

Research Paper

Preparation and characterization of activated carbon from chickpea stalk by chemical activation

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ABSTRACT

The research was conducted to valorize the raw chickpea stalk by producing activated carbon. Activated carbon was produced through a process of chemical activation utilizing potassium hydroxide (KOH), conducted at 750 °C in an environment of nitrogen (N₂) gas. The BET surface area, scanning electron microscopy (SEM), proximate analysis, elemental analysis, Adsorption analysis, and calorific value of raw chickpea stalk and activated carbon were investigated and critically compared. The finding showed that the specific surface area, total pore volume, methylene blue value, and iodine value were 1176 m²/g, 0.5 cm³/g, 315 mg/g, and 368 mg/g, respectively. The synthesized activated carbon had high porosity. The pores were interconnected like a honeycomb structure as investigated by scanning electron microscopy analysis. Thus, it was concluded that the raw chickpea stalk was valorized by thermochemical activation, and enhancement in activated carbon properties was achieved. The activated carbon can be used for dye removal, water purification, gas storage, etc.

Key words: Chickpea stalk, Activated carbon, Chemical activation, Iodine value, Methylene blue value, Specific surface area.

INTRODUCTION

Currently, a major fraction (84.3%) of the world's energy supply comes from fossil fuels (World Energy Outlook 2021). This is a worrisome scenario since the burning of petroleum and coal harms the planet by emitting greenhouse gases that lead to climate change and global warming. To address the issues related to burning fossil fuels, several attempts have been undertaken to create sustainable energy systems. Biomass energy has demonstrated significant potential for creating environmentally friendly energy sources (Ghritalahre *et al.* 2023). Considering the advantages of inexpensiveness, simple availability, zero carbon emissions, and a brief life cycle of non-woody biomass, biomass is plentiful globally. There has been scant study work done on lignocellulosic biomass from an energy perspective. This is brought on by biomass's hydrophilic character, high moisture content, low bulk density, lower calorific value, and low energy output (Mohd Safaai and Pang 2021). The use of various techniques such as drying, torrefaction, combustion, pyrolysis, activation, and densification in biomass enhanced these encouraging properties (Jagadale *et al.* 2024).

In this context, activated carbon produced via thermochemical activation methods and from inexpensive, easily accessible precursors are being viewed as essential components for environmental remediation as well as for the production and storage of sustainable energy (Sevilla and Mokaya 2014). The extensive category of carbon that are presently the subject of extensive research for use as electrode materials for supercapacitors or as gas storage materials (hydrogen, CO₂, methane, etc.) are examples of this (Sevilla and Mokaya 2014, Wang and Kaskel 2012). The intention for the next generation of carbon material is to develop materials that are easier to make using economical methods and that may employ renewable resources as precursors with the potential to valorize the raw biomass. Among carbon materials, activated carbon has attracted much attention due to its superior chemical resistance, porosity, and adjustable pore size (Balahmar *et al.* 2017). Activated carbons are notably interesting since they may be produced from renewable biomass resources that are typically regarded as waste materials (Balahmar *et al.* 2017). Activated carbon can be produced through direct activation of raw biomass, or it can be made in two

steps, namely carbonization followed by activation (Heidarinejad *et al.* 2020). Pyrolysis is the process of carbonization, which involves heating raw biomass (crop residue) to a high temperature (600–1000 °C) absence of oxygen in an inert environment (Sahu *et al.* 2023, Sahu *et al.* 2025). The activation stage follows to improve the activated carbon's structural properties. This process aims to increase the pore size, volume, and diameter, as well as the surface area, resulting in the formation of well-distributed microscopic cavities throughout the material. There are three methods for this process: hydrothermal, chemical, and physical (Sahu *et al.* 2023).

Cheng *et al.* (2021) compared activated carbon produced from one-stage and two-stage H_3PO_4 chemical activation process. The results showed that, compared with the activated carbon produced using the two-step activation method, the activated carbon produced using the one-step activation process at 600 °C exhibited a high proportion of mesopores pore volume (97 %) and a good specific surface area (1342.93 m^2/g) (Chen *et al.* 2021). Genli *et al.* (2021) obtained activated carbon from chickpea stalks by hydrothermal method followed by chemical activation method using $ZnCl_2$. They recorded iodine number, methylene blue number, and BET surface area as 734 mg/g, 105 mg/g, and 572 m^2/g , respectively (Genli *et al.* 2021). Genli *et al.* (2022) synthesized mesoporous activated carbon from chickpea stem by hydrothermal carbonization followed by the chemical activation method (KOH). They noted a high BET surface area (455 m^2/g) and methylene blue value (887 cm^3/g) (Genli *et al.* 2022). Previous investigations on chickpea waste show that new technology for producing activated carbon is still being explored. The author is aware of no research that has been undertaken for chickpea stalk by one stage chemical activation using potassium hydroxide.

Therefore, this study aims to introduce an innovative technique for valorizing raw chickpea stalk, transforming it into activated carbon, with a detailed characterization of its unique structural and adsorptive properties. This study concentrated on a quick and simple method for converting chickpea stalk into activated carbon. This process reduced the time required for both the two-step and conventional approaches.

MATERIALS AND METHODS

Collection of chickpea stalk biomass

Chickpea stalk (CS) was collected from

a nearby village located in Bhopal. Following cleaning with hot water, the chickpea stalk was left outside to dry in the sun. Then, it was ground into a powder, and the fraction between 0.7 to 1.2 mm was separated by sieve analysis for the experiment. Further, the sample went on for 24 hours at 105 °C in a hot air oven to remove unnecessary moisture before conducting any experiment.

Process of activated carbon synthesis

Raw chickpea stalk was carbonized by KOH chemical activation. The chickpea stalk was treated with KOH chemical on a 1:1 by weight basis and left for 48 hours. After that, it was dried in a hot air oven at 105 °C until it was completely dry. The impregnated sample was carbonized in a horizontal reactor chamber at 750 °C temperatures with a heating rate of 10 °C/min in the presence of N_2 gas (flow rate 60 mL/min) for 90 min. After the experiment was completed, the carbonized sample was washed with hot distilled water and 1M HCl until the pH of the washed solution reached about 7. Finally, the prepared carbon was dried in a hot air oven at 105 °C for 24 h. Thus, the carbon sample was prepared.

Analysis of raw chickpea stalk and activated carbon

The chickpea stalk and carbon content were analyzed using various types of characterization. The proximate analysis identified moisture (ASTM E1756-08), ash (ASTM E1755-01), volatiles (ASTM E872-82), and fixed carbon content. Elemental analysis included the determination of elements such as carbon, nitrogen, oxygen, hydrogen, and sulfur. Additionally, a bomb calorimeter was used to estimate the higher heating value (ASTM D5865-13).

Adsorption analysis of the iodine value (ASTM D4607-94,2006) was carried out for both raw and activated carbon. The following is the calculating formula:

$$\text{Iodine value (mg/g)} = C \times \text{Conversion factor}$$

$$\text{Conversion Factor} =$$

$$= \frac{\text{Mol wt. of iodine} \left(127 \frac{g}{mol}\right) \times \text{normality of iodine} \left(\frac{mol}{L}\right) \times 40 \text{ ml}}{\text{Wt. of carbon (g)} \times \text{Blank reading (ml)}}$$

$$C = \text{Blank reading (ml)} - \text{Standard reading (ml)}$$

The investigation of methylene blue (MB) adsorption was carried out for raw and activated carbon. The calculation formula is as follows:

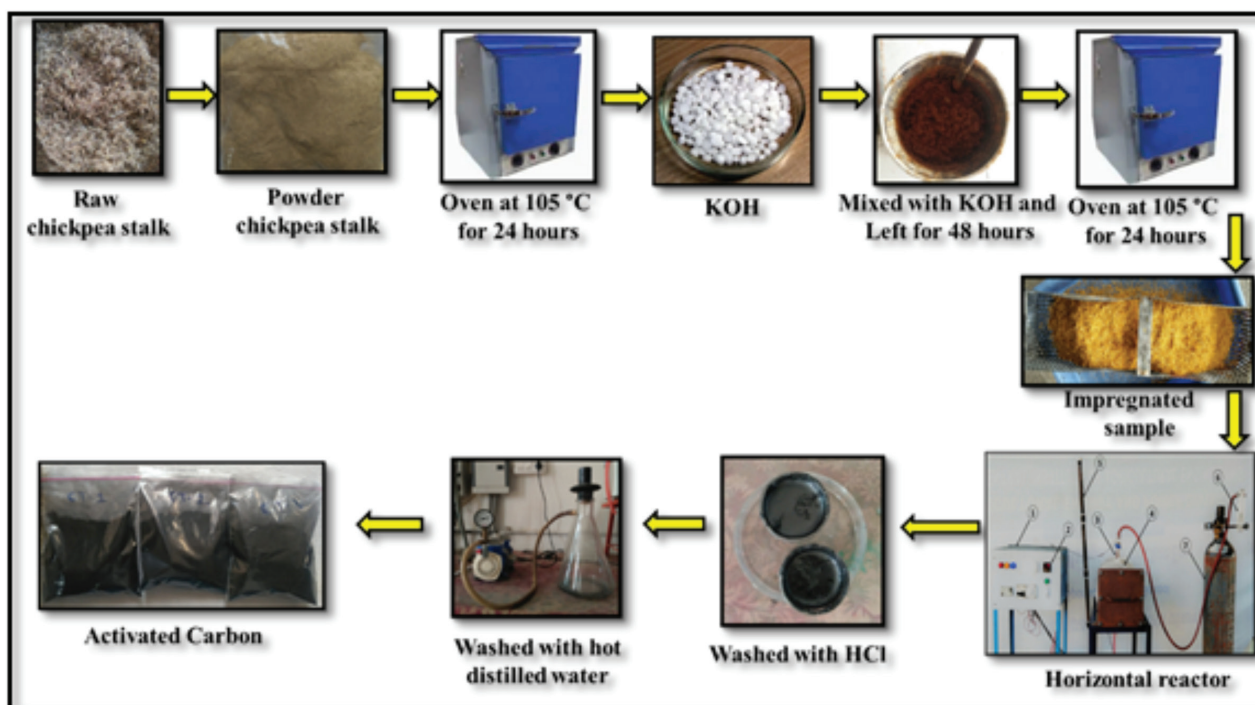


Fig. 1. Flow chart of preparation of activated carbon

$$MB \text{ Value} = \frac{C_o - C_e}{w} \times V$$

Where V is a volume (ml), w is the weight of the sample (g), C_o is the initial concentration (mg/ml), and C_e is the equilibrium concentration (mg/ml).

The specific surface area, pore volume, and pore size distribution of raw chickpea stalk and activated carbon were determined by nitrogen adsorption/desorption isotherms at 77 K using a surface area analyzer (Microtrac, Belsorp Max G). The sample underwent vacuum degassing prior to analysis. The surface morphologies of the activated carbon and raw chickpea stalk were examined by the use of field emission scanning electron microscopy (Carl Zeiss, Germany) operated at 20 kV.

RESULTS AND DISCUSSION

Physicochemical analysis

The primary components of physicochemical analysis for biomass include proximate, elemental, and calorific value assessments. Table 1 presents the results of these analyses for raw chickpea stalk (CS) and activated carbon. Proximate analysis revealed that volatile matter, moisture content, and ash content were 65.63%, 9.02%, and 7.96%, respectively.

The fixed carbon content, calculated by difference, was 17.39%. In activated carbon, the fixed carbon content increased to 68.91% due to the removal of volatile matter from raw chickpea stalks (Durga *et al.* 2022). The ash content slightly increased as a result of metal oxide and mineral salt formation during the thermochemical process. The moisture content in the biomass was within an acceptable range for energy production (Sahu *et al.* 2022). The high percentage of volatile matter (65.63%) indicates that the biomass is suitable for bioenergy production. Additionally, the low ash content positively impacts energy production from biomass. Previous studies have shown that both high and low volatile matter content can be beneficial for energy production from biomass (Durga *et al.* 2022).

The total carbon content of raw chickpea stalk was higher (58.89%) as compared to activated carbon (52.64%). This reduction in total carbon content during activation is mainly due to the decomposition and volatilization of unstable components like hemicellulose and lignin. However, the activating process led to the breakdown of biopolymer bonds, thus increasing the amount of stable organic carbon fractions contained within the biomass matrix. This resulted in a substantial 49.81% enhancement of the total organic content in the activated carbon produced. This enrichment means the formation of

Table 1. Physiochemical analysis of chickpea stalk and activated carbon.

Sample	Proximate (%)				TC/ToC Analysis (%)		Elemental (%)					Calorific Value (MJ/kg)
	MC	VM	Ash	FC	TC	ToC	C	H	N	S	O	
Chickpea Stalk	9.02	65.63	7.96	17.39	58.89	41.99	40.62	6.55	0.83	0.31	51.69	16.46
Activated carbon	8.06	14.51	8.52	68.91	52.64	49.81	62.16	4.13	0.32	0.17	33.22	20.45

Note: TC = Total carbon, ToC = Total organic carbon, MC = Moisture content, VM = Volatile matter, FC = Fixed carbon.

a carbonaceous structure with enhanced stability and higher functionality.

The calorific value of raw chickpea stalk was measured at 16.46 MJ/kg. The activated carbon exhibited a higher heating value of 20.45 MJ/kg, attributed to the increased fixed carbon content percentage (Anshariah et al. 2020). Elemental analysis examined the chemical composition of raw chickpea stalk and activated carbon. The elemental composition of raw chickpea stalk was determined to be carbon (40.62%), nitrogen (0.83%), hydrogen (6.55%), and sulfur (0.31%), with oxygen (51.69%) calculated by subtraction. The higher percentages of carbon (40.62%) and hydrogen (6.55%) suggest greater energy content in the biomass, while the lower nitrogen content (0.83%) indicates reduced hazardous gas emissions (Durga et al. 2022). In activated carbon, the carbon percentage increased while other elements decreased. This change occurs during thermochemical conversion when complex bonds break down, releasing CO₂, H₂O, and other gases (Gangil 2014).

Adsorption analysis

The iodine number and methylene blue (MB) measurements were utilized to assess Adsorption characteristics. This Adsorption process enables the detection of typical pores found in biomass. Iodine values can identify micropores (< 2 nm), while methylene blue values can detect mesopore structures (2-50 nm) (Ektepe et al. 2017). The methylene blue value and iodine number of raw chickpea stalk and activated carbon are illustrated in Fig. 2.

Results indicated a methylene blue adsorption capacity of 78.73 mg/g and an iodine number of 88.54 mg/g. These values suggest that raw chickpea stalk has a limited micropore and mesopore structure (Durga et al. 2022). In comparison, the activated carbon exhibited a higher iodine number and methylene blue value, indicating a greater proportion of mesopores and micropores (Manocha 2003). The study concluded that raw chickpea stalk possesses limited surface area and porosity, with

few micropores and mesopores. These properties can be enhanced through chemical or thermal activation techniques.

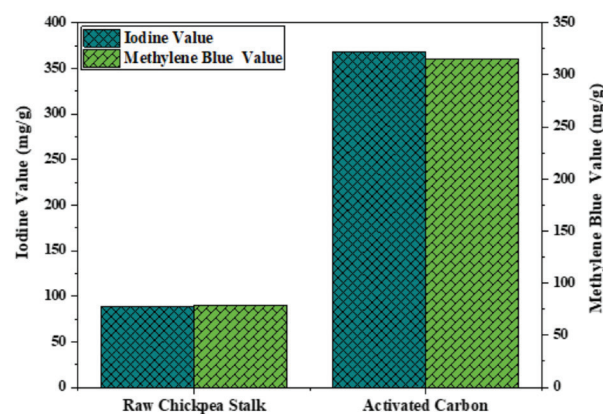


Fig. 2. Methylene blue and Iodine value analysis of raw chickpea stalk and activated carbon.

BET Surface area analysis

The Brunauer-Emmett-Teller (BET) surface area analyzer was employed to determine surface area, pore volume, and pore size distribution. Figure 3 displays the Adsorption desorption curve of raw chickpea stalk. According to the IUPAC classification, this curve follows a Type III

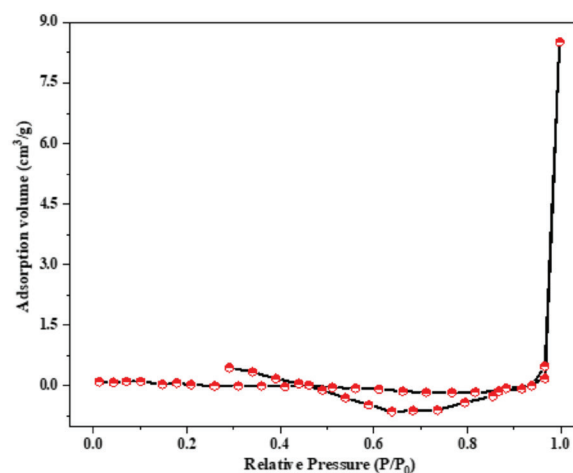


Fig. 3. Adsorption desorption isotherm curve of raw chickpea stalk.

isotherm. Such isotherms are typically associated with macroporous or nonporous materials where adsorbent-adsorbate interactions dominate the adsorption process rather than adsorbate-surface interactions (Manocha 2003). This suggests that raw chickpea stalk may lack a well-developed pore structure, explaining its relatively low surface area and pore volume.

The specific surface area of chickpeas stalk was determined to be notably lower ($0.45 \text{ m}^2/\text{g}$), suggesting a scarcity of active sites for Adsorption. This was attributed to the absence of significant mesoporous or microporous characteristics in the raw material. The limited porosity of the material was further evidenced by its pore volume, measured at $0.01 \text{ cm}^3/\text{g}$. The average pore width of 45.50 nm indicates that the pores present in this material fall within the macropore category ($>50 \text{ nm}$). The pore size distribution curve shows that the chickpea stalk had mainly macropores with some mesopores (Fig. 5a). The pore size distribution prominent peak was observed between 25 and 50 nm pore diameter, which indicates a significant concentration of mesopores within this size range. Whereas, most of the pores are concentrated within a relatively narrow range of $25\text{--}75 \text{ nm}$, suggesting a relatively uniform macropore structure. The chickpea stalk's low surface area and pore volume collectively imply that it may need additional processing to enhance its Adsorption capacity and expand its potential applications across various industrial sectors.

Table 2. BET surface area characteristics of raw chickpea stalk and its activated carbon.

Sample	Surface area (m^2/g)	Total pore volume (cm^3/g)	Average pore diameter (nm)
Raw Chickpea Stalk	0.45	0.01	45.50
Activated carbon	1176	0.50	0.85

The activated carbon derived from raw chickpea stalk demonstrated high microporosity and mesoporosity, along with a large surface area. Its isotherm adsorption and desorption curve exhibit a type IV pattern (Fig. 4) (Üner and Bayrak 2018). A sharp increase in the curve up to 0.01 indicates the rapid Adsorption of nitrogen into the micropores. The subsequent curve edge turns, and gradual increase with relative pressure signify the onset of mesopore adsorption, while the slow rise reflects the gradual filling of micro and mesopores on the surface (Sahu and Gangil 2024). In the final stages, adsorption increases significantly, approaching nitrogen saturation pressure due to active capillary

condensation. The presence of a hysteresis loop in the curve confirms the availability of mesopores on the surface of the activated carbon (Manocha 2003).

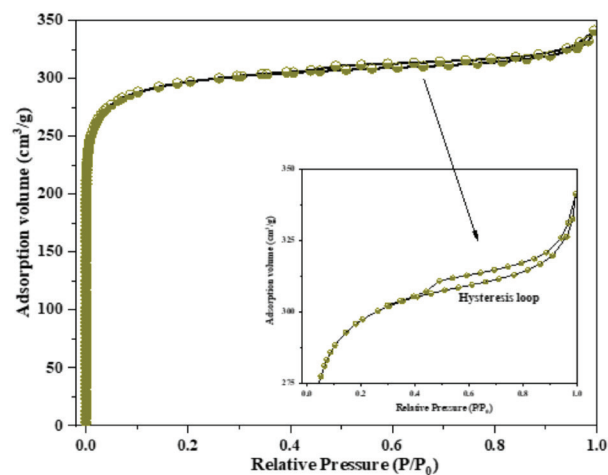


Fig. 4. Adsorption-desorption isotherm curve of activated carbon.

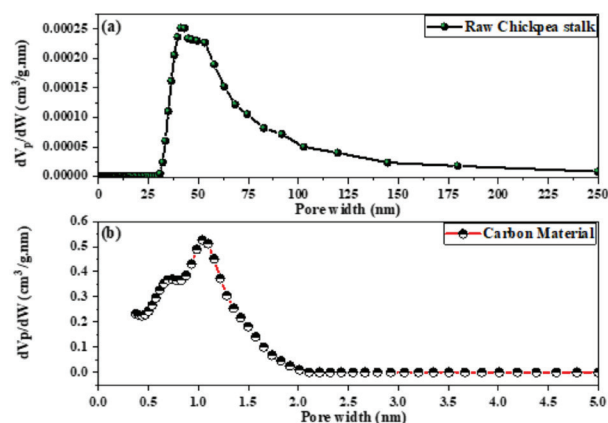


Fig. 5. Pore size distribution of (a) Raw chickpea stalk (b) activated carbon.

To ascertain the pore size distribution of the activated carbon, the non-local density functional theory (NLDFT) model was employed (Yang *et al.* 2010). The pore size distribution showed that the peak of the curve was observed in the micropore range, suggesting the concentration of micropores present in the activated carbon structure (Fig. 5 b). Whereas, the distribution range of pores varying from 2 to 50 nm confirms the presence of mesopore-activated carbon. Overall, high surface area and pore volume were recorded in activated carbon-containing micropores (less than 2 nm) and mesopores (2 to 50 nm) (Yang *et al.* 2010). The activated carbon was recorded to have a high specific surface area ($1176.10 \text{ m}^2/\text{g}$) and pore volume

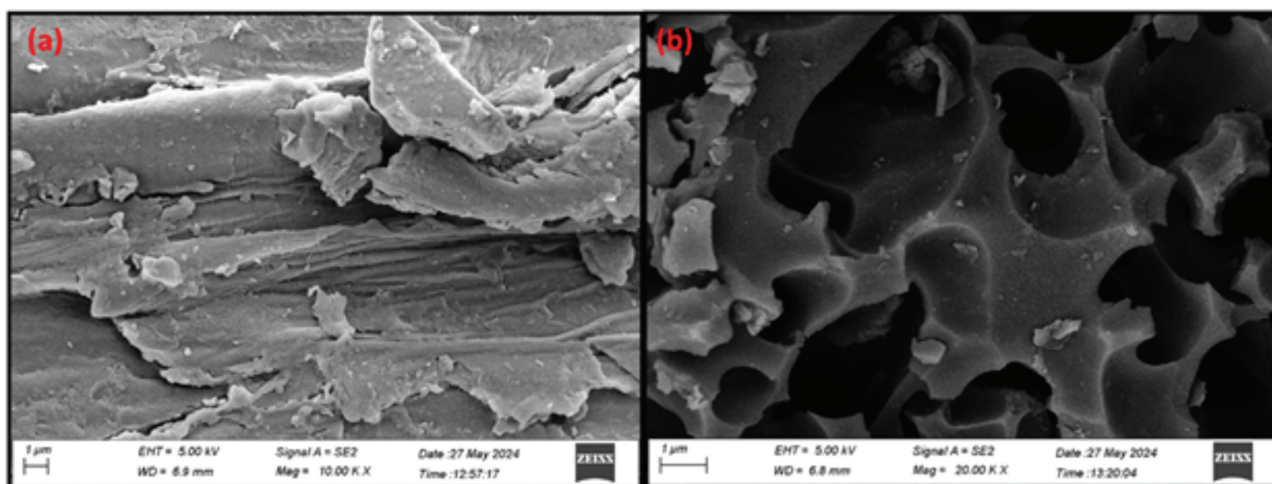


Fig. 6. Scanning electron microscopy (SEM) image (a) raw chickpea stalk (b) activated carbon.

($0.50 \text{ cm}^3/\text{g}$), indicating that the material has a high number of micro and meso pore active sites that are available for Adsorption. During thermochemical conversion, due to the evaporation of several gases and liquid biofuels, it forms pores on the surface of the activated carbon (Wang and Kaskel 2012). The average pore width (0.85 nm) confirms that the material had the presence of micropores ($< 2 \text{ nm}$).

Scanning electron microscopy analysis

The Fig. 6 displays scanning electron microscopy (SEM) images of both raw chickpea stalk and activated carbon. The SEM image of raw chickpea stalk (Fig. 6a) shows a surface devoid of pores. In contrast, the activated carbon exhibits micro- and mesopores on its surface, which shows a clear difference from raw gram stalk. This pore development is the result of thermal degradation and evaporation of volatile matter in the chickpea stalk. The formation and transformation of pores are significantly affected by the activation time and temperature. The image clearly shows that an optimal activation temperature and duration generated numerous pores on the surface (Fig. 6b). As thermal degradation progresses and volatile matter begins to evacuate, certain catalysts interact with the activated carbon (González-García 2018). As a result, a high proportion of micropores develop in the activated carbon. Furthermore, the extended exposure to high temperature allows the catalyst (KOH) ample time to react with carbon and other volatile substances (González-García 2018, Wang and Kaskel 2012). This reaction facilitates the formation of both micro and mesopores on the surface of the activated carbon, further increasing the number of pores.

At this stage, pore metamorphosis occurs, with micropores evolving into mesopores, and mesopores transforming into macropores (González-García 2018, Wang and Kaskel 2012). The activated carbon exhibits a honeycomb-like structure, comprising micro, meso, and macropores interconnected in a tunnel-like configuration. These observations lead to the conclusion that thermochemical action plays a crucial role in pore formation within the activated carbon.

CONCLUSIONS

This study presents activated carbon prepared from chickpea stalks by chemical activation method. The prepared activated carbon has a high specific surface area ($1176 \text{ m}^2/\text{g}$), with a high iodine value ($368 \text{ mg}/\text{g}$) and methylene blue value ($315 \text{ mg}/\text{g}$), as well as a nanoporous nature. It is anticipated that the chemical activation method has a simple and less time-consuming process to prepare highly porous carbon compounds from chickpea stalks. It will reduce the impact of global warming and provide a more convenient and environmentally friendly route. It is possible to use the produced activated carbon for gas storage, gas Absorption in air purification, efficient water purification filters, removing impurities, etc.

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