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Development of a Low-Cost Biosorbent from *Daphniphyllum glaucescens* Seeds for the Sorption of Methylene Blue

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Abstract

Recent years have seen increased interest in using natural adsorbents as alternatives to conventional ones, from both economic and environmental viewpoints. This study aims to produce a natural adsorbent from a locally available plant: *Daphniphyllum glaucescens* seeds (DG), and to assess its potential for removing Methylene Blue (MB) from wastewater. The biosorbent material was prepared by grinding and pyrolysing DG at three different temperatures: 300 °C, 400 °C, and 500 °C. The material was characterised using XRD, EDX, and SEM. Proximate analysis, including ash, moisture, and carbon content, was conducted in accordance with ASTM-recommended protocols. Results showed that the adsorbent material prepared from DG had an optimal pH value of approximately 9.77. The ash content was 28.57%, and the carbon content was 59.67%. Additionally, the effects of process parameters such as contact time, initial concentration, and temperature on MB biosorption were examined. DG-based material achieved an adsorption efficiency of 96.1 mg/g, demonstrating its potential as an effective, sustainable biosorbent at Room temperature. Furthermore, the adsorption equilibrium data were best fitted to the Langmuir isotherm model ($R^2 = 0.9875$), indicating monolayer adsorption of MB onto DG-based activated carbon. The maximum adsorption capacity was 10.5 mg/g with a Langmuir constant of 1.70 L/mg. This work represents the first detailed investigation of DG seeds as a precursor for adsorbent synthesis, highlighting their untapped potential as a low-cost biosorbent, to the best of our knowledge. Owing to its cost-effectiveness, simple synthesis, and sustainable origin, the developed adsorbent shows potential for practical application in wastewater treatment.

Keywords: Adsorption, Biosorption, Methylene blue, *Daphniphyllum glaucescens*, Wastewater treatment.

1. Introduction

Environmental pollution has become a major worldwide concern due to its steady increase. One of the most important resources for sustaining life, water is constantly contaminated by a variety of sources, including sewage discharge, industrial effluents, agricultural runoff containing pesticides and fertilizers, metal plating, and mining operations¹⁻⁴. Since many parts of the world lack access to clean drinking water, the issue of water pollution is especially concerning. Every day, human activity releases significant volumes of both organic and inorganic contaminants. Another significant cause of water contamination is dyes, which are frequently used as coloring agents in sectors like textiles, paper, plastics, food processing, cosmetics, and pharmaceuticals⁵. Approximately 70–80% of illnesses in underdeveloped nations are associated with contaminated water, with women and children most affected, according to the World Health Organization (WHO) and UNICEF (2000)⁶.



Water containing colored organic molecules hinders photosynthesis in aquatic plants by reducing sunlight penetration. Iron (3 mg/L), copper (1.5 mg/L), cadmium (0.005 mg/L), lead (0.05 mg/L), zinc (5 mg/L), and chromium (0.05 mg/L) are among the WHO-set allowable limits for heavy metals in water, highlighting the significance of stringent regulation. In addition to hazardous heavy metals including Pb, Cd, Hg, and As - many of which are carcinogenic - industrial effluent frequently contain colours^{7,8}. Adopting efficient techniques for wastewater decolorization is crucial given the ecological significance of aquatic systems.

There are various methods for treating wastewater, which can be broadly categorized into biological, physical, and chemical approaches. Among these, the application of

agricultural waste products as adsorbents has drawn a lot of interest⁹⁻¹². There are several benefits to turning plant-based wastes into effective adsorbents, including affordability, chemical stability, accessibility, quick regeneration cycles, and environmental sustainability, as presented in Fig. 1. Effective adsorption of dyes and heavy metals is made possible by their porous structure, high surface area, and variety of functional groups^{13,14}. In addition to lowering waste disposal expenses, activated carbon made from plant materials, whether in its raw or modified form, offers a sustainable solution to environmental issues¹⁵. To ensure their practical usefulness, these adsorbents must frequently be modified to improve their stability, recyclability, and adsorption effectiveness.

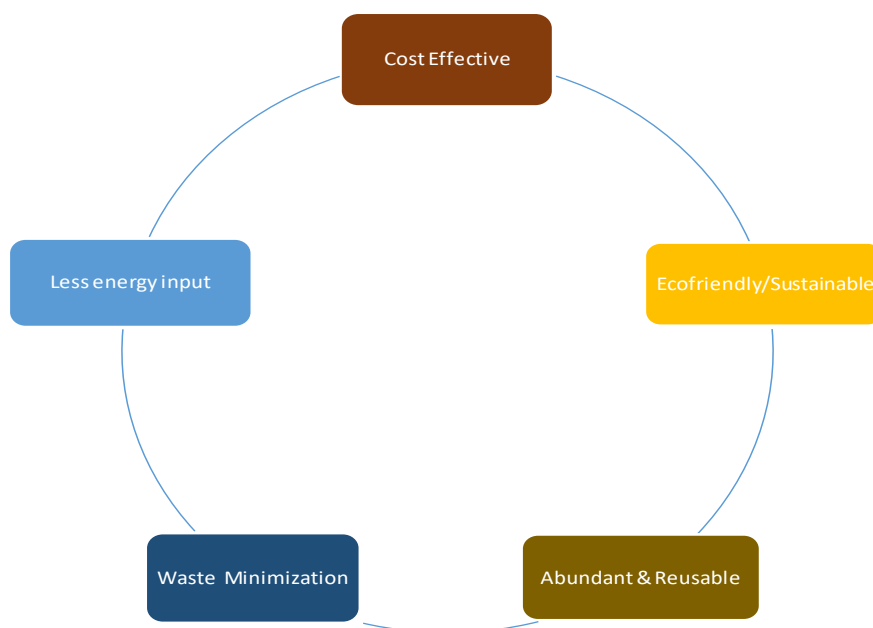


Figure 1. Advantages of Plant-Based Adsorbents

One of the most promising methods for removing dye from wastewater is adsorption, which involves the accumulation of dye molecules on the surface of the adsorbent through interactions like van der Waals forces, hydrogen bonding, ionic attraction, and occasionally covalent linkages¹⁶. The main benefit of adsorption is its cost-effectiveness, mainly because sorbents are less expensive than other treatment methods. The efficiency of adsorption depends on the type of sorbent and operating

conditions like pH, temperature, stirring rate, and ionic strength, all of which affect how well dyes bind to the adsorbent surface¹⁷.

The primary aim of this paper is to calculate the efficacy of adsorption for the removal of methylene blue (MB) from aqueous solution using *Daphniphyllum glaucescens* Blume (family Daphniphyllaceae) seed-based activated carbon powder (DGAC) as a potential adsorbent for first-time application. Adsorption data were analyzed by isotherm models, and the results

were correlated with some thermodynamic parameters. The adsorbent was characterized before MB adsorption, and it removed roughly 96.1% of the MB, making it a potential low-cost biosorbent material.

2. Materials and Methods

2.1 Preparation of the Adsorbent

DG seeds were gathered from rural Ansuya village, Mandal, Gopeshwar, and Chamoli (Uttarakhand), India. The samples were cleaned with distilled water to get rid of any physical

contaminants, like dirt particles. They were then sun-dried for a considerable amount of time. The samples were dried in an oven after the procedure. In 10 minutes, the oven's temperature increased from room temperature to 100 °C. The sample was kept at a constant temperature of 100 °C in the oven for two hours. To cool, the dried seeds were put in a desiccator. As a result, the following formula was used to determine the weight loss:

$$\text{Percent weight loss} = \frac{\text{weight of sample taken for physical activation}}{\text{weight of physically activated sample obtained}} \times 100$$

A mixer was used to smash and grind dried materials into a powder. 50 g of the pre-treated sample (obtained in the previous stage) were put in a muffle furnace to create a physically activated sample. Its temperature was raised from room temperature (RT) to 100 °C in five minutes and held there for an hour. Then, at a rate of 25 °C every 20 minutes, it was raised to 300 °C, 400 °C, and 500 °C in four hours, respectively. It was

maintained at the same temperature for an hour after reaching the target temperature. In this manner, *Daphniphyllum glaucescens* seeds are used to create the physically (thermally) activated sample (DGAC-1 at 300 °C, DGAC-2 at 400 °C, and DGAC-3 at 500 °C). Fig. 2 shows a schematic illustration of the preparation of the adsorbents at different temperatures.



Figure 2. Schematic diagram for DGAC preparation

2.2 Preparation of the dye solution

MB is a cationic dye with a molar weight of 319.85 g/mol and the chemical formula $C_{16}H_{18}N_3SCl$. At 293 K (room temperature), this cationic dye has great water solubility and is positively charged on the S atom, whose chemical structure is depicted in Figure 318. The remaining

aqueous solutions were made using deionized water. A suitably weighed quantity of MB hydrate reagent (bought from Sigma-Aldrich, India) was dissolved in 1 L of deionized (DI) water to create an MB stock solution (1 g/L). By diluting MB stock solution with DI water, the experimental

solutions of the required initial concentrations were produced.

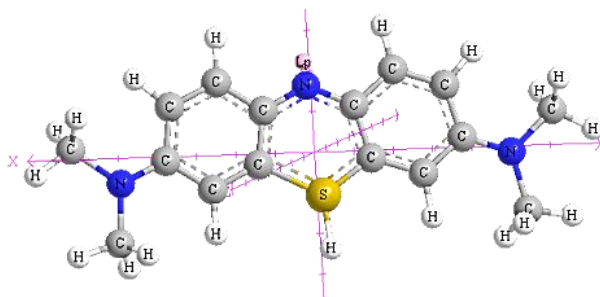


Figure 3. Chemical structure (2D) of Methylene Blue dye

2.3 Proximate analysis

2.3.1 Moisture content

The standard procedure ASTM D2867 was used to determine the moisture content. Using this approach, three crucibles were filled with one gram of activated carbon. The crucibles' weights were recorded both before (G) and after (B) the

activated carbon was added. After that, these crucibles were heated to 150 °C for three hours in a hot air oven. The crucibles were cooled for three hours before being weighed once again (F). The following formula yields the moisture content:

$$\text{Percent moisture content} = \frac{(B - F)}{(B - G)} \times 100$$

2.3.2 Ash content

The ash content of the adsorbent was established using the ASTM D2866 standard methodology. Three crucibles were needed for this procedure; each crucible's weight (G) was recorded before 1 g of activated carbon was added. After that, each

crucible's weight was recorded (B). The crucibles were placed in a muffle furnace in an oxygen-depleted environment at 750 °C for three hours. The weight of each crucible was measured (F) after it had cooled. The following formula yields the amount of ash in each crucible:

$$\text{Ash content} = \frac{(F - G)}{(B - G)} \times 100$$

2.3.3 Volatile content

The ASTM D5832 standard methodology was used to determine the biosorbent's volatile content. Three crucibles were needed for this approach; each crucible's weight (G) was recorded before 1 g of activated carbon was added. The weight of each crucible was then

recorded (B). These crucibles were then heated to 650 °C for ten minutes in an oxygen-depleting muffle furnace. Each crucible's weight was determined after it had cooled (F). Each crucible's volatile content can be found using the following formula:

$$\text{Volatile content} = \frac{[100 \times (B - F) - M(B - F)]}{(B - G)(100 - M)} \times 100$$

2.3.4 Fixed Carbon content

The determination of fixed carbon content was done using the ASTM D 7633-13 standard protocol by the following equation:

$$\begin{aligned} \text{Fixed carbon content} &= 100 \\ &- [\text{Percent moisture content} + \text{Percent volatile content} \\ &+ \text{Percent ash content}] \end{aligned}$$

2.3.5 Bulk Density

In accordance with the ASTM D2854-09 standard technique, the bulk density was determined. This process involved weighing and recording (X) a clean, dry measuring cylinder with a volume of 10 mL. The weight of the cylinder containing the sample was measured (Y)

$$\text{Bulk density} = \frac{(Y - X)}{V} \times \frac{(100 - Z)}{100}$$

2.4 Characterization of the adsorbent

To determine whether active functional groups were present, the biosorbent was examined using X-Ray Diffraction (P-XRD). The wavelength of X-rays used in XRD is between 0.07 and 0.2 nm, and they diffract across the specimen. The diffraction pattern is used to determine the properties of the crystalline phases. Due to the non-destructive nature of this technique, sample preparation is not necessary. An XRD apparatus determines XRD data in the 2θ range between 10 and 80° in this study. To comprehend the adsorbent properties in terms of the biosorbent's porous structure, scanning electron microscopy was also used. Prior to imaging, the sample was sputter coated with a thin layer of gold to enhance conductivity and image resolution, SEM has an image magnification of between 10 and more than 2000. In addition, Energy Dispersive X-ray Spectroscopy (EDX) was used in conjunction with SEM to ascertain the biosorbent's elemental makeup.

2.5 Dye adsorption experiments

MB was used in an aqueous solution for adsorption investigations on produced DGACs. To make a stock solution of 200 mg/L of MB, a stoichiometric amount of dye was dissolved in 500 mL of distilled water. Using pure water, the stock solution was diluted as needed. The air atmosphere was used for all experiments. After the desired sample preparation was finished, the flasks were placed on a magnetic stirrer set to shake at 200 rpm. A spectrophotometer (Shimadzu UV-Vis) was used to measure the dye concentration at λ max 662 nm after the biosorbent was separated by decantation. Before the measurements, the solutions were diluted to known concentrations to provide absorbance.

after activated carbon was added to the cylinder up to the 10 mL mark. The activated carbon was then placed inside the aluminium foil and cured for sixty minutes at 105 °C. The dry sample's weight (Z) was determined after drying. The adsorbent's bulk density can be found using the following formula:

2.5.1 Effect of Contact Time on Biosorption of MB

For adsorption tests, a series of Erlenmeyer flasks were filled with a known beginning concentration of methylene blue, a salt used as a dye and medicine. The solvent utilized was distilled water. Adsorbents (0.25 g/L) were then added to a succession of Erlenmeyer flasks that hold 200 mL of Methylene Blue solution apiece. Next, a magnetic stirrer was used to agitate the flasks while also regulating the temperature. The stirrer unit was kept at a constant temperature of 30 °C. The flasks were shaken at 200 rpm until they reached equilibrium. Finding the equilibrium contact time for MB dye is the goal of this experiment.

2.5.2 Effect of Initial Concentration on Biosorption of MB

For adsorption studies, a succession of Erlenmeyer flasks is filled with artificially generated dye solutions with different starting concentrations. Next, a series of Erlenmeyer flasks containing 100 mL of methylene blue solution are filled with adsorbents (2.0 g). A magnetic stirrer is then used to agitate the flasks and regulate the temperature. The stirrer's temperature was kept at 30 °C. Until equilibrium was established, the flask was shaken at 200 rpm. The relationship between the concentration used and the adsorption equilibrium reached is provided by this experiment.

2.5.3 Effect of Temperature on Biosorption of MB

For adsorption tests, a succession of Erlenmeyer flasks is filled with an artificially generated dye solution with a known initial concentration. The adsorption experiment in this study was conducted at three distinct temperatures: 30 °C,

40 °C, and 50 °C. A series of Erlenmeyer flasks with 100 mL of dye solution each were filled with adsorbents (0.5 g). A magnetic stirrer was then used to agitate the flasks and regulate the temperature. The stirrer was kept at three different temperatures: 30, 40, and 50 °C. Until equilibrium is achieved, the flask is shaken at 200 rpm.

3. Results and discussion

The experimental findings confirmed that activated carbons (DGACs) derived from DG seeds are insoluble in water, consistent with their predominantly aromatic and graphitic carbon framework. The yield of DGACs was strongly dependent on pyrolysis temperature. At 300 °C, the yield was comparatively high (49%), whereas increasing the temperature to 400 °C and 500 °C reduced the yield to 26% and 25%, respectively. This decline can be attributed to thermal decomposition and volatilization of non-carbonaceous components that progressively break down at higher temperatures. Elevated pyrolysis temperatures promote the release of volatile gases, thereby reducing the solid carbon

residue. Similar yield reductions with increasing temperature have been reported in biomass derived carbon studies, supporting this mechanism.

3.1 Proximate Analysis

3.1.1 Moisture content

The fraction of moisture in Precursor, DGAC-1, DGAC-2, and DGAC-3 was determined to be 6.713%, 3.727%, 3.1827%, and 1.2461, in that order. A high moisture content suggests a rather moderate particle density, making the biomaterial an excellent raw material for adsorbents utilized in various applications. The outcome supported the findings of Babatunde et al.¹⁹, which indicate that certain activated carbons will absorb a significant amount of moisture over several months when kept in humid circumstances. Although it clearly dilutes carbon, this moisture concentration has no effect on adsorptive power for many purposes. Among the variables, carbon dosage is especially crucial since it dictates the degree of decolonization and can be utilized to forecast carbon costs²⁰.

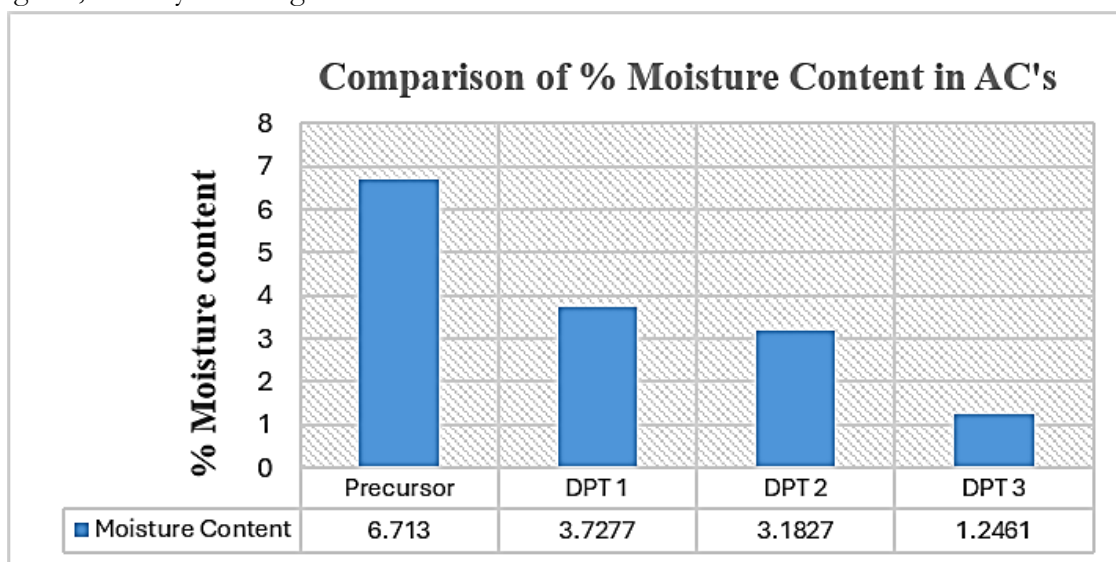


Figure 4. Comparison of Moisture Content in DGACs

3.1.2 Ash content

The residue left over after burning off carbonaceous material is known as carbon ash content. Ash content is mostly determined by measuring the carbon precursor of the minerals as impurities in the carbons, which are non-combustible contaminants found in coal that

remain after burning. Precursor, DGAC-1, DGAC-2, and DGAC-3 all had average ash contents of 3.77, 11.76, 21.05, and 28.57, respectively, as shown in Fig. 5. Ash content hurts coal's value and is expressed as a percentage of the coal's weight.

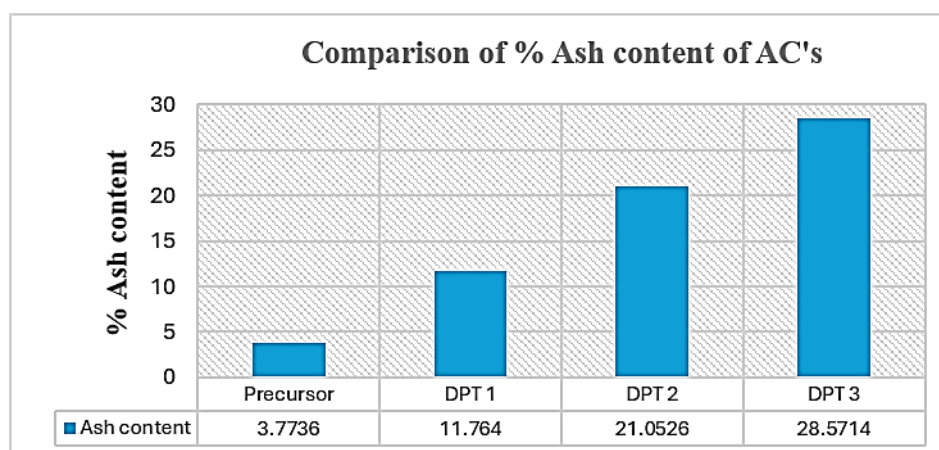


Figure 5. Comparison of Ash Content in DGACs

3.1.3 Fixed Carbon Content

Precursor, DGAC-1, DGAC-2, and DGAC-3 had average carbon contents of 45.78%, 56.983%, 54.06%, and 59.67%, respectively (represented in Fig. 6). The carbonization temperature needs to be carefully regulated to activate carbon to have the appropriate qualities. All non-carbon materials must be dried and

volatilized during carbonization, which requires a high enough temperature. An excessively high carbonization temperature has a significant impact on carbon's activity. Increased surface area and interior openings result from the carbonized material's molecular architecture being influenced by the temperature of carbonization and activation.

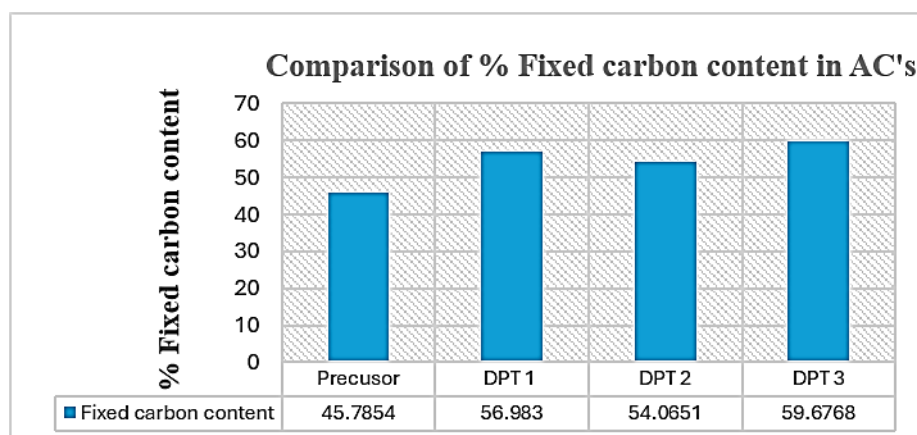


Figure 6. Comparison of Carbon Content in DGACs

3.1.4 Volatile content

To gauge the degree of thermal breakdown and the release of non-carbonaceous chemicals during carbonization, the volatile matter content of the precursor and activated carbons (DGAC-1, DGAC-2, and DGAC-3) was measured (Fig. 7). The amount of material that evaporates when a sample is heated without oxygen is known as volatile matter, and its value is directly correlated with the stability and purity of the carbon that is

formed. From precursor to activated samples, the volatile content gradually decreased, indicating the gradual elimination of organic components and the formation of a more ordered carbon structure. Because it increases the fixed carbon percentage and helps to improve surface area and pore formation, this decrease in volatile matter is desired.

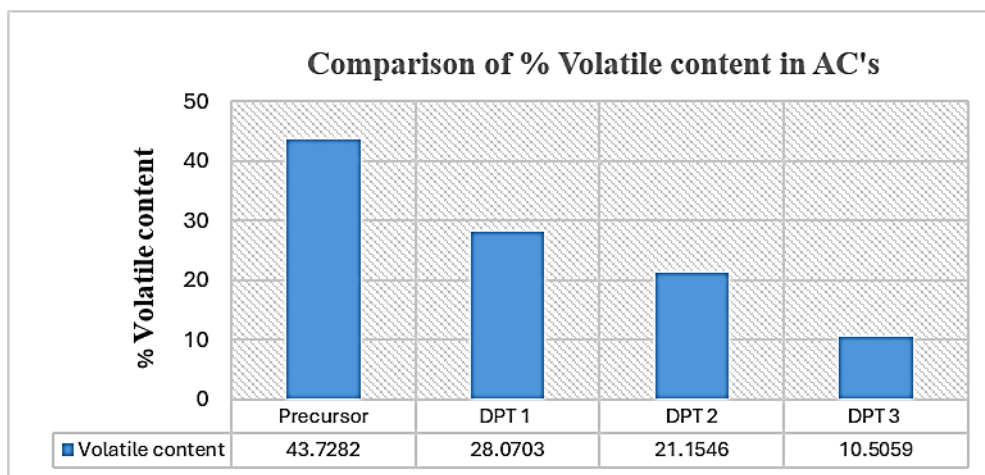


Figure 7. Comparison of Volatile Content in DGACs

3.1.5 Bulk Density

Bulk densities for precursor, DGAC-1, DGAC-2, and DGAC-3 were 610.9, 300.47, 228.75 & 216.31 Kg/m³, respectively (Figure 8). One important factor affecting the packing, porosity,

and adsorption efficiency of activated carbons is bulk density. While overly high values may restrict available surface area, higher bulk densities often suggest superior packing and mechanical strength.

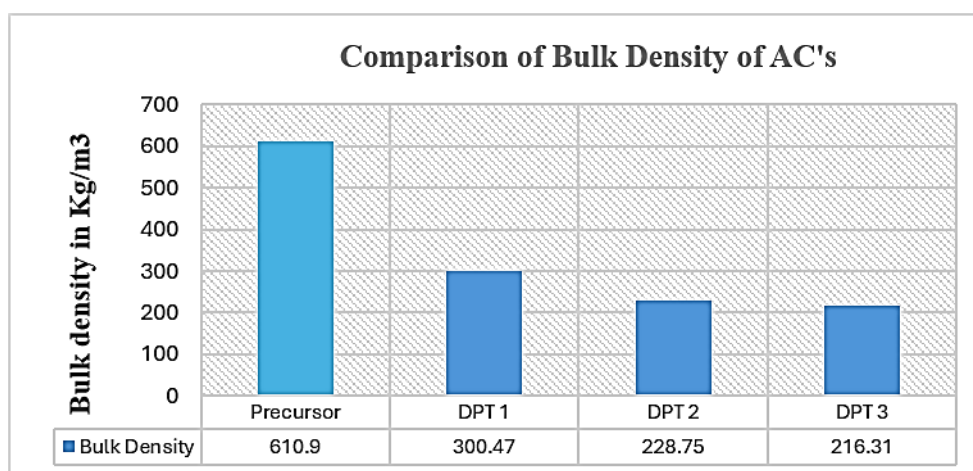


Figure 8. Comparison of Bulk Density in DGACs.

3.2 Characterization of Adsorbent:

3.2.1. X-ray diffraction (XRD):

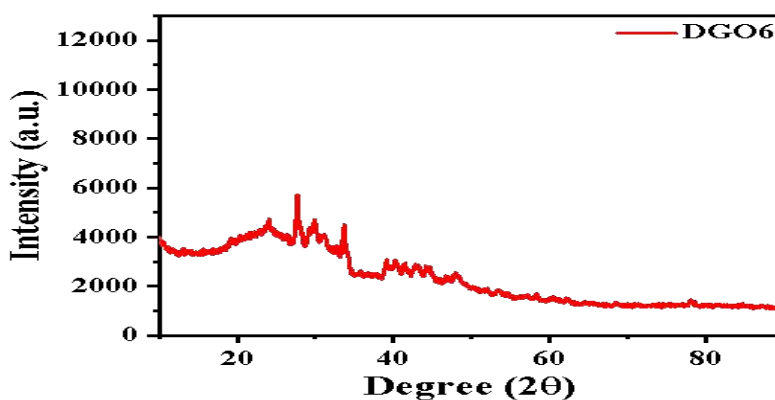


Figure 9. XRD diffractogram showing amorphous phases of DGAC-3

The XRD patterns, presented in Figure 9, showed that there were noisy, broad bumps with a smaller number of Sharp peaks. This suggested that the prepared AC (DGAC-3) had a mostly amorphous form. This is one of the benefits of AC. A wide maximum hump at 2 θ between 20 and 30 degrees indicates that the material is not well crystalline. Partial ordering in some domains may be the cause of the small, pointed feature on top of the broad hump. A peak of about 25° closely corresponds to the (002) plane, suggesting

tiny domain sizes or poor ordering. The inclusion of materials such as potassium oxides results in very low crystallinity. Both the crystallinity of biomass and the diffraction peaks may be impacted by the pyrolysis temperature. Because alkaline oxyhydroxides are created during the synthesis of biochar, the other peaks seen in biochar corresponded to Calcite (Cc) and Plagioclase.

3.2.2. Field emission scanning electron microscopy (FE-SEM):

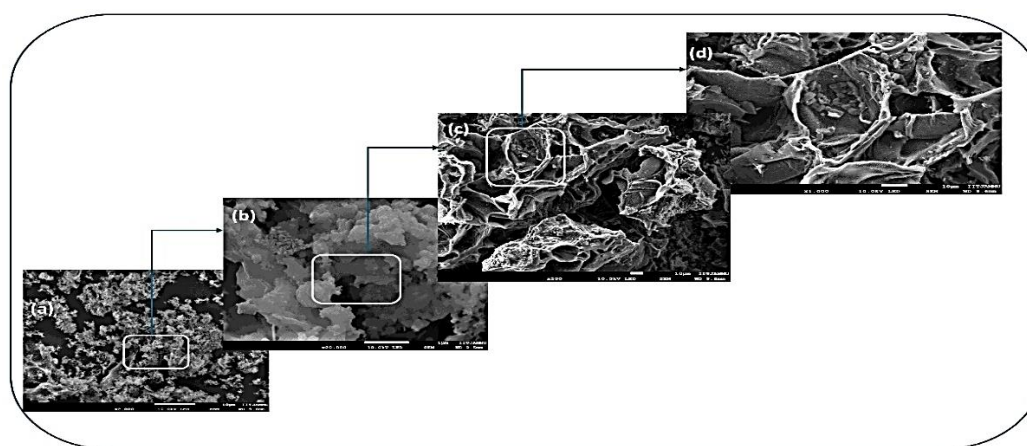


Figure 10. SEM micrographs of DGAC-3 before dye adsorption

Before MB biosorption, the energy-dispersive spectrum (EDS) examination of the biochar material along scanning electron micrographs (SEM) at various magnifications was analyzed as shown in Figs. 10 and 11. The activated material (DGAC-3) is displayed at various magnification scales in Figure 10 (a–d), displaying its porous and cavity-containing shape. The precursor material that is not activated is shown in Figure 3e. As a result, it has a very compact, layered

surface with ridges and folds, suggesting restricted pore growth. DGAC 3, on the other hand, is physically activated at 500 °C and, as a result, has a highly porous structure with large cavities, thin, wrinkled sheets, and angular particles embedded within the cavities (Figure 3f). This change demonstrates how well activation produces pore channels and surface roughness, both of which are essential for adsorption performance.

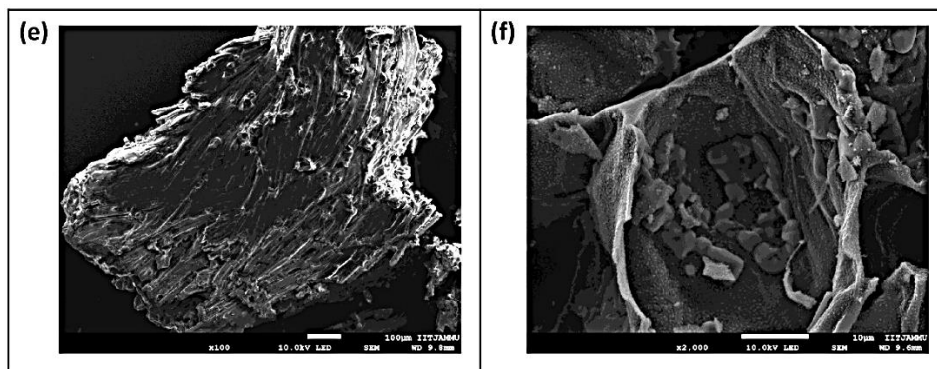


Figure 11. (e) SEM micrographs of Precursor material without activation; (f) SEM image of DGAC-3

3.2.3. Energy-dispersive X-ray spectroscopy (EDX):

Energy-dispersive X-ray (EDX) spectroscopy was used to examine the elemental makeup of the precursor and activated materials. The precursor material's spectra (Figure 12a) showed weak signals of potassium (1.98%) and significant peaks of carbon (74.56%) and oxygen (62.16%). The conductive coating used during SEM/EDX preparation is responsible for the presence of gold (6.46%) peaks. With a small inorganic input, this composition represents the raw biosorbent's organic structure. On the other hand, the

elemental profile of the activated material at 500 °C (Figure 4b) was significantly different. Strong carbon and oxygen peaks were seen once more, but along with potassium and gold, there were also signals for calcium (Ca), magnesium (Mg), phosphorus (P), and Sulphur (S). These elements' appearance upon activation points to structural alteration and the exposure of mineral phases that are embedded in the biosorbent matrix. The higher surface heterogeneity and possible active sites created during activation are highlighted by the greater diversity of elemental peaks, which should increase adsorption efficiency

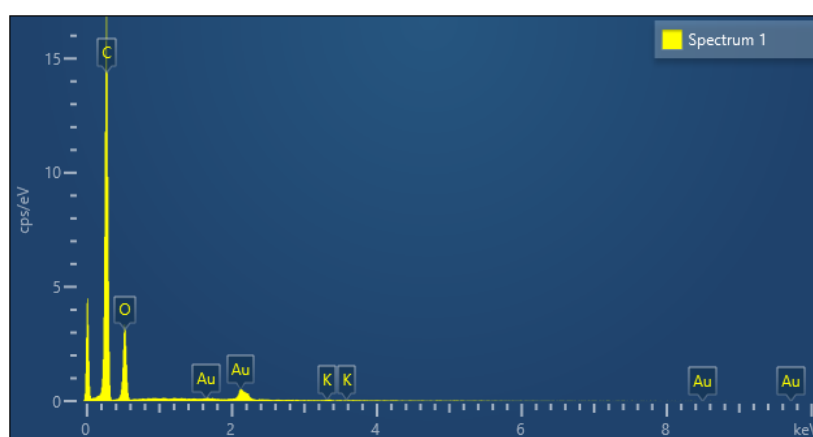


Figure 12 (a). EDX spectra of Precursor material

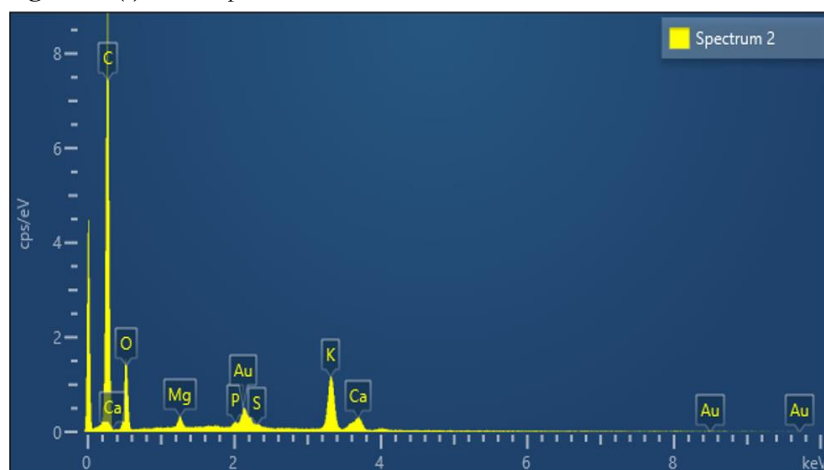


Figure 12 (b). EDX spectra of DGAC-3.

3.3 Dye adsorption experiment

3.3.1. Effect of Contact Time on Adsorption of MB

To determine the equilibrium period, the impact of contact time on the degree of MB adsorption was examined between 0 and 90 minutes. The results for the variance in MB removal

effectiveness by DGAC-3 biosorbent are shown in Fig. 13. It has been noted that MB removal efficiency rises with time and eventually reaches equilibrium, beyond which MB removal is impossible. In the case of MB, it takes 20 minutes to reach equilibrium. This pattern of initially faster adsorption followed by equilibrium is

caused by the fact that, at the beginning of the adsorption, all of the sites are empty, resulting in a significant rate of adsorption; as the treatment time increases, the sites become occupied,

resulting in a decrease in the adsorption rate. After a while, the adsorption ends when equilibrium is attained.

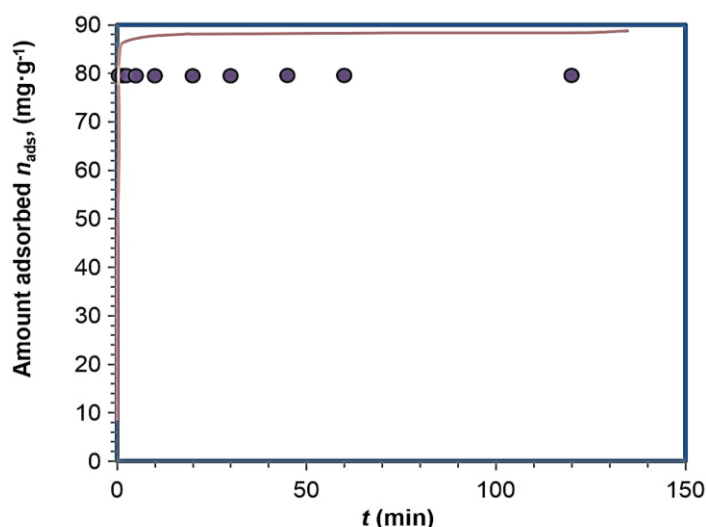


Figure 13. Variation of Adsorbed Amount with Contact Time.

3.3.2 Effect of Concentration on Adsorption of MB

By adjusting the initial concentration between 10 and 40 mg/L and maintaining the temperature at 30 °C, experiments were carried out to investigate the impact of initial concentration on the elimination of MB. The acquired results are displayed in Figure 14, and the rate of adsorption depends on the initial metal ion concentration. A greater driving force in terms of the available binding sites is responsible for the larger adsorption extents seen at lower concentrations. More MB molecules remain un-adsorbed in the solution because of rapid adsorption site

saturation at higher concentrations because of competition for adsorption. A lower percentage of adsorption results from a higher ratio of the initial quantum of MB molecules to the available adsorption sites. Additionally, it was found that while the amount of compound adsorbed increases as the initial concentration rises, the removal effectiveness (measured in percentage removal) falls because of the adsorbent surface becoming more quickly saturated. Therefore, diluting the solution or increasing the dosage of the biosorbent would be necessary to treat water with high MB concentrations.

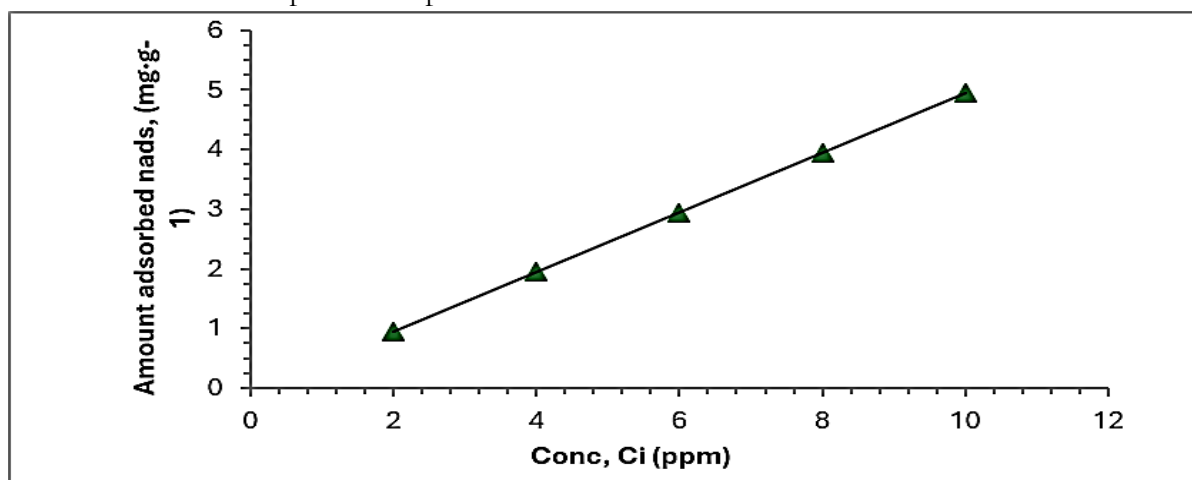


Figure 14. Variation of Adsorbed Amount with Concentration.

3.3.3 Effect of Temperature on Adsorption of MB

To assess the thermodynamic behavior of the process, the impact of temperature on the adsorption of methylene blue (MB) was investigated in the range of 30–50 °C. The removal efficiency rose with temperature, according to the data (Fig. 15), suggesting that the adsorption of MB onto the DGAC-3 biosorbent is endothermic. Because MB molecules had less kinetic energy and were less mobile toward the

active sites at lower temperatures, the adsorption rate was comparatively mild. Increased interaction between MB molecules and the biosorbent surface was made possible by the increased molecular mobility as the temperature rose, increasing the adsorption capacity. The process is thermodynamically favorable and controlled by endothermic interactions, as demonstrated by the observed rise in adsorption efficiency with temperature.

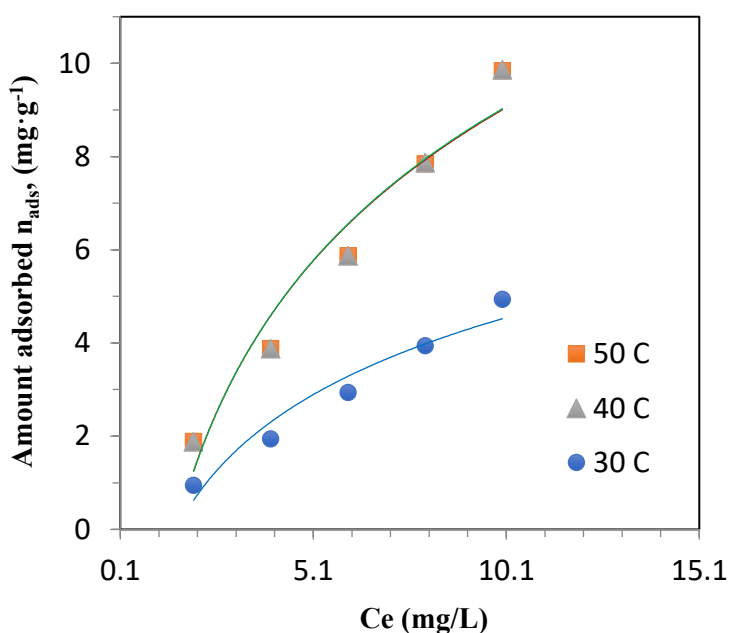


Figure 15. Variation of Adsorbed Amount with Temperature.

3.4 Adsorption isotherms

Equilibrium data were analyzed by Langmuir, Freundlich and Temkin isotherm models. The

Langmuir model is described by the following equation²¹:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m}$$

Where q_m is the amount adsorbed in mg/g (maximum adsorption capacity); K_L is the Langmuir constant in L/mg. The graph between $1/q_e$ and $1/C_e$ was plotted (Figure 16), which yielded q_m and K_L values of 10.0 mg/g & 0.95 L/mg, 10.5 mg/g & 1.25 L/mg, and 11.1 mg/g

& 1.70 L/mg for 30 °C, 40 °C, and 50 °C, respectively. The corresponding values of R^2 are 0.986, 0.988, and 0.989, respectively. R_L , the separation factor, was calculated with K_L values using the following expression:

$$R_L = \frac{1}{1 + K_L C_i}$$

Where C_i is the initial concentration in mg/L. The Value of R_L predicted the nature of

adsorption. Here, RL values were in the range of 0.3448 – 0.913 at approximately room temperature (30 °C), demonstrating $0 < RL < 1$;

$$\log q_e = \log K_f + \frac{1}{n} \log C_e$$

Where K_f and n are related to adsorption capacity and intensity of adsorption, respectively. The graph between $\log Q_e$ and $\log C_e$ were plotted (Figure 17), yielding K_f , n and R^2 values of 2.1, 1.85, and 0.95; 2.5, 1.70, and 0.96; & 2.9, 1.55, and 0.96 for 30 °C, 40 °C, and 50 °C, respectively, demonstrating favorable

$$q_e = B \log A + B \log C_e$$

where B is related to the heat of adsorption, A is the Temkin equilibrium binding constant (L/g), R is the universal gas constant, and T is the absolute temperature. The plots of q_e vs. $\ln C_e$ (Fig. 18) yielded values of A , B , and R^2 at different temperatures. The Temkin constants

hence, the adsorption was favorable at room temperature.

Freundlich isotherm model²² was also investigated using the following equation:

adsorption. The values of $R^2 > 0.9999$ were found for both Langmuir and Freundlich isotherm models. Therefore, both models were able to describe the adsorption process very well. The equilibrium data was further analyzed using the Temkin isotherm model. The Temkin equation is expressed as²³:

indicated positive values of B , confirming that adsorption is endothermic in nature. The relatively high correlation coefficients (R^2 values close to unity) demonstrated that the Temkin model could adequately describe the adsorption process.

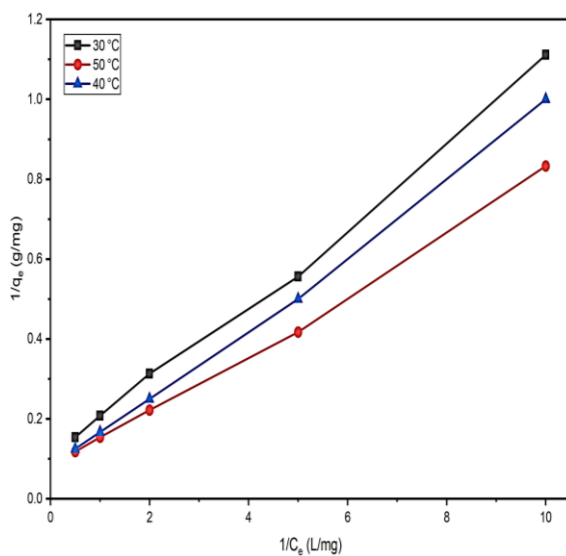


Figure 16. Langmuir adsorption isotherm for the adsorption of MB at different temperatures.

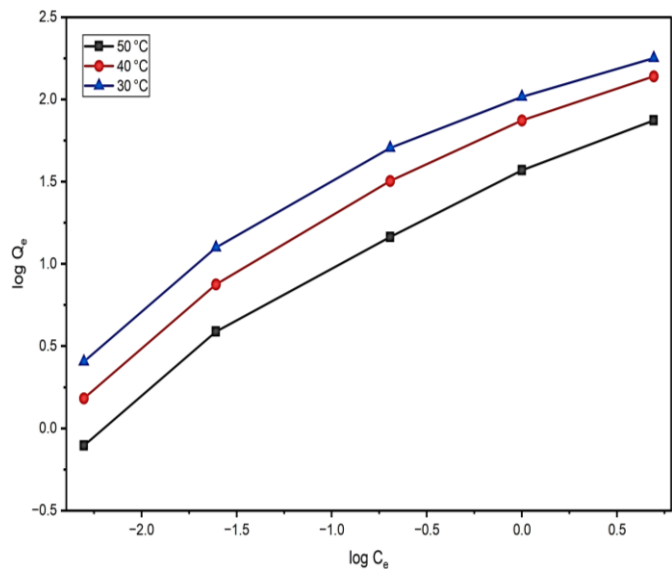


Figure 17. Freundlich adsorption isotherm for the adsorption of MB at different temperatures.

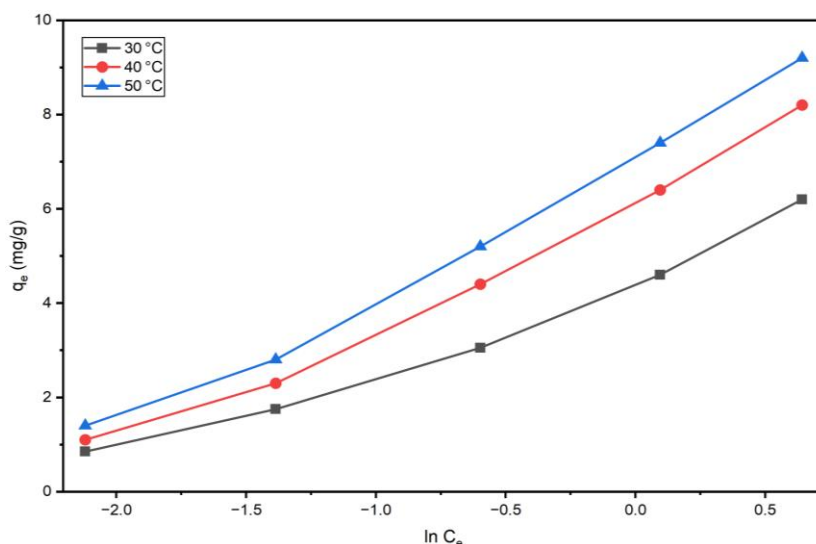


Figure 18. Temkin adsorption isotherm for the adsorption of MB at different temperatures.

3.5. Response Surface Modelling

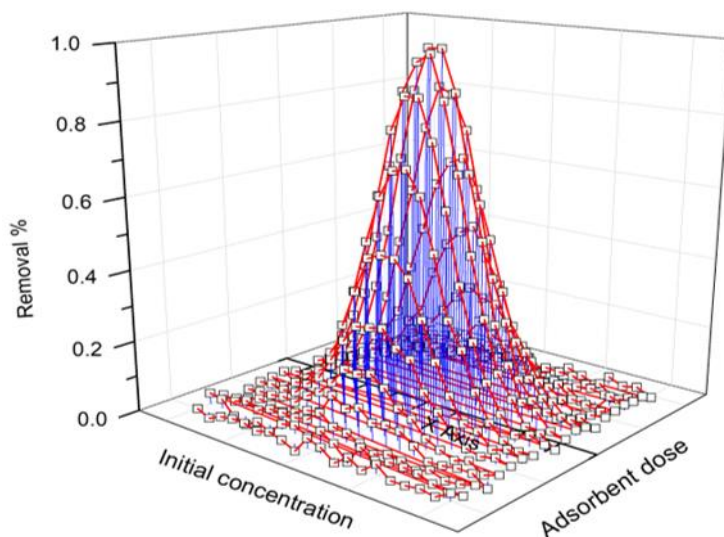


Figure 19. Response surface plot showing the effect of adsorbent dose and initial concentration.

The adsorption efficiency of methylene blue was optimized using a quadratic response surface model. The developed equation successfully predicted the removal percentage as a function of initial concentration and adsorbent dose. The 3D surface plot (Figure 19) illustrates the combined effect of these parameters, with the model normalizing the maximum removal efficiency to 100%. Experimentally, the highest removal achieved was 96.1%, which closely matches the model prediction, thereby validating the reliability of the statistical approach. The sharp peak observed in the plot highlights the optimum

operating condition, confirming that adsorption efficiency is strongly dependent on the synergistic influence of both variables.

The comparative adsorption efficiency of DGACs prepared at different pyrolysis temperatures is visually demonstrated in Figure 20. The untreated solution (Without Adsorbent) retained its deep blue coloration, indicating the original concentration of methylene blue. In contrast, DG-based adsorbents progressively reduced the dye intensity with increasing pyrolysis temperature. The sample prepared at 300 °C (DGAC-1) showed partial decolorization,

while the 400 °C material (DGAC-2) exhibited a more pronounced adsorption effect. The adsorbent obtained at 500 °C (DGAC-3) achieved the highest removal efficiency among the DG samples, with a color reduction comparable to that of standard activated carbon

(S.A.C.). This clear trend highlights the role of pyrolysis temperature in enhancing surface properties and adsorption capacity, and the visual evidence corroborates the quantitative adsorption data presented elsewhere in this study.

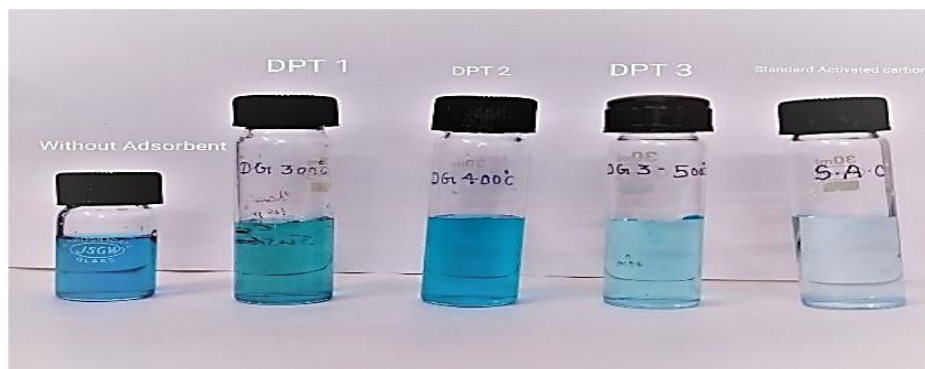


Figure 20. Comparative adsorption capacities of Precursor and DGACs.

3.6 Thermodynamic studies:

Thermodynamic parameters such as the free energy change (ΔG°), Enthalpy change (ΔH°)

and entropy change (ΔS°) of the adsorption processes were calculated from the following equation:

$$\Delta G^\circ = -RT \ln K_c$$

$$\Delta G^\circ = -RT \ln K_c$$

R is the gas constant (8.314 J/mol K), and T is the temperature in Kelvin (K). The negative value of ΔG indicated the feasibility and spontaneous nature of MB adsorption onto DGAC-3. The positive value of ΔH° confirmed that the MB

adsorption process was endothermic & spontaneous in nature. The positive value of ΔS° reflected an increased degree of disorderliness at the solid-liquid interface during the adsorption of MB onto DGAC-3.

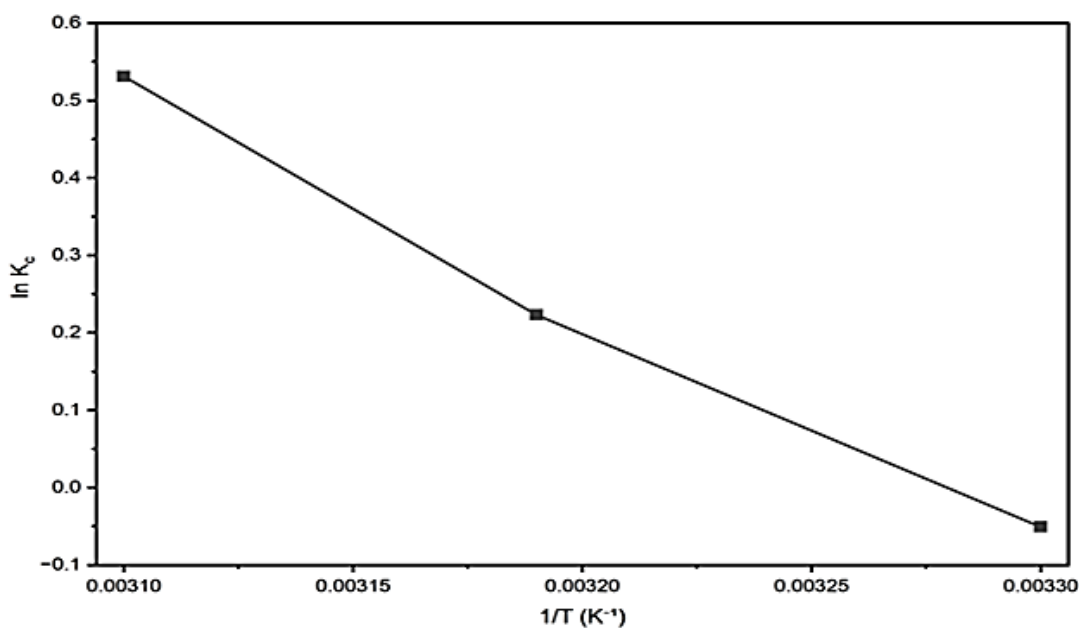


Figure 21. Thermodynamic study of Adsorption of MB onto DGAC-3.

4. Conclusions

The low-cost biosorbents were synthesized from the seeds of *Daphniphyllum glaucescens*. The optimum adsorption conditions were achieved at an adsorbent dosage of 0.5 g/100 mL solution, contact time of 90 min, pH of nearly 9, and initial Methylene Blue concentration of 10 mg/L, under which a maximum removal efficiency of 95% was obtained. The adsorption results fit with Pseudo-Second-Order kinetics and Langmuir isotherm theory, with a maximum adsorption capacity of 96 mg/g. This study shows that these locally available seed-derived biosorbents might be used as effective, low-cost, and eco-friendly adsorbent material to remove Methylene blue from wastewater. Many materials are continuously being tested in terms of cost-effectiveness, availability for commercial production, reusability & harmless nature. The present selected seed biomass fulfils most of the parameters and lacks experiments for reusability with natural water samples. Moreover, the experimental conditions did not include industrial effluents, as the Methylene Blue solutions were artificially prepared in the laboratory.

Authorship contribution statement

Anuj Gaur: Writing – original draft, Data curation, Formal analysis, Methodology; **Rohit Verma:** Conceptualization, Writing – review & editing, Supervision.

Financial & Ethical disclosure

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. All the co-authors have seen the final manuscript and agreed with the submission to the journal. The manuscript has not been published elsewhere. The article is not under consideration by any other journal in full or in part. All further responsibilities will be undertaken by the corresponding author.

Data Availability

All data generated or analyzed during this study are included in this published article.

Conflicts of interest

Authors state that there is no conflict of interest.

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