American Academic Scientific Research Journal for Engineering, Technology, and Sciences
ISSN (Print) 2313-4410, ISSN (Online) 2313-4402
http://asrjetsjournal.org/

Production and Characterization of Paw-paw Trunk Activated Carbon

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Abstract

Low-cost adsorbents were prepared from matured Pawpaw trunk by chemical activation method using $ZnCl_2$ at various impregnation ratios of 1:2, 1:3 and 1:4 ($ZnCl_2$/pawpaw). Unwashed Pawpaw Trunk activated Carbon (UPTAC) and Raw Pawpaw Trunk carbon (Raw PTC) (not impregnated) were all carbonized at 400^{o}C for one hour. The produced carbons were characterized in terms of surface area, porosity, bulk density, ash content, moisture content, iodine number and carbon yield. Proximate analysis was also conducted using Scanning Electron Microscopy (SEM) to detect the surface morphology and Fourier Transform Infra-red (FTIR) to identify the kind of functional groups present on the activated carbon. The results showed that surface area, bulk density, porosity, iodine number, moisture content and carbon yield of UPTAC decreased significantly with increase in impregnation ratio with ratio 1:2 having the highest surface area of 1575m^2/g, porosity of 0.89, carbon yield of 69%, bulk density of 0.68g/cm^3 and iodine number of 231.1mg/g while Raw PTC had the lowest surface area of 652m^2/g. The results of the SEM micrograph showed that UPTAC has a well-developed porous surface with different pores diameter than the Raw PTC indicating the impact of chemical activation in creating well-developed pores. Furthermore, the results of the FTIR transmittance spectra showed several peaks belonging to different functional groups including O-H group in either alcohol, phenol or carboxylic acid; R-OH group and C≡C stretching of the alkynes; weak C=O stretching of the carboxylic groups and the twisted region. Comparing the FTIR transmittance spectra of Raw PTC to UPTAC, showed a clear shift in band indicating the effect of $ZnCl_2$ activation and this shows that UPTAC may be very useful in adsorption processes.

Keywords: Low-cost; adsorbent; pawpaw trunk; activated carbon; surface area; impregnation.

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1. Introduction

Activated carbon (AC) is a black carbonaceous material which is porous and can be distinguished from other elemental carbon by the oxidized surface and its wide application in the treatment of waste. Due to the special pore structure of activated carbon, they have super adsorption capacity and are generally used in variety of industrial and domestic fields, such as water treatment, solvent decolourization, and catalyst supports of fuel cell [1].

In recent years, there is growing interest in the production of activated carbons from agricultural by-products and residual wastes. Activated carbons can be produced from coal, wood or agricultural wastes such as coconut and palm shell, corncob, rich husk, etc., activated by physical or chemical process. The carbonization process enriches the carbon content and creates an initial porosity in the char while activation further develops the porosity and enhances ordering of the structure, thereby generating a highly porous solid as the final product [2].

Activated carbon has been prepared from Pawpaw leaf [3] while the Pawpaw seed and stem has been used for the removal of heavy metals from wastewater without activation [4,5,6]. Very few studies have been reported on the carbonization and activation of pawpaw trunk as adsorbent. The choice of pawpaw trunk as precursor for the production of low-cost adsorbent is due to its relative abundant, cheap, excellent porous structure and renewability.

Pawpaw (Carica papaya) belongs to the family of Caricaceae. It is an African native commercial tree whose trunk becomes useless at the end of its productive life cycle. To make economic use of this agricultural waste, there is need to research on how it can be converted to activated carbon and to substitute to the high cost commercial activated carbon. This paper aimed at reporting the production and characterization of pawpaw trunk activated carbon with zinc chloride ($ZnCl_2$) as the activating agent.

2. Materials and methods

2.1 Preparation and Activation of Pawpaw Trunk Carbon

Pawpaw trunk obtained from felled matured pawpaw tree was sourced from Lubara in Khana LGA of Rivers state. The Pawpaw trunk was cut into smaller sizes, washed and sun dried to reduce the moisture content. One part of the dried pawpaw trunk was activated with $ZnCl_2$ at different impregnation ratios of 1:2, 1:3 and 1:4 ($ZnCl_2$/Pawpaw) and then carbonized at 400^{o}C for one hour. It is then cooled and crushed to powder and named Unwashed Pawpaw Trunk Activated Carbon (UPTAC). The other part of the dried pawpaw trunk was not impregnated but was also carbonized at 400^{o}C for hour and then cooled and crushed into powder and named Raw Pawpaw Trunk Carbon (Raw PTC). This nomenclature was used to identify the two variations of pawpaw trunk activated carbons prepared.

2.2 Characterization of the Pawpaw Trunk Activated Carbon (UPTAC and Raw PTC)

The characterization of the Pawpaw trunk activated carbon was based on determination of the surface area, porosity, carbon yield, moisture content, bulk density, ash content, pH, iodine number, scanning electron microscopy (SEM) and Fourier Transform Infra-Red (FTIR).

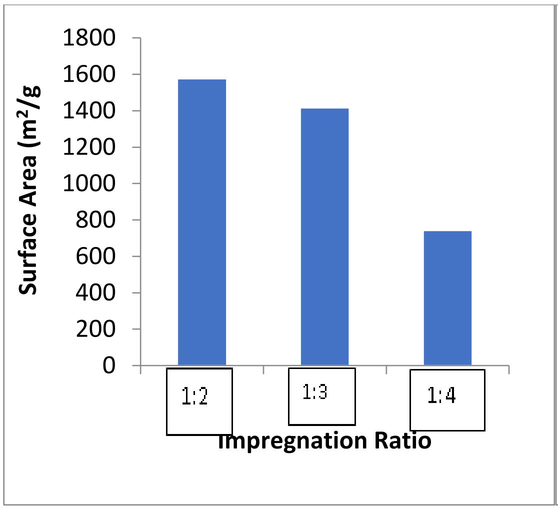
3. Results and discussions

3.1. Characterization of UPTAC and Raw PTC

The results of the characterization of Raw PTC and UPTAC at different impregnation ratios are shown in Table 1. The surface area of Raw PTC (unimpregnated) was 625m^2/g. After being treated with $ZnCl_2$ at various ratios, a drastic increment was achieved. The highest surface area was obtained at impregnation ratio of 1:2 (1575m^2/g). This showed that the activating chemical promoted pore formation and enlargement of small pores within the carbon matrix which subsequently enhanced the surface area. Similar observations were discovered by [7,8,9]. However, a sudden drop in the surface area to 743m^2/g was observed when impregnation ratio was further increased to ratio 1:4. This might be due to limited formation of new micropores and the conversion of existing micropores into mesopores [10,11]. The effects of impregnation ratios on the other properties of Pawpaw Trunk carbon showed that porosity (0.89), carbon yield (69%), bulk density (0.68g/cm^3) and iodine number (231.1mg/g) increased after activation/impregnation than the Raw PTC as shown in Table 1. This may be caused by the activation process, in which the organic substances in the sample became unstable and the bonding or linkage of the molecules break causing the volatile matter to be discharged as gas and liquid products leave the material with high carbon content [10]. However, their ash contents increased with decrease in impregnation ratio. Effects of impregnation ratio on surface area, porosity, iodine number and pH are shown in Figures 1 to 4.

Table 1: Comparison of UPTAC Characterization with Raw Pawpaw Trunk Carbon (Raw PTC)

S/n	Properties	Raw PTC	UPTAC Samples at different Ratios		
		No impregnation	1:2	1:3	1:4
1	Surface area	652m^2/g	1575m^2/g	1415m^2/g	743m^2/g
2	Bulk Density	0.34 g/cm^3	0.68 g/cm^3	0.64g/cm^3	0.62g/cm^3
3	Specific Gravity	1.08	1.32	1.25	1.19
4	Moisture Content	1.5%	3.5%	2.1%	1.9%
5	Porosity	0.78	0.89	0.83	0.82
6	Carbon Yield	25%	69%	55%	53%
7	Ash Content	12.7%	10.2%	10.6%	10.8%
8	pH	10.13	6.30	6.24	6.20
9	Iodine Number	193.7 mg/g	231.1 mg/g	207.54 mg/g	205 mg/g



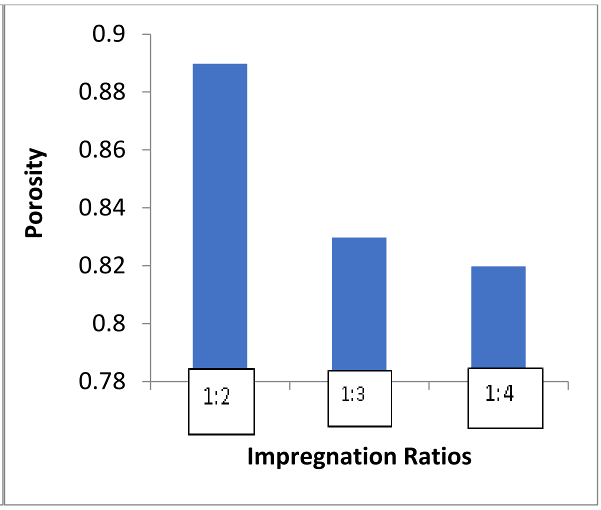
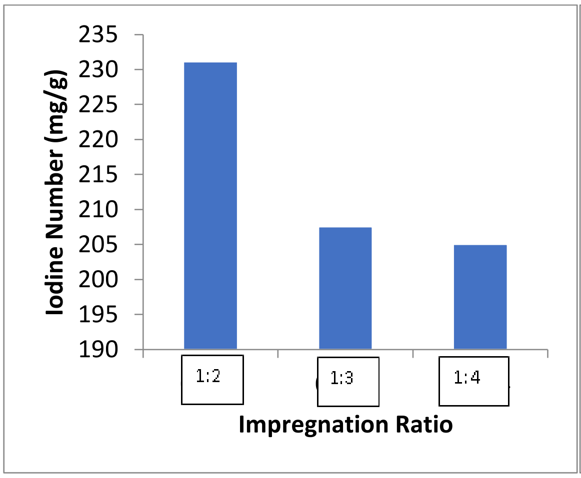


Figure 1: Impregnation Ratio as function of Surface Area. **Figure 2**: Impregnation Ratio as a function of porosity



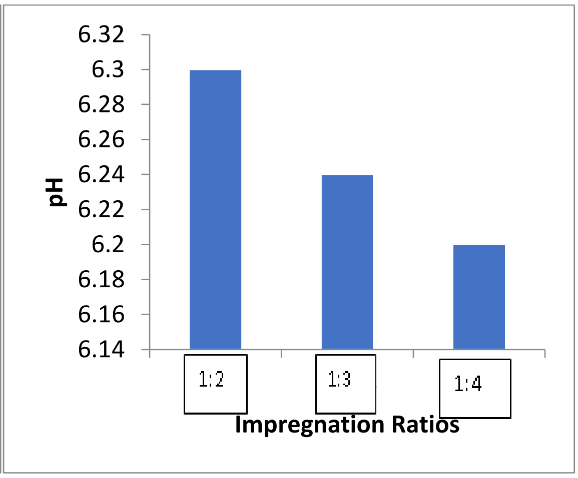


Figure 3: Impregnation Ratio as function of iodine number. **Figure 4:** Impregnation Ratio as a function of pH

The effect of pH on the impregnated carbon (UPTAC) were found to be 6.30, 6.24 and 6.20 for ratios 1:2, 1:3 and 1:4 respectively as shown in Table 1. It has been reported that for most applications, carbon pH of 6 – 8 is acceptable [12]. The pH values obtained are in the range of acceptable limit except for Raw PTC sample with no impregnation which has a pH value of 10.13.

3.2 Results of Scanning Electron Microscopy (SEM) Characterization

The surface structural composition of the Pawpaw trunk carbon samples was examined using the Scanning Electron Microscopy (SEM). Figure 1 and 2 shows the SEM images of Pawpaw trunk activated carbon (UPTAC) with impregnation ratio of 1:2 and Raw PTC with no impregnation respectively. A well-developed porous surface with different pores diameter which appeared rocky and unevenly distributed was observed on SEM image in Figure 1 indicating the impact of the chemical activation in creating well-developed pores on the surface of UPTAC. Similar observation was reported by [13,14]. However, the SEM image obtained for Raw PTC sample in Figure 2 has shown less developed porous surface and fewer pore confirming the low porosity value seen in Table 1. Similar kind of SEM surface structure was reported by [15] for pawpaw seed modified with Feldspar clay. These pores may allow a good surface for dyes, heavy metals and waste effluents to be trapped and adsorbed into it.

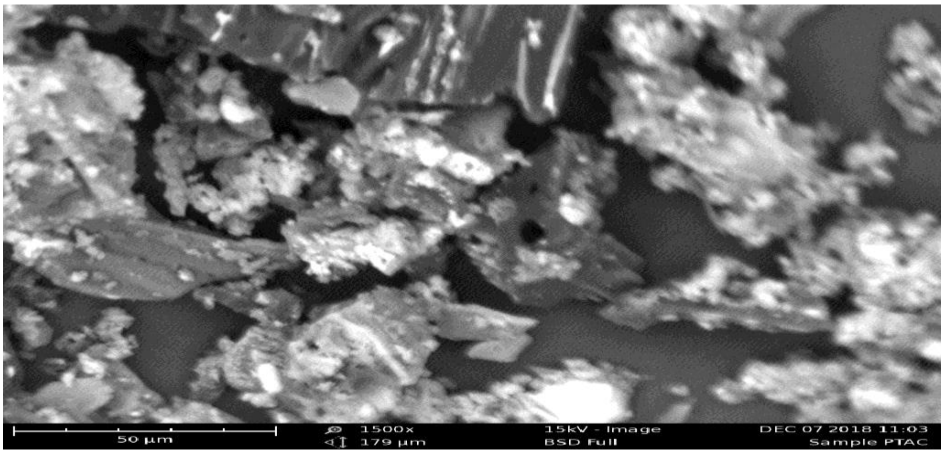


Figure 5: SEM Micrograph of UPTAC Sample

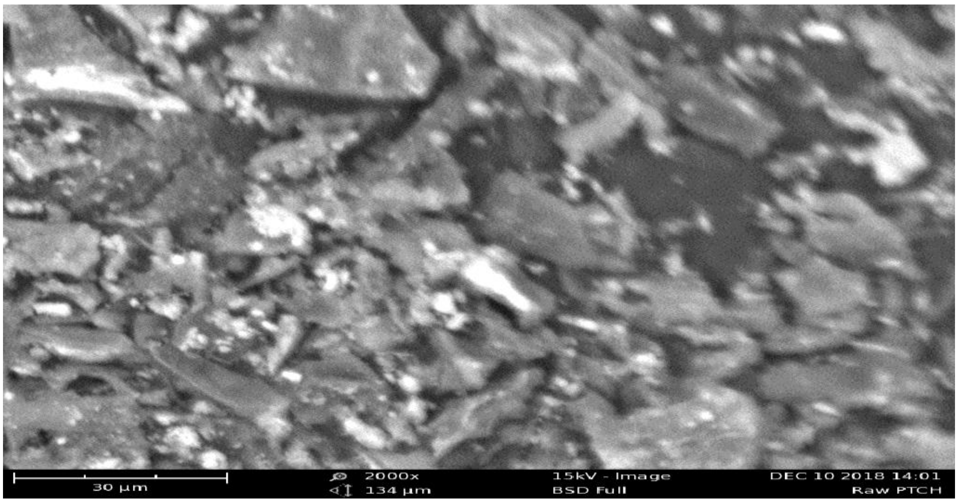


Figure 6: SEM Micrograph of Raw PTC Sample

3.4 Results of Fourier Transform Infra-Red (FTIR) Analysis

The transmittance spectra of the Raw PTC and UPTAC samples are shown in Figures 3 and 4. The spectra displayed a large number of peaks on each spectrum representing different functional groups. In the spectrum of the Raw PTC sample shown in Figure 3, the strong broad band at 3468.3cm^{-1} represents the hydrogen bonded (-OH) groups in either alcohol, phenol or carboxylic acid. Also, the peaks at 2346.4cm^{-1} and 2111.6cm^{-1} correspond to R-OH group and C≡C stretching in the alkynes respectively. The peaks observed at 1735.1cm^{-1} and 1910.3cm^{-1} were weak and strong C=O stretching of carboxylic acid respectively. Other peaks seen at 1543.1cm^{-1}and 1507cm^{-1} are the N-H bending of the amides group while the peaks at 1459.3cm^{-1} and 1474.2cm^{-1} represents the C-H bending of the alkyl groups. Most importantly, the peak at 659.7cm^{-1} has shown that there was overlapping of band at this region.

In the spectrum of UPTAC sample shown in Figure 4, a shifting in band was observed from 3468.3cm^{-1} to 3343.4cm^{-1} forming a medium broad band of O-H stretching of the alcohol groups when compared with the Raw PTC sample while the narrow band formed at 1610.2cm^{-1} disappeared completely. However, the medium narrow band at 1436.9cm^{-1} represents a ring of C=C bond stretching of the aromatic groups while the overlapping band seen in the Raw PTC sample has shifted to 682.1cm^{-1} in the UPTAC sample.

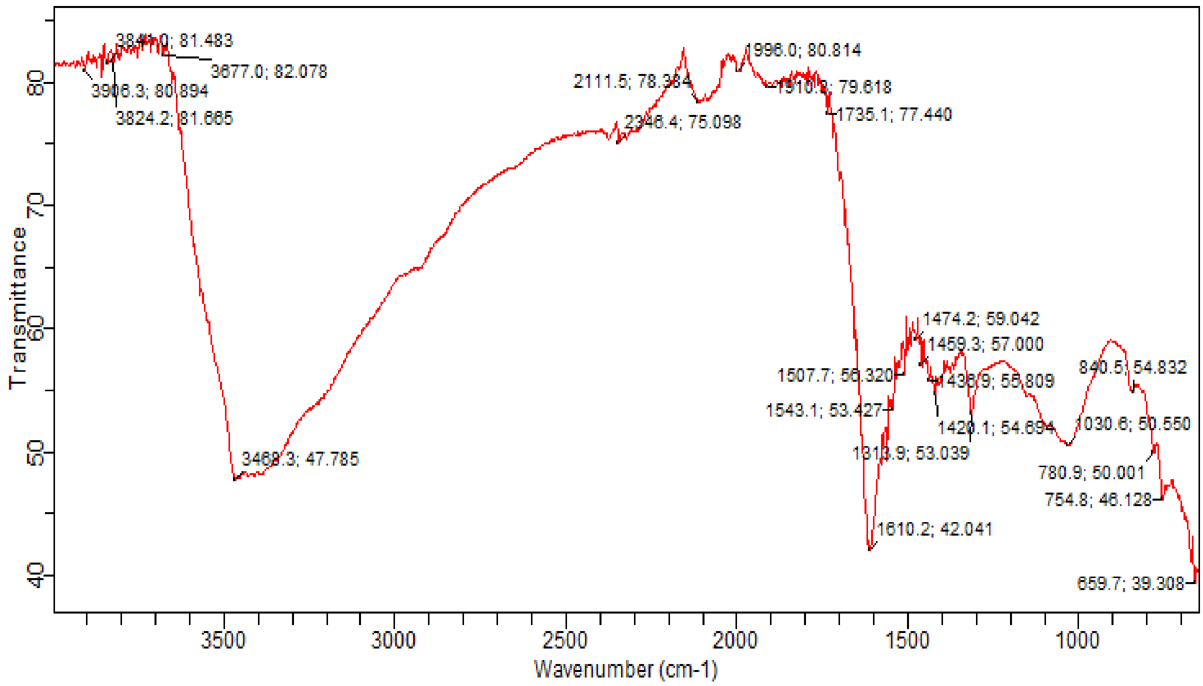


Figure 7: FTIR Spectrum of Raw PTC Sample.

The changes in FTIR spectra (Figures 3 and 4) confirmed the effect of chemical activation on UPTAC indicating that the bonded O-H groups vibrations are due to inter- and intramolecular hydrogen bonding of polymeric compounds (macromolecular association) such as alcohols phenol and carboxylic acids as in pectin, cellulose and lignin, thus showing the presence of free hydroxyl groups on the prepared adsorbents. Similar trends were observed by [16,17,18]. Thus, the FTIR analysis pronounced a clear band shift for Raw PTC when compared to UPTAC. All due to the impact of $ZnCl_2$ activating agent.

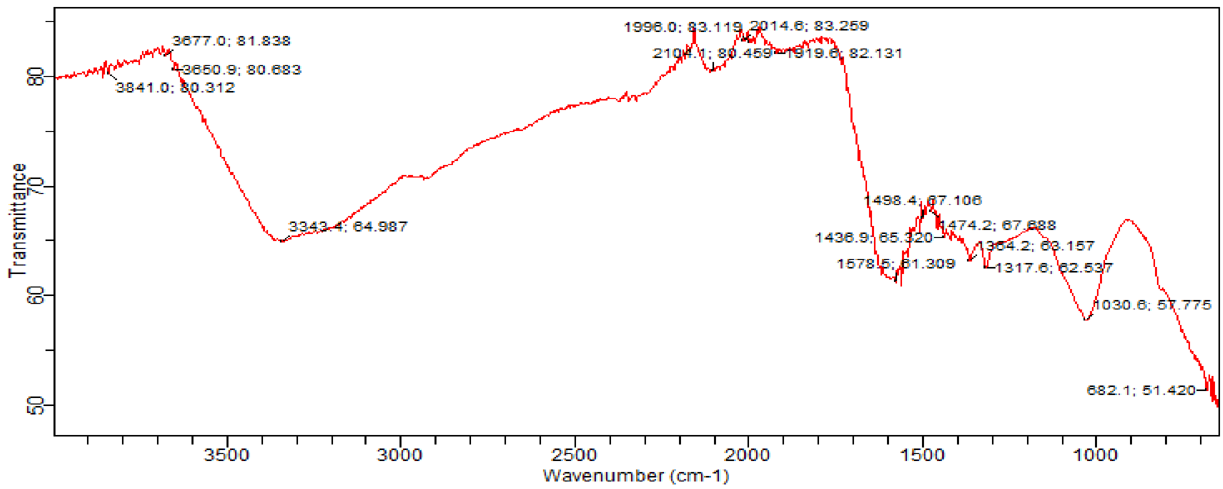


Figure 8: FTIR Spectrum Activated PTAC Sample

4. Conclusion

This study shows that UPTAC sample, activated with $ZnCl_2$ at an impregnation ratio of 1:2, gave the highest surface area, porosity, bulk density and iodine number of 1575m^2/g, 0.89, 0.68g/cm^3 and 231mg/g respectively. Also, the results of the SEM micrograph showed that UPTAC has a well-developed porous surface with different pores diameter than the Raw PTC indicating that chemical activation may have influenced the porosity of the Pawpaw trunk carbon. Furthermore, the results of the FTIR transmittance spectra showed several peaks belonging to different functional groups including O-H group in either alcohol, phenol or carboxylic acid; R-OH group and C≡C stretching of the alkynes; weak C=O stretching of the carboxylic groups and the twisted region. However, comparing the FTIR transmittance spectra of Raw PTC to UPTAC, there is a clear shift in the broad band transmitted by the UPTAC sample from 3468.3cm^{-1} to 3343.6cm^{-1}, the overlapping band formed at 659.7cm^{-1} changed position to 682.1cm^{-1} and the narrow band observed at 1610.2cm^{-1} disappeared completely. The pronounced shift may be due to the effect of $ZnCl_2$ activation, indicating that UPTAC would be very useful in adsorption processes.

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