



Efficient Removal of Dye Compounds from Wastewater Using Activated Carbon Derived from *Albizia lebbek* Leave

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ABSTRACT

The considerations of activated *Albizia lebbek* leaves by synthesis with auto phosphoric acid for environmental remediation as a cost-effective and highly efficient pollutant removal technique is a stepping stone for further research. This research evaluated the removal efficiency of activated *Albizia lebbek* leaves on the reduction of methylene blue concentration from solution through a series of batch adsorption processes. The *Albizia lebbek* leaves were carbonized at temperature of 300°C for 2 hours and 30 minutes. The synthesized *Albizia lebbek* leaves-derived adsorbent was characterized by Fourier Transform Infrared spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET). The effect of contact time, adsorbent dosage, solution temperature, initial concentration and pH on the reduction of chromium from solution was investigated. The BET surface area, pore volume and pore size of the activated adsorbent was found to be greater than that of the blank adsorbent. The FTIR analysis established the presence of sharper peaks in the activated adsorbent compared to the blank adsorbent, and the presence of more surface functional groups. Batch adsorption results revealed that the removal of chromium from the solution was dependent of contact time, solution temperature, pH, and adsorbent dosage with the optimum value of 120 minutes, 60°C, pH of 6, and 0.2g respectively within the design space. It can be inferred that activated *Albizia lebbek* leaves is an effective adsorbent for removal of dye. Hence it may be explored for removal of heavy concentration of dye in different solutions from various industrial wastewater sources.

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INTRODUCTION

The relentless quest to meet escalating human demands in an industrial era continues to pose significant environmental challenges, particularly in the realm of waste disposal. Industries, at the forefront of this era, bear the crucial responsibility of adhering to environmental sustainability standards, especially in wastewater management (Nguyen and Garcia 2023). As stringent regulations evolve, industries have been adapting various methods for proper

wastewater treatment, with a notable focus on research into cost-effective and innovative solutions (Renge *et al.* 2012; Agarwal and Singh, 2017). Although significant progress has been made in the removal of heavy metal contaminants from wastewater, the challenge of dye pollution, particularly from industrial sources, remains a critical environmental concern (Chandra and Thompson 2023). The prevalent methods for dye removal often involve costly and complex processes, leaving a gap in the search

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for efficient, economical, and sustainable solutions (Boretti 2023). The escalating industrial activities, fueled by population growth, continue to release a variety of non-biodegradable and toxic dyes into water bodies. These dyes not only pose a threat to aquatic life but also affect the quality of water for human consumption. Therefore, the development of innovative and cost-effective methods for dye removal from industrial wastewater is imperative (Johnson and Patel (2024). In this regard, the potential of Albizia lebbeck leaves as an adsorbent material for dye removal in wastewater presents a promising avenue for research. Albizia lebbeck, a widely available and underutilized natural resource, offers an eco-friendly and economical alternative for dye adsorption (Kaur, and O'Connor (2024). The leaves of this species are known for their significant adsorptive properties, which could be harnessed to effectively remove various dyes from contaminated water (Wagner and Lopez 2024). This approach aligns with sustainable environmental practices and offers a potentially viable solution for developing countries, where industrial growth is rapid and the need for affordable wastewater treatment methods is high. The exploration into the effectiveness, feasibility, and implementation of Albizia lebbeck leaves in dye adsorption and removal from wastewater is therefore a critical area of study that warrants in-depth research and analysis.

The Albizia lebbeck tree, thriving in various climatic conditions and widely grown through natural propagation and human cultivation, presents a valuable resource in the realm of environmental remediation. The abundance of this tree on the University campus and its frequent occurrence as biomass waste on forest floors and school pathways underscores its potential utility. While numerous efforts have targeted the reduction of heavy metals like Cr, Cd, Pb in wastewater, the challenge of dye pollution, particularly from industrial sources, necessitates the exploration of new, effective adsorbents. The leaves of Albizia lebbeck, known for their porosity and adsorption capacity, offer a promising, low-cost solution for the adsorption of

dyes from wastewater, thereby mitigating environmental contamination. The aim of this research is to synthesize, characterize, and apply adsorbents derived from Albizia lebbeck leaves for the removal of dyes from wastewater.

METHODOLOGY

Batch experiments were conducted to determine the adsorption isotherms of industrial waste water. In order for the research to be indicative of climatic conditions, it was conducted at room temperature (Desta, 2013). Wastewater samples were poured into various graduated flasks. The flasks were shaken steadily at the rate of 50rpm to 300rpm for 120 minutes to give enough time for adsorption equilibrium to be achieved and for all the surface area to come into touch with heavy metal ions. The effect of five factors on methylene blue concentration were examined. The factors are interaction duration, temperature, adsorbent dosage, solution concentration, and pH.

Determination effect of Temperature on percentage removal of dye

The effect of temperature on percentage removal of dye was investigated at different temperatures of 30 °C, 35 °C, 45 °C, 55 °C and 60 °C

Determination of the effect of adsorbent dosage on percentage removal of dye

Six distinct masses of adsorbent dosages (0.02, 0.04, 0.06, 0.08, 0.1 and 0.2) were separately mixed with the industrial wastewater in the flask and were used to check for the effect of amount of dosage on the percentage removal of dye.

Determination of the effect of concentration on percentage removal of dye

Six standard solutions of varying concentrations were prepared using a stock solution of potassium dichromate and used to study the effect of concentration on dye removal. The six concentrations were 120ppm, 80ppm, 60ppm, 50ppm, 40ppm and 20ppm.

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Determination of pH effect on percentage removal of dye

The effect of pH was investigated for the adsorption of methylene blue from a solution of potassium dichromate solution of 50 ppm. The pH parameters were varied between the values of 2 and 12 over a wide range of acidic and basic pH regions, with a pH interval of 2 (i.e., 2,4,6,8,10,12). A uniform volume of 20mL of 50ppm potassium dichromate solution was measured and added to 0.02g of modified *Albizia lebbbeck* leaves derived activated adsorbent (MALL). The six conical flasks of varying pH values were left inside a water bath at a constant temperature of 25°C and were agitated for 120 minutes. Hence, while all other factors were kept constant the pH values were varied.

Effect of time on percentage removal of dye

The effect of contact time was investigated on the adsorption of Methylene blue on the MALL at various time intervals by varying the contact time from 5 to 120min (at intervals of 20 minutes) using 0.2g of adsorbent in 20mL of wastewater and at 25°C for the reduction of chromium content in the solution.

Carbonization of *Albizia lebbbeck* Leaves

Carbonization was carried out at temperature of 300°C for 2 hours 30 minutes duration. Analysis of synthesized *Albizia lebbbeck* leaves and the unmodified calcined *Albizia lebbbeck*

The synthesized *Albizia lebbbeck* leaves (MALL) and the unmodified calcined *Albizia lebbbeck* (ALL) were analyzed for their surface area, pore volume and pore size using Brunauer-Emmett-Teller (BET) under N₂ adsorption-

desorption study. The FTIR technique was applied to unravel the intricacies of functional groups present in the adsorbent and to predict the interaction with the adsorbate as a result of the functional groups present. The spectra were collected in the range from 400 cm⁻¹ to 4000 cm⁻¹.

RESULTS AND DISCUSSION

Characterization of Adsorbent BET characterization

The results of Brunauer-Emmett-Teller (BET) characterization are presented in Table 1 and Figure 1. The results showed that the synthesized adsorbent has higher surface area which serves as an indicator that it may have higher adsorption capacity. It was also observed that the modified *Albizia lebbbeck* leaves (MALL) adsorbent has a higher surface area compared to the unmodified *Albizia lebbbeck* leaves (ALL) adsorbent (O'Neil, and Kumar (2023).. The enhanced surface area of MALL may be attributed to incorporation of the auto phosphoric acid as a synthesizing agent, which may have improved the MALL because the pore size of this adsorbent was about 5.902 nm which indicated that they are more mesoporous in nature compared with the unmodified adsorbent (ALL) with pore size 6.014nm. It can be deduced that the introduction of acid during the synthesis of the adsorbent can alter the surface properties of the adsorbent, increasing the porosity and surface area. It is expected that due to such desirable properties adsorption capacity and reactivity is enhanced, making it more effective for capturing ions from a solution (Van Erp and Martens, 2011).

Table 1: BET Analysis of Modified and Unmodified Adsorbents

Sample	Surface Area (m ² /g)	Pore Volume (cc/g)	Pore Size (nm)
ALL	231.617	0.215	6.014
MALL	326.436	0.305	5.902

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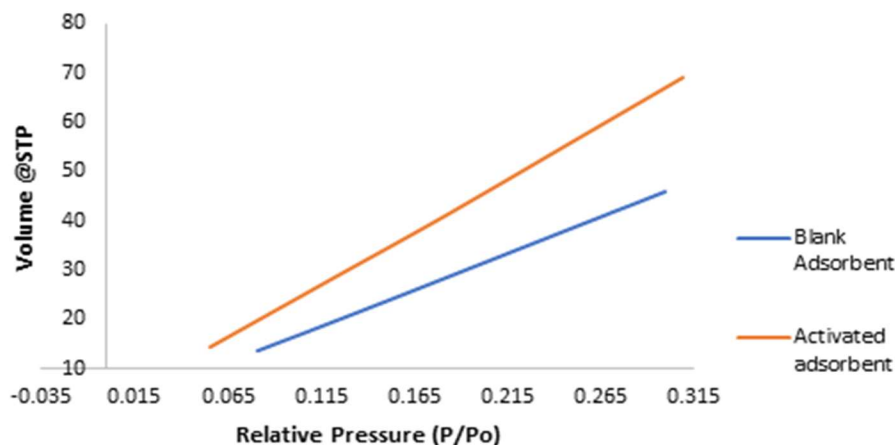


Figure 1: BET analysis result for the blank and activated prepared adsorbent.

Fourier Transform Infrared Spectroscopy Analysis (FTIR)

From FTIR analysis, it was observed that the while that unmodified spectrum has about 10 peaks, the modified adsorbent (MALL) possessed more than 20 peaks within its spectrum. The presence of more peaks in the modified adsorbent's spectrum can be said to be a function of the synthesis of the MALS with an acidic agent.

A higher number of peaks in the FTIR spectrum of the modified adsorbent suggests greater chemical complexity and diversity within the sample. Diversity and complexity depicted in the spectrum of the modified adsorbent reflect heavily on the expected broader selectivity

affinity of the modified adsorbent to target contaminants.

The important peaks represented are the C-H bond, the O-H bond, the C=C bond and C-O bonds (Ullah *et al.*, 2019). Figure 2 shows the plots of the activated and blank adsorbent. The high range of values for transmittance on the FTIR plot for the activated adsorbent can be attributed to several factors related to the changes in the adsorbent's structure, composition, and surface properties during the activation process. The activation process generated more active sites with functional groups. This increased availability of functional groups contributed to higher transmittance values due to enhanced adsorption (Yurdakal *et al.*, 2019).

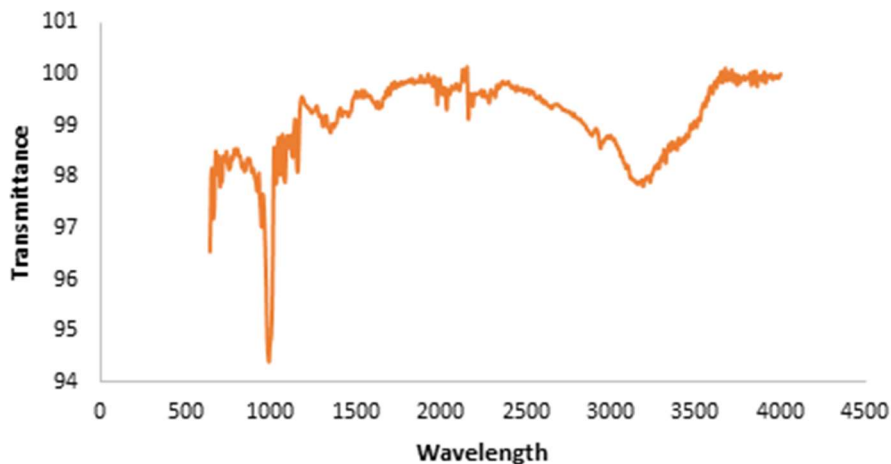


Figure 2: FTIR of the activated Albizia lebeck leaf adsorbent

It is clear that for the unmodified Albizia leaves adsorbent the absorption peaks observed at about 3201cm^{-1} indicate the presence of the O-H group due to alcohol or phenol (Mahananda *et al.*, 2021). The peaks observed at about 1118cm^{-1} indicates the presence of C-O bond within the unmodified Albizia seed adsorbent. The peak observed at the range of 13700 to 1400 for the unmodified adsorbent indicates the presence of C-H bond within the adsorbent.

The peak number between 3000 cm^{-1} and 3980 cm^{-1} indicate a high density of various functional groups in the fingerprint section of the spectra. The peaks observed between 3200 cm^{-1} and 3260 cm^{-1} indicate the presence of the O-H bond in the modified adsorbent. The observable peaks seen between 2850 cm^{-1} and 2960 cm^{-1} indicated the presence of C-H functional group in the adsorbent compound. The observable peaks between 1650 cm^{-1} and 1800 cm^{-1} indicate the presence of C=O functional group in the

adsorbent Between 1400 cm^{-1} and 1600 cm^{-1} , the observable peaks detected signify the presence of C-C bond within the adsorbent spectra. Observable peaks around 2300 cm^{-1} were attributed to the presence of C≡C bonds in that range of the spectra. Peaks observed between 1100 cm^{-1} and 1300 cm^{-1} could also serve as an indication of the presence of phosphates (P=O) as a result of synthesis of the adsorbent with phosphoric acid.

The greater number of peaks seen in the modified Albizia seed adsorbent as compared to the unmodified Albizia leaf adsorbent elaborates on the effect of the synthesizing agent that has been added to the Albizia lebeck leaf adsorbent to increase its adsorption efficiency. Hence, the modified adsorbent contains more functional groups than the unmodified adsorbent ensuring greater adsorption efficiency during the experiment.

Physiochemical Study of Albizia lebeck Leaves Adsorbent Biomass

Table 2: the summary of the carbonization process

Parameters	Value
Temperature	300°C
Time	2 hours 30 minutes

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Initial weight (g)	112.62
Final weight (g)	46.13

It was observed, as shown in Table 2, that the weight of the Albizia lebeck Leaves Adsorbent Biomass reduced by 52% percent

after carbonization at 300°C for 2 hours 30 minutes which was determined as the optimum factor setting after preliminary investigation.

Table 3: The physiochemical analysis of the *Albizia lebeck* leaves derived adsorbent

Parameters	Value
Bulk density (g/cm ³)	0.732
Ash content (%)	0.56
Moisture content (%)	6.00239
Dryness content (%)	94.3188

As it can be seen in Table 3, the bulk density of the Albizia leaves adsorbent is 0.732 g/cm³ meaning that the biomass has undergone carbonization at high temperature, and it can be deduced that it has high packing quantity of solid and can hold much adsorbate. The Bulk density is a measure of the mass of the Albizia lebeck leaves adsorbent per unit volume. The Bulk density measures how tightly the adsorbent particles are packed together. Adsorbents with higher bulk densities generally have more tightly packed particles. Proper packing ensures uniform flow distribution of the fluid (gas or liquid) through the adsorbent bed, reducing channeling and improving the adsorption efficiency (Brahma et. al 2018).

An ash content of about 0.56% was observed as shown in Table 3 and it is satisfactory as it shows that there is a very low amount of ash and possible impurity accompanied with it. The Ash content in the Albizia leaves adsorbent refers to the non-combustible residue left behind after the adsorbent material has been subjected to high temperatures. This residue typically consists of minerals, inorganic impurities, and other non-volatile substances (Renge, 2012). Impurities in the ash may interfere with the adsorption process

by occupying active sites on the adsorbent surface, reducing the available surface area for adsorption, and affecting the selectivity of the adsorbent (Ahmed and Balakrishnan 2010). A moisture content of about 6% was observed as shown in Table 3 and it is fairly satisfactory. The Percentage Dryness of about 94% was observed, this strongly suggests that the adsorbent is quite suitable for storage and the adsorption process.

Moisture content can affect the adsorption capacity of the Albizia leaves adsorbent. When an adsorbent contains moisture, some of its surface area and pore space may be occupied by water molecules. This can reduce the available sites for adsorption of the target substances. In some cases, moisture itself can be the target of adsorption (Mahananda et. al 2021).

Batch Experiment Studies

The bath adsorption studies were carried out with five predominant factors in consideration: pH, time, adsorbent dosage, temperature and initial concentration. Figure 3 depicts the calibration curve before adsorption takes place. It is instrumental in order to determine the slope E that will be used for further calculations.

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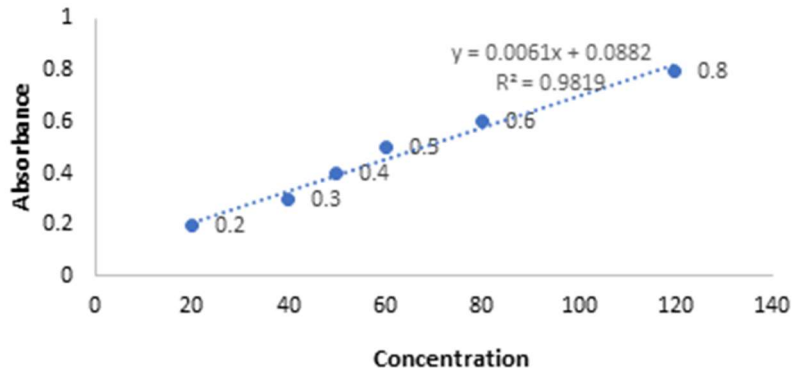


Figure 3: Calibration curve from the result for the initial UV spectrum test before adsorption

Effect of pH

The effect of adsorption with respect to varying pH values is depicted in Figure 4. It was observed that the optimum pH for adsorption was a pH value of 6. The trend observed can be attributed to several factors, including the

adsorbate and the surface charge of the adsorbent. The maximum level of percentage removal observed around a pH of 6 is due to charges that attract or facilitate binding of the adsorbate at top the surface of the adsorbent, enhancing the adsorption efficiency.

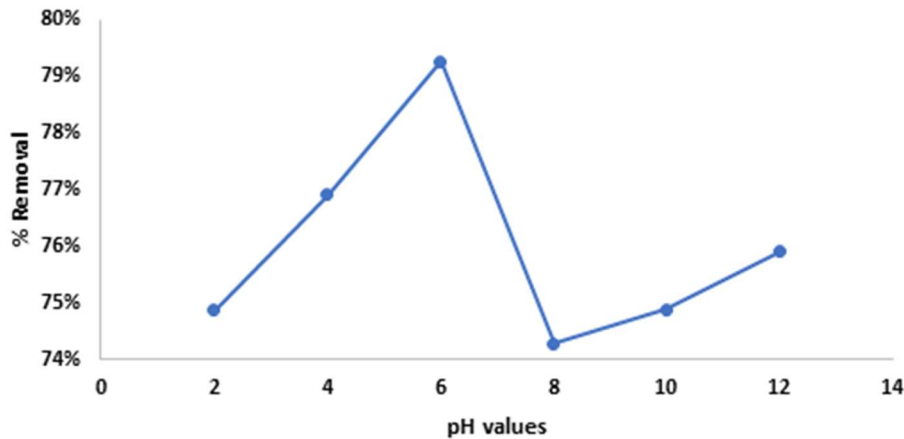


Figure 4: The effect of pH on percentage removal

As the pH increases gradually from 6 and into the basic range, the surface charge of the adsorbent may change. Electrostatic repulsion between the adsorbate and the adsorbent could be a significant factor that accounts for the sharp decline in the percentage removal as it progresses into the basic region. The increase in percentage removal of

methylene blue as the pH value increases from 8 to 12 is as a result of the surface charge of the adsorbent becoming positively charged, reaching a point where it can favorably interact with the adsorbate. As a result of possible changes, the adsorbate could undergo changes in ionization state, leading to improved adsorption efficiency due to more favorable interactions between the

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positively charged adsorbent surface (Azouaou et. al 2010).

Effect of Initial Concentration

Figure 5 shows the relationship between the percentage removal and the initial concentration of the Six standard solutions of

varying concentrations. It was observed that the percentage removal of methylene blue increased with an increase in initial concentration of the potassium dichromate solution. It was further observed that equilibrium was approached as the plot progressed towards 120ppm against percentage removal.

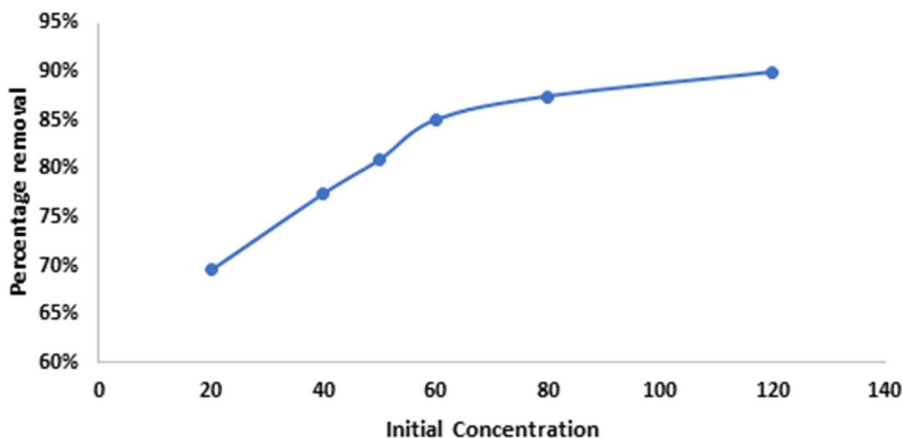


Figure 5: Effect of Initial concentration against percentage removal

It has been ascertained from previous researches that adsorption processes generally follow equilibrium kinetics (Mohammad et al. 2011). When the system reaches equilibrium, the percentage removal stabilizes. It can be seen from the plot of initial concentration against percent removal that the percent removal begins to stabilize at around 120ppm. At lower concentrations, it may take longer for the system to reach equilibrium, which can result in a lower percentage removal when compared to higher concentrations where equilibrium is reached more quickly (Padmavathy et al. 2016). Another reason for the trend on the plot in Figure 5 may be attributed to the fact that at lower concentrations, the adsorption sites may not be fully utilized, resulting in a lower percentage

removal. As the concentration increases, the adsorption sites become saturated, and the percentage removal approaches a maximum value.

Effect of Adsorbent Dosage

The general increase in the percentage removal of methylene blue from the solution as the dosage of the adsorbent increased, as shown in Figure 6, can be attributed to the available surface area for adsorption. Adsorption occurs at the surface of the adsorbent, so a greater surface area provides more sites for the target substance to adsorb to. This leads to higher adsorption capacity and, therefore, greater percentage removal (Mustapha et al. 2019).

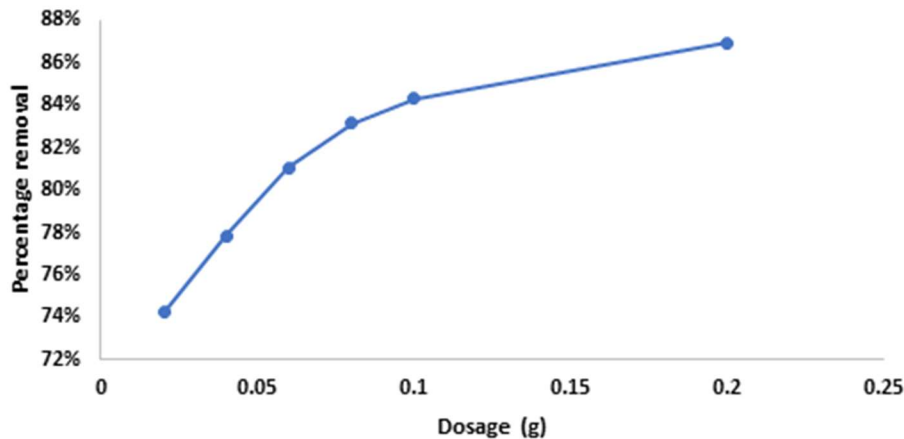


Figure 6: The effect of adsorbent dosage on removal of dye

It is evident that as the dosage increases, more sites become available for adsorption, resulting in increased percentage removal. At lower dosages, some adsorption sites are apparently unoccupied, which may be the resulting factor for limit in the percentage removal. Hence, the maximum adsorption as seen in Figure 6 occurred at an adsorbent dosage of 0.2g.

Effect of Time

Figure 7 shows the removal of methylene blue from the solution with respect to contact time. It was observed that the removal efficiency of MALL adsorbents increased with an increase in contact time. Equilibrium started to be attained as it approached towards 120 minutes. The increase in the percentage removal may be due to the presence of functional group for long periods being in contact with the adsorbate (Ullah et al. 2019).

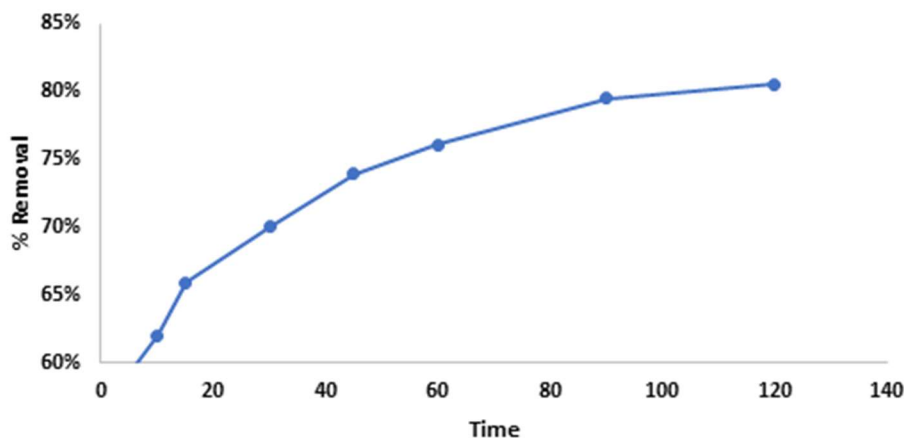


Figure 7: The effect of contact time on removal of dye

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Effect of Temperature

The Figure 8. The effect of temperature on the adsorption of chromium from the solution

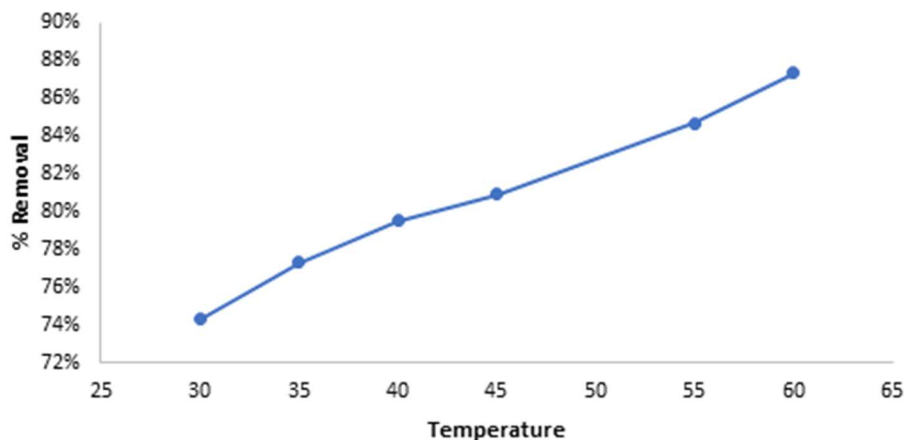


Figure 8: Effect of temperature on removal of dye

Temperature has strong effect on the intermolecular force between the adsorbate and the adsorbent particles. Effect of temperature on the adsorption process was investigated and it was observed that an increase in the temperature causes an increase in the efficiency of MALL removal. This proportional relationship indicated that the adsorption process is an endothermic reaction.

CONCLUSION

The carbonization of *Albizia lebbek* leaves at 300 degrees for 2 hours and 30 minutes, as well as the activation of the adsorbent with acid resulted in developing effective active sites and mesopores necessary for the adsorption process. Characterization of the prepared *Albizia lebbek* leaves using BET and FTIR shows variation in the surface area and functional groups present in the activated adsorbent as compared to the blank adsorbent. The activation resulted to more purification effect of the adsorbent. Adsorption tests using activated *Albizia lebbek* leaves showed high percentage removal of methylene blue in the range of 80-90%. Thus, activated *Albizia lebbek* leaves is recommended to be adopted for, or as part of, waste water purification process.

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