

**SYNTHESIS OF VARIOUS VALUE-ADDED
PRODUCTS USING WASTE BIOMASS AND
THEIR APPLICATION IN ENERGY AND
ENVIRONMENT**

Thesis submitted by

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List of Publications:

1. **Ghosh, S.**, Basak, D., Manna, S., Bhowal, A., & Das, P. (2025). Removal of naphthalene utilizing synthesized silica doped PVA aerogel: removal, optimization and mechanism. *Discover Chemistry*, 2(1), 82.
2. Samanta, A., **Ghosh, S.** & Das, P. (2025). Extraction of silica and biochar from biomass waste for the synthesis of aerogel and its application for the removal of dye. *Biomass Conv. Bioref.* <https://doi.org/10.1007/s13399-025-06776-2>.
3. **Ghosh, S.**, Roy, S. & Das, P. Beyond waste: waste rice husk to value-added products using sonic waves and chemical treatment and principal component analysis of extraction. *Biomass Conv. Bioref.* (2024). <https://doi.org/10.1007/s13399-024-05855-0>.
4. **Ghosh, S.**, Chakraborty, P., Bhowal, A. *et al.* Synthesis of polymeric aerogels with different fillers and their application for the removal of emerging pollutants: a comparative study. *Biomass Conv. Bioref.* (2024). <https://doi.org/10.1007/s13399-024-05715-x>.
5. **Ghosh, S.**, Sarkar, S., Mukherjee, S. *et al.* Silica-coated cellulose using *Shorea robusta* sawdust biomass and its application for methylene blue dye removal from aqueous solution. *Biomass Conv. Bioref.* (2024). <https://doi.org/10.1007/s13399-024-05616-z>.
6. Roy, S., Mukherjee, S., **Ghosh, S.**, & Das, P. (2024). Synthesis of rice husk-derived cellulose for efficacious removal of malachite green from aqueous solution. *Sādhanā*, 49(1), 15.

List of Patents:

1. A method of preparation of a microbe-doped polymeric aerogel for wastewater treatment (KB ID: KBP54). (Filed)

List of Conferences:

1. **International Conference on Energy Transition: Challenges and Opportunities (IChE-Chemcon- 2023).**

Organized by: Indian Institute of Chemical Engineers.

Venue: Heritage Institute of Technology, Kolkata.

Topic: Synthesis of silane-treated aerogel for the treatment of naphthalene-containing solution.

Date: 27-30 December 2023.

2. 3rd International Conference on Water Technologies (ICWT- 2023).

Organized by: Water Innovation Center: Technology, Research & Education in association with Asian Universities Alliance.

Venue: Indian Institute of Technology Bombay.

Topic: Bacterial Bioremediation of Acenaphthene and Phenol using Isolated Bacteria: A Promising Technology for Water Treatment.

Date: 04-07 December 2023.

3. International Conference on Chemical Engineering Innovation and Sustainability (ICEIS- 2023).

Organized by: Department of Chemical Engineering, Jadavpur University, Kolkata.

Venue: Jadavpur University, Kolkata.

Topic: Cellulose from acid pre-treated lignocellulosic biomass and its application in dye removal.

Date: 26-27 February 2023.

4. International Conference on Sustainable Resilient Remediation (ICSRR- 2023).

Organized by: Centre for Environmental Studies (CES), Anna University, Chennai.

Venue: Anna University (Online).

Topic: Production of Biochar from Lignocellulosic Biomass and Its Application in Dye Removal.

Date: 02-03 February 2023.

5. Himalaya Calling: Global Summit on Challenges & Opportunities in the Himalayan Region.

Organized by: University of Petroleum and Energy Sciences (UPES), Dehradun.

Venue: UPES (Online).

Topic: Preparation of silica-doped polymeric aerogel and its' application for Phenol removal from wastewater.

Date: 10 September 2024.

6. 39th Indian Engineering Congress

Organized by: The Institution of Engineers (India)

Venue: Novotel, Kolkata

Topic: Preparation of Activated Biochar doped Polyvinyl Alcohol (PVA) Aerogel in Wastewater Treatment.

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SYNOPSIS

1. Introduction:

With the development of civilisation, industrialisation is increasing rapidly. Especially in developing countries, industrialisation and urbanisation are reaching their peak as these nations serve both as a cheap production hub and a large market. Due to the establishment of various industries, waste is also being generated and discarded exponentially. Organic pollutants, like polycyclic aromatic hydrocarbons (PAHs) from petrochemical industries, dyes from textile industries, heavy metals from tannery industries, and solid biotic wastes from agricultural sectors, hospitals, and residential places are generating more concerns nowadays. These pollutants are carcinogenic and mutagenic in nature, cause several diseases, and accumulate in biotic organisms via processes like biomagnification and bioaccumulation. As most of the time these materials are being discarded into the environment without any proper treatment, soil, water, and air are being contaminated and producing phenomena like acid rain. On the other hand, the biotic wastes are causing problems in landfilling sectors, by occupying a massive space and leaching through the soil layers, contaminating groundwater. Though policies and technologies exist to manage these issues, they have their drawbacks.

Only rules and regulations for disposing of waste safely are not enough to cure the environmental damage. As the human population is increasing, another problem society is facing is a resource shortage. So, rather than using a material, generating waste, and treating the waste before disposal, in the age of circular economy, the recycling and reusing waste materials of one sector as the raw material in another industry is a more accepted approach. Solid biotic wastes, like rice husk and sawdust, are rich with usable components, viz., cellulose, hemicellulose and lignin that can be extracted by various treatment methods.

Also, the treatment of wastewater includes technologies, viz., membrane filtration, adsorption, advanced oxidation, and biological treatment, that are being studied in a detailed manner. Though the main focus is gained by adsorption, because of its easy setup, cost effectiveness, and convenient applications on a large scale. The main problem associated with adsorption is the desorption of pollutants after the equilibrium, and the disposal of used adsorbents. To mitigate these problems, the reusability of different kinds of adsorbents is being investigated.

In the current study, the extraction of cellulose, hemicellulose, and lignin was investigated by the sonochemical method from rice husk and sawdust, and the synthesis of furfural and bioethanol was conducted. Also, silica and biochar were produced from the raw materials, and the biochar, cellulose, and silica were utilised to synthesise PVA aerogels for wastewater treatment. Bacteria were also doped into the PVA matrix of the aerogels to increase their efficiency, along with phytotoxicity studies were conducted to understand the environmental effects of the treated solutions.

2. Methods of experiment:

To extract cellulose, hemicellulose, and lignin, sodium hydroxide (NaOH), and sulphuric acid (H₂SO₄) solutions were used in three different concentrations, viz., 2%, 4%, and 6%. The raw materials were treated with the solutions for 2h of a magnetic stirrer, followed by sonication. After treatment, the solid residue was collected, and an anthrone test was performed to estimate the cellulose. On the other hand, the liquid hydrolysate was mixed with absolute ethanol in a 1:1 (v/v) ratio to precipitate out hemicellulose. After collecting the hemicellulose by filtration, the hydrolysate was again treated with concentrated hydrochloric acid (HCl) to precipitate out lignin. The lignin was collected by filtration, in the next step, and the produced xylan content in the hydrolysate was analysed by UV-VIS spectrophotometer at 275 nm. The extracted hemicellulose and lignin were dried in a hot air oven and collected.

On the other hand, after estimating the cellulose content by the anthrone test, the treated biomass with the highest cellulose yield was selected for saccharification, a process to break down cellulose fibre to simple sugar molecules. From this process, *Aspergillus sp.* was used as the source of cellulase, an enzyme to breaks down cellulose fibre. Different parameters, viz., inoculation time (5 days- 10 days), pH of the medium (4-10), temperature (298 K- 313 K), microbial dose (1 mL/100 mL- 3 mL/100 mL), and substrate concentration (1 g/100 mL -3 g/100 mL) were varied to study their effect on the reducing sugar production. 3,5-Dinitrosalicylic acid was used to estimate the reducing sugar content and was measured by a UV-VIS spectrophotometer at 540 nm. The saccharified media was then inoculated with *Saccharomyces Cerevisiae* to produce bioethanol from the sugar by fermentation. Different parameters, viz., inoculation time (5 days- 10 days), pH of the medium (4-10), temperature (298 K- 313 K), microbial dose (1 mL/100 mL- 3 mL/100 mL), and substrate concentration (1 g/100 mL -3 g/100 mL) were varied to study

their effect on the ethanol production. The produced ethanol was estimated by the dichromate method. To produce furfural from the extracted cellulose, the cellulose was treated with an H₂SO₄ solution under high pressure, using a hydrothermal reactor and was detected by an HPLC machine.

Silica and biochar were produced from the raw materials by conventional methods. To produce biochar, the materials were pyrolysed at 600°C and for silica acid precipitation method was followed.

The cellulose, biochar, and silica were used as filler materials of the aerogels. To produce PVA aerogels, the sol-gel method was used. Glutaraldehyde was used as the gelation agent, and HCl was used as the catalyst. After dissolving PVA in water at 90°C, the crosslinker and catalyst were added to the solution, and the hydrogel was produced. Fillers were added to the network before producing the hydrogel. The hydrogel was dried by lyophilisation and stored in a dry container. To dope bacteria into the aerogel network, the produced aerogels were treated with nutrient broth, inoculated with the bacteria. After five days of treatment, they were again dried using lyophilisation.

Naphthalene, acenaphthene, and phenol were considered as the pollutants of interest in this study. As naphthalene and acenaphthene are insoluble in water, they were dissolved in acetone, and then water was added. The acetone was eliminated from the solution by applying heat. Different parameters, viz., the pollutant concentration (1 mg/L- 10 mg/L), dose of adsorbents (0.5 g/L- 1.5 g/L), pH (2-10), and temperature (298 K- 313 K) were varied to study their effect on the adsorption process. The final concentration of the pollutant was detected by a UV-VIS spectrophotometer and an HPLC machine. The concentration was calculated by formula 1.

$$Removal \% = \frac{C_0 - C_t}{C_0} \times 100 \dots \dots \dots (1)$$

For the modelling of this study, the Langmuir and the Freundlich isotherm models, along with pseudo-1st-order and pseudo-2nd-order kinetic models, and thermodynamics were analysed. A Response Surface Methodology (RSM) study was performed using the Box-Behnken model.

3. Result and conclusion:

In this study, it was observed that sonication has a high influence on the cellulose content extracted from biomass. The highest cellulose content was found in the case of SD_4%_NaOH_S, and the value was 958.88 mg/g, and the lowest cellulose concentration was found to be 101.48 mg/g in the case of RH_2%_NaOH_NS. As sonication broke the chemical bonds present in the biomass, the materials became easily accessible for delignification. Also, as mentioned before, the sonochemical reaction of water produces hydroxyl ions and caused a right shift of reaction equilibrium in NaOH-containing pre-treatment media and a greater yield of cellulose. Like alkali-treated biomass, acid treatment with sonication also showed a higher yield compared to a sample without sonication. Though the cellulose yield of acid-treated sonicated biomass was less than alkali-treated sonicated biomass, which could be explained by the reaction of -OH ions produced during the sonochemical reaction of water, and the proton released from acid, this medium also affected the yield for cellulose production. During sonication, as water molecules broke into hydroxyl ions, they reacted with the proton released by the sulfuric acid present in the media. Unlike alkaline media, this condition caused a left shift of the reaction equilibrium and slowed down the chemical process. In the case of TRS, the highest TRS yield was observed in the case of SD_4%_H₂SO₄_S and was found to be 26.82 mg/g with 3 mL/ 100 mL of microbial concentration. As the microbial concentration increased in the medium, the availability of cellulase also increased, increasing TRS yield. But in higher concentrations, the competition of microorganisms increased, resulting in a decline in the TRS content. In this study, the highest ethanol concentration was found in the case of SD_6%_H₂SO₄_NS with 1 mL microbial dose/100 mL solution, and the ethanol concentration was 3.24 mg/g of biomass. From the RSM study of bioethanol production, it was found that the model was significant with a P-value of 0.0006 (<0.05) and an F-value of 17.1 (>12). The correlation coefficient (R²) value of the mentioned model was 0.95. The R² value also indicated that the predicted and observed data of this experiment were in reasonable agreement, with the value of 4.84 mg/g and 4.32 mg/g, respectively, indicating the significance of the model. In the case of furfural production, RH_4%_NaOH_S generated the highest furfural production yield, with the value of 17.35% after treating for 30 min.

The pollutant removal study was conducted by the aerogels, bacteria, and bacteria-doped aerogels, respectively. In the case of naphthalene, the highest removal of naphthalene was

found in the case of PVA-Si aerogel, at 313 K temperature, with the value of 96.32% removal. For the same aerogel, the removal at 298 K was 79.42%. This phenomenon can be explained by the increase in kinetic energy of the samples. As the kinetic energy increased, the diffusivity of the molecules in the water also increased, causing their increased movement towards the aerogel pores. For phenol and acenaphthene, the highest removal was 97.44% and 94.47%, respectively, by the same aerogel. From the modelling study, it was found that the processes were endothermic and spontaneous. They were observed to follow the pseudo-2nd-order kinetic model. The isotherm model, though, was found to depend on the adsorbent type. In general, the aerogels containing sawdust materials as filler followed the Langmuir isotherm model; on the other hand, the rest of the aerogels were found to better fit the Freundlich isotherm model, allowing multilayer adsorption. From the multipollutant removal study of the aerogels, it was found that the adsorbents are capable of removing multiple organic pollutants simultaneously from a mixed pollutant solution. It was also observed that the adsorbents have a higher affinity to organic pollutants and dyes compared to metal ions, as found in the selectivity study.

In the case of the bacteria, it was shown that *Bacillus sp.* showed an inhibition zone for 100 mg/L naphthalene, and *Lysinibacillus sp.* and *Pseudomonas sp.* showed the zones for 10 mg/L and 100 mg/L naphthalene concentration, confirming the tolerance of these organisms against these pollutants. During the removal of these pollutants, in case of naphthalene, the highest removal was achieved by *Bacillus sp.*, at 2 mL/ 100 mL dose, and the value was 99.52%. Similarly, for phenol, the highest removal achieved in this study was 84.19% by *Pseudomonas sp.* after 5 days of treatment, and for acenaphthene, the highest removal achieved in this study was 74.18% by *Lysinibacillus sp.* after 5 days of treatment.

Bacteria-doped aerogels also showed significant removal of the pollutants. For naphthalene, the highest removal was achieved by PVA-Si-*Bacillus sp.* showed the highest removal with the value of 97.39% after five days of treatment, as well as for phenol and acenaphthene, with the removal of 94.20% and 96.35%. From the GC-MS analysis of the pollutants after treatment, it was found that 2-methyl-eicosane, 3,3-dimethylhexane, and 1-iodo-tridecane were the major produced components, confirming the metabolism of the pollutants. From the toxicity analysis, it was observed that all the phytotoxicity indices improved after the remediation of the solutions by bacteria.

In conclusion, it can be stated that this study investigated the effect of sonochemical treatment on the valorisation of biomass. This research also explored the development of aerogels by the extracted biomass products and the doping of microorganisms for wastewater treatment. The results confirm that the processes significantly improved the value-added product extraction and adsorption quality by combining it with bioremediation.