	0.0531	0.1268	0.0358	2.452E-06	2.689E-07	6.418E-07	1.813E-07	4.82E+12	5.28E+11	1.26E+12	3.56E+11	1.501	1.76866E-18	5.09E-19	1.25965E-18	6.067E-06	6.654E-07	1.588E-06	4.486E-07
1.125	1	0.6919	0.0759	0.1811	0.0512	3.502E-06	3.841E-07	9.168E-07	2.590E-07	4.70E+12	5.16E+11	1.23E+12	3.48E+11	1.635	2.28589E-18	7.45E-19	1.54123E-18	7.249E-06	7.949E-07	1.898E-06	5.360E-07
1.279	1.36	0.9409	0.1032	0.2463	0.0696	4.763E-06	5.224E-07	1.247E-06	3.522E-07	4.35E+12	4.77E+11	1.14E+12	3.22E+11	1.789	2.99456E-18	1.09E-18	1.90032E-18	8.272E-06	9.072E-07	2.165E-06	6.117E-07
1.453	1.75	1.2108	0.1328	0.3169	0.0895	6.129E-06	6.722E-07	1.604E-06	4.532E-07	3.82E+12	4.19E+11	1.00E+12	2.82E+11	1.963	3.95606E-18	1.6E-18	2.35171E-18	8.984E-06	9.853E-07	2.352E-06	6.644E-07
1.651	2.17	1.5013	0.1646	0.3930	0.1110	7.600E-06	8.335E-07	1.990E-06	5.620E-07	3.23E+12	3.54E+11	8.45E+11	2.39E+11	2.161	5.27796E-18	2.35E-18	2.9243E-18	9.443E-06	1.036E-06	2.472E-06	6.983E-07
1.875	2.58	1.7850	0.1958	0.4673	0.1320	9.036E-06	9.909E-07	2.365E-06	6.682E-07	2.62E+12	2.87E+11	6.86E+11	1.94E+11	2.385	7.09524E-18	3.45E-18	3.64773E-18	9.561E-06	1.048E-06	2.503E-06	7.070E-07
2.131	2.96	2.0479	0.2246	0.5361	0.1514	1.037E-05	1.137E-06	2.714E-06	7.666E-07	2.05E+12	2.25E+11	5.36E+11	1.51E+11	2.641	9.63401E-18	5.06E-18	4.57282E-18	9.367E-06	1.027E-06	2.452E-06	6.926E-07
2.421	3.31	2.2901	0.2511	0.5995	0.1693	1.159E-05	1.271E-06	3.035E-06	8.572E-07	1.56E+12	1.71E+11	4.09E+11	1.16E+11	2.931	1.31689E-17	7.42E-18	5.74749E-18	8.978E-06	9.846E-07	2.350E-06	6.639E-07
2.75	3.59	2.4838	0.2724	0.6502	0.1837	1.257E-05	1.379E-06	3.291E-06	9.298E-07	1.16E+12	1.27E+11	3.03E+11	8.55E+10	3.26	1.81198E-17	1.09E-17	7.24308E-18	8.373E-06	9.182E-07	2.192E-06	6.191E-07
3.125	3.81	2.6360	0.2891	0.6900	0.1949	1.334E-05	1.463E-06	3.493E-06	9.867E-07	8.36E+11	9.17E+10	2.19E+11	6.18E+10	3.635	2.51197E-17	1.6E-17	9.15903E-18	7.658E-06	8.398E-07	2.005E-06	5.662E-07
3.55	3.95	2.7328	0.2997	0.7154	0.2021	1.383E-05	1.517E-06	3.621E-06	1.023E-06	5.91E+11	6.48E+10	1.55E+11	4.37E+10	4.06	3.50009E-17	2.34E-17	1.16025E-17	6.860E-06	7.523E-07	1.796E-06	5.073E-07
4.034	4.02	2.7813	0.3050	0.7281	0.2057	1.408E-05	1.544E-06	3.688E-06	1.041E-06	4.10E+11	4.50E+10	1.07E+11	3.03E+10	4.544	4.90701E-17	3.43E-17	1.47373E-17	6.044E-06	6.628E-07	1.582E-06	4.469E-07
4.583	4.03	2.7882	0.3058	0.7299	0.2062	1.411E-05	1.548E-06	3.695E-06	1.044E-06	2.80E+11	3.07E+10	7.34E+10	2.07E+10	5.093	6.90912E-17	5.03E-17	1.87488E-17	5.256E-06	5.764E-07	1.376E-06	3.886E-07
5.207	3.98	2.7536	0.3020	0.7208	0.2036	1.394E-05	1.529E-06	3.649E-06	1.031E-06	1.89E+11	2.07E+10	4.94E+10	1.40E+10	5.717	9.77251E-17	7.38E-17	2.38898E-17	4.510E-06	4.946E-07	1.181E-06	3.335E-07
5.916	3.88	2.6844	0.2944	0.7027	0.1985	1.359E-05	1.490E-06	3.557E-06	1.005E-06	1.25E+11	1.38E+10	3.28E+10	9.28E+09	6.426	1.38779E-16	1.08E-16	3.04896E-17	3.826E-06	4.196E-07	1.002E-06	2.829E-07
6.722	3.76	2.6014	0.2853	0.6810	0.1924	1.317E-05	1.444E-06	3.447E-06	9.738E-07	8.29E+10	9.09E+09	2.17E+10	6.13E+09	7.232	1.97823E-16	1.59E-16	3.89694E-17	3.231E-06	3.543E-07	8.457E-07	2.389E-07
7.637	3.6	2.4907	0.2731	0.6520	0.1842	1.261E-05	1.383E-06	3.301E-06	9.323E-07	5.41E+10	5.94E+09	1.42E+10	4.00E+09	8.147	2.8281E-16	2.33E-16	4.98561E-17	2.698E-06	2.959E-07	7.064E-07	1.995E-07
8.677	3.44	2.3800	0.2610	0.6230	0.1760	1.205E-05	1.321E-06	3.154E-06	8.909E-07	3.53E+10	3.87E+09	9.23E+09	2.61E+09	9.187	4.0553E-16	3.42E-16	6.3857E-17	2.252E-06	2.469E-07	5.894E-07	1.665E-07
9.858	3.29	2.2762	0.2496	0.5958	0.1683	1.152E-05	1.264E-06	3.016E-06	8.521E-07	2.30E+10	2.52E+09	6.02E+09	1.70E+09	10.368	5.8289E-16	5.01E-16	8.1855E-17	1.883E-06	2.064E-07	4.928E-07	1.392E-07
11.201	3.16	2.1863	0.2398	0.5723	0.1617	1.107E-05	1.214E-06	2.897E-06	8.184E-07	1.51E+10	1.65E+09	3.94E+09	1.11E+09	11.711	8.40008E-16	7.35E-16	1.05034E-16	1.582E-06	1.735E-07	4.140E-07	1.170E-07
12.726	3.08	2.1309	0.2337	0.5578	0.1576	1.079E-05	1.183E-06	2.824E-06	7.977E-07	1.00E+10	1.10E+09	2.62E+09	7.40E+08	13.236	1.21275E-15	1.08E-15	1.34854E-16	1.350E-06	1.480E-07	3.533E-07	9.980E-08
14.458	3.05	2.1102	0.2314	0.5524	0.1560	1.068E-05	1.171E-06	2.796E-06	7.899E-07	6.76E+09	7.41E+08	1.77E+09	5.00E+08	14.968	1.75385E-15	1.58E-15	1.73236E-16	1.171E-06	1.284E-07	3.065E-07	8.657E-08
16.427	3.08	2.1309	0.2337	0.5578	0.1576	1.079E-05	1.183E-06	2.824E-06	7.977E-07	4.65E+09	5.10E+08	1.22E+09	3.44E+08	16.937	2.54104E-15	2.32E-15	2.22702E-16	1.036E-06	1.136E-07	2.713E-07	7.663E-08
18.664	3.16	2.1863	0.2398	0.5723	0.1617	1.107E-05	1.214E-06	2.897E-06	8.184E-07	3.25E+09	3.57E+08	8.52E+08	2.41E+08	19.174	3.68672E-15	3.4E-15	2.86428E-16	9.323E-07	1.022E-07	2.440E-07	6.894E-08
21.205	3.26	2.2555	0.2473	0.5904	0.1668	1.142E-05	1.252E-06	2.989E-06	8.443E-07	2.29E+09	2.51E+08	5.99E+08	1.69E+08	21.715	5.35527E-15	4.99E-15	3.6853E-16	8.438E-07	9.253E-08	2.209E-07	6.239E-08
24.092	3.35	2.3177	0.2542	0.6067	0.1714	1.173E-05	1.287E-06	3.071E-06	8.676E-07	1.60E+09	1.76E+08	4.20E+08	1.19E+08	24.602	7.78777E-15	7.31E-15	4.74351E-16	7.610E-07	8.346E-08	1.992E-07	5.627E-08
27.373	3.38	2.3385	0.2564	0.6121	0.1729	1.184E-05	1.298E-06	3.099E-06	8.754E-07	1.10E+09	1.21E+08	2.89E+08	8.16E+07	27.883	1.13376E-14	1.07E-14	6.10807E-16	6.741E-07	7.392E-08	1.765E-07	4.985E-08
31.1	3.31	2.2901	0.2511	0.5995	0.1693	1.159E-05	1.271E-06	3.035E-06	8.572E-07	7.37E+08	8.08E+07	1.93E+08	5.45E+07	31.61	1.65187E-14	1.57E-14	7.86713E-16	5.797E-07	6.358E-08	1.518E-07	4.287E-08
35.335	3.12	2.1586	0.2367	0.5651	0.1596	1.093E-05	1.198E-06	2.860E-06	8.080E-07	4.74E+08	5.19E+07	1.24E+08	3.50E+07	35.845	2.40873E-14	2.31E-14	1.01358E-15	4.800E-07	5.264E-08	1.257E-07	3.550E-08
40.146	2.8	1.9372	0.2124	0.5071	0.1432	9.807E-06	1.075E-06	2.567E-06	7.252E-07	2.90E+08	3.18E+07	7.59E+07	2.14E+07	40.656	3.5146E-14	3.38E-14	1.30612E-15	3.785E-07	4.151E-08	9.908E-08	2.799E-08
45.613	2.38	1.6466	0.1806	0.4310	0.1218	8.336E-06	9.141E-07	2.182E-06	6.164E-07	1.68E+08	1.84E+07	4.40E+07	1.24E+07	46.123	5.13162E-14	4.96E-14	1.68352E-15	2.827E-07	3.101E-08	7.401E-08	2.091E-08
51.823	1.88	1.3007	0.1426	0.3405	0.0962	6.585E-06	7.221E-07	1.724E-06	4.869E-07	9.05E+07	9.92E+06	2.37E+07	6.69E+06	52.333	7.49598E-14	7.28E-14	2.17023E-15	1.963E-07	2.153E-08	5.139E-08	1.452E-08
58.88	1.36	0.9409	0.1032	0.2463	0.0696	4.763E-06	5.224E-07	1.247E-06	3.522E-07	4.46E+07	4.89E+06	1.17E+07	3.30E+06	59.39	1.09557E-13	1.07E-13	2.79824E-15	1.248E-07	1.369E-08	3.268E-08	9.232E-09
66.897	0.88	0.6088	0.0668	0.1594	0.0450	3.082E-06	3.380E-07	8.068E-07	2.279E-07	1.97E+07	2.16E+06	5.15E+06	1.46E+06	67.407	1.60183E-13	1.57E-13	3.60839E-15	7.103E-08	7.789E-09	1.859E-08	5.252E-09
76.006	0.48	0.3321	0.0364	0.0869	0.0246	1.681E-06	1.844E-07	4.401E-07	1.243E-07	7.32E+06	8.03E+05	1.92E+06	5.41E+05	76.516	2.34293E-13	2.3E-13	4.65371E-15	3.407E-08	3.736E-09	8.918E-09	2.519E-09
86.355	0.2	0.1384	0.0152	0.0362	0.0102	7.005E-07	7.682E-08	1.834E-07	5.180E-08	2.08E+06	2.28E+05	5.44E+05	1.54E+05	86.865	3.42796E-13	3.37E-13	6.00248E-15	1.248E-08	1.369E-09	3.268E-09	9.231E-10
98.114	0	0.0000	0.0000	0.0000	0.0000	0.000E+00	0.000E+00	0.000E+00	0.000E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	98.624	5.01706E-13	4.94E-13	7.74302E-15	0.000E+00	0.000E+00	0.000E+00	0.000E+00
111.473	0	0.0000	0.0000	0.0000	0.0000	0.000E+00	0.000E+00	0.000E+00	0.000E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	111.983	7.34443E-13	7.24E-13	9.88891E-15	0.000E+00	0.000E+00	0.000E+00	0.000E+00
Total		69.1583	7.5842	18.1036	5.1139	3.501E-04	3.839E-05	9.165E-05	2.589E-05	4.24E+13	4.65E+12	1.11E+13	3.13E+12					1.411E-04	######	3.695E-05	######

Overall Recipe		Paste Recipe		1
Ingredient	%	Ingredient	%	kg
Crystal Sugar	50.5	Crystal Sugar	54.30	0.54
Cocoa Powde	6.5	Cocoa Powde	7.00	0.07
SMP	11	SMP	11.80	0.12
WMP	0	WMP	0.00	0.00
Lactose	6.5	Lactose	7.00	0.07
CBD	22.28	CBD	19.90	0.20
AMF	2.5	AMF	0	0.00
YN	0.52	YN	0	0.00
PGPR	0.2	PGPR	0	0.00
		Total	100.0	1.00

Conch Recipe		5
Ingredients	%	kg
RMC	93	4.65
CBD	3.80	0.190
AMF	2.5	0.125
YN	0.52	0.026
PGPR	0.2	0.01
Total	100.0	5.00
Coating Thickn	0.255	micron

Fat in RMC	kg	ensity (kg/m3)
CBD	0.199	974.000
Fat from SMP	0.001	920.000
Fat from Cocoa	0.008	974.000
Total	0.208	

6.1236E+13

Total Fat in LI 0.558

Total Fat in	0.000204009
Total Fat in	0.198704301
% Free Fat	19.87043013

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