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Effect of Activation Time on Physicochemical Properties of Activated Carbon Prepared from Coconut Shell

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Abstract

The Characteristics of activated carbon derived from coconut shells (CS) are influenced by the activation process parameters, which include activation temperature, time, and the application of different activation agents. This study intends to evaluate the impact of activation time on the physicochemical properties of activated carbon produced from coconut shells. Coconut shells were used as a precursor to prepare activated carbon, with phosphoric acid serving as the activation agent. Different samples were prepared using activation times of 6, 12, and 24 hours, with a carbonization temperature of 500 °C for 60 minutes. The prepared activated carbon samples were characterized using a gas sorption analyzer, SEM, and FT-IR. The activated carbon's specific surface area increased from 189 to 505.8 m^2/g, and micropore volume increased from 0.146 to 0.311 cm^3/g, as the activation time was extended from 6 to 24 hours, respectively. This property improvement was also confirmed by SEM and FTIR. The FTIR analysis depicts the presence of different functional groups. In conclusion, the properties of the prepared activated carbon are affected by the activation time. The results confirmed that coconut shell is a promising low-cost precursor for activated carbon production, which is useful for environmental applications.

Keywords: *Coconut shell, activated carbon, surface area, SEM/EDX, FTIR.*

1.0 Introduction

Activated carbon is a widely utilized material in the chemical industry owing to its exceptional physicochemical properties, including a large surface area and thermal stability [1]. The use of activated carbon (AC) for the adsorption of organic and inorganic contaminants dissolved in aqueous media or from the gaseous environment has dramatically increased [2]. However, due to its high operating costs and non-renewable nature, the use of commercially available activated carbon made from coal, a highly carbonaceous precursor, is limited [2]. These disadvantages have prompted researchers in search of inexpensive, renewable carbonaceous materials, such as agricultural waste, to prepare activated carbon. Forest residues, crops, wood, herbaceous plants, and animal waste are examples of these types of waste [3]. Because of their low cost, abundance of sources, and large production scale, Nigeria's yearly production of these waste materials, which are mostly underutilized, could be used as a new source of precursors to make activated carbon [4, 5]. Worldwide, the processing of coconuts (*cocos nucifera L*) for the production of coconut milk and coconut oil produces biomass from coconuts. These waste materials, shells, husks, and coir, have the capacity to serve as fuel for sustainable energy sources and as a source of activated carbon for environmental applications [6]. Because CS is used to make charcoal by open burning and landfilling, it pollutes the air and the land, respectively. Improper handling of coconut waste also pollutes the water and the soil [6]. Because coconut shell (CS) is an inexpensive substrate and an eco-friendly material, it was chosen for this research. CS is also a superior carbonaceous precursor due to its sustainability and versatility in adsorbing contaminants across a range of concentrations, abundantly available, ease of operation, low initial moisture content, and less feedstock preparation effort [6, 7].

Utilising biomass as a precursor, activated carbon is made using two activation techniques: physical activation, which entails first carbonizing the starting precursor, and then activating it with steam, oxygen, or CO_2. While chemical activation involves the use of several activating agents, such as KOH, H_3PO_4, $ZnCl_2$, NaOH, $CaCl_2$, and $KMnO_4$. Chemical activation is more preferable than physical activation because it is employed at a lower temperature and activation time, higher yield with an improved porous structure [1]. The parameters of the activation process affects the characteristics of activated carbon derived from CS . Key parameters include activation temperature, time, and the utilization of several activation agents.

The objective of this research is to investigate how activation time impacts the surface properties; surface area and pore size, surface morphology, and functional groups found on the surface of coconut shell-derived activated carbon, with phosphoric acid as an activating agent. The analysis of the activated carbon was performed using a gas sorption analyzer, Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy and energy-dispersive X-ray (SEM/EDX).

2.0 Materials and Methodology

2.1 Materials

The chemicals used in this study were of analytical grade. Phosphoric acid, H_3PO_4 (98% purity) (BDH Chemicals, Poole, England) was used for the activation process, glassware, Whatman filter papers, and distilled water, oven, furnace, a weighing balance, a ball mill, and funnels.

2.2 Sourcing and Preparation of Coconut Shell

The CS used in this study were collected from Bakindogo Central Market, Kaduna State, Nigeria. The shells were washed with thorough several times by using tap water after selecting the foreign materials, and then subsequent washing with distilled water, sundried for 5 days, and then oven dried overnight; this is followed by crushing with an industrial crusher. The ground coconut shell was sieved with a mesh size of 280 microns to obtain a uniform particle size.

2.3 Preparation of Activated Carbon from Coconut Shell Powder

The method described by [8] was modified and then utilized to prepare CS activated carbon. The ground coconut shell was placed in a crucible and subsequently in a muffle furnace to carbonize at 500 ^{o}C for 60 minutes. After cooling in a desiccator, the carbonized shell was treated with a 1M solution of phosphoric acid in a ratio of 2:1 (volume of acid per gram of precursor) for 6, 12, and 24 hr at room temperature to obtain activated carbon. Using distilled water, the paste was washed to a neutral pH, followed by oven drying overnight at 120 ^{o}C. The oven-dried sample was cooled using a desiccator and stored in an air-tight labelled sample bottle for characterization. The samples were labelled as CS, A1, A2, and A3 for coconut shell powder, activated carbon prepared at activation times of 6, 12, and 24 hr, respectively.

3.0 Results and Discussion

3.1 Textural Characterization

The surface area of an adsorbent is important as it gives an insight into its adsorption capacity [9]. The pore characteristics, surface area of the CS and the activated carbon prepared from CS at different activation time of 6, 12 and 24 hr were analyzed using a gas sorption analyzer (Quartracrome NOVA 4200e) and from the results displayed in Table 1, it was observed that an increase in the activation time from 6 to 24 hr, the surface areas; BET, BJH and DH, increased from 165.045 to 355.357, 177.385 to 473.131 and 189 to 505.8 m^2/g respectively. Except for the BET surface area, the CS powder has nearly the same surface area as the produced activated carbon in this research at 6 hr activation time. This shows that lower activation time prevents the optimum development of surface area and porosity. The pores within porous solids are classified into micropores (< 2nm diameter), mesopores (2-50 nm), and macropores (>50 nm) [2]. While the total and micropore volume increased with activation time, the pore diameter has a value between 2.1 and 2.4 nm, which implies that the samples of activated carbon produced are within the mesopore region. A similar pattern was observed by Tan et al. [2]. An increase in activation time can result in a more developed structure, resulting in higher surface areas and pore volume, which can impact the adsorption capacity of the activated carbon. However, excessive activation can cause a decline in adsorption performance due to pore enlargement or destruction, which causes the wall structure to become thin and weak [10].

Table 1: Surface Area and Pore characteristics of coconut shell and prepared activated carbon

Sample	S_{BET} (m^2/g)	S_{BJH} (m^2/g)	S_{DH} (m^2/g)	Micropore vol. cm^3/g	Pore $Volume_{BJH}$ (cm^3/g)	Pore $Size_{BJH}$ (nm)
CS	141	177.1	189	0.127	0.08666	2.132
A1	165.045	177.385	189	0.146	0.087	2.453
A2	280.126	289.295	311.1	0.246	0.139	2.111
A3	355.357	473.131	505.8	0.311	0.226	2.411

3.2. SEM/EDX Characterization

A scanning electron microscope (PhenomWorld Pro X) was used in determining the porosity and surface texture of the sample. The morphological structure of the CS, prepared activated carbon from CS at activation times of 6, 12, and 24 hr, is presented in figures 1 (a, b, c, and d, respectively). The SEM micrographs were recorded at 1500x magnification. The SEM of the raw coconut shell revealed a compact structure with no visible pores. However, there is a change in the form and structure of the prepared activated carbon, which is ascribed to the breakdown of lignocellulosic materials due to carbonization [8] and activation of the sample. The exterior surfaces of the samples of activated carbon become rough, irregular, and full of various shapes and sizes of cavities. This change in morphology, which persists with activation time, is attributed to the oxidative effect of the phosphoric acid, which leads to enlargement of the pore. At an activation time of 24 hrs, larger pores are present and noted in the produced activated carbon.

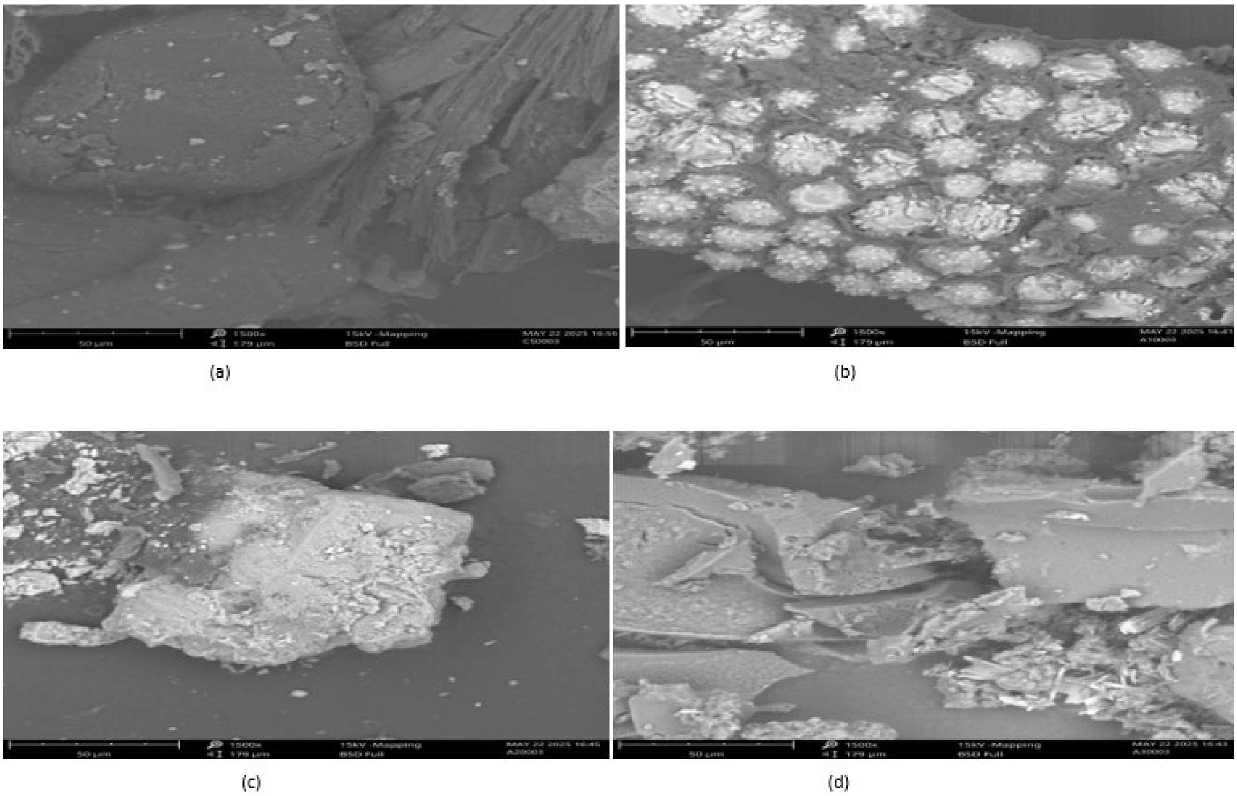

Figure 1: Scanning electron microscope image of (a) CS, (b) (c) (d) CS activated carbon prepared at activation times of 6, 12, and 24 hr, respectively.

In combination with Scanning Electron Microscopy (SEM, the Energy Dispersive X-ray Analysis (EDX) technique is used for performing fundamental element analysis or chemical characterization of a sample. The results of the EDX spectrum in Table 2 and Figure 2 showed that the weight and atomic percentages of all the samples mainly consist of carbon and oxygen at diverse and varied proportions, with sample A3 having the highest ratio of carbon to oxygen as a result of prolonged activation time. The weight percent of oxygen is higher in the CS than in the samples of activated carbon produced. This can be attributed to the reaction of oxygen to create the functional groups responsible for adsorption in the prepared activated carbon samples. The following elements such as C, O, N, K, Al, are present in weight% of 69.51%, 19.33%, 3.95%, 1.01%, 0.9%, for CS sample, 79.76,%, 4.84%, 3.27%, 1.3%, 1.01%, for A1 sample 79.32%, 9.12%, 2.69%, 1.48%, 1.15% for A2 and 77.78%, 3.85%, 1.41%, 1.19%, 5.45% for sample A3, respectively. Some of the elements found on the exterior of the prepared activated carbon samples can combine with oxygen to form metal oxides, which aid in adsorption [11, 12]. Contamination of the samples during the preparation or the quality of the coconut shell samples can be attributed to the presence of elements like Ag, P, and N. [13]. It can also be a result of contaminants from the apparatus used, an incomplete washing process, or the reagent used [1].

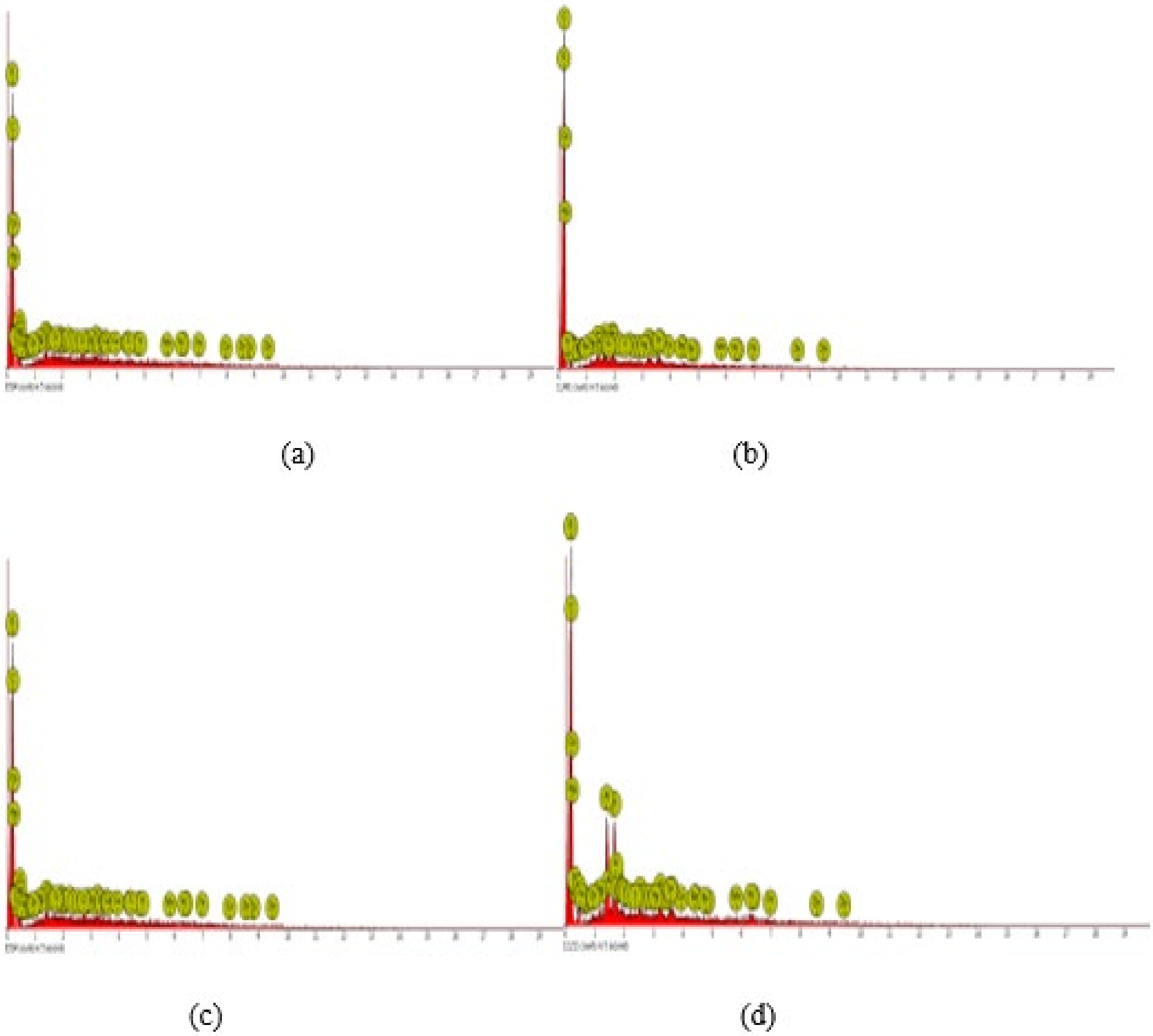

Figure 2: EDX Spectra of (a) CS, (b), (c), (d) CS activated carbon prepared at activation times of 6, 12, and 24 hr, respectively.

Table 2: Chemical composition from EDX analysis of CS, A1, A2, and A3.

Element	Atomic Concentration (%)				Weight Concentration (%)			
	CS	**A1**	**A2**	**A3**	**CS**	**A1**	**A2**	**A3**
C	78.05	88.96	86.63	88.13	69.51	79.76	79.32	77.88
O	16.29	4.05	7.47	3.27	19.33	4.84	9.12	3.85
N	3.8	3.13	2.52	1.37	3.95	3.27	2.69	1.41
Si	ND	0.53	1.0	2.64	ND	1.11	2.15	5.45
Al	0.45	0.5	0.56	2.74	0.9	1.01	1.15	5.45
Ca	ND	0.48	0.53	0.44	ND	1.42	1.61	1.28
Mg	0.23	0.26	0.08	0.22	0.41	0.47	0.15	0.4
K	0.35	0.44	0.5	0.41	1.01	1.3	1.48	1.19
P	0.12	0.57	0.47	0.11	0.28	1.32	1.1	0.24
Others	0.71	1.08	0.24	0.67	4.61	5.5	1.23	2.81

*ND=Not Detected.

3.3 FTIR Characterization

In determining the presence of functional groups on the surface of the samples, a Fourier Transform Infrared (FT-IR) spectrometer (Shimadzu 8400s) was used. The IR spectra of raw CS and prepared activated carbon from CS at activation times of 6, 12, and 24 hr are displayed in Figures 3, 4, 5, and 6, respectively. Interpretation of these spectra is based on the work cited in reference [14]. The analysis indicated bands at 3324, 3537.2, 3552.2, and 3589.4 cm^{-1} for CS, A1, A2, and A3, respectively, indicating the presence of -OH functional groups due to hydrogen bonding in cellulose [15, 17]. This peak is broader in the coconut shell. The band at 720-590 cm^{-1} for

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all the samples is attributed to the out-of-plane bend of the -OH group [4, 14]. The FTIR analysis revealed aromatic ring vibrations between 2000-1660 cm⁻¹ [16], the peak observed at 1026.9, 1058.6 and 1032.5 cm⁻¹ for samples CS, A2 and A3 respectively can be linked to silicate or phosphate ion or -C-N, while asymmetric stretch of –C-H was observed at 2992.2 cm⁻¹ for CS and symmetric stretch of –C-H at 2885 cm⁻¹ for samples A1 and A3. The bands observed in the range of 2325.9 cm⁻¹ and 2478.7 cm⁻¹ indicated the presence of –COOH groups on A2 and A3 surfaces, respectively [17]. Similar trend in peaks were observed for all the samples of activated carbon, with shifts in the spectra. However, vibrations at 1720.2 cm⁻¹ for sample CS only are probably due to the stretching vibration of the C-O bond in the phenolic group, and the peak at 1459.2 cm⁻¹ refers to the -C-H group of lignin [10]. The FT-IR analysis depicts the presence and existence of a variety of functional groups (such as hydroxyl, carboxyl, and aromatic compounds), some of which can serve as important adsorption sites for pollutants.

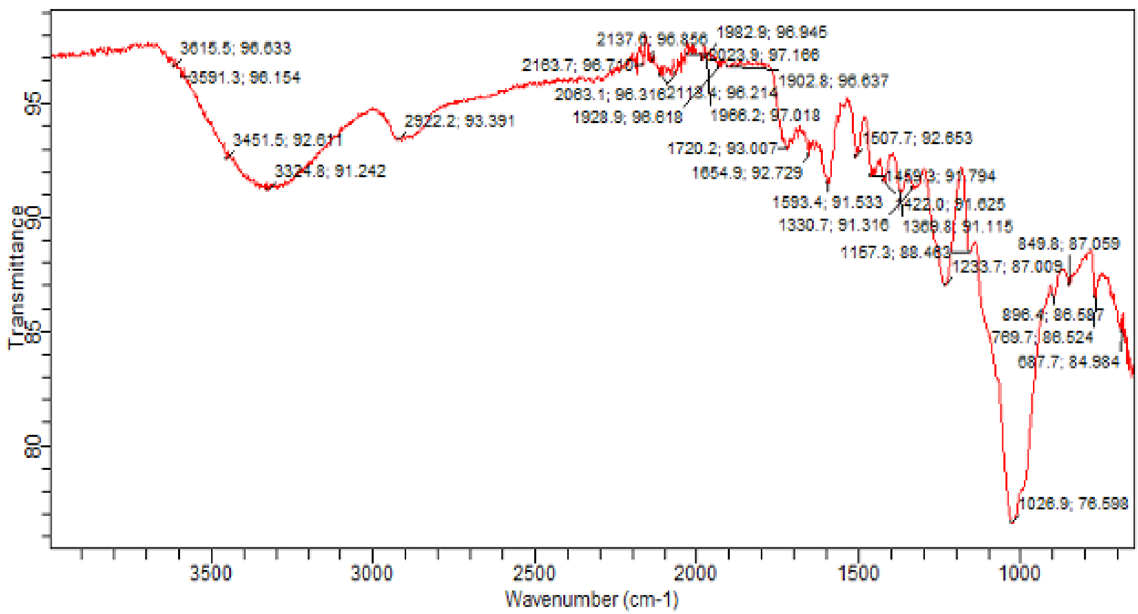

Figure 3: The FTIR spectrum of raw CS powder.

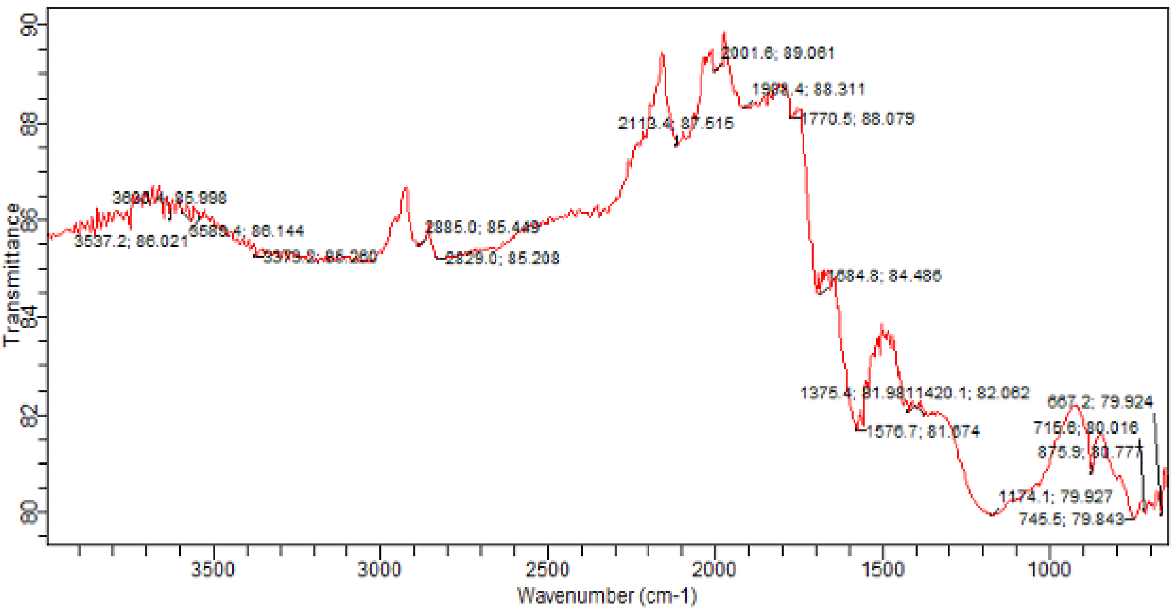

Figure 4: The FTIR spectrum of Activated carbon (activation time of 6hr) (Sample A1)

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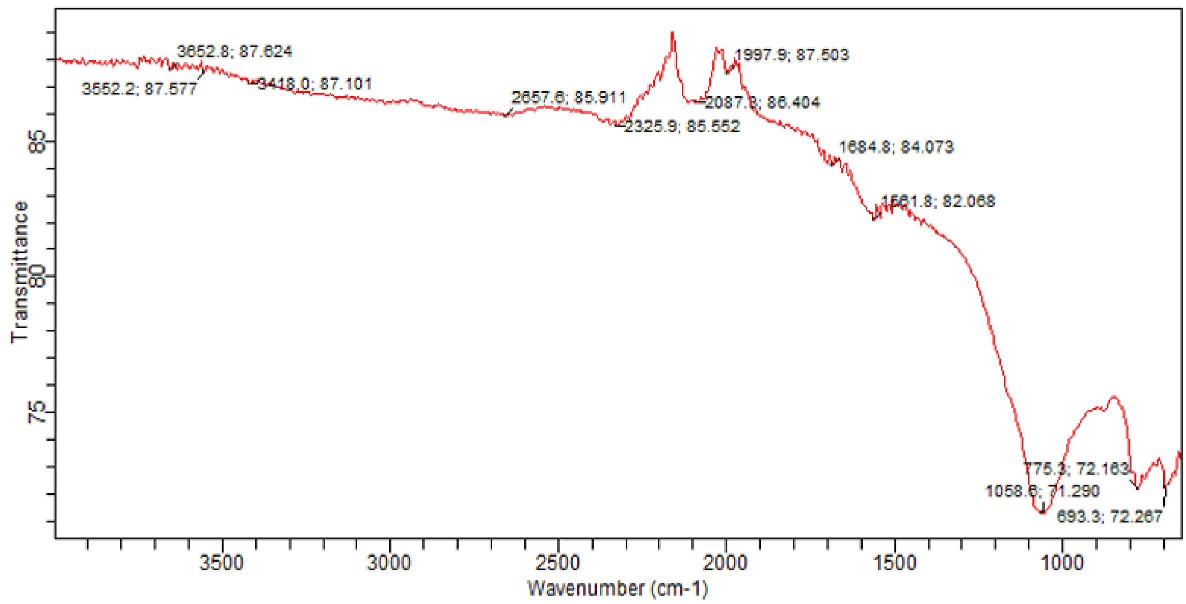

Figure 5: The FTIR spectrum of Activated carbon (activation time of 12 hours) (Sample A2)

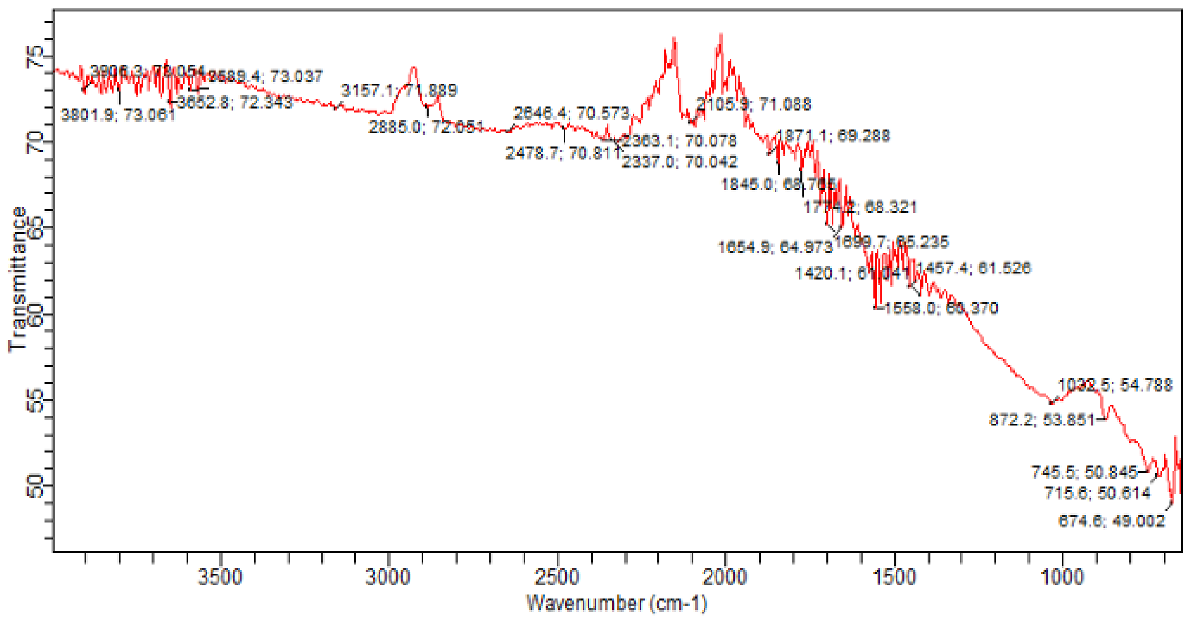

Figure 6: The FTIR spectrum of Activated carbon (activation time of 24 hours) (Sample A3)

4.0 Conclusion

Utilizing CS by using phosphoric acid as chemical activating agent, activated carbon was successfully prepared. Textural analysis was conducted with a gas sorption analyzer, SEM, and FTIR spectroscopy. Findings from the result of the surface area analysis suggest that prepared activated carbon at a higher activation time of 24 hr exhibits the highest surface area, and micropore volume of 505.8 m^2/g and 0.311 cm^3/g, respectively. The pore diameter has similar value between 2.1 and 2.4 nm, which implies that the activated carbon samples prepared are in the mesopore region. Larger pores are present in the activated carbon produced at 24 hr activation time. This finding is corroborated by the result of the SEM micrograph. The existence and occurrence of several functional groups, such as hydroxyl, carboxyl, and aromatic compounds, is shown by FT-IR. Some of which can serve as important adsorption sites for pollutants. The variation of activation time significantly affects the characteristics of the prepared activated carbon. The obtained results confirmed that coconut shell is a promising low-cost precursor to activated carbon utilization and production, which is useful for environmental applications.

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Declaration of Conflicting Interest
The authors declare that they have no known conflicting interests that could have affected the publication of this research work.

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