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Preparation and Characterisation of Activated Carbon from Pomegranate Waste Using Chemical with Microwave Activation

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Abstract

This study presents the preparation method and characterisation of activated carbon (AC) from pomegranate waste by using chemical with microwave activation. Pomegranate peels in powder form were impregnated in 40–60 %wt sulphuric acid with different ratios (1:3 of pomegranate to sulphuric acid) and times (2–6 h) for chemical activation. The impregnated samples were activated in microwave under inert atmosphere at different powers (450–900 W) and activation times (7–21 min) to produce AC. Design-Expert software (version 13) was selected with the I-optimal method to analyse the effects of AC preparation variables. The preparation variables, including acid concentration, impregnation time, impregnation ratio, microwave power and activation time, with their effects on the adsorption capacity of methylene blue dye, were investigated. On the basis of the obtained maximum methylene blue number, the AC and raw material were characterised via Brunauer–Emmett–Teller test, field emission scanning electron microscopy with energy-dispersive X-ray analysis, Fourier transform infrared spectroscopy and X-ray diffraction analysis. Results indicate that the AC produced from pomegranate peels achieves a sufficient surface area of 1431.8 m^2/g and a total pore volume of 1.6236 cm^3/g with a spongy and amorphous nature with minor crystal characteristics. It can be used as a low-cost product with desirable surface characteristics. These results promote the use of AC prepared from pomegranate waste as an obtainable and low-cost adsorbent.

Keywords: *Pomegranate peels; Activated carbon; Sulphuric acid; Microwave activation*

1. Introduction

Activated carbon (AC) is a material that is similar to black charcoal; it exhibits a good pore structure and high surface area with significant mechanical durability [1]. AC is widely used in different applications, such as gas cleaning and separation [2], water purification [3-5], industrial effluent treatment [6-9] and pharmaceutical [10], catalyst, supercapacitor and electrode [11] applications. AC is popular because of its versatility in adsorbing various types of contaminants that range from dyes [4, 5, 8, 12], heavy metals [3, 6, 13], phenolic compounds [14], antibiotics [10], hydrogen sulphide [15] and carbon dioxide [16].

AC derived from agricultural by-products is a viable and affordable substitute for conventional AC derived from hardwood or petroleum-based sources. Agricultural by-products are widely accessible and abundant, making them desirable as raw materials for the manufacture of AC. Given its high adsorption capacity, AC has a substantial specific surface area, i.e., the total surface area of a material per unit of mass. Considering its large surface area, AC can be applied to processes, such as adsorption, which involves the removal of contaminants from gases or liquids. [17]. These

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properties are significantly affected by raw material selection, activation process and activating substance [18]. Therefore, many researchers have investigated AC production from a variety of organic materials that are affordable and sustainable precursors, such as *Alhagi* [14], buckthorn twigs [19], plum seed [6], water hyacinth [20], saw dust [3, 21], *Cycas* leaves [12], apricot seeds [10], date palm seeds and fronds [8, 22], date stones [23, 24], orange peel [25], cassava peel [26], banana peel [27], garlic peel [28], potato peel [29], waste tea [30, 31], rice husk [32], walnut shell [33], oil palm fibre [34] and coconut shell [35].

Pomegranate is one of the most well-known fruits due to its rich nutritional contents and delicious flavour. It has peel and internal edible seeds. Pomegranate peel is a nonedible part that comprises about 5%–15% of its weight; it is considered a waste product without commercial benefits [36]. Pomegranate peel is a low-cost and eco-friendly precursor that can be used for AC preparation; the produced AC can be utilised to remove pollutants from gases and aqueous solutions [37-40]. The preparation of AC from various precursors can be conducted using physical and chemical activation techniques. Physical activation is performed in two stages. The first stage comprises the pyrolysis process of the carbonaceous material; the second stage involves gasification with oxidant agents, such as CO_2, steam or a combination of CO_2 and steam, at high temperatures [41, 42]. Chemical activation is achieved by impregnating a carbonaceous material with different chemical agents, followed by either conventional or microwave carbonisation [43, 44]. Chemical activation necessitates the use of activating agents, such as sulphuric acid [6, 45], phosphoric acid [4, 10, 19, 29], zinc chloride [46-48] potassium carbonate [49] and potassium hydroxide [14]. Microwave carbonisation is preferred due to its heating method (low temperature), lower gas consumption, shorter carbonisation time, higher productivity and higher product specification (surface area, pore diameter and pore volume) [50]. However, conventional heating or carbonisation is performed using a conventional furnace, which requires high temperature with a long period of heating (high energy consumption) [18]. In this regard, microwave is a renewable alternative to conventional carbonisation [51]. Given its many benefits over conventional heating methods, the use of microwave in the synthesis of AC is garnering growing study interest. Its benefits include an internal heating rate, a high heating rate, selective heating, effective process regulation, short heating time that results in lower energy consumption and indirect contact with the heat source [10, 29, 52].

In addition, microwave heating creates a uniform temperature distribution, reducing the temperature gradient in the material and leading to the uniformity of pore size [50].

The objective of the current research is to prepare AC by using pomegranate peels as a natural and low-cost source and microwave activation as an efficient technique for preparation. The effects of the parameters and conditions for preparation, including impregnation ratio (IR), activation energy and activation time, on the specific surface are investigated to achieve optimum conditions. Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM) with energy-dispersive X-ray (EDX) analysis and Brunauer–Emmett–Teller (BET) test are used for the characterisation of pomegranate peels and the AC produced under optimum conditions.

2. Materials and Methods

2.1. Preparation of AC

Pomegranate peels were collected from a local plantation area in Baghdad. Firstly, the pomegranate peels were chopped to 1–2 cm pieces, washed with plenty of water to clean dirt and then dried for 4 h at 105 °C by using an air dryer. The dried material was crushed and sieved to the required size (2 mm). The sample (approximately 20 g) was impregnated into sulphuric acid (40–80 wt%) for 2–6 h at room temperature with different IRs: 1–3. Then, the samples were filtered to remove excess acid from each and the microwave method was used for AC preparation [4]. Each sample was poured into a flask and fixed in a microwave oven under a nitrogen flow of 200 cm^3/min at different activation levels (450–900 W) and activation times (7–21 min). After activation, the samples were allowed to cool under nitrogen atmosphere. The final product was washed with hot water and filtered until its pH reached 6–7. Finally, the product was dried at 105 °C for 4 h, pulverised and sieved to the required size range. The schematic of the AC preparation steps is presented in Figure 1.

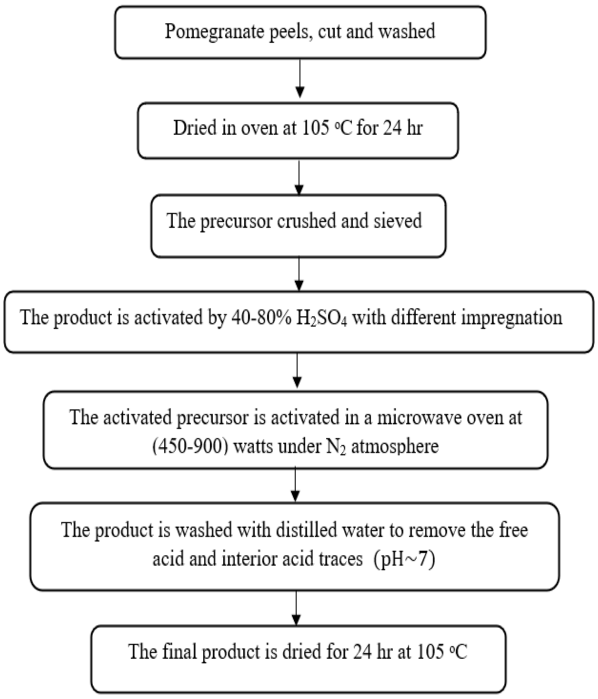

Fig. 1. Schematic of AC preparation from pomegranate peels

2.2. Methylene Blue Test

Methylene blue dye powder was used to prepare methylene blue stock solution (50 mg/L) by dissolving 50 mg of the powder in 1 L of distilled water. Ten flasks (10 cm^3) were used to prepare samples with the desired concentrations of 0.025, 0.05, 0.1, 0.2, 0.4, 0.5, 0.8, 1, 2 and 4 mg/L [7]. Ultraviolet–visible (UV–Vis) spectroscopy (wavelength = 660 nm) was used for the calibration curve, as shown in Figure 2. To determine the concentration after adsorption, 0.1 g of each of the prepared AC sample was shaken for 60 min with 50 mL of stock solution. Absorbance was measured via UV–Vis spectroscopy at wavelength λ = 660 nm. Adsorption capacity, given as methylene blue number (MBN), was calculated using Equation 1.

$$MBN = \frac{(C_o - C_e)V}{W}, \qquad \dots(1)$$

where

C_o is the initial concentration (mg/L),

C_e is the equilibrium concentration (mg/L),

MBN is the adsorption capacity of the dye to AC weight (g/g),

V is the volume of the dye sample (mL),

W is the AC weight (g).

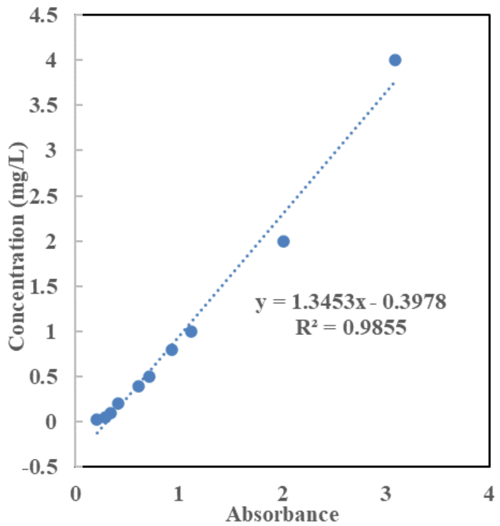

Fig. 2. Methylene blue calibration curve

3. Experimental Design

Design-Expert software (version 13.0) is a helpful statistical programme for creating models and designing experiments by considering the interactions amongst different parameters. The effects of acid concentration, IR with impregnation time and microwave power with activation time as independent variables on methylene blue dye adsorption capacity were investigated using the I-optimal method, as indicated in Tables 1 and 2.

Table 1,
Independent variables of AC preparation

Symbol	Variables	Level		
A	Acid concentration (%)	40	60	80
B	IR (ratio)	1:1	1:2	1:3
C	Impregnation time (h)	2	4	6
D	Activation power (W)	450	720	900
E	Activation time (min)	7	14	21

Table 2,
Design-Expert variables and results by using the I-optimal method

Run #	A: Concentration %	B:IR ratio	C: Impregnation time h	D: Power W	E: Activation time min	MBN g/g
1	80	3	4	720	7	24.713247
2	40	3	6	450	7	24.356002
3	40	1	2	900	7	25.13029
4	80	1	6	450	21	25.18612
5	80	1	6	900	7	25.126254
6	40	1	6	900	7	25.05159
7	60	1	4	720	14	25.120873
8	60	1	4	720	14	24.964818
9	40	1	2	450	21	24.804054
10	80	1	2	900	14	24.928629
11	60	3	6	900	14	23.626244
12	80	2	6	450	7	24.938584
13	40	2	4	720	14	24.904952
14	**60**	**3**	**6**	**450**	**21**	**25.198227**
15	40	3	2	900	21	23.677366
16	60	3	2	450	14	24.103826
17	80	1	2	450	7	24.520869
18	60	2	4	900	7	24.376922
19	40	2	4	720	14	25.188138
20	40	1	6	450	14	25.188138
21	40	1	6	900	21	24.522887
22	80	2	4	900	21	23.090949
23	80	2	2	720	21	24.524905
24	40	1	2	450	7	23.678711
25	60	2	4	900	7	24.574008
26	60	2	2	720	7	25.18612
27	60	1	4	720	14	25.079841
28	40	3	4	720	7	24.524232
29	80	3	2	900	14	23.694182
30	40	3	4	450	14	24.470353

4. Results and Discussion

4.1. BET Test

Table 2 clearly shows that the AC sample (run number 14) produce the maximum MBN value, indicating that this sample achieves the maximum specific surface area and pore volume. The BET technique (HORIBA, SA-900 series, USA) was selected to estimate the specific surface area, average pore diameter and pore volume based on the liquid nitrogen adsorption–desorption method at an adsorption temperature of 77 K, saturated vapor pressure of 86.75 kPa and adsorption cross-section area of 0.162 nm^2. The results are provided in Table 3. The specific surface area, average pore diameter and pore volume of pomegranate peels were 8.9671 m^2/g, 5.6405 nm and 0.012645 cm^3/g, respectively. Meanwhile, the specific surface area, average pore diameter and pore volume of AC increased to 1431.8 m^2/g, 2.5934 nm and 1.623601 cm^3/g, respectively.

Table 3,
BET test for the raw material and AC

Samples	Surface area [m^2/g]	Average pore diameter [nm]	Total pore volume (p/p_0 = 0.990) [cm^3/g]
Pomegranate peel	8.9671	5.6405	0.012645
AC	1431.8	2.5934	1.623601

4.2. Field Emission SEM (FESEM) and EDX Analysis

To investigate the morphological surface and elemental species composition of pomegranate peels and AC, FESEM with EDX analysis was conducted, and the results are presented in Figures 3–6 and Tables 4 and 5. As shown in Figure 3, the pomegranate peels exhibit a homogenous surface, large pore diameter and low pore volume. Meanwhile, Figure 4 shows a spongy surface with many porous structures and pores with different volumes and shapes. This finding is attributed to the decomposition and volatilisation of non-carbonaceous substances in pomegranate peels due to chemical and microwave activation. In addition, the EDX spectra of pomegranate peels and AC exhibit an evident variation in their elemental composition due to chemical and microwave activation, as shown in Figures 5 and 6 and Tables 4 and 5. These tables indicate that the carbon content of pomegranate peels is increased from 38.6% to 74.5% for AC due to the carbonisation and activation processes. Consequently, their elemental compositions were changed.

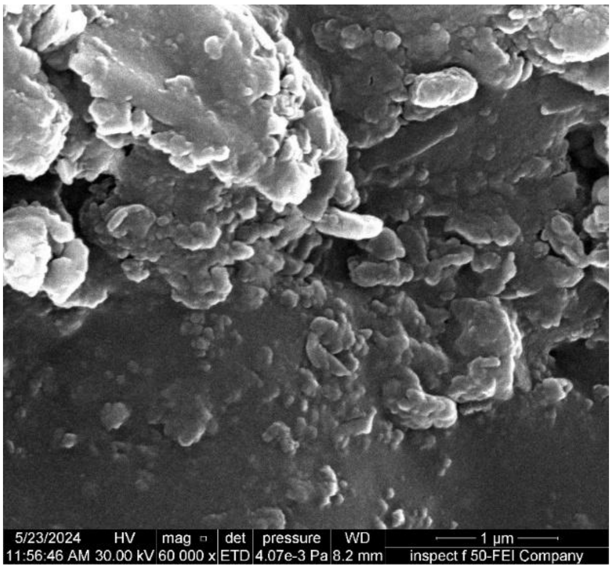

Fig. 3. Images of pomegranate peels

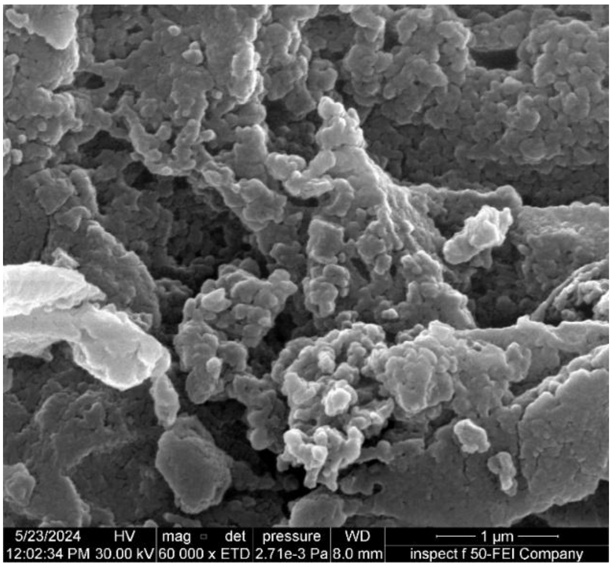

Fig. 4. Images of AC

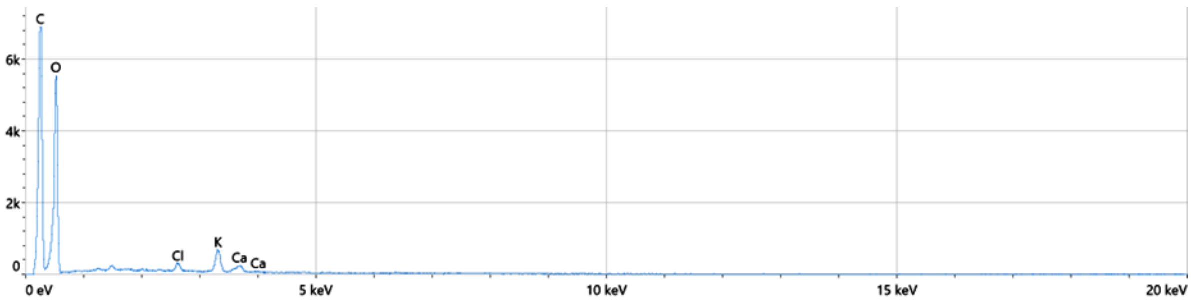

Fig. 5. EDX spectroscopy of pomegranate peels

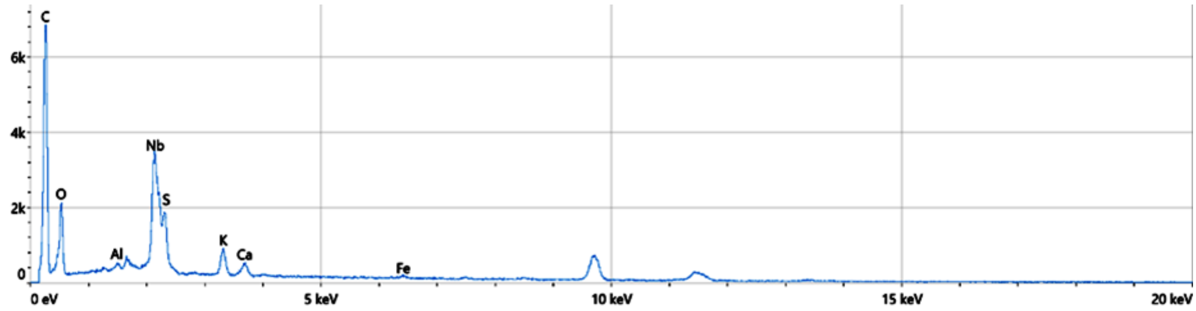

Fig. 6. EDX spectroscopy of AC

Table 4,
Composition of pomegranate peels

Element	Atomic %	Atomic % Error	Weight %	Weight % Error
C	46.2	0.3	38.6	0.3
O	52.8	0.4	58.8	0.4
Cl	0.2	0.0	0.5	0.0
K	0.6	0.0	1.5	0.0
Ca	0.2	0.0	0.5	0.0

Table 5,
Composition of AC

Element	Atomic %	Atomic % Error	Weight %	Weight % Error
C	72.8	0.4	74.5	0.4
O	25.4	0.5	20.3	0.6
Al	0.4	0.0	0.7	0.1
K	0.8	0.0	2.1	0.1
Ca	0.4	0.0	1.2	0.0
Fe	0.1	0.0	0.5	0.1
Nb	0.1	0.1	0.7	0.6

5. FTIR Analysis

The FTIR analysis spectra of the pomegranate peels (PP) and AC (PPAC) between the wavelengths of 4000 cm^{-1} and 500 cm^{-1} are shown in Figure 7. The raw materials have different peaks, reflecting their complex properties. The peaks between 3000 cm^{-1} and 3500 cm^{-1} indicate the OH stretching of alcohols, phenols and carboxylic acids [53, 54]. Similarly, the peaks between 2500 cm^{-1} and 3000 cm^{-1} are denoted by the C-H group [55]. Meanwhile, the spikes between 1000 cm^{-1} and 1500 cm^{-1} refer to phenolic C=C and aldehydic C=O, whilst the peaks between 500 cm^{-1} and 1000 cm^{-1} indicate aromatic chemicals [56, 57]. Furthermore, the peaks appeared due to the formation of a new bond during chemical and microwave activation. Consequently, the peaks around 3000 cm^{-1} and 3500 cm^{-1} indicate the OH stretching of alcohols, phenols and carboxylic acids [53, 54]. Similarly, the peaks between 2500 cm^{-1} and 3000 cm^{-1} are indicated by C-H bending stretching [55, 58]. The peaks between 1500 cm^{-1} and 1750 cm^{-1} are mostly signified by alkene C-C and ketone C=O. Conversely, the peaks around 1000 cm^{-1} and 1300 cm^{-1} are assigned to C-O bonds due to the existence of of hydroxy and esters. Similarly, the peaks between 600 cm^{-1} and 900 cm^{-1} are assigned to = C-H due to the existence of alkenes [8].

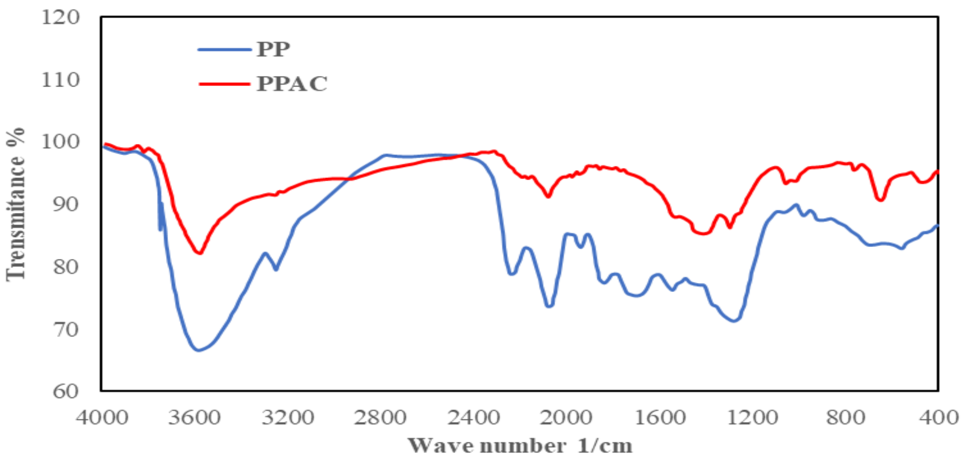

Fig. 7. Spectra of pomegranate peels and AC

6. X-ray Diffraction (XRD)

XRD analysis with a range of 2θ of 10°–80° for pomegranate peels and AC are depicted in Figures 8 and 9, respectively. Pomegranate peels and AC have amorphous structures. Sharp peaks at 2θ of 21.3°, 26.8° and 29.5°, as shown in Figure 9, indicate that AC exhibits mostly amorphous characteristics with a minor crystalline structure [59].

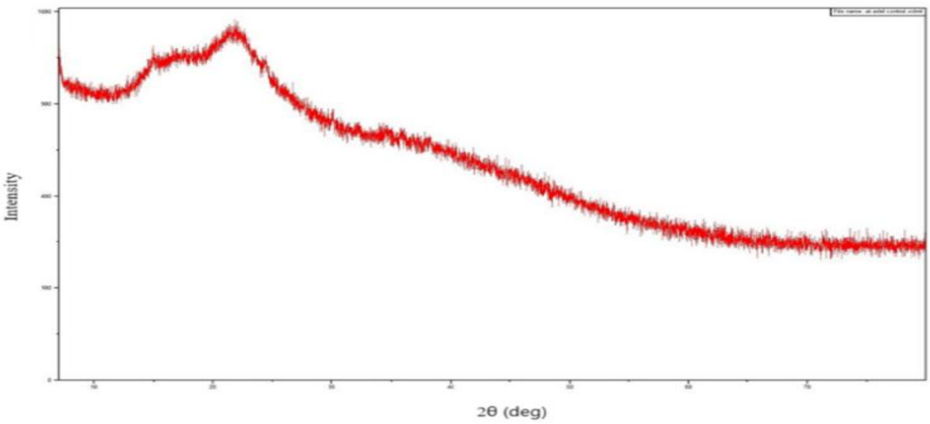

Fig. 8. XRD profile of pomegranate peels

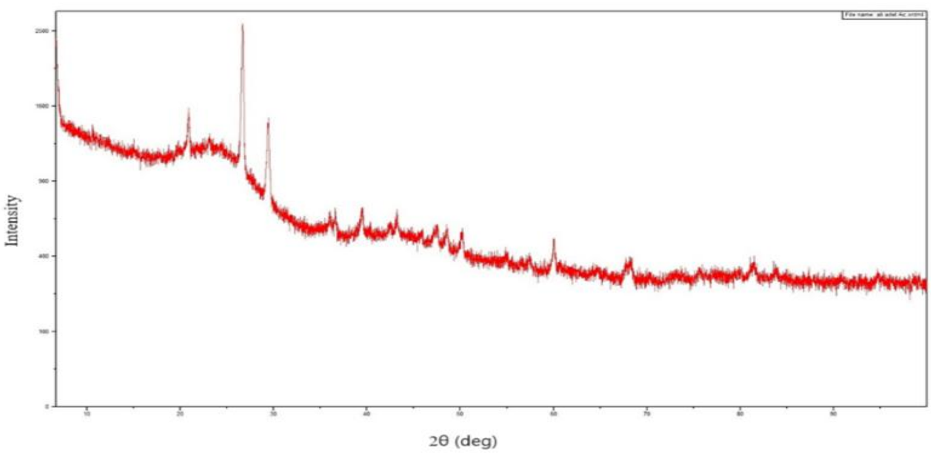

Fig. 9. XRD profile of AC

7. Conclusion

The major results of the current research demonstrated the suitability of AC prepared from pomegranate peels with significant specific surface area, average pore diameter and pore volume via chemical and microwave activation. Sulphuric acid with different concentrations (40%–80%) and IRs (1–3) was used as an activation agent with different impregnation times (2–6 h), microwave power (450–900 W) and radiation times (7–21 min) to prepare AC. Design-Expert software (version 13) with the I-optimal method was selected to analyse the AC preparation data. The data were analysed using MBN, and the results showed that sulphuric acid with a concentration of 60%, IR of 1:3, impregnation time of 6 h, microwave power of 450 W and radiation time of 21 min emerged with the highest MBN value. On the basis of the maximum MBN value, which elaborated the maximum specific surface area and pore volume, pomegranate peel and AC samples were selected for characterisation. The results demonstrated that the specific surface area, average pore diameter and pore volume of pomegranate peels were 8.9671 m^2/g, 5.6405 nm and 0.012645 cm^3/g, respectively. For AC, these parameters were enhanced to 1431.8 m^2/g, 2.5934 nm and 1.623601 cm^3/g, respectively. In addition, FESEM with EDX and XRD characterisation showed that AC exhibits a spongy property with different micro pores, indicating an increase in carbon content from 38.6% to 74.5% due to activation and carbonisation.

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Conflict of Interest

The authors declare that they have no conflicts of interest.

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تحضير وتوصيف الكربون المنشط من مخلفات الرمان باستخدام طريقة التنشيط الكيميائي والمايكروويف

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المستخلص

يقدم هذا البحث طريقة تحضير وتوصيف الكربون المنشط من قشور الرمان باستخدام التنشيط الكيمياوي مع تقنية الميكروويف. حيث يتم غمر مسحوق قشور الرمان بحامض الكبريتيك كعامل تفعيل كيميائي وبنسب وزنية مختلفة (قشور رمان إلى حامض الكبريتيك) وبأوقات غمر مختلفة. كما تم تنشيط العينات المفعلة بالحامض باستخدام تقنية الميكروويف تحت جو خامل بقدرة وأوقات تنشيط مختلفة لإنتاج الكربون المنشط. تم اختيار برنامج تصميم التجارب (الإصدار ١٣) باستخدام طريقة I-Optimal لتحليل تأثير متغيرات تحضير الكربون المنشط بما في ذلك تركيز الحامض، زمن التفعيل ونسبة التفعيل وقدرة فرن الميكروويف وزمن التنشيط على قدرة امتزاز صبغة الميثيلين الزرقاء (MBN). بناءً على الحد الأقصى امتزاز صبغة الميثيلين الزرقاء اتي تم الحصول عليها، تم توصيف قشور الرمان والكربون المنشط باستخدام Brunaure-Emmett-Teller (BET)، ومجهر المسح الإلكتروني الانبعاثي (FESEM) مع تحليل الأشعة السينية المشتتة للطاقة (EDX)، ومطيافية الأشعة تحت الحمراء (FTIR)والحيود بالأشعة السينية .(XRD) وقد أثبتت النتائج أن الكربون المنشط المنتج من قشور الرمان له طبيعة إسفنجية وغير متبلور بشكل أساسي مع خصائص بلورية قليلة ويمتلك أحجام مختلفة من المسام الدقيقة ويمكن استخدامه كمنتج منخفض التكلفة بخصائص مرغوبة.