

Characterization of Chemically Activated Carbons Produced from Coconut and Palm Kernel Shells Using SEM and FTIR Analyses

Boadu Kwasi Opoku¹, Asiamah Isaac², Anang Akrofi Micheal¹, John Kwesi Bentum², Wanjala Paul Muyoma³

¹Industrial Chemistry Unit, Department of Chemistry, School of Physical Sciences, University of Cape Coast, Cape Coast, Ghana

²Department of Chemistry, School of Physical Sciences, University of Cape Coast, Cape Coast, Ghana

³Bomet University College, School of Pure and Applied Sciences, Bomet County, Kenya

Email address:

koboadu@ucc.edu.gh (B. K. Opoku)

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Abstract: Chemically activated carbons generated from coconut (CS) and palm kernel (PKS) shells soaked with 1M solution of K_2CO_3 and $NaHCO_3$ at $1000^\circ C$ using the Carbolite Muffle Furnace were examined using scanning electron microscopy (SEM) and Fourier Transformation Infrared Spectroscopy (FTIR). Results from the FTIR analyses revealed that the coconut and palm kernel shells manufactured were successfully chemically activated. Several chemical compounds and functional groups, such as hydroxyl groups, carbonyl groups, ethers, alkanes, alkenes, and aromatic groups, were detected in chemically activated carbon produced from palm kernels and coconut shells as proof of the lignocellulose structure in them. Chemically activated carbon made from coconut shells exhibited nine distinct spectra, while palm kernel shells exhibited six distinct spectra. The pores were larger in the chemically activated carbons produced at a higher temperature ($1000^\circ C$), demonstrating that temperature is an essential process parameter in the development of surface porosity in chemically activated carbons. The chemical carbonization activation methods used provided porosity, a large surface area, and precise morphology for absorption in both the coconut and palm kernel shells, indicating that they can be turned to high-performance adsorbents. Both organic and inorganic contaminants can be removed from the environment using the chemically activated carbons produced.

Keywords: SEM and FTIR Analyses, Pollutants, Chemically Activated Carbons, Palm Kernel Shell and Coconut Shell

1. Introduction

Activated carbon has been demonstrated to be the most effective and beneficial adsorbent for removing pollutants from contaminated gas and liquid flow after extensive investigation. This is due to the activated carbon's huge active surface area, which allows for a well-developed porous structure with high absorption capacity and excellent mechanical qualities. [1]. Arami-Niya *et al.*, 2012, [2] also reported that activated carbon has a wide active surface area, high adsorption capacity, porous morphology that is well developed, and superior mechanical qualities. Because of their chemical (e.g. surface groups) and functional features, activated carbons are the most widely used (e.g. morphology

and pore size distribution) [3]. They are typically designed and altered to meet the needs of their application. [4, 5]. Furthermore, adsorption on activated carbon appears to be one of the most preferred ways because the adsorbents can be made exceedingly efficient, stress-free to handle, and, in some cases, regenerated [6].

Activated carbons generated from coconut and palm kernel shells can be used as a high-performance adsorbent with improved adsorption capacity, according to Boadu *et al.*, 2020. They also discovered that certain types of the heavy metal contents in used lubricating oil samples might be decreased to acceptable levels by re-refining with chemically activated carbons.

On the other hand, the prices of 1 kg commercially available

chemically activated carbon obtained from Sigma-Aldrich with product number 242276 Darco®-100 mesh particle size powder was 236.00 euros, which is expensive and therefore limit their global applications. This sparked a surge in research into the manufacture of chemically activated carbons from abundant and low-cost precursors, such as coconut shell, which are primarily industrial and agricultural by-products [7] and palm kernel shells [8] which have been successfully used. Chemically activated carbon has been developed from a variety of agricultural wastes, including corn cobs, palm kernel shells, coconut shells, nuts kernels, and rice husks, according to previous research [8]. SEM and FTIR studies were used to assess chemically activated carbons prepared from palm kernel shell and coconut shell that had been chemically activated with potassium carbonate (K_2CO_3) and sodium bicarbonate ($NaHCO_3$).

2. Raw Materials and Methods



Figure 1. Palm kernel shell.



Figure 2. Coconut shell.

Coconut shell (CS) and palm kernel shell (PKS) were employed for chemically activated carbon synthesis among the numerous agriculture by-products. Both PKS and CS were collected from the Aluu based Oil Palm Mill Industry and the Choba Local Community Area, both in Port Harcourt, Rivers State, Nigeria. These ingredients (palm and coconut shells collected) were carefully washed with tap water, then extra with purified water for numerous times to get rid of dirt and all other pollutants. PKS and CS samples were then dried

for 24 hours at $110^\circ C$ in a spray dryer oven to remove all exterior moisture. The dried samples remained were pulverised based on ASTM-D2862-(2016) [9]. The powdered samples were then sieved with 500 μm mesh and then stored in an airtight container for future analyses.

2.1. Preparation of Chemically Activated Carbon

Preparation of chemically activated carbons from coconut and palm kernel shells were carried out using the chemical activation method by Boadu et al., 2018 [8]. 30 g of powdered samples (coconut and palm kernel shells) were impregnated with a 1 M solution of K_2CO_3 and $NaHCO_3$ and allowed to sit at room temperature for three hours. The samples were chemically activated for 40 minutes in a Carbolite Muffle Furnace at a high carbonization temperature of $1000^\circ C$. After being treated with a 0.5 M glacial acetic acid solution, the chemically activated carbons were thoroughly rinsed with distilled water until the pH values were between 6-7. Sun-dried samples were sieved with a 500 μm mesh sieve. The chemically activated carbons that remained on the mesh were dried for 1 hour in the oven, then removed and stored in airtight containers. Chemically activated carbons have a surface area ranging from 250 to 2500 $m^2 g^{-1}$, according to the literature [8], depending on the precursor and the type of chemical treatment utilized. The most typical values are in the range of 600 to 1000 $m^2 g^{-1}$ [10, 11].

2.2. Scanning Electron Microscopy (SEM)

The morphology of the materials (chemically activated carbons from palm kernels and coconut shells) was determined using a JSM-7610F scanning electron microscope (Tokyo, Japan). The equipment is an ultra-high resolution Schottky Field Emission Scanning Electron Microscope which has a semi-in-lens objective lens and high power optics that can provide high throughput as well as high-performance analyses. The morphology of the adsorbents was investigated using scanning electron microscopy analysis (i.e. carbons from palm kernels and coconut shells that have been artificially activated).

2.3. Fourier Transform Infrared Spectra (FTIR)

The Fourier transform infrared spectra (FTIR) of chemical activated carbons from palm kernels and coconut shells (in the form of KBr pellets) were analyzed in the range 400-4000 cm^{-1} with a resolution of 4 cm^{-1} using a Digilab Excalibur FTS 3000 series spectrometer. FTIR spectroscopy was used to analyze and identify the primary functional groups discovered in the structures of chemically activated carbons from palm kernels and coconut shells.

3. Results

The findings of the tests are provided in the form of figures and tables, which are followed by in-depth scientific explanations.

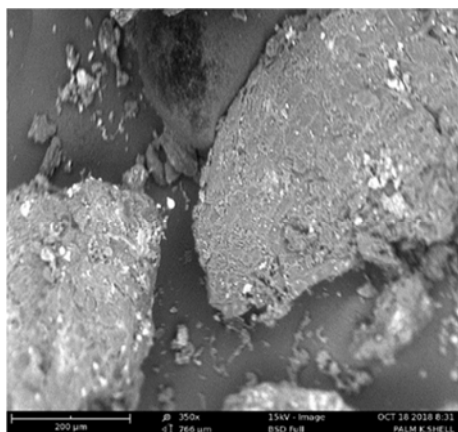


Figure 3. SEM micrograph of Palm Kernel Shell.

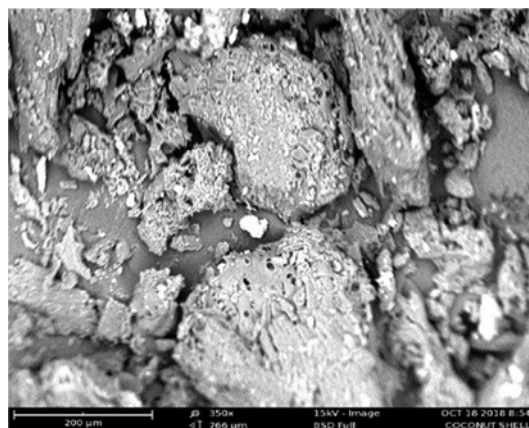


Figure 4. SEM micrograph of Coconut Shells.

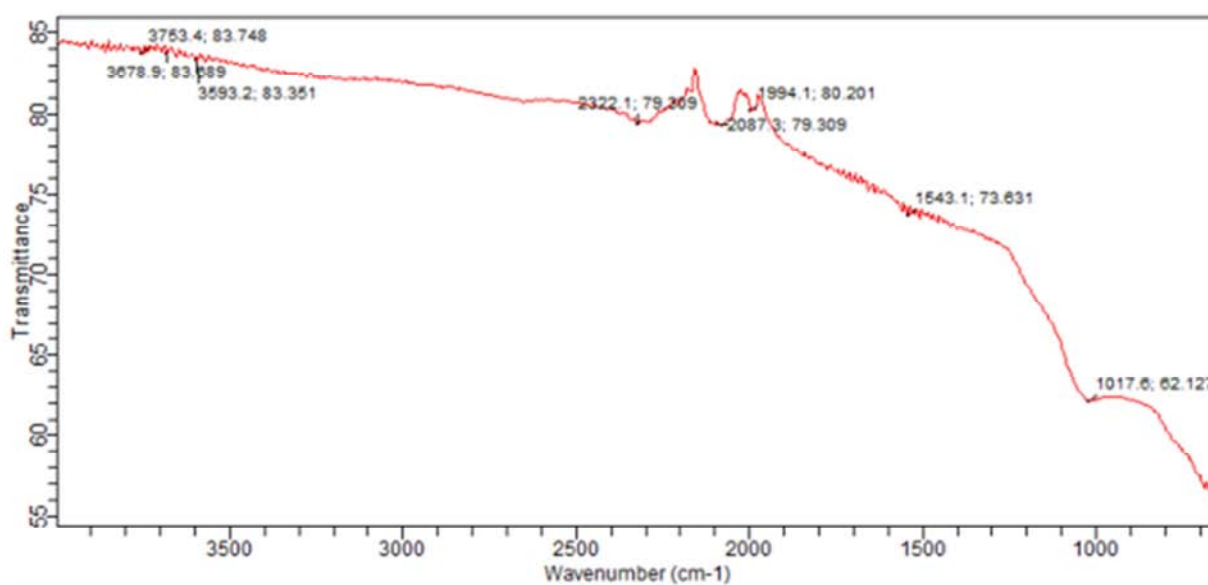


Figure 5. FTIR spectra of Palm kernel Shell.

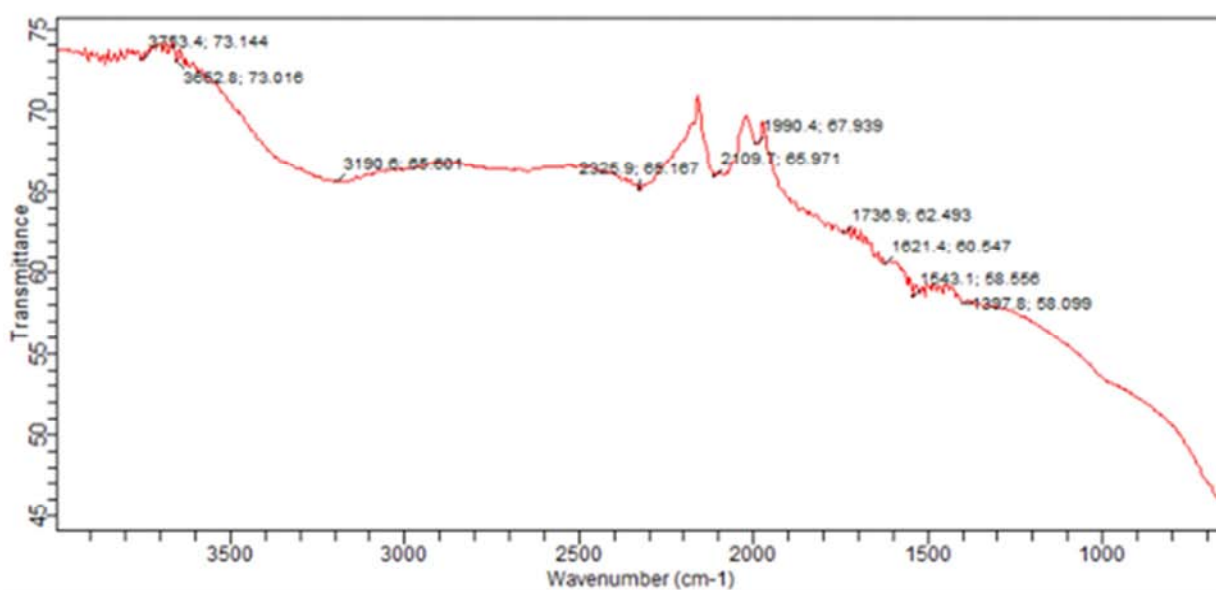


Figure 6. FTIR spectra of Coconut Shell.

Table 1. FTIR study revealed functional groups and classifications of chemicals in chemically activated carbon from palm kernel shell.

The range of wavenumbers shows the transmittance peaks. (cm ⁻¹)	Samples per wavenumber (cm ⁻¹)	Functional groups	Classification of compounds
3800-4000	3753	O-H broadening	Alcohol, Phenol and Hydroxyl group
3600-3800	3678	O-H extending	Alcohol, Phenol and Hydroxyl group
3000-3600	3593	O-H elongating	Alcohol, Phenol and Hydroxyl group
2300-2800	2322	-COOH broadening	Carboxylic acid group
2000-2300	2087	C=N broadening	Nitriles
1700-2000	1994	CH ₂ =CH ₂	Alkynes
1300-1600	1543	CH ₂ and CH ₃ twisting	Alkanes
1000-1200	1017	C-O Bending	Alcohol; Ether; Carboxylic acid

Table 2. Functional groups and the classification of compounds identified in chemically activated carbon from Coconut shell by FTIR analysis.

The range of wavenumbers shows the transmittance peaks. (cm ⁻¹)	Samples per wavenumber (cm ⁻¹)	Functional groups	Classification of compounds
3800-4000	3753	O-H stretch	Hydroxyl group
3500-3700	3652	O-H stretch	Hydroxyl group
3100-3400	3190	≡ C-H stretch	Alkyne
2200-2400	2325	C≡C stretch	Aldehyde
2100-2300	2109	C=C=C band	Allene group
1900-2000	1990	C=C=C band	Allene group
1700-1800	1736	C=C Broadening	Pyrene
1600-1700	1621	C=O axial deformation	Aldehyde; Ketones; Carboxylic acid
1500-1600	1543	C=C stretch	Aromatics
1300-1500	1397	C-H Twisting (CH ₂ and CH ₃)	Alkanes

4. Discussions

4.1. Chemically Activated Carbons Made from Palm Kernels and Coconut Shells Have Different Surface Morphologies

Figure 3 depicts the surface morphology of chemically activated carbons produced from palm kernel shell at 200 μm magnification in the Carbolite Muffle Furnace at 1000°C. After the carbonization process, pores were discovered on the surface of the chemically activated carbon. This means that the carbonization process made released volatile organic molecules from the palm kernel shell, while the non-volatile components were changed into chemically activated carbon with pores of varied shapes and sizes. The chemically activated carbons generated at a higher temperature (1000°C) had larger pores, implying that temperature is a critical process variable in the formation of surface porosity in chemically activated carbons. Within the PKS, more heat energy was created when the temperature was raised, which aided in the degradation of lignocellulosic components into more volatile materials. As a result of the volatile organic matter that has been released during carbonization process at a higher level of temperature, more pores were obtained.

Also, the pores on the surface of chemically activated carbons from palm kernel shells at high temperature were observed with fewer impurities as shown in the figure 3, with 200 μm magnification. This could be owing to the fact that the carbolite muffle furnace pyrolysis technology used in this study was based on a vacuum rather than a continuous flow of nitrogen gas. Compared to standard chemical activation, vacuum condition requires less energy, simultaneity, and biomass-oil is collected more easily in the method, lowering

the volatile matter's residence time inside the reactor. Clogging is to hinder or obstruct with thick or sticky matter; choke up: especially so that movement is impeded. As a result, the pore-clogging, re-condensation reaction of volatile molecules on the surface of chemically activated carbon from palm kernel shell can be prevented, resulting in the formation of "cleaner" pores on the chemically activated carbon's surface [12, 13].

In contrast to the above, figure 4 shows the surface image or morphology of chemically activated carbons made from coconut shells using Carbolite Muffle Furnace set at 1000°C. During the carbonization process, the nature of chemically activated carbon is known to be a key component in determining the extent of carbon-richness [14]. Carbonization of carbon materials has a combined action that results in hierarchically porous characteristics [5]. The SEM image further supports this conclusion. Micropores in the exterior and interior of chemically activated carbons are shown by a translucent mesopore and a micropore with a rough surface. According to the previous studies [8, 10, 12, 14], more active sites and a larger BET surface area are both features of chemical activated carbon with a suitable grainy surface. These characteristics explain the adsorption mechanism and increase the adsorbate removal capacity per unit of sorbent [15]. Jalani *et al.*, 2016 [15] reported that a quick adsorption process is usually observed because there are more bare pores and active sites available at the start of the adsorption phase. This method may have a foggy appearance due to the deposited adsorbate [16].

Also, the image of the SEM shows that the activation stage produced a sizeable external surface with quite, pores and very high porosity. The amorphous character of the carbons, the smoot characteristic, and the presence of carbon-rich big molecules were shown by SEM patterns. As a result of the

micro and nanoscale discoveries, many researchers [17] now see activated carbon as a structure made up of fragments. The chemically activated carbon micrograph also shows the modification of the shell matrix as a result of continuing heating during the activation. In the magnified SEM cross image, the composite coal materials between activated carbon and binder composite material can be observe. Surface images of chemically activated carbons from coconut shells reveal a rough and layered carbon surface with a disorder defined as amorphous, which becomes more obvious as magnification increases. The SEM morphology of the coconut shell reveals visible pores in chemical activated carbon samples from the coconut shells, showing that the activated carbon processes were successful.

4.2. Analyses of Chemically Activated Carbon Samples from Coconut and Palm Kernel Shells using Fourier Transform Infrared (FTIR)

The chemically activated carbons produced from coconut and palm kernel shells were examined using the FTIR. This was carried out because FTIR is a well-known state of the art method for analysing surface area chemistry. Surface functional groups in the various chemically activated carbons were characterized by FTIR spectroscopy, figures 5 and 6 showing palm kernel and coconut shell FTIR spectra respectively. Surface area functional groups containing oxygen have a significant impact on chemically activated carbon features such as surface hydrophobicity and charges [18].

The FTIR spectra of chemically activated carbons from palm kernel (PKS) and coconut shells (CS) produced from Carbolite Muffle Furnace at a temperature of 1000°C for 1 hour are shown in Figures 5 and 6, as well as Tables 1 and 2. The wavenumbers and functional group classifications in chemically activated carbons from palm kernels and coconut shells were remarkably alike. The majority of the activated carbon peaks were seen at several wavenumber ranges, including C-H stretching, C-C stretching, O-H stretching, C=C stretching, C-H bending, C-O bending, and so on. Some of the peaks seen in the PKS and CS spectra (i.e. O-H stretching, C-H stretch, C-H bending, and C-O bending) were also seen in the spectra of the chemically activated carbons.

PKS and CS had a broad peak at 3000–3800 cm^{-1} (i.e. peak (a) in the figures). The O-H stretching was attributed to the presence of chemical compounds with hydroxyl functional groups such as phenolic or aliphatic alcohol and carboxylic acid in both the activated carbons from PKS and CS, which indicates the presence of chemical compounds with hydroxyl functional groups such as phenolic or aliphatic alcohol and carboxylic acid in both the activated carbons from PKS and CS. After further carbonization, the hydroxyl group was found to be undetectable in the chemically activated carbons spectra, implying that hydroxyl-containing compounds (e.g. CH_3OH and CH_3COOH) were released as volatile matter (most likely via the fragmentation reaction of hemicellulose [19, 20] and cracking of the alkyl-hydroxyl chain present in lignin [21]).

However, as revealed in the tables, the peak present in this

range (i.e. peak (b) in figure 6) was created from chemical compounds with a C-H stretching functional group in CS alone, not in PKS.

Also, the peak presence in this range was obtained from chemical compounds having -COOH stretching and CC stretching functional groups in PKS and CS, respectively, at 2300–2800 cm^{-1} (i.e. peak (b) in figures 5 and 6.). The peak present in this range (i.e. peak (c) in figures 5 and 6) was formed from chemical compounds having C=C=C band for CS and C=N stretching for PKS, respectively. A similar pattern of peaks was observed at peak ranges 1900–2000 cm^{-1} for chemical compounds with C=C=C band for CS and $\text{CH}_2=\text{CH}_2$ stretch for PKS respectively. The distinct peaks obtained at these ranges indicated the uniqueness of the chemically activated carbons created from coconut and palm kernel shells. The difference in the peaks recorded in both CS and PKS shows a clear indication that each activated carbon have peculiar characteristics and adsorption pattern.

The presence of compounds with C=C stretching and C=O axial deformation was demonstrated by transmittance peaks at 1700–1800 cm^{-1} and 1600–1700 cm^{-1} (i.e. peak (d) in figure 6. However, these peaks were only found in CS spectra. In both CS and PKS, however, chemical compounds with C=C stretching and CH_2 and CH_2 twisting each had a peak in the 1500–1600 cm^{-1} range. In addition, in the ranges 1000–1200 cm^{-1} , a substantial transmittance peak (nearly 30%) of the PKS spectra for C-O bending (peak (f) in figure 5.) was detected, indicating that alcohols, carboxylic acids, esters, or ethers were abundant inside the PKS. In contrast, only in the ranges 1300–1500 cm^{-1} did a peak from chemical compounds with C-H bending appear for CS, indicating the presence of alkanes.

Furthermore, the absence of C-O stretching and decreased peak intensity of C-O bending observed in chemically activated carbon spectra could have come from the removal of oxygenated molecules during PKS carbonization conversion [22, 23]. These oxygenated compounds were obtained during carbonization through the release of volatile matter from the decomposition of lignocellulosic components, leaving the residual carbon as chemically activated carbon with low oxygen content. As a result, after the carbonization procedure, both the PKS and the CS were changed into chemically activated carbons with greater aromaticity. This transition can be achieved by releasing H_2 to form the unsaturated product, demethylation of short substituents (e.g. $-\text{OCH}_3$ and $-\text{CH}_3$) to produce polycyclic aromatic compounds, or dehydration to produce alkene and aromatic compounds by releasing H_2O .

Finally, sample from palm kernel shells showed six well-defined signals (i.e. spectra). The broad- bands at 3593 cm^{-1} , 3678 cm^{-1} and 3753 cm^{-1} were due to the O–H stretching mode of hydroxyl, alcohol and phenol groups as well as adsorbed water [24, 25]. The band at 2322 cm^{-1} can be assigned to –COOH stretching mode of carboxylic acid groups [26]. A band with 2087 cm^{-1} could be attributed to $-\text{C}=\text{N}$ stretch mode of nitriles [10]. The band at 1994 cm^{-1} can be attributed to the $\text{CH}_2\equiv\text{CH}_2$ stretchingmode of alkynes [10]. A band with 1543 cm^{-1} can be assigned $-\text{CH}_2$ and CH_2 twisting mode of alkanes

[18] while a band with the lower definition at 1017 cm^{-1} can be attributed to C-O bending mode of alcohol, ether and carboxylic acid [27]. The presence of numerous chemical compounds and their functional groups such as hydroxyl groups, carbonyl groups, ethers, alkanes, alkenes, and aromatic groups, as well as other components identified in Brazilian coconut shells [28], is proof of the lignocellulose structure of coconut shells.

However, the sample from the coconut shells showed nine well-defined signals (i.e. spectra). The broad bands at 3652 cm^{-1} and 3753 cm^{-1} were due to the O-H stretching mode of hydroxyl, alcohol and phenol groups [29]. The band at 3190 cm^{-1} can be assigned to $\equiv\text{C-H}$ stretch mode of the alkyne [24]. A band with 2325 cm^{-1} can be attributed to $\text{C}\equiv\text{C}$ stretching mode of aldehyde [30]. The bands at 2109 cm^{-1} and 1990 cm^{-1} were assigned to $\text{C}=\text{C}$ band mode of allene [24]. A band at 1736 cm^{-1} can be allotted to $\text{C}=\text{C}$ stretching mode of pyrone [28]. The band at 1621 cm^{-1} can be attributed to C=O axial deformation modes of aldehyde, Ketone, Carboxylic acids [24, 25]. A band with a higher definition at 1543 cm^{-1} may be attributed to aromatics' $\text{C}=\text{C}$ stretching mode [30], whereas a band with a lesser definition at 1397 cm^{-1} can be attributed to alkanes' C-H bending mode.

5. Conclusions

Finally, FTIR analyses of chemically activated carbon generated from palm kernel shells revealed six well-defined signals (i.e. spectra), whereas examinations of the coconut shell sample revealed nine well-defined signals (i.e. spectra). Also, SEM morphology of the palm kernel and coconut shells shows that visible pores and apparent pores were present, indicating that the chemically activated carbon processes were successful. When it comes to eliminating pollutants, chemically activated carbon adsorbents made from palm kernel and coconut shells are a good substitute for synthetic adsorbents like silica gel, according to this study.

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