Available online at sjuoz.uoz.edu.krd

Science Journal of University of Zakho
Vol. 10, No. 4, pp. 159 –162, October-December 2022

p-ISSN: 2663-628X
e-ISSN: 2663-6298

PREPARATION OF ACTIVATED CARBON FROM HAWTHORN CORE STONES BY CHEMICAL ACTIVATION

Rami N. Saleem[a], Payman A. Abduljabar[a, *], Samara S. Faraj[a]

[a] Dept. of Environmental Sciences, Faculty of Sciences, University of Zakho, Kurdistan Region of Iraq
(payman.abdulsatar@uoz.edu.krd)

Received: *14 Jun., 2022 /* ***Accepted:*** *31 Jul., 2022 /* ***Published:*** *09 Oct., 2022* ***https://doi.org/10.25271/sjuoz.2022.10.4.954***

ABSTRACT:
The objective of this study is the characterization of activated carbon prepared from hawthorn core natural residue which is a vegetation waste. By using zinc chloride ($ZnCl_2$) as a chemical agent 40 for 25 hours at (25 °C) as a raw material in this work. The best conditions were determined to be a carbonization temperature of 400 °C for 1 hour and a maximum yield is 82 %. Different operational factors such as carbonization time and temperature were used to prepare activated carbon from hawthorn core stones, pH, ash content, density, humidity content, conductivity, iodine number, and methylene blue dye absorption were all investigated in the generated activated carbon. The obtained characterization of the activated carbon was performed by using scanning electron microscopy (SEM), X-ray diffraction (XRD) and energy dispersive X-ray (EDX). Finally, the activated carbon synthesized in this study acquired good properties that enable it to play an important function in a variety of environmental applications, including eco-protection, water treatment, and wastewater treatment and it was compared to a commercial reference sample from the B. D. H Company in industrial applications.

KEYWORDS: Activated Carbon, Chemical activation, hawthorn core, Zinc chloride.

1. INTRODUCTION

Activated carbon is mainly found in amorphous carbonaceous materials with high porosity and surface area (ElShafei and *et al*, 2014). It performs well in a variety of environmental applications, including eco-protection, water treatment, and wastewater treatment (Juan and Qiang, 2009). In practice, it is made from any carbonaceous substance containing a significant quantity of natural or manufactured carbon element. Many criteria, such as availability, cost, purity, and manufacturing technique, can have a significant impact on the choice of the starting material or precursor, which are both utilized to manufacture activated carbon. Carbonizing precursors in a static environment shorten the physical activation by bringing out the non-carbonic components, which is then followed by activation within the cell. (Liu *et al*, 2016; Hayashi *et al*, 2000; Lua and Guo, 2000).

Due to its highly developed porous structure and high adsorption capacity, activated carbon plays a significant role in adsorption and catalytic processes. Activated carbon preparation has also been the subject of numerous investigations (Kumar and Jena, 2015; Jain *et al*, 2016). Among them, hawthorn core is a well-known plant that thrives in the desert. Mountains near Zakho, Kurdistan Region, Iraq, were used as herbal medication to heal ailments and bladder infections. Hawthorn core is also high in vitamin C, which is essential for human immunity.

Hawthorn core stones were employed as a raw material in this study to optimize the manufacture of activated carbon. The chemical agent zinc chloride was utilized for chemical activation ($ZnCl_2$). Temperature and the carbonization process had their own impacts that needed to be investigated.

2. MATERIALS AND METHODS

2.1 Materials

The natural raw material, hawthorn core, was harvested in Iraq's Kurdistan Region's Zakho Mountains. BDH chemical firm, UK, provided iodine (0.1 N), potassium iodide (> 98 %), $Na_2S_2O_3$ (> 99 %), methylene blue dye (20 ppm), HCl (0.1 M), commercial activated carbon, and zinc chloride ZnCl> 98%).

2.2 Methods

2.2.1 Preface: The synthesis of activated carbon from hawthorn core stones was investigated in this work. These stones were first cleaned with deionized water to remove pollutants before being dried in a 100 °C oven for 24 hours.

2.2.2 Preparation of Activated Carbon: As an activation agent, 20g of dry precursor was combined with 250 mL of 40 % $ZnCl_2$ (Ziyad *et al*, 2018). At 25 °C, 150rpm stirring was carried out for 24 hours, followed by filtration and washing with deionized water. Finally, the sample was dried at 100 °C. This was then carbonized in a muffle furnace (MTF12/38/400) at a steady rate of 5 °C /min. (1-3 h) and (400-700 °C) were employed as time and temperature, respectively. When the timer ran out, the sample was removed from the oven and allowed to cool. It was steeped in (0.1 M) HCl solution (at 25°C) for 24 hours after cooling to remove the left over zinc chloride. Filtration and washing with deionized water were performed on the sample until the filtrate reached a pH of 6.5-7Tan *et al*, 2007). The product was then packaged. After drying for 24 hours at 110°C, the samples were placed in a glass vial to analyze.

* Corresponding author

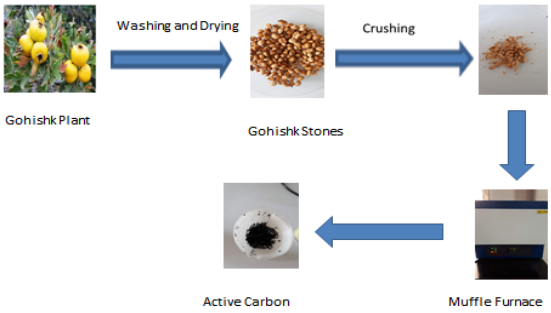


Figure1. Preparation of activated carbon from hawthorn core stones

2.3 Activated carbon measurements

2.3.1 Activated Carbon Yield: The per cent of activated carbon was estimated as follows:

$$\textbf{Yield percent} = \textbf{Wt/W0} \times \textbf{100} \quad (1)$$

Where Wt represents the weight (g) of the dry final sample of activated carbon and W0 is the weight (g) of the dry preface.

2.3.2 Activated Carbon Density: a specified amount of activated carbon is placed in a graduated cylinder or a volumetric bottle of 5 mL so that the activated carbon occupies its volume and the carbon particles are at one level, the carbon in the cylinder is weighed using a sensitive scale, and the density is measured.

$$g/cm3density = \text{weight/volume} \quad (2)$$

Where D is the bulk density, M is the dry activated carbon mass, and V is the dry activated carbon cylinder volume (mL).

2.3.2 Total Ash Content: (Masmoudi *et al*, 2021)

One gram of activated carbon was placed in a crucible, which was then placed in an electric oven at 1000 °C for 3 hours, after which the sample was cooled and weighed using a sensitive balance, and the percentage of ash was calculated using the difference in weights.

$$Ash\% = Wc - Wt/W0 \quad (3)$$

Where Wc denotes the weight of an ash-filled crucible (g), Wt denotes the weight of an empty crucible (g), and W0 is the weight of the previously utilized activated carbon (g)

2.3.4 Humidity Content: (Ariany *et al*, 2018)

The humidity content of a given weight of the sample was measured by heating it in a prepared oven for 3 hours at 150 °C and then calculating it as follows

$$Humidity\% = Wc - Wt/W0 \times 100 \quad (4)$$

Wc stands for the weight of the crucible with the original sample (g), Wt stands for the weight of the crucible with dry activated carbon (g), and W0 stands for the weight of the original sample (g).

2.3.5 pH Measurement: For 1.5 hours, a half gram of the sample was immersed in 50 mL of deionized water and stirred at 150 rpm. Finally, a pH meter was used to determine the pH of the slurry (Cyber Scan 510 pc).

2.3.6 Iodine Number Measurement (Machrouhi *et al*, 2019)

To determine the activated carbon's iodinenumber. Weigh one gram of dry activated carbon and place it in a 250 mL conical flask.

To the flask, pipette (10) mL of a 5 % hydrochloric acid solution. The contents of the beaker are heated till they boil for half an hour, and then cooled to room temperature. pipette is added to the beaker 100 mL of iodine solution at laboratory temperature (1.0 N). After putting the flask in the electric shaker for half an hour, the contents are filtered. Using a clean, dry filter paper, discard about 20-25 mL of the filter at the start of the filtration process and collect the rest in a clean beaker. Transfer 50 mL of the filtrate to a 250 mL conical flask and titrate with a standard solution.

From the activated carbon models, the weight of the adsorbed iodine is determined using the equation:

$$X = A - [2.2\ B \times mL\ of\ thiosulfate\ used] \quad (5)$$

$$A = N1 \times 12693 \quad (6)$$

$$B = N2 \times 126.93. \quad (7)$$

So:

X = weight of iodine in mg adsorbed by activated carbon

N1 = titer of iodine solution (0.1 N)

N2 = sodium thiosulfate titer (0.1 N)

$$In=D/M \quad (8)$$

So: M = weight of the used activated carbon model

D = Correction Factor.

The normalcy of Na2S2O3 is represented by N, the atomic weight is represented by (126.9), and the mass of the activated carbon is represented by M.

2.3.7 Conductivity Measurement: For conductivity testing, 1 g of activated carbon was mixed with 100 mL of deionized water for 30 minutes while stirring at 150 rpm. At 25 °C, electrical conductivity was measured using a (Wagtech international) conductivity meter with micro Siemens per meter (µS/cm) values.

2.3.8 Adsorption of Methylene Blue Dye: The adsorption capacity of methylene blue dye from its aqueous solution is used to calculate carbon's outer surface area. Taking (0.1 g) of activated carbon and placing it in a conical flask is the basis for this approach. It is then placed in the shaker with a known amount of (20 ppm) methylene blue added to it at room temperature for 24 hours, and if necessary, when the colour fades, a new amount is added. The clear solution is obtained and placed in an absorption cell and measured absorbance at a wavelength (665 nm) which a dye is absorbed methylene), then the concentration of the dye is calculated. By taking different concentrations of dye solution (5, 10, 15, 20, 25 ppm) and measuring its absorbance at the wavelength (665 nanometres) and drawing a line diagram between the values of absorption and concentration, the dye which is removed from its aqueous solution is calculated using the curve standard prepared for this purpose.

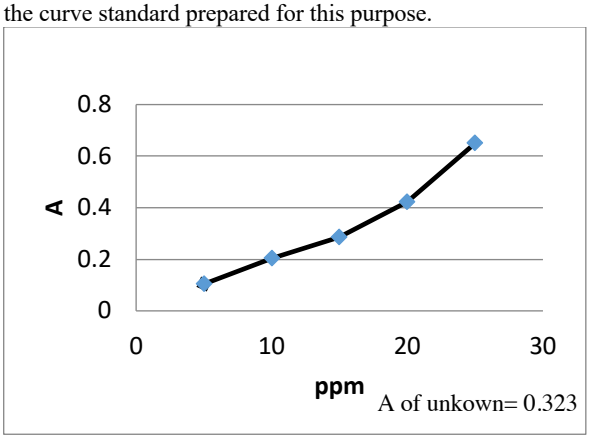


Figure 2. Calibration curve of prepared activated carbon

3. RESULTS AND DISCUSSION

3.1 Effect of temperature on the carbonization process

The influence of carbonization temperature on the yield of activated carbon that has been prepared from 400°C to 600 °C, the temperature of carbonization rises dramatically. The yield peaked at 82 % after 60 minutes at 400°C, and then began to decline after 2 hours, reaching 34% at the same temperature, suggesting that it was totally burned. However, at 600°C, the yield was at its highest. After 1 hour, the value was increased by 36 %. After 2 hours, it dropped dramatically to 16 %. To put it in another way, as a stable structure was developed, the yield rate decreased. It has been observed that when the temperature rises, the adsorption capacity decreases (Algidsawi, 2011).

High activation temperatures, on the other hand, were reduced in a noticeable manner in microspore volume.

As a result of the burning of some microspore walls to manufacture microspore, the specific surface area of the resulting activated carbon will be reduced. Reduced temperatures, on the other hand, cause an activated carbon's micro porosity to form rapidly, resulting in a large specific surface area (Yan, and Yang,2008). This supports the theory that the higher the temperature, the broader the microspores (Bae *et al*, 2018). As a result, 400 °C temperature is seen as the optimum choice.

3.2 Effect of Carbonization Time

The carbonization temperature treatment was done over several hours, ranging from 1-3 hours. As a result, it was determined that the optimal period at the best temperature (400 °C) resulted in an 82% maximum yield percentage. However, after two or three hours, the percentages dropped to 70% and 37%, respectively. Furthermore, increasing the activation time reduces the microspore volume of activated carbon. While the macrospore volume increased, the mesoporous volume remained stable (Bae *et al*, 2018). In conclusion, all trials were started at the optimum temperature of 400 °C, with a heating duration of 1hour.

3.3 Characterization of Activated Carbon Prepared at Optimum Conditions

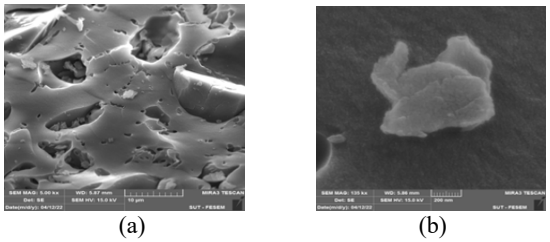
Table 1. Characterizations of prepared activated carbon and commercial activated carbon.

Characteristic	Prepared	Commercial
Yield (%)	82	
Bulk density (g/ml)	0.43	0.302
Ash content (%)	5	3.2
Humidity Content (%)	5	8.05
pH	5	6.8
Iodine number (mg/g)	627.04	889.7
Conductivity (µs/cm)	850	430
Methylene Blue Dye	17	

The activated carbon obtained from hawthorn core has a density of 0.43 g/mL. When the density of the activated carbon decreases, the pores grow larger and the surface area becomes more available. As a result, as density increased, the adsorption capacity decreased correspondingly. Another feature of the preparation is the ash level of activated carbon with a satisfactory value of 5 %, indicating its high purity. On the other hand, a high ash level has a deleterious effect on activated carbon, reducing mechanical strength and hence affecting adsorption capacity (Abdullah *et al*, 2001). The activated carbon had a 5 percentage-point humidity content. It did not dilute the activated carbon in any way. Influence on adsorption capacity the pH of the activated carbon suspension in water is represented by this acidity (pH) of activated carbon. As can be seen, lowering the pH causes an increase in capacity (Senthilkumaar *et al*, 2019). This pH was measured at 5 in this study. This activation procedure increased the surface area and porosity of the material, as well as the surface basicity. To conduct the activation of carbon to acquire the dry weight, the additional moist weight of the carbon was required. The activated carbon's iodine number was estimated to be 627.04 mg/g. The activated carbon's ability to adsorb low molecular weight a chemical was demonstrated when it was used to remove iodine. The more iodine that was extracted by the activated carbon, the more surface area was available. The increased iodine adsorption resulted in a large surface area and microspore structure (Baccar *et al*, 2009). At 35 °C, the activated carbon had a conductivity of 850 µs/cm. The activated carbon absorbance value was determined to be 17 mg/g, indicating that it has a strong adsorptive ability. As a result, the methylene blue dye was absorbed by the activated carbon, and the dye's blue colour faded process of adsorption. This adsorption capacity provided the activated carbon with its large area surface.

3.4 Textural Characterization by SEM

A SEM image of prepared activated carbon sample is shown in Figure 3a and b. It can be seen from the micrographs that the activated carbons are relatively heterogeneous. Thus, it is used in a variety of environmental applications used as fertilizer, absorbent of impurities from water, and wastewater treatment.



(a) (b)

Figure 3. SEM micrographs of prepared activated carbon

3.5 XRD analysis

Figure 3 illustrates the X-ray diffraction profile of the active carbon. This activated carbon exhibit very broad diffraction peaks and the absence of a sharp peak reveals a predominantly amorphous structure (Wang *et al*, 1997. There are two broad diffraction peaks around 2θ = 24° and the other one is less broad 7. The appearance of the peak at around 24° at activation temperature (400 °C) signifies an increasing regularity of crystalline structure (Kasaoka *et al*, 1989).

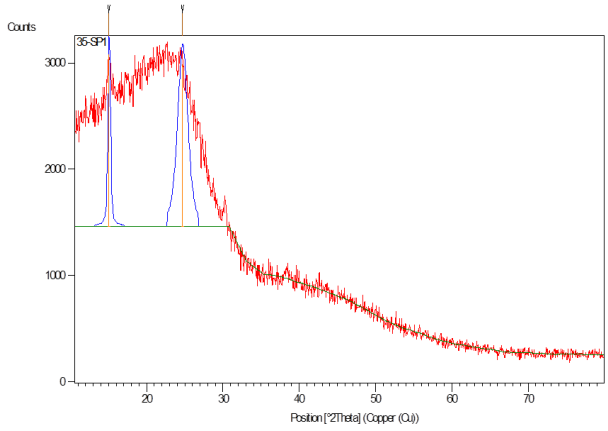


Figure 4. X-ray diffraction profile of prepared activated carbon

3.6 EDX analysis

The EDX is an analytical technique used for the analysis of elemental composition and chemical characterization of samples. The EDX spectra of activated carbon showed that on the surface of all the prepared activated carbon samples, carbon, and oxygen were mostly present. Carbon in the samples had a greater ratio than that of oxygen. The EDX spectra also showed the presence of other elements, such as nitrogen, calcium and chlorine. It is clear from the chart as provide the activated carbon does not include a significant amount of impurities.

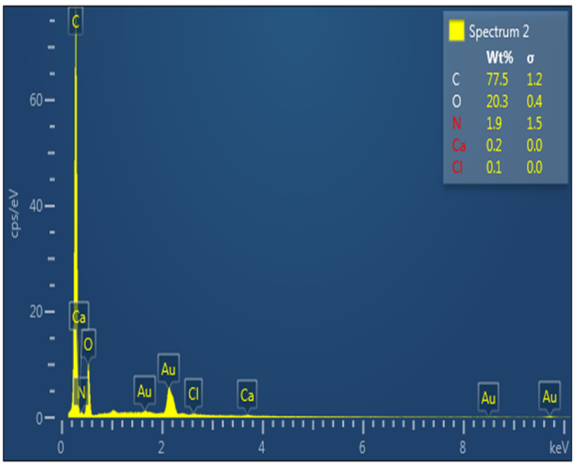


Figure5. EDX analysis of prepared activated carbon

4. CONCLUSION

Different operational factors such as carbonization time and temperature were used to prepare activated carbon from hawthorn core stones using zinc chloride as a chemical agent. They were investigated to find the best circumstances, and it was discovered that $ZnCl_2$ was an effective activating agent for creating activated carbon with an 82 % yield in 60 minutes at 400 °C. As it is mentioned above, it is crystal clear then the characterization of new prepared activated carbon has a good pores and regular patron also showed a high degree of purity as conformed by EDX technique.

ACKNOWLEDGEMENTS

The authors would like to thank the Environmental Sciences Department for providing the necessary materials and laboratory for this research.

REFERENCES

Abdullah, A. A. Kassim, Z. Zainal, M. Hussien, and D. Kuang. (2001) "Preparation and characterization of activated carbon from gelam wood bark (Melaleuca cajuputi)," Malaysian J. Anal. Sci., vol. 7, no. 1, pp. 65–68.

Algidsawi, A. "A Study of ability of adsorption of some dyes on activated carbon from date's stones," Aust. J. Basic Appl. Sci., vol. 5, no. 11,pp. 1397–1403, 2011.

Ariany Zulkania*, Ghina Hanum F., and Amelia Sri Rezki. (2018).Corresponding author:ariany.zulkania@uii.ac.id the potential of activated carbon derived from bio-char waste of bio-oil pyrolysis as adsorbent . MATEC Web of Conferences 154, 01029.

Baccar, R. J. Bouzid, M. Feki, and A. Montiel. (2009)."Preparation of activated carbon from Tunisian olive-waste cakes and its application for adsorption of heavy metal ions," J. Hazard. Mater., vol. 162, no. 2–3, pp. 1522–1529.

Bae, W. J. Kim, and J. Chung. (2014). "Production of granular activated carbon from food-processing wastes (walnut shells and jujube seeds) and its adsorptive properties," J. Air Waste Manage. Assoc., vol. 64, no. 8, pp. 879–886.

ElShafei, G. I. ElSherbiny, A. Darwish, and C. Philip. (2014). "Silkworms' feces-based activated carbons as cheap adsorbents for removal of cadmium and methylene blue from aqueous solutions," Chem. Eng. Res. Des., vol. 92, no. 3, pp. 461–470.

Hayashi, J. A. Kazehaya, K. Muroyama, and A. Watkinson. (2000). "Preparation of activated carbon from lignin by chemical activation," Carbon N. Y., vol. 38, no. 13, pp. 1873–1878.

Jain, A. R. Balasubramanian, and M. Srinivasan. (2016). "Hydrothermal conversion of biomass waste to activated carbon with high porosity: A review," Chem. Eng. J., vol. 283, pp. 789–805..

Juan Y. and Q. Ke-Qiang. (2009). "Preparation of activated carbon by chemical activation under vacuum," Environ. Sci. Technol., vol. 43, no. 9, pp. 3385–3390.

Kasaoka, S. Y. Sakata, E. Tanaka, R. Naitoh. (1989).Int. Chem. Eng., 29, 101.

Kumar A. and H. Jena. (2015). "High surface area micro porous activated carbons prepared from Fox nut (Euryale ferox) shell by zinc chloride activation," Appl. Surf. Sci., vol. 356, pp. 753–761.

Liu, Z. Y. Huang, and G. Zhao. (2016)."Preparation and characterization of activated carbon fibers from lique wood by ZnCl2 activation," BioResources, vol. 11, no. 2, pp. 3178–3190.

Lua A. and J. Guo. (2000). "Activated carbon prepared from oil palm stone by one-step CO2 activation for gaseous pollutant removal," Carbon N. Y., vol. 38, no. 7, pp. 1089–1097.

Machrouhi, A.; Alilou, H.; Farnane, M.; El Hamidi, S.; Sadiq, M.; Abdennouri, M.; Tounsadi, H.; Barka, N. (2019).Statistical optimization of activated carbon from Thapsiatran stagana stems and dyes removal efficie using central composite design. J. Sci. Adv. Mater. Devices, 4, 544– 553.

Masmoudi, M.; Baccouche, A.; Borchani, M.; Besbes, S.; Blecker, C.; Attia, H. (2021). Physico-chemical and antioxidant properties of oils and by-products obtained by cold press-extraction of Tunisian Opuntia spp. seeds. Appl. Food Res., 1, 100024..

Senthilkumaar, S. P. Kalaamani, K. Porkodi, P. Varadarajan, and C (2019). Subburaam, "Adsorption of dissolved reactive red dye from aqueous phase onto activated carbon prepared from agricultural waste," Bioresour. Technol., vol. 97, no. 14, pp. 1618–1625,20.

Tan, I. B. Hameed, and A. Ahmad. (2007). "Equilibrium and kinetic studies on basic dye adsorption by oil palm fibre activated carbon," Chem. Eng. J., vol. 127, no. 1–3, pp. 111–119.

Wang, S.B. G.Q. Lu, Ind. Eng. Chem. Res. (1997).36, 5103

Xu, B.Y. Chen, G. Wei, G. Cao, and H. Zhang. (2010). "Activated carbon with high capacitance prepared by NaOH activation for supercapacitors," Mater. Chem. Phys., vol. 124, no. 1, pp. 504–509.

Yan, Y. and L. Yang. (2008.) "Preparation of activated carbons from sawdust by chemical activation," Adsorpt. Sci. Technol., vol. 26, no. 7, pp. 533–543. .

Ziyad. A. T, M. Salim. H , and P. Abdulsatar. A . (2018). Preparation of activated carbon from helhelok stones by chemical activation" Science journal of university of zakho., vol. 6, no. 3, pp. 100–103.