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Physicochemical and Adsorption Properties of Guava Leaves-Activated Carbon by Hydrochloric Acid on Adsorption of Methylene Blue

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ABSTRACT

This study emphasizes the biomass waste which is dried guava leaves as a natural low-cost adsorbent precursor of activated carbon for methylene blue (MB) dye solution due to its high abundance and availability in Malaysia. MB dyes which were largely consumed by the textiles industry pose several hazardous complications on human health without proper wastewater treatment. Thus, adsorption for water treatment serves as a cost-effective method to adsorb the dyes using low-cost adsorbent from dried leaves biomass. The dried leaves underwent chemical activation with hydrochloric acid (HCl) with ratio 1:1 and were carbonized at 700 °C for an hour and then neutralized for characterization and batch adsorption study. The physicochemical properties of guava leaves HCl activated carbon (GL-HAC), was characterized using ash content, moisture content, bulk density and iodine number. Percentage yield of GL-HAC was apparently high at 89.90 %. The GL-HAC reported had relatively low percentage of ash content and moisture content. The bulk density of the activated carbon was also low at 1.6 g/mL. The iodine number was calculated at 339.9 mg/g. The pH_{PZC} *for GL-HAC was obtained at 4.3 indicating that this activated carbon is efficient for the removal of MB dye with the pH solution above the* pH_{PZC} *value. The results of the FTIR study shows that the functional groups of hydroxyl, amine, carbonyl, and carboxylic acid are present in GL-HAC. Batch adsorption studies were carried out to determine the most favourable condition for the activated carbon to adsorb methylene blue*

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dye. The operating variables studied were adsorbent dosage, initial pH of solution, initial dye concentration and contact time. The results revealed that the effect of initial concentration and contact time were directly proportional to the adsorption capacity of MB dyes on the activated carbon. Thus, this study has demonstrated the potential of dried guava leaves as low-cost precursor of activated carbon for the removal of MB dyes.

Keywords: Low-cost adsorbent; activated carbon; proximate analysis; bulk density; adsorption capacity.

INTRODUCTION

Today, numerous manufacturing sectors, including those in the dye, paper, textile, and plastic industries, use dyes like methylene blue (MB) to colour their finished goods. According to Jawad et al. [1], dyes, which are coloured substances, can be categorised as anionic, cationic, and non-ionic, which typically produces a significant volume of coloured effluent. As a result, more dye effluent is being discharged into wastewater on a regular basis, endangering aquatic life and other living things. More than 10,000 different types of dyes are commercially accessible, and most of them are difficult to biodegrade [2]. This is because those dye chemicals have complex aromatic molecular structures and come from synthetic sources. Many people have focused their attention on this issue while thinking about the long-term effects of the water's toxicity due to the presence of these dissolved pollutants.

Several conventional approaches, including photocatalysis, cation exchange membranes, Fenton chemical oxidation, bioremediation, and electrochemical degradation, have been studied for their ability to remove colours from industrial effluents and wastewaters [3]. Due to technological and economic constraints, the majority of these solutions, however, have a limited application. Adsorption, in contrast, is frequently employed in water purification techniques. When the right materials are used as the adsorbent, the adsorption technique for treating water shows extremely selective and effective operation with no dangerous secondary products. Most notably, wastewater treatment via adsorption has been shown to be economically cost-effective [4].

The adsorption method is widely used in the treatment of wastewater because of its simple design, easy operation, and high adsorption efficiency [5]. Adsorbent material with high affinity for pollutant analytes is required for an effective adsorption process. Many organic, inorganic, or hybrid materials have been developed to produce high-energy adsorption processes [6]. Researchers have been drawn to the development of carbon-based adsorbent materials to conduct research. This interest stems from the fact that the carbon precursor material has several beneficial properties, including significant natural abundance, high renewability, and low cost [7,8]. Activated carbon is another carbon-based adsorption material with a large surface area, internal pore structure, different pore size distributions, and a wide range of oxygenated functional groups [9]. Activated carbons are widely used as an adsorbent in numerous applications because of the carbon's porous structure that allows it to absorb materials from the liquid and gas phases. However, commercially activated carbon that is available nowadays may not be economical enough due to the higher cost. Alternatively, to solve this problem activated carbon can be prepared by using a low-cost material such as agricultural waste or biomass waste. Recently, researchers have focused on a variety of cash crops to produce the activated carbon. Guavas or its scientific name Psidium guajava are tropical trees originating in Central America. In Malaysia, guava called Jambu Batu. Due to demand, guavas now cultivated, and its fruit is commercially available in all market area in Malaysia. However, the guava leaf which traditionally used as medicine are abundant, thus make it a low-cost material to create the activated carbon due to its high carbonaceous materials.

In this study, the dried fallen guava leaves were activated with hydrochloric acid (HCL) to produce the activated carbon. The physicochemical properties of guava leaves-HCL activated carbon (GL-HAC) were determined, which includes bulk density, iodine number, moisture and ash content, and zero point of charge (pH_{PZC}). The FTIR analysis was carried out to identify the functional groups present in the activated carbon. Furthermore, batch adsorption will be used to examine the effects of the starting pH of the solution, initial dye concentration, and contact time on the ability of the activated carbons to adsorb MB dye.

METHODOLOGY

Preparation and Characterization of GL-HAC

The dried fallen guava leaves were gathered from a guava plantation in Felda Batu 8, Changlun, Kedah, Malaysia. The process for making the activated carbon was modified from Rashid et al. [10]. The leaves were activated using concentrated hydrochloric acid (HCl) at a ratio of 1:1 (wt%). The sample was washed with hot distilled water until the pH of the filtrate was neutral. The activated carbon was dried in an oven for 24 hours at 105 °C. The activated carbon powder was then sieved to produce particles with a size range of 150-212 µm, and it was then stored in closed bottles for later usage. The functional groups present in the GL-HAC before and after the adsorption process was performed by Perkin Elmer-Frontier Fourier Transform Infrared (FTIR) in the 4000 cm^{-1} - 500 cm^{-1} wavenumber range.

Bulk Density

To determine the bulk density, a 10 ml measuring cylinder was filled to a specified volume with activated carbon sample and guava leaves powder sample that had been dried in an oven at 80 °C for 24 hours. The bulk density was then calculated as follows:

$$Bulk\ density = \frac{W_c}{V_c} \tag{1}$$

Where Wc (g) is the weight of dried activated carbon and Vc (ml) is cylinder volume pack with dried activated carbon.

Moisture Content

The moisture content was determined by using an oven drying method. 0.5 g of activated carbon sample and guava leaves powder sample were put into a pre-dried ceramic crucible with lid, and the weight were recorded. After that, the lid was removed, and the crucible and the lid were heated in a ventilated drying oven at 150 °C for 3 hours. After the heating process, the plate was quickly covered and cooled in desiccators and the weight was recorded. The loss in weight of the adsorbent represented the moisture content. The moisture content was calculated by the following equation:

$$Moisture\ content,\ (wt\%) = \frac{W_{m3} - W_{m2}}{W_{m1}} \times\ 100 \qquad (2)$$

Where W_{m3} (g) represents the weight of crucible containing original sample, W_{m2} (g) represents the weight of crucible containing dried sample, and W_{m1} (g) represents the weight of original sample used.

Ash Content

This method covers the determination of the total ash content of activated carbon sample and the guava leaves powder sample. Both samples were dried to constant weight at 150 °C for 3 hours. Then, 0.5 g of both samples were placed in crucibles and ignited in a muffle furnace for 1 hour at 650 °C. After the heating process, the crucibles were placed in a desiccator and underwent cooling process at room temperature. The ash was taken from 3 to 16 hours depending on the type of activated carbon used and its particle size. Ashing was considered complete when a constant weight was achieved. Then the crucibles were kept in a desiccator and cooled to ambient temperature and the weight was recorded. During cooling process, the air was admitted slowly to avoid loss of ash from the crucible. The percent of ash was calculated as follows:

$$Ash\ content,\ (wt\%) = \frac{W_{s3} - W_{s2}}{W_{s1}} \times 100 \qquad (3)$$

Where W_{s3} (g) is the weight of crucible containing ash, W_{s2} (g) is the weight of crucible, and W_{s1} (g) is the weight of original activated carbon used.

Iodine Number

The first step of the iodine number test involved titrating 0.1 N sodium thiosulfate solution with 10 mL of 0.1 N iodine solution in a conical flask while using 2 drops of a 1% starch solution as an indicator. The titration procedure was continued until the solution lost its colour. This titration will determine the volume of titration for the blank solution. The following stage was the titration of the sample. About 1 g of GL-HAC was added to a conical flask that already contained 15 mL of a 0.1 N iodine solution. The mixture was then agitated for approximately 4 minutes before filtering.

Finally, 10 mL of the filtrate was titrated with 0.1 N sodium thiosulphate until the solution was turned to a pale yellow. A few drops of starch indicator were added and the titration with sodium thiosulphate was continued until a colourless solution produces at first drop. This test was done to determine the amount (in unit mg) of iodine absorbed by 1 g of activated carbon and it was calculated using the following equation:

$$Iodine\ number = \frac{(V_b - V_s) \times N \times (126.9) \times \left(\frac{15}{10}\right)}{M} \tag{4}$$

Where V_b and V_s (mL) are volumes of sodium thiosulfate solution required for blank and sample titrations, respectively, N (mol/L) is the normality of sodium thiosulfate solution, 126.9 is atomic weight of iodine, and M (g) is the mass of activated carbon used.

Zero Point of Charge (pH_{PZC})

pH_{PZC} of an adsorbent is important because it indicates the net surface charge of the carbon in solution. Besides, the pH_{PZC} was the point where the curve of pH (final) versus pH (initial) intersects the line pH (initial) = pH (final). To determine the pH of point of zero charge, 0.5 g of activated carbon sample and the guava leaves powder samples were added to 200 ml solution of 0.1M NaCl where the initial pH had been measured and adjusted with NaOH or HCl. The containers were then sealed and placed on a shaker for 24 hours. After that, the pH for both samples was measured.

Batch Adsorption Experiments

All the batch adsorption experiments were done in a set of 250 mL sealed conical flask containing 100 mL of MB solution. Batch adsorption experiments were carried out by varying several experimental variables such as adsorbent dosage (0.02 - 0.20 g), pH (3 - 11), initial dye concentration (50 - 300 mg/L) and contact time (0 - 1200 min) to determine the best uptake conditions for adsorption. After mixing and shaking, all the samples were filtered using 0.45 μm syringe filter into cuvette to monitor the concentrations of MB using Shimadzu UV-1001 UV-Visible Spectrophotometer at maximum wavelength of absorption of MB. The adsorption capacity at equilibrium, q_e (mg/g) and the colour removal percentage, CR (%) were calculated by using the following formula:

$$q_e = \frac{(C_o - C_e)/V}{W} \tag{5}$$

$$\% \, CR = \frac{(C_o - C_e)/V}{C_o} \times 100 \tag{6}$$

where C_o and C_e (mg/L) are the initial and equilibrium concentrations of MB, respectively, V (L) is the volume of the solution, and W (g) is the mass of dry adsorbent used.

RESULTS AND DISCUSSION

Physicochemical Properties of Activated Carbon

The physicochemical properties of GL-HAC were calculated through bulk density, moisture content, ash content, percentage yield of GL-HAC, volatile matter content, fixed carbon content, point of zero charge (pH_{PZC}), and iodine number. Table 1 shows the characterization result for both GL-HAC and untreated guava leaves sample. The results indicate that GL-HAC has a relatively high iodine number, low values of bulk density, ash content and moisture content along with a relatively high yield of carbon. These physicochemical properties results have been proved by a few researchers in previous studies such as Rehman *et al.*,[11] and Gaikwad & Misal [12] which confirm that a good characteristic of GL-HAC has relatively low bulk density and moisture content.

Table 1: Physiochemical properties of dried fallen guava leaves and GL-HAC

Characteristics	Dried Guava Leaves	GL-HAC
Percent yield (%)	-	89.90
Bulk density (g/mL)	1.29	1.65
Iodine number (mg/g)	198.51	339.91
Moisture content (%)	8.68	9.54
Ash content (%)	17.80	5.80

Analysis of pH_{PZC} was used to determine the pH of the GL-HAC at zero point of charge. It is one of the key factors that controls the adsorption process on carbon materials where the adsorbent net surface charge corresponds to zero and shows the electrostatic interaction between the adsorbent and adsorbate [10]. Figure 1 shows the plot of pH difference (ΔpH) versus initial pH for GL-HAC. The plotted graph indicates an inverse linear relationship. Based on the graph, the value of pH_{PZC} for GL-HAC was obtained at pH 4.3. According to Jawad *et al.*, [13], when pH solution below the pH_{PZC} value, adsorption of anion is more favoured as the surface of GL-HAC is positively charged due to protonation whereas adsorption of cation is more preferred when pH solution above the pH_{PZC} value as the surface of GL-HAC is negatively charged [1]. Thus, by increasing the pH solution above the pH_{PZC} value, the amount of dye uptake increases. Therefore, the pH solution above the pH_{PZC} value is efficient for the removal of MB dye.

Interestingly, the percent yield of GL-HAC was 89.90 % which is considered higher. The reason for high percentage yield was due to the HCl activation, which caused more potential sites to penetrate and occupy the activating agent. This enhances the pore opening and widening [14].

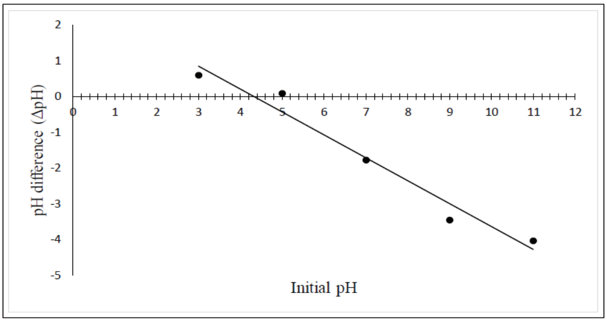
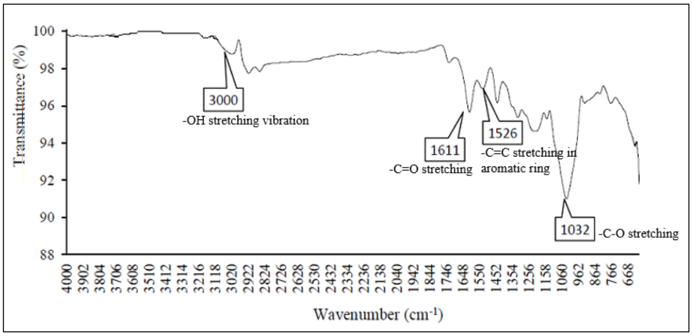


Figure 1: pH of the GL-HAC at zero point of charge (Under operating condition of 0.5 g of GL-HAC, pH 3-11, 24-hour, 0.01 mg/L NaOH concentration, 303 K temperature and 100 mL of NaOH solution)

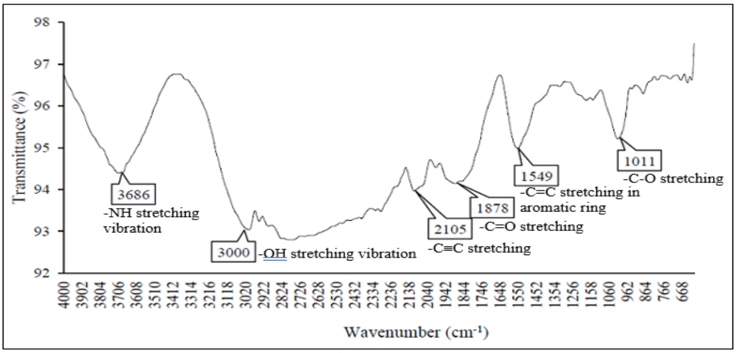
FTIR Analysis

Figure 2 shows the FTIR spectra of (a) raw guava leaves and (b) GL-HAC samples. As can be seen in Figure 2(b), the broad band at 3686 cm^{-1} showed the stretching vibration of -NH group while the bands at 3000 cm^{-1} in both figures ascribed the stretching vibration of hydrogen-bonded hydroxyl group which indicates the presence of carboxylic acids, phenols or alcohol groups as in lignin and cellulose on the GL-HAC surface. The broad-range O-H stretching vibrations occur indicates the presence of free hydroxyl groups and bonded O-H bands such as carboxylic acids as has been described by Jawad et al., [3]. Additionally, the adsorption band at 2105 cm^{-1} in Figure 2(b) corresponds to the C≡C alkyne stretching for the GL-HAC adsorption surface, similar result was obtained by Ojedokun & Bello [15].

The bands at 1878 cm^{-1} in GL-HAC and 1611 cm^{-1} in raw guava leaves showed the stretching of C=O probably from ester or carboxylic acid. The existence of bands at 1549 cm^{-1} and 1526 cm^{-1} in Figure 2(a) and (b) respectively, indicated the presence of C=C stretching in aromatic rings. Finally, the sharp peaks observed at 1011 cm^{-1} and 1032 cm^{-1} were assigned to the C-O stretching (acids, alcohols, phenols, ethers, ester group) that can be found in Figure 2(a) and (b), respectively. In conclusion, the activity of hydroxyl, amine, carbonyl, carboxylic acid and C=C functional groups may act as a possible active site on the GL-HAC surface for adsorption which occur after chemical treatment. It can be suggested that the activity of hydroxyl, amine, carbonyl, carboxylic acid and C=C functional groups may act as a possible active site on the GL-HAC surface for adsorption which occur after chemical treatment.



(a)



(b)

Figure 2: FTIR spectra of (a) Raw sample of guava leaves (b) GL-HAC

Adsorption Batch Study

Effect of adsorbent dosage

Adsorption of MB dye is influenced by adsorbent dosage. Figure 3 showed the percent of MB removal at different values of adsorbent dosage ranging from 0.02 to 0.2 g. The percentage of MB removal showed an increasing trend from 91.8 % to 94.7 % with increasing adsorbent dosage of 0.02 to 0.10 g which then it decreased and became constant. Thus, as the adsorbent dosage increases, the adsorption capacity also increases. This is due to the increasing number of active sites with greater surface area produce and more availability of adsorption sites on the adsorbent [16,17]. In this research, the optimum adsorbent dosage of 0.10 g was selected for further study since the increase of dosage did not show much difference, and the trend of this result is consistent with the previous research conducted by Rehman et al., [11].

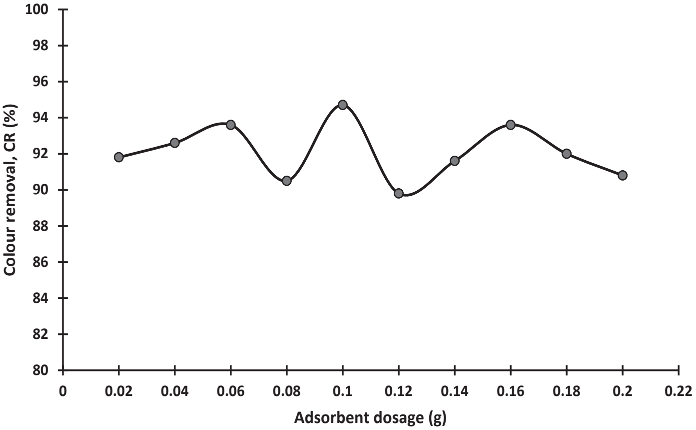


Figure 3: Effect of adsorbent dosage on MB removal by GL-HAC (Under operating condition of unadjusted pH (4.84), 1 hour contact time, 100 mg/L of MB solution, 303 K temperature and 100 mL of MB solution)

Effect of initial pH

The pH of the solution may influence the speciation of the dyes whether it increases or decreases the uptake of dye which is due to the surface charge of the adsorbent. Figure 4 shows the effect of pH variable from 3 to 11 on the percentage removal of the MB dye. As can be seen in the figure, the removal of MB increased slowly when the pH of the solution increased from 3 to 5 which then slightly started to decrease from pH 7 and then became constant up to pH 11 where no remarkable change was observed when increasing the pH of the solution to an alkaline environment. The lower adsorption of MB at acidic pH could be due to the competition of excess H^+ ions with MB cations for potential adsorption sites [18], while as the pH increases, the adsorption capacity of MB will increase whereby the increasing of pH solution will favour the adsorption of cation due to the increasing number of negatively charged site [3,4]. Moreover, the surface of the adsorbent was positively charged, since the $pH < pH_{PZC} = (4.30)$, contributing to repulsion of MB cations. Thus, the decrease in repulsion between the adsorbent surface and the dye will eventually increase the adsorption.

MB removal was not affected by pH within the range from 5 to 11 due to buffering effect of the adsorbent [19]. The optimum value of pH is 5 and this pH value was used for further experiment. This result is supported by the other researchers which stated that the optimum pH for MB removal is within the range of pH 5 to pH 6 [1,10].

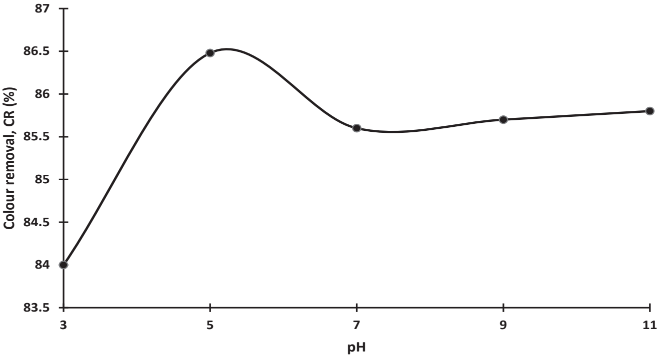


Figure 4: Effect of initial pH on MB removal by GL-HAC (Under operating condition of 0.10 g of GLAC, 1 hour contact time, 100 mg/L of MB solution, 303 K temperature and 100 mL of MB solution)

Effect of initial dye concentration and contact time

The effect of adsorption capacity with contact time is done by ranging the initial MB dye concentration from 50 to 300 mg/L as shown in Figure 5. The adsorption uptake of MB dye solution increases with time at the initial stage (0 – 30 minutes) before slowly reached the equilibrium (30 – 90 minutes). The increase in the driving force of mass transfer between the liquid phase and the solid phase is responsible for the improvement of adsorption capacity. High concentrations also resulted in an increase in the frequency of collisions between MB molecules and the GL-HAC surface. The interior surface of the GL-HAC was successfully penetrated by the MB dye, causing the absorption to stabilise. The adsorption uptake then increased once more, probably as a result of the MB dye's attempt to occupy more active adsorption sites of the GL-HAC, but the increase was too slight and not particularly significant.

Renugadevi *et al.,* [20] suggested that the first step leads to surface adsorption and the second step leads to intraparticle transport from bulk fluid to the external surface of the porous adsorbent. This trend agrees with the report of other investigators [21]. The stages of sorption of MB on activated carbon might be controlled by the diffusion process from bulk to the surface or the heterogeneous nature of the adsorbent [22].

From Figure 5, the amount of MB adsorbed by GL-HAC at equilibrium increases significantly from 5.8 to 59.4 mg/g as the initial MB concentration increases from 50 to 300 mg/L. This effect creates a strong driving force for dye molecules to overcome mass transfer resistance between aqueous and solid phases [4]. As the initial dye concentration increases, so does the collision rate between the MB dye and the GL-HAC surface, resulting in more MB cations being transferred to the GL-HAC surface.

The initial dye concentrations for 250 mg/L and 300 mg/L took 90 to 120 minutes to reach the required equilibrium state due to the higher driving force that allow MB dye molecules to penetrate deeper into the porous site within the interior surface of the GL-HAC to be adsorbed at active sites [10]. Hence, it is shown that initial dye concentration plays an important role in the adsorption capacity of MB by GL-HAC.

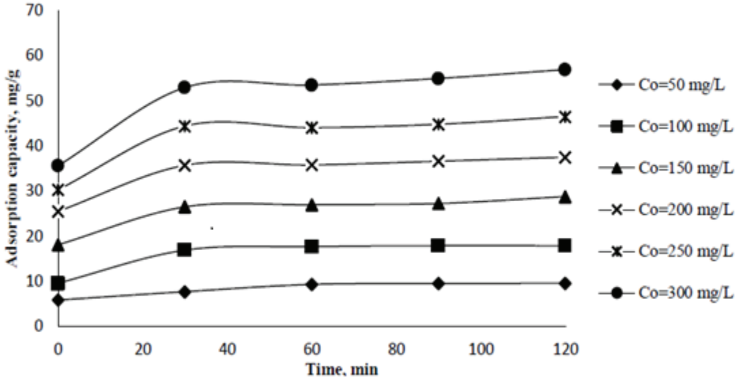


Figure 5: Effect of initial MB dye concentration on MB removal by GL-HAC (Under operating condition of 0.10 g of GLAC, pH 5 of MB solution, 303 K temperature and 25 mL of MB solution)

CONCLUSION

The Guava Leaves HCL Activated Carbon (GL-HAC) was successfully produced from agricultural waste using dried guava leaves as the raw material and hydrochloric acid as an activator. The outcome demonstrated that the GL-HAC has the potential to contribute as an alternative and affordable removal technology. The findings show GL-HAC has a comparatively high iodine number, low values for bulk density, ash content, and moisture content, as well as a reasonably high carbon yield. These physicochemical results demonstrate that GL-HAC is an effective type of activated carbon for dye adsorption. FTIR study shows that the functional groups of hydroxyl, amine, carbonyl, and carboxylic acid are present in GL-HAC. The activation by HCl led to more potential sites penetrating and occupying the activating agent, increasing the yield for GL-HAC to 89.90 %, which is considered high. It was discovered that 0.10 g of adsorbent dosage, an initial pH of 5, and 30 minutes of contact time were the best conditions for the removal of MB utilising GL-HAC. In addition, the increasing in the initial MB dye concentration boosted the trend for the adsorption process. The finding suggest that the GL-HAC offers more effective usage absorption capacity, which leads to superior performance as MB adsorption.

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