Development of nano-activated carbon and apply it for dyes removal from water

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ABSTRACT

The present study investigates the production of nano-activated carbon from banana peels mixed with nylon 6.6 and polyethene. The carbonization process was carried out by mixing accurate percentages of the banana peels with different ratios of nylon 66 and a suitable amount of potassium hydroxide. The fusion carbonization, without solvents, was used in this paper to decompose the nylon mixture, releasing amino and carboxylate roots that can easily react with the carbon chains. The prepared nano-activated carbon was characterized using different technologies, including SEM, AFM, FT-IR, and EDX technologies. The results showed the produced carbon has spherical particles with a pore size of 1.21 nm and a surface area of 1,071.7 m²/gm. Additionally, it was noticed, from the FT-IR spectrum, the prepared carbon does not contain any active groups, which means it is an inert material. X-ray analysis showed the new carbon is made from carbon (78.57%) and oxygen (21.43%). After optimizing the wavelength, the prepared carbon was used to adsorb methylene blue and Eirochrom black T dyes from solutions. The results showed the best equilibrium time, dose of carbon and concentration of dyes was 40–50 minutes, 0.04 g and 20 ppm, respectively.

Key words: dyes removal, nano-activated carbon, water

HIGHLIGHTS

- Nano-activated carbon was made from banana peels, nylon 6.6 and polyethylene.
- The new carbon has a surface area of 1071.7 m²/gm.
- Nano-activated carbon removed dye after 40–50 minutes.

1. INTRODUCTION

The global population is increasing at a fast pace, especially in the last centuries due to the enhancement in medication and the economy. This increase in the population was accompanied by a similar increase in industry and agriculture (Abdulhadi et al. 2019; Aqeel et al. 2020) and the urbanization process (use of the land) (Al-Jumeily et al. 2019; Farhan et al. 2019, 2021). Thus, as a result of this increase, the production of wastewaters either from agriculture (Hashim et al. 2018; Al-Saati et al. 2021) or industry (Zubaidi et al. 2019; Emamjomeh et al. 2020b). In fact, the effects of this increase are not limited to water pollution; these activities (agricultural and industrial ones) have seriously contributed to air pollution (Grmasha et al. 2020; Al-Sareji et al. 2021), which in turn resulted in global warming and droughts (Salah et al. 2020b, 2020c). For example, the recent investigations
are indicating serious changes in rain patterns (Zubaidi et al. 2020a, 2020b) and also a shortage in water (Salah et al. 2020a; Zubaidi Salah et al. 2020). Therefore, the need for water/wastewater treatment technologies increased more than ever before (Mohammed et al. 2020; Omran et al. 2021). For example, electrocoagulation (Hashim et al. 2019a), filtration (Abdulla et al. 2020), coagulation using chemical coagulants (Alenazi et al. 2020), or natural coagulants (Al-Saati et al. 2019), assisted filtration (Ahmedal et al. 2020), such as the combination of electrocoagulation and filtration (Emamjomeh et al. 2020a). Although some methods, such as electrocoagulation (Hashim et al. 2020c) and filtration (Hashim et al. 2021b), the adsorption method has brought more attention due to its affordability and effectiveness (Abdulraheem et al. 2020; Alenazi et al. 2020).

Several natural and waste materials are currently used to develop activated carbon, such as wood, bones, brown and dark coal, peels of coconuts, wastes of refinery products, sugar, and sludges of water treatment plants. Additionally, some synthetic materials, such as polymers, are currently used as adsorption media. Generally, high carbon content materials are favourable in the production of activated carbon as these materials do not require expensive processes to produce the activated carbon (Al-Hashimi et al. 2021). While low carbon content materials require a carbonization process at elevated temperatures ranging between 400 and 500 °C to remove unwanted materials (will be volatilized), then the materials pass through the activation process to increase the adsorption capacity. For example, Stoeckli (1990) developed activated carbon from charcoal by reacting it with sodium and potassium hydroxides and then activated the carbon at a temperature of 950–1,000 °C in the presence of argon gas. The developed carbon showed excellent adsorption properties and high surface area. Zhongfu (1991) investigated the preparation of activated carbon from the by-products of the furfural industry; the latter was dried first at a temperature between 80 and 200 °C for 2–8 hours. Then, the dried materials were carbonized at a temperature of 300–500 °C for 2–5 hours, followed by an activation process using water vapour at 800–1,000 °C. Zhong et al. (2012) used the peels of peanuts to prepare activated carbon; the peels were cleaned and heated using a microwave oven (500 W) for 8.9 minutes in the presence of phosphoric acid, a similar trial was made by Foo & Hameed (2009) to develop activated carbon from agricultural wastes. The authors used peels of pineapples to prepare the activated carbon; the peels were heated in a microwave oven (600 W) for 6 minutes in the presence of potassium hydroxide and potassium carbonate. The preparation of activated carbon was not limited to agricultural wastes, but petroleum wastes were also used in this field. For example, asphalt plant wastes with polymers to develop activated carbon. The mixture was subjected to thermal and chemical activations; the thermal activation included the addition of potassium hydroxide, while the heating process was carried out using conventional and microwave sources.

The activated carbon is divided into two types, according to the shape of its particles, which are powder activated carbon and spherical activated carbon; the spherical type is usually used in adsorption of gases, while the powdered one is used in liquids (Abatan et al. 2019). According to the activity, the removal of pollutants on the activated carbon is usually divided into four types, namely adsorption, surficial reactions, ion exchange, and mechanical filtration (Taha et al. 2016).

Generally, activated carbon is manufactured from materials with high carbon content, such as wood, brown and dark charcoals, petroleum charcoal, cellulosic materials, and some polymers (Lim et al. 2010). Wood is the most used material in activated carbon production because of its availability and high content of cellulose, lignin, and hemicellulose. Therefore, many studies have employed wood in different industries, such as petrochemical products, fuel and activated carbon (Ekpete et al. 2017). For example, Yamaguchi et al. (2019) produced activated carbon by adding some alkaline materials to the lignin, then treated the mixture thermally at a temperature of 600 °C. The produced activated carbon had excellent adsorption properties. Activated carbon was also produced from wood by adding sodium acetate to softwood and treated them thermally at a temperature of 450 °C; the products of the thermal treatment were activated using water vapour at 1,000 °C (Rahimian & Zarinabadi 2020).

The problem with activated carbon production is the appearance of different-sized pores in the structure of the activated carbon during the manufacturing process, which affects the adsorption process. Generally, the activated carbon has a large surface area ranging between 300 to 2,000 m²/gm and could reach 5,000 m²/gm in some cases (Ozdemir et al. 2014; Efievokhokan et al. 2019).

Dyes are considered one of the main sources of water pollution due to their wide use in different industries, such as textile, printing, petroleum, backing and food industries. Additionally, the amounts of dyes used in the mentioned industries are relatively high compared to other materials, where the wasted dyes represent 10 to 15% of the total industrial wastes. The problem of dye contamination of water is the variety of dyes' chemical
compositions. Indeed, this problem is one of the most significant problems at the current time due to the importance of water for humans and also for industry. Therefore, water pollution with dyes needs special attention and reliable treatment.

Many treatment methods have been used to remove dyes from water, including electrocoagulation (Hashim et al. 2019b), filtration (Abdulaheem et al. 2020), coagulation using chemical coagulants (Omran et al. 2019), or natural coagulants, and assisted filtration, such as the combination of electrocoagulation and filtration (Alyafei et al. 2020). Although many researchers claimed electrocoagulation as very efficient (Hashim et al. 2020b), affordable and able to eliminate many pollutants in a short time (Hashim et al. 2020a; Zanki et al. 2020), there are many serious facts reported about the performance of electrocoagulation, such as the effects of organic matter on the removal of pollutants (Abdulhadi et al. 2021; Hashim et al. 2021a). That forces the researchers to combine the electrocoagulation method with other technologies, such as ultrasonic (Al-Marri et al. 2020; Alnaimi et al. 2020). Therefore, the adsorption method has brought more attention.

The adsorption method is the most widely used in this field of treatment due to its high efficiency and affordable cost in comparison with other methods (Alyafei et al. 2020). The application of the adsorption method in removing dyes from water dates back to the 70 s of the last century when activated carbon and charcoal were used to remove acidic and alkaline dyes from solutions.

For example, El-Sayed et al. (2014) used the activated carbon from different sources to adsorb dyes from solutions. The used activated carbon was derived from charcoal, silica, alumina and kaolin. The results showed the excellent efficiency of the activated carbon to remove dyes, and it was noticed that the best equilibrium time ranged between 90 and 100 minutes, and the adsorption was exothermic and with low randomness. Abdullah et al. (2020) produced activated carbon from lemon leaves and used it for the removal of Eriochrome dye from water and found the equilibrium time was 30 to 35 minutes when the dose was 0.05 grams. The results showed the adsorption increases with the increase of the contact time, and the adsorption was exothermic. Additionally, nano adsorption is widely used in other applications, such as human health, sterilization and purification of water.

In this context, the present study aims at the production of activated carbon from banana peels by mixing them with nylon 6.6 and polyethylene. The main physical properties of the produced activated carbon will be examined, in this study, using SEM, AFM, FT-IR, and EDX technologies. Then the activated carbon will be used to adsorb methylene blue and Eirochrom black T (EBT) dyes from solutions.

2. EXPERIMENTAL WORK

2.1. Materials and chemicals

The used materials and chemicals in this study were banana peels, sodium hydroxide (0.1 N), hydrochloric acid (0.1 N), nylon polyethylene and methylene blue dye, EBT dye, [1-hydroxy-2 naphthol azo]-6-nitro, Naphthol-4-sulfonic acid sodium salt, and HOC10H6N=NC10H4(OH)(NO2)SO3Na. These chemicals were supplied from Aldrich and Fluka companies.

Several devices were used in this study to achieve the planned goals. A UV spectrophotometer (Shimadzu, 1650PC) and FT-IR spectrophotometer (Shimadzu, 1S-IR Affinity) were used to measure the wavelengths. Additionally, a shaking water bath (YCW012S), drying and incineration ovens, Atomic Force Microscope (AFM), Scanning Electron Microscope (SEM), Energy-dispersive X-ray spectroscopy and Fourier-transform infra-red spectroscopy (FT-IR).

2.2. Preparation of activated carbon

Initially, the peels of banana were cleaned and dried in their natural states; then they were ground into a powder. The powder was firstly placed in a clean steel container, then NaOH solution and nylon 66 were added to the container at different ratios 0.1:2:2, 0.2:2:2, 0.3:2:2, and 0.4:2:2 (NaOH: Nylon 66: Peels of banana). The mixtures were heated at 300 °C for 30 minutes with continuous stirring. This process is the initial carbonization, which was followed by heating the mixture at 550 ± 25 °C for 90 minutes. Then, the mixture was left to cool down. The produced activated carbon was washed several times with deionized water to remove the alkaline materials, then washed with hydrochloric solutions to remove unwanted ions. Finally, the sample was washed with deionized water to make sure that all unwanted materials were removed. The clean activated carbon was dried at a temperature of 110 °C before grinding it. The activated carbon powder was saved in airtight containers to be used later, see Figure 1.
2.3. Measurements of the activity of the activated carbon

2.3.1. Surface area

2.3.1.1. Measurement of the surface area using iodine adsorption method. The surface area of the prepared activated carbon was measured using the iodine adsorption from the solution. This method was used in this study because it is widely used in the literature. This method measures the surface area depending on the number of the adsorbed ions of iodine on 1 gram of activated carbon. In this study, 0.2 grams of the activated carbon was mixed with 2 mL of HCl (5%) and heated for 30 minutes, cooled down to room temperature, and mixed with 100 mL (0.1 N) of iodine solution mixed for 30 minutes using a shaker. Then, 50 mL of the mixed solution was filtered and pipetted out with anhydrous sodium thiosulfate (0.1 N) in the presence of a Starch indicator; the used volume of anhydrous sodium thiosulfate from the pipette. Iodine number is calculated by calculating the adsorbed weight of iodine by the activated carbon as follows:

\[ X = A - (2.2B \times V) \]
\[ A = N1 \times 12,695 \]
\[ B = N2 \times 126.93 \]

where \( X \) is the adsorbed weight of iodine (in mg), \( V \) is the volume of anhydrous sodium thiosulfate (0.3), \( N1 \) is the molarity of the iodine solution (0.1), and \( N2 \) the molarity of the anhydrous sodium thiosulfate (0.1).

Then, the iodine number (1N) is calculated as follows:

\[ 1N = \frac{1.6X}{MD} \]

where \( M \) and \( D \) are the weight of the activated carbon and the correction coefficient (which has a value close to 1), respectively.

2.3.1.2. Measurement of the surface area using methylene blue adsorption method. This method is also widely used in the literature; it was performed by mixing 0.1 mg of the activated carbon in a container and then 20 ppm of dye solution and mixed using a shaker for 25 hrs at room temperature. When no change in the colour is noticed, the solution is separated by leaving it to settle down. The separated solution is placed in the adsorption cell and measure the adsorption at a wavelength of 665 nm. Then, the adsorbed amount of dye on the activated carbon using the pre-prepared standard calibration curve. The latter was prepared by measuring the adsorption for different concentrations of methylene blue dye (5, 10, 15, 20 and 25 ppm) at the same wavelength (665 nm).
2.3.2. Measurement of the moisture content
The moisture content was measured by exposing the activated carbon to the atmosphere of the laboratory for 24 hrs and then drying it at 110 °C for 2 hrs. Then, the sample was left to cool down and the difference between the wet and dry weights measured.

2.3.3. Percentage of ash
A measured weight of the activated carbon (1 gram) was placed in a ceramic container and heated at 1,000 °C for 60 minutes and then cooled down to the temperature of the laboratory. The weight of the residual weight was calculated, which represented the weight of the ash.

2.3.4. Measurement of carbon density
A measured weight of the activated carbon was placed in a bottle, 5 mL capacity, and pressed gently to reduce and remove spaces between activated charcoal particles. The surface of the activated carbon in the bottle was levelled according to the mark on the side of the bottle, then the weight of carbon was measured, and the density was calculated as follows:

\[
\text{Density} \left( \frac{g}{cm^3} \right) = \frac{\text{Mass}}{\text{Volume}} \tag{5}
\]

2.3.5. Adsorption
The adsorption of EBT dye from the water was done using the developed activated carbon, taking into account the effects of several factors, such as the equilibrium time and dose of the adsorbent. Initially, the maximum wavelength was measured using the dye solution, and the results were used to develop a standard calibration curve. The results showed the maximum wavelength was 528 nm for the tested solution (containing 5–65 ppm). The relationship between the wavelength and concentration of dye is a linear relationship according to the Lambert-Beer law.

3. RESULTS AND DISCUSSION

3.1. Characterization of the nano-activated carbon
The characterization of the nano-activated carbon was done using different technologies, such as:

3.1.1. Fourier transformed infrared (FT-IR)
The FT-IR technology was used in this study to characterize the surface of the developed carbon. This test helps to identify the active groups on the surface of the carbon. The results of characterization are shown in Figure 2.

The FT-IR spectrum results show there are no active groups that may affect the adsorption process; thus, it can be concluded that the nano-activated carbon is chemically inert. This fact makes the developed activated carbon favourable for the adsorption process.

3.1.2. Atomic force microscope (AFM)
The results of the AFM analysis of the surface of the nano-activated carbon are shown in Figure 3, which shows a 3D picture of the activated carbon. It can be seen from this figure many sharp protrusions with heights that could reach up to 99.83 nm, which gives a high surface area for the carbon.

The SEM technology, with a magnifying ratio of 500, was used to study the structure of the new activated carbon; the results of the SEM are shown in Figure 4.

3.1.3. Scanning electron microscope (SEM)
It was noticed from the SEM images that the surface of the prepared activated carbon was rich in voids, cracks, cuts and different sizes of particles were noticed on the surface of the activated carbon, making the structure of this material porous. These voids, cuts and cracks are attributed to the effect of the arsenic chloride that acts to remove water from the fresh material (of cellulose, lignin, and hemicellulose) during the carbonization process.
Figure 2 | The spectrum of FT-IR for the nano-activated carbon.

Figure 3 | AFM analysis of the surfaces of the developed activated carbon.

Figure 4 | SEM analysis of the surface of the activated carbon.
3.1.4. Energy-dispersive X-ray spectroscopy

Additionally, X-ray technology was used to characterize the new activated carbon. The results of this analysis show in Figure 4 that the activated carbon’s chemical composition is made from carbon (78.57%) and oxygen (21.43%), which confirms the activity of the activated carbon as an adsorbent Figure 5.

The results of the measurement of sizes of pores and voids on the surfaces of the activated carbon that was prepared using the BJH (Barrett-Joyner–Halenda) method are shown in Table 1. It can be seen from these results that the average width of the pores is 1.21 nm, and the surface area (after carbonization) is about 1,071.7 m²/mg. The presence of hyperbranched in the structure of the activated carbon gives it an important feature: the captured ions or atoms in these pores will not be able to escape these pores.

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![Figure 5](image-url) | Results of the X-ray analysis of the activated carbon.

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<table>
<thead>
<tr>
<th>Table 1</th>
<th>BET measurement before and after mixing the banana peels, nylon 66 and polyethene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Banana peels only (BET)</td>
<td>Width of pores before carbonization</td>
</tr>
<tr>
<td>1,389.9 m²/mg</td>
<td>1.21 nm</td>
</tr>
</tbody>
</table>

It should be mentioned that the purpose of mixing polyethylene and nylon 66 with the peels of banana is decomposing of nylon and removal of hydrogen from activated carbon, and reconnection of the formed parts of the mixture with the free amine. These reactions occur in alkaline media and help to decompose the original materials and reform the produced parts.

3.2. Dye adsorption

Results of Table 2 and Figures 5–8 deliver essential information about the efficiency of the activated carbon. For example, it can be seen that adding polyethylene and nylon 66 to the banana peels increases the voids ratio on the surface of the new activated carbon. Previous studies showed that adding polyethylene alone to the mixture could decrease the pores on the surface of the peels of banana, while other studies stated that adding nylon 66 alone to the peels of banana increases the pores on the surface of the produced carbon (Awalgaonkar et al. 2020).

The present study showed that mixing polyethylene and nylon 66 and adding them to the peels of banana significantly increased the pores on the surface of the activated carbon, which was proved by the adsorption of methylene dye, as shown in Table 2. Additionally, the iodine number during the adsorption of iodine dye was good.
The measurement of activated carbon density showed that the density was not stable (increase and decrease) due to the difficult separation of groups and their carrier media.

An increase in the moisture indicates the activity of the produced carbon.

Table 2 | Values of key parameters obtained from mixing nylon 66, polyethene and banana peels

<table>
<thead>
<tr>
<th>Sample</th>
<th>The ratio of banana: KOH: nylon - poly</th>
<th>IN mg/gm</th>
<th>Methylene Blue 2 mg/gm</th>
<th>λ</th>
<th>Ash %</th>
<th>Density gm/cm³</th>
<th>Cinders %</th>
<th>Humidity %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1 : 2 : 0.1</td>
<td>297.7</td>
<td>24</td>
<td>665</td>
<td>0.264</td>
<td>0.25</td>
<td>0.68</td>
<td>12.8</td>
</tr>
<tr>
<td>2</td>
<td>1 : 2 : 0.2</td>
<td>417.7</td>
<td>50.7</td>
<td>665</td>
<td>0.390</td>
<td>0.52</td>
<td>0.61</td>
<td>26.4</td>
</tr>
<tr>
<td>3</td>
<td>1 : 2 : 0.3</td>
<td>599.1</td>
<td>61.87</td>
<td>665</td>
<td>0.465</td>
<td>0.27</td>
<td>3.02</td>
<td>13.5</td>
</tr>
<tr>
<td>4</td>
<td>1 : 2 : 0.4</td>
<td>620.6</td>
<td>76.7</td>
<td>665</td>
<td>0.243</td>
<td>0.294</td>
<td>1.61</td>
<td>14.7</td>
</tr>
</tbody>
</table>

Figure 6 | Methylene Blue dye adsorption on the produced activated carbon.

Figure 7 | Effect of nylon addition on the adsorption capacity of activated carbon.

Figure 8 | Effect of nylon addition on the density of activated carbon.
The results of this study, in summary, proved the activity of the produced carbon, its high porosity, its high efficiency in the removal of pollutants, and also indicated the produced activated carbon is free of toxic elements, such as sulfate and nitrogen.

Also, the results showed that by using different concentrations of activated charcoal before using the nanomaterials, the removal of methylene and EDT increased with the increase in the adsorbent dose.

The spectrum method was also used to assess the adsorption by the new activated carbon; the results of this method showed a linear relationship between the adsorption and the concentrations of the dye, as shown in Figure 9. Also, the results of this study showed the adsorption time for the new activated carbon ranged between 40 and 50 minutes, as shown in Table 3. Increasing the contact time was not useful because of the depletion of the pores on the surface of the new activated carbon.

\[ \text{Adsorption} = \frac{C_0 - C_e}{C_0 \times 100} \]  

\[ Q_e = \frac{C_0 - C_e}{M \times V} \]  

Temperature effects on the equilibrium constant, as listed in Table 6, show increasing the temperature decreases the adsorption capacity. The thermodynamic functions were calculated, as listed in Table 7, to


Define the nature of the adsorption system:

\[ \Delta G = \Delta H - T \Delta S \]  
\[ \log K = (\frac{-\Delta H}{2.303 \, RT}) + C \]  

where \( K \), \( R \) and \( C \) represent the highest adsorbed amount, gases constant, and integration constant, respectively.

The free energy value was calculated using the following equation:

\[ \Delta G = -RT \ln(Qe/Ce) \]  

where \( Ce \) and \( Qe \) represent the concentration at the equilibrium, and the adsorbed amount, respectively.

Figure 10 shows the best peak at which the dye is absorbed. Through the best wavelength (530 nm), the calibration curve (Figure 10) found that the adsorption process is subject to the pseudo-second-order equation according to the correlation coefficient R2 with a value of (0.997).
From the results obtained from the UV tests, it was found that the best peak at which the EBT dye was absorbed took place at a wavelength of 530 nm, and according to the calibration curve (Figure 10), the adsorption process is subjected to a second-order false equation (according to the correlation coefficient \( R^2 \), which has a value of 0.9978).

The negative sign of \( \Delta H \) indicates the adsorption process of the EBT dye on the new nano-carbon was an exothermic process; the adsorption physically occurs because it is less than 40 KJ/mole. Additionally, the change in the value of the free energy (\( \Delta G \)) indicates the reaction happens automatically (spontaneously), and the negative value of \( \Delta S \) indicates the reduction in the randomness of the process, as shown in Table 7.

Any adsorption media is subjected to depletion after a certain period of service; therefore, it is important to use an efficient monitoring tool to avoid the effects of the depletion on the performance of the adsorption unit. In this study, microwave sensing systems are recommended for the monitoring of depletion in the nanoparticle filters. The microwave sensing system was recommended because of its past successful applications; for example, it was used in the monitoring of health structures (Gkantou et al. 2019; Teng et al. 2019; Kot et al. 2021a), non-destructive sensing (Kot et al. 2021b; Omer et al. 2021), communications (Ryecroft et al. 2019a; Ryecroft et al. 2021), water pollution monitoring (Ryecroft et al. 2019b).

4. CONCLUSION

The present study focused on the development of nano-activated carbon from banana peels that mixed some additives (nylon 6.6 and polyethene). Additionally, this study aims at the application of the developed nano-activated carbon to remove dyes from water. The findings of this study indicated the nano-activated carbon has spherical particles with a pore size of 1.21 nm and a surface area of 1,071.7 m²/gm. Additionally, it was noticed that the prepared carbon does not contain any active groups, which means it is an inert material. Additionally, the results showed the nano-activated carbon has a good ability to remove dyes from water; the best equilibrium time dose of carbon and concentration of dyes was 40–50 minutes, 0.04 g and 20 ppm, respectively. It was also noticed that the adsorption efficiency decreases with the increase of the temperature; the adsorption was exothermic and with low randomness.

For future studies, a detailed investigation of the effects of solution pH on the adsorption of dyes by nano-carbon should be performed.

DATA AVAILABILITY STATEMENT

Data cannot be made publicly available; readers should contact the corresponding author for details.

REFERENCES


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