Research Article

Synthesis and Characterisation of Activated Carbon Obtained from Marula (*Sclerocarya birrea*) Nutshell

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Globally, a ninth of people use polluted water sources because an estimated 300–400 Mt of waste and 90% of sewage are discharged into water bodies from industries and developing countries, respectively. The utilisation of indigenous fruit pits in producing novel adsorbents will greatly benefit in wastewater treatment. In most underdeveloped countries, activated carbon (AC) is imported at a high cost. The study was aimed at synthesising and characterisation of AC obtained from Marula nutshell. Carbonization of organic matter from Marula nutshell was carried out at 200°C, 400°C, 500°C, and 600°C. Sulphuric (H_2SO_4) and phosphoric (H_3PO_4) acids were used as activating agents at concentrations of 20–60% (v/v). Physicochemical characteristics of the AC, such as bulk density, moisture, ash, pH, and iodine number, were analyzed using standard methods. Functional groups and total carbon content were determined using the FTIR spectroscopy and Nitrogen Carbon Sulphur (NCS) analyzer, respectively. The values of carbon yield and total carbon in activated samples with H_2SO_4 and H_3PO_4 were 32.2–93.2%, 26.9–95.8%, and 46–79%, 20.8–69.8%, respectively. The pH, ash, moisture, and bulk density of activated high carbon samples with H_2SO_4 ranged from 2.4–6.1, 0.65–3.49%, 1.3–8.4%, and 0.42–0.62 gcm^{-3}, respectively. Activated high carbon samples with H_3PO_4 had 2.7–3.2, 11.3–29.8%, 4.7–14.6%, and 0.39–0.54 gcm^{-3} pH, ash, moisture, and bulk density, respectively. The synthesised AC samples with 40% H_3PO_4 at 500°C had the highest iodine value of 1075.7 mg/g. FTIR results showed the presence of aliphatic carboxylic acid salt, inorganic nitrate (NO^{3-}), and phosphate groups in the synthesised AC and were not significantly different ($p < 0.05$) from commercial AC. The untreated Marula nutshell had some aliphatic hydrocarbon (alkanes), inorganic phosphate (PO_4^{3-}), aliphatic ester (–COO), and aliphatic carboxylic acid salt (–C(=O)O–) groups. A novel adsorbent, AC was produced from Marula nutshell with the potential to be used in water treatment.

1. Introduction

Activated carbon is a term that refers to a family of carbonaceous solid material resulting from biomass, coal, and polymer scrap through thermal or thermochemical processes [1]. AC are nanoporous adsorbents in nature and play an important role in both gas and liquid phase separation processes [2]. AC is characterised by a well-formed pore morphology, high surface area, electron-conducting amphoteric property, and high adsorptive capacity [3]. The chemical properties of AC are well-defined by the composition of its surface groups and the chemical bonding of heteroatoms [1]. Characteristics of AC are influenced by the physical and chemical properties of the raw materials plus the method of activation [4, 5]. The functional groups determine the important characteristics of the activated carbon, such as acidity, hydrophobicity, polarisation intensity, and adsorption properties [6]. AC is widely used in different

fields further than adsorption to wastewater treatment, solvent recovery, air pollution control, and other industrial effluents [1, 6], which pose major health risks [7].

Marula (*Sclerocarya birrea subsp. caffra*) is an indigenous fruit tree (IFT) in Africa and belongs to the family Anacardiaceae [8, 9]. Marula is a deciduous tree with a height of 7–18 m and grows well in semi and dry areas in sub-Saharan Africa [10, 11]. The tree is most abundant in eastern Africa (Kenya, Tanzania), southern Africa (Angola, Botswana, Namibia, Malawi, Mozambique, South Africa, and Swaziland) [12, 13]. The tree produces about approximately 500 kg of ripe fruits per year which have a gelatinous thick, gluey substance with a sweetly acid taste [14]. The fruits and seed have been used in producing cooking oils, jam, jelly and other tree parts have medicinal properties for treatment of diseases [15]. The extensive use of Marula fruit and seed has resulted in the nutshells (seed husk) often discarded as an agricultural waste material which causes pollution of the environment [15].

In recent years, the demand and marketing of AC has grown due to the problems of water pollution globally [16, 17]. Adsorption has been reported to be an efficient separation technique for removing harmful heavy metals from water [17].

The increased demand for adsorbents has led to much researches on the utilisation of available carbonaceous materials in the synthesis of activated carbon. Studies have been conducted on the use of different agricultural by-products in the syntheses of activated carbon [1, 18–20]. Pavlenko et al. [21] indicated other benefits of AC in hemoperfusion, dressing of wounds with pus, and removal of endogenous and exogenous toxins in uraemia.

Commercial activated carbon (CAC) on the market remains an expensive material for wastewater treatment. The hard shell of the Marula nut can be processed into a novel carbonaceous adsorbent. Therefore, this research was aimed at synthesising and characterising AC produced from Marula nutshell with the potential to be used in water purification in Zimbabwe.

2. Materials and Methods

2.1. Sampling and Treatment of Marula Nutshells. Marula nuts were collected from Shurugwi district (a semidry area located 30°S 19°E in Agro farming region 3 and has sandy soils in Zimbabwe) as shown in Figure 1. The collected nuts were crushed and the germ separated manually from the shell. The shells were then washed with distilled water and rinsed with 0.3 M HCl to remove impurities such as metal ions and surface adhering dirt particles. The shells were then soaked in 0.1 M NaOH solution for 24 hr to remove lignin, followed by soaking for 2 hr in 0.1 M acetic acid to balance the pH. The shells were then washed with distilled water and then dried at 105°C in a Gallenkamp oven for 72 hr to completely remove the moisture and other volatiles [5]. The dried shells were then pulverized into a fine powder using a laboratory mill and sieved using an 1180 μm sieve to achieve homogeneity [22]. The milled Marula nutshell powder was

then placed in tightly closed, clean polythene bags and stored at room temperature, awaiting further use.

2.2. Carbonization and Chemical Activation of Milled Marula Nutshell. The milled Marula nutshell powder (5 g) was weighed and mixed with 15 ml of 0%, 20%, 40%, and 60% H_3PO_4 and left to soak for 2 hr. The same process was carried out using H_2SO_4. The mixtures were then placed in a Gallenkamp oven set at 100°C for a further 2 hr. The activation process of the samples was then conducted by heating at 300°C for 2 hr, 400°C for 2 hr, 500°C for 1 hr, and 600°C for 1 hr [5, 23]. The synthesised AC samples were then cooled to room temperature (26°C) in a desiccator. The cooled AC samples were then soaked in 1 M NaOH to neutralize the acid and thoroughly washed with distilled water. The samples were then dried in a Gallenkamp (Plus II Oven, UK) oven at 105°C for 24 hr and packed in tightly closed clean polythene bags.

2.3. Carbon Yield Analysis. The carbon yield was calculated by weighing the mass of the AC and dividing it by the total weight of the sample before the carbonization process. It was expressed as a percentage on a dry matter basis as follows:

$$\text{yield (\%)} = \frac{\text{weight of activated carbon}}{\text{weight of raw material}} \times 100. \quad (1)$$

2.4. Total Carbon Content. A milled sample of 2 mg was weighed into a tin container using a microspatula. The tin container was then folded lightly to ensure it does not break using roller tongs. Two blank containers were also folded and transferred into the first Nitrogen Carbon Sulphur (NCS) analyzer cells (Flash 2000 NCS, Thermo Scientific, Waltham, MA USA). A suitable method profile was selected on the NCS analyzer method sheet provided and samples were imputed into the sample table. Conditions for analyses were set following NCS analyzer operating instructions and analyses were then initiated. After a full combustion cycle, results were automatically transmitted in a digital result spreadsheet.

2.5. Physicochemical Characterisation of Activated Carbon Samples. The samples that contained high carbon contents were analyzed for the following physicochemical properties:

2.5.1. Bulk Density. The density of the AC was determined following a method (D2854-96, 2000) by ASTM [23] with slight modifications. The sample was poured into a glass container of a known volume and the mass measured. The density was calculated as follows:

$$\text{density} \left(\frac{g}{\text{ml}}\right) = \frac{\text{mass of activated carbon } (g)}{\text{volume of activated carbon (ml)}}. \quad (2)$$
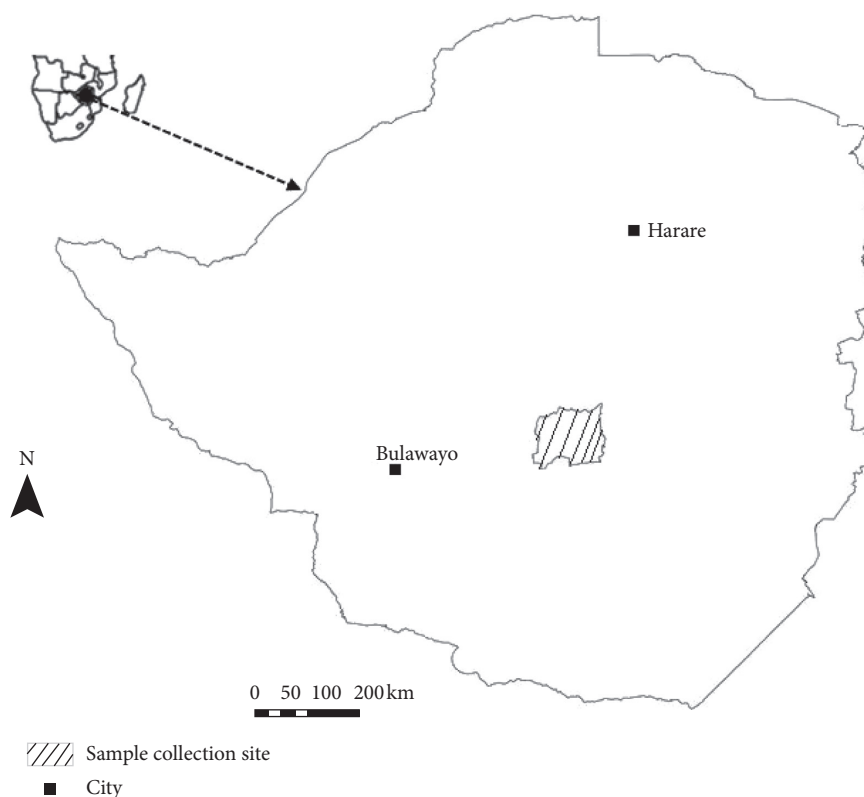
FIGURE 1: Map showing Marula nuts collection area of Shurugwi district, Zimbabwe.

2.5.2. Moisture Content. Moisture was determined using a standard method for analyzing moisture in AC by ASTM D 2867 [24]. Moisture content was calculated as follows:

$$\text{moisture } (\%) = \left(\frac{C - D}{C - B}\right) \times 100, \qquad (3)$$

where B = Weight of empty crucible, C = weight of crucible + sample (g), D = weight of crucible with cover + dry sample (g).

2.6. Ash Content (on a Dry Basis). Ash content was determined following a method ASTM D2866-11 by ASTM [25] as follows:

$$\text{total ash } (\%) = \left(\frac{D - B}{C - B}\right) \times 100, \qquad (4)$$

where B = weight of crucible (g), C = weight of crucible + original sample (g), and D = weight of crucible + ashed sample (g).

2.7. pH Analysis. The pH was measured using a method described by Abdullah et al. [26] using an Ion and pH meter (GLP[22], Crison instruments, South Africa).

2.8. Iodine Number. The iodine number was measured following a method described by Abdullah et al. [26] with slight modifications. A sample of 1 g AC was mixed with 10 mL of 5% (w/v) of HCL and heated up to a boiling point.

The mixture was then cooled to room temperature and 100 mL of 0.1 N Iodine was added. The resulting mixture was then vigorously shaken and filtered. The filtrate (25 mL) was then titrated against a 0.1 N Sodium thiosulphate in the presence of a starch indicator.

2.9. FTIR Analysis. The Fourier transform infrared (FTIR) analysis of the samples was carried out by FTIR equipment (Nicolet 6700, Thermo Scientific, Waltham, MA USA) incorporated with software (Perkin Elmer Instruments version 3.02.01) for the examination of the spectra. The high carbon sample with the highest iodine value was analyzed. A 1 mg of synthesised AC sample was collected mixed with 100 mg KBr and ground into a fine powder. The powder was then compressed into a pellet and then introduced into the spectrophotometer for analysis. The spectra were scanned over the wavenumber range of 4000 and 500 cm^{-1}. The same procedure was also carried out for the untreated Marula nutshell and commercial AC.

2.10. Statistical Analysis. The results of physical and chemical characteristics of AC were expressed as mean-± standard deviation (SD). All experiments were conducted in triplicate and analysis was carried out using SPSS package version 18.0 (Coakes and Ong, John Wiley & Sons, Queensland, Australia). The least significant difference (LSD) test was conducted and one-way Analysis of Variance (ANOVA) was performed at $p < 0.05$.

3. Results and Discussion

Yield results of AC from different treatments are shown in Tables 1 and 2. The observed results indicated that the yield of activated carbon tends to increase with the increase in the concentration of reducing agents. Research by Chen [27] has shown that the activating agent digests the amorphous lignin in the plant material. In AC synthesis, the occurrence of hemicellulose, a cross-linked polymer with other sugars such as arabinose, glucose, galacturonic acid, mannose, O-methyl-glucuronic acid, and xylose is of importance [1]. The presence of hydrogen bonding and crystallinity in natural cellulose has been reported to contribute to the lower adsorption capacity; hence, its capacity can be improved by modifying its structure using chemicals [28].

The AC synthesised at 200°C and activated using H_2SO_4 had a decreased yield as the concentration of the reducing agent was increased. The yield of AC is affected by the temperature at which the carbonation process occurs. Danish and Ahmad [28] reported that the thermal decomposition of hemicellulose occurs above 200°C, and in the range of 250–400°C, CO and CO_2 are released from the glucopyranose rings. This is then followed by the decomposition of cellulose [1].

During the synthesis of activated carbon, the activating agents inhibit tar formation and hence lead to an increased yield with increasing concentration [29]. Also, an increase in the concentration of the reducing agent reduces excessive sample burn-off and hence leads to high yield [30]. This could support the observed results on the high carbon yield on samples activated using H_2SO_4 at 200°C. The reactivity of the chemical activating agent under high heating conditions creates an intermolecular reaction on the biomass and result in an efficient activated carbon production [1].

The carbon yield for the three treatments with 20%, 40%, and 60% H_2SO_4 ranged between 15.6 and 93.2% with a high yield obtained at 200°C (Table 1). The lowest carbon yield was obtained at 400°C using H_2SO_4. It was observed that the carbon yield for samples activated using 60% H_2SO_4 decreased with an increase in temperature from 200°C to 400°C and then gradually increased thereafter. This implies that an increase in acid concentration and the activation temperature have a positive effect on the yield of AC. However, the results of H_3PO_4 showed a different trend (Table 2). Though the highest carbon yield was also obtained at 200°C, a further increase in temperature resulted in a decrease and later an increase in carbon yield. It can, however, be reported in this study that samples activated using H_3PO_4 at 500°C produced a high carbon yield. H_3PO_4 undergoes polymerisation with epoxides, other polymerisable mixtures, and can break lignocellulose into lignin and cellulose [31]. The H_3PO_4 can directly react with lignin and breaks down hemicellulose and cellulose found in the biomass of the plant into monomeric sugars [1]. Örkün et al. [32] reported the effectiveness of using different concentrations of H_3PO_4 on the development of pores, improved morphology, surface chemistry, and size distribution of AC synthesised from hazelnut shells. Huang et al. [33] showed that H_3PO_4 could break bonds, dissociates aliphatic, and aromatic molecules in biomasses, thereby promoting the removal of volatile substrates and causing partial aromatisation and carbonization.

Nwabanne and Igbokwe [34] reported a high carbon yield from nipa palm nuts that were treated with H_3PO_4 (172.4 g) as compared to H_2SO_4 (196.6 g). Our results also indicated no carbon in Marula nutshell samples that were not activated with acid but heated at 400°C, 500°C, and 600°C. It can be explained that at such high temperatures, burning off of the samples occurred because of the absence of acids (activating agents). More so, the acid (activating agents) tends to bind to the carbon and other compounds in the samples and prevent burn-off.

The total carbon content of the synthesised AC samples is shown in Table 3. Results observed indicated that samples activated using H_2SO_4 and H_3PO_4 at 200°C had a high carbon content of 79% and 69.8%, respectively (Table 3). The low carbon content (20.8% and 46%) was obtained in samples synthesised at 400°C with 40% H_3PO_4 and H_2SO_4, respectively. The heating process led to the burning off of noncarbon material and might have led to an increased carbon content per weight of the sample used. Also, during the synthesis of AC, the acids were critical in retaining the carbon in the samples. This is supported by Nicolae et al. [35] who reported that dehydrating agents such as acids used during the carbonization stage tend to promote bond cleavage reactions and the formation of cross-linkages with organic compounds in the sample while the noncarbon material undergoes the process of devolution.

3.1. Physicochemical Characterisation of Activated Carbon.

The untreated Marula nutshell samples had a carbon content, pH, moisture, ash, bulk density, and iodine value of $38.2 \pm 0.01\%$, 5.5 ± 0.1, $8.4 \pm 0.02\%$, $12.1 \pm 0.1\%$, 0.64 ± 0.01 gcm^{-3}, and 943.8 ± 0.01 mg/g, respectively, as shown in Table 4. There was no significant difference ($p < 0.05$) in the bulk density of the untreated Marula nutshell and commercial AC on the market.

The pH of the Marula nutshells and commercial type AC were 5.8 and 4.8, respectively, as shown in Table 4. The pH values for the synthesised high carbon-containing samples activated using H_2SO_4 and H_3PO_4 at different temperatures (200–600°C) ranged from 2.4–6.1 and 2.8–3.2, respectively (Table 5). High carbon samples activated with 40% H_2SO_4 at 400°C had a high ash content of 51% and samples activated using 40% H_3PO_4 at 500°C had a 29.8% ash content. The ash contents in samples activated using H_2SO_4 were lower as compared to the commercial sample. The low ash and moisture content in the AC showed a low particle density and the biomaterial could be a good raw material for adsorbents in reactors [20, 36]. The moisture content of all high carbon samples activated using both H_3PO_4 and H_2SO_4 was higher as compared to commercial AC samples (0.1). This study also observed that the bulk density and iodine values of the synthesised high carbon samples were not significantly different ($p < 0.05$) from the commercial AC and Marula nutshell samples.

The ash content of the synthesised carbon samples was generally low. This study also noted a relationship between

TABLE 1: Yield of activated carbon from H_2SO_4 treatment.

Acid (%)	Temperature (°C)				p value
	200	400	500	600	
Control*	83.0 ± 0.01	NY	NY	NY	ns
20	82.9 ± 0.01[a]	15.6 ± 0.01[b]	33.1 ± 0.02[c]	39.8 ± 0.01[c]	<0.05
40	83.4 ± 0.01[a]	19.8 ± 0.02[b]	32.2 ± 0.01[c]	40.6 ± 0.01[c]	<0.05
60	93.2 ± 0.02[a]	28.5 ± 0.01[b]	38.0 ± 0.02[c]	41.5 ± 0.01[c]	<0.05

* = distilled water, NY = no yield; ns = not significant; mean ± standard deviations are reported. Means in a row with different superscripts ([a,b,c]) are significantly different at $p < 0.05$.

TABLE 2: Yield of activated carbon from H_3PO_4 treatment.

Acid (%)	Temperature (°C)				p value
	200	400	500	600	
Control*	95.8 ± 0.01	NY	NY	NY	ns
20	73.6 ± 0.02[a]	38.4 ± 0.02[b]	45.1 ± 0.01[b]	26.9 ± 0.02[c]	<0.05
40	77.0 ± 0.01[a]	52.9 ± 0.01[b]	67.9 ± 0.01[c]	45.8 ± 0.01[b]	<0.05
60	68.4 ± 0.01[a]	52.8 ± 0.02[b]	66.6 ± 0.02[a]	60.6 ± 0.02[a]	<0.05

* = distilled water, NY = no yield; ns = not significant; mean ± standard deviations are reported. Means in a row with different superscripts ([a,b,c]) are significantly different at $p < 0.05$.

ash content, bulk density, and total carbon content. Activate samples with low ash and bulk density tend to have a high total carbon content. Ekpete et al. [20] reported the effects of ash content on the ignition point of the carbon and it reduces the overall activity and efficiency of the reactivation of the AC. More so, a low ash value would improve the property of the activated carbon as an adsorbent. The lower ash content in the synthesised AC would possibly make it a good adsorbent. Abdullah et al. [26] reported that a high ash content is undesirable for AC since it reduces the mechanical strength of carbon and affects adsorptive capacity. Also, high ash content can reduce the adsorption power of AC due to the presence of minerals such as Ca, Na, K, and Mg, which tend to fill the pores of activated carbon [1].

The iodine value ranged between 915–1075 mg/g in the synthesised AC samples (Table 5). AC samples activated using 40% H_3PO_4 at 500°C had the highest iodine value of 1075.7 mg/g. The iodine number can be used to estimate the surface area of the AC in m^2/g and it measures the porosity within the dimensions [20]. The higher iodine value of AC has been ascribed to the occurrence of a large micropore structure that can result in the carbon having a large surface area because of the enlargement of its pore structure [37]. The observed iodine values make it possible for use in water treatment. Verla et al. [5] reported that AC with an iodine range of 600–1100 mg/g could be recommended for use in water treatment. The iodine values of the synthesised AC were comparable to that of the commercial AC (1034.3 ± 0.02 mg/g) on the market.

Furthermore, the iodine value, however, tends to increase with an increase in carbon content in samples activated with H_2SO_4 and heat-treated at 200°C but was different in samples heated at 500°C. This could be explained by the extent of pore formation in the synthesis of the activated carbon at these different conditions. AC samples activated using H_3PO_4 had higher carbon content and also high iodine

values when compared to samples activated using H_2SO_4. This could be explained by the differences in reactivity rate with the activating agents [6]. The differences in the iodine number between untreated Marula nutshell carbon and synthesised AC could be attributed to the chemisorption which might have taken place in the pores of the carbons during carbonization and activation, resulting in the development of large micropores [6, 37].

Novel AC from Marula nutshell had a significantly high iodine value (1075.7 mg/g) and yield of the AC (45.8–77.0 wt%) when compared with adsorbents produced from tire and tamarind seeds. Li et al. [38] observed an iodine value and carbon yield of 352 mg/g and 39.8 wt% in AC from tire char, respectively. Another study by Sharada et al. [39] reported that AC synthesised from tamarind seeds has a pH (7.0), moisture content (2.9%), ash (1.5%), bulk density (0.4 g/cm^3), iodine number (995 mg/g), and specific surface area (912 m^2/g). Furthermore, Mopoung et al. [40] reported a carbon yield of 54.09–82.03 wt% from AC produced from tamarind seeds activated with KOH. Although the carbon content was lower in the synthesised AC as compared to other adsorbents, it has the potential to be used as a good adsorbent in removing harmful compounds in water.

3.2. FTIR Characterisation. FTIR analysis results are indicated in Figures 2–4. FTIR analysis was used to identify the functional groups and other chemical compounds present in the activated carbon, which are important in its characterisation and forecasting adsorption performance [20]. The surface chemistry of untreated Marula nutshell samples was observed to have an FTIR spectrum within 3400 cm^{-1} correlated to a primary amine group (N–H), and the small band at 2950 cm^{-1} showed the presence of an alkane (Figure 2). More so, untreated Marula nutshell indicated the presence of an anhydride group (CO–O–CO) at a band of

TABLE 3: The total carbon content of the synthesised activated samples.

Acid (%)	Treatments											
	200°C			400°C			500°C			600°C		
	H_3PO_4 (%) Carbon	H_2SO_4 (%) Carbon	p value	H_3PO_4 (%) Carbon	H_2SO_4 (%) Carbon	p value	H_3PO_4 (%) Carbon	H_2SO_4 (%) Carbon	p value	H_3PO_4 (%) Carbon	H_2SO_4 (%) Carbon	p value
20	66.5 ± 0.01^a	69.3 ± 0.02^a	ns	62.1 ± 0.02^a	49.9 ± 0.03^b	<0.05	53.6 ± 0.01^a	59.3 ± 0.02^a	ns	65.9 ± 0.01^a	67.0 ± 0.01^a	ns
40	42.9 ± 0.01^a	57.2 ± 0.01^b	<0.05	20.8 ± 0.03^a	56.1 ± 0.02^b	<0.05	61.8 ± 0.02	—	ns	50.7 ± 0.02^a	46.0 ± 0.05^a	ns
60	69.8 ± 0.02^a	63.2 ± 0.02^a	ns	56.1 ± 0.01	—	ns	60.24 ± 0.03^a	79 ± 0.03^b	<0.05	58.3 ± 0.03	—	ns

Mean ± standard deviations are reported. Means in a row with different superscripts ([a], [b]) are significantly different at $p < 0.05$. ns = not significant.

TABLE 4: Physicochemical properties of Marula nutshell and commercial carbon.

Sample	Carbon (%)	pH	Moisture (%)	Ash (%)	Bulk density (gcm^{-3})	Iodine value (mg/g)
Marula nut shell	38.2 ± 0.01^a	5.5 ± 0.1^a	8.4 ± 0.02^a	12.1 ± 0.1^a	0.64 ± 0.01^a	943.8 ± 0.01^a
Commercial carbon	76.9 ± 0.01^b	4.8 ± 0.1^a	0.1 ± 0.01^b	13.8 ± 0.2^a	0.54 ± 0.02^a	1034.3 ± 0.02^b
p value	<0.05	<0.05	<0.05	ns	ns	<0.05

Mean ± standard deviations are reported. Means in a column with different superscripts ([a,b]) are significantly different at $p < 0.05$.

TABLE 5: Physicochemical characterisation of the synthesised high carbon samples.

Temperature (°C)	Acid (%)	Physicochemical properties				
		pH	Moisture (%)	Ash (%)	Bulk density (g/cm^3)	Iodine value (mg/g)
H_2SO_4 treatments:						
	20	2.9 ± 0.1^a	2.4 ± 0.2^a	0.65 ± 0.01^a	0.62 ± 0.02^c	1036.1 ± 0.01^c
200	40	3.9 ± 0.1^b	1.3 ± 0.1^a	3.49 ± 0.01^b	0.53 ± 0.01^a	938.5 ± 0.11^a
	60	2.4 ± 0.1^a	10.7 ± 0.2^c	0.97 ± 0.01^a	0.54 ± 0.02^a	947.4 ± 0.12^a
	40	6.1 ± 0.1^c	3.9 ± 0.1^b	51.0 ± 0.01^c	0.43 ± 0.01^a	1024.3 ± 0.10^c
400	20	5.7 ± 0.1^c	2.8 ± 0.2^a	3.79 ± 0.01^b	0.42 ± 0.01^a	1036.1 ± 0.12^c
	40	5.7 ± 0.1^c	3.3 ± 0.1	4.62 ± 0.2^b	0.43 ± 0.02^a	915.2 ± 0.01^a
500	60	6.1 ± 0.1^c	2.5 ± 0.2^a	4.53 ± 0.01^b	0.45 ± 0.01^a	990.8 ± 0.15^a
p value		<0.05	<0.05	<0.05	<0.05	<0.05
H_3PO_4 treatments:						
200	60	2.8 ± 0.1^a	6.4 ± 0.2^a	14.2 ± 0.01^a	0.54 ± 0.01^a	1042.6 ± 0.10^a
400	20	3.2 ± 0.1^b	6.6 ± 0.1^a	14.2 ± 0.01^a	0.42 ± 0.02^a	1048.3 ± 0.14^a
500	40	2.7 ± 0.1^a	14.6 ± 0.1^b	29.8 ± 0.01^b	0.82 ± 0.01^b	1075.7 ± 0.11^a
600	20	$3.2 \pm 0.^b$	4.7 ± 0.2^a	11.3 ± 0.01^a	0.39 ± 0.02^a	1131.9 ± 0.13^b
p value		<0.05	<0.05	<0.05	<0.05	<0.05

Mean ± standard deviations are reported. Means in a column with different superscripts ([a, b, c]) are significantly different at $p < 0.05$.
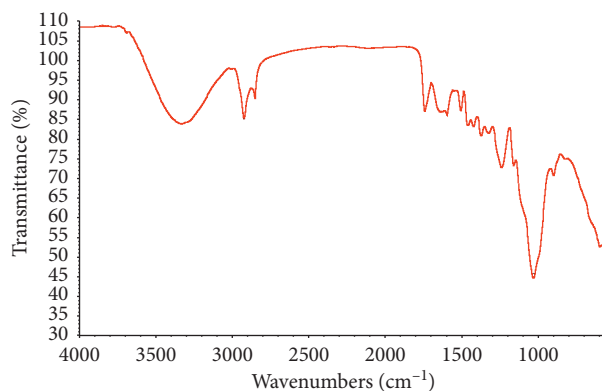

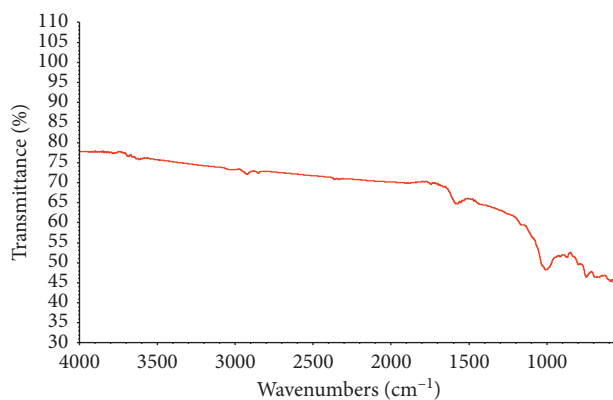FIGURE 2: FTIR wavenumbers for untreated Marula nutshell.
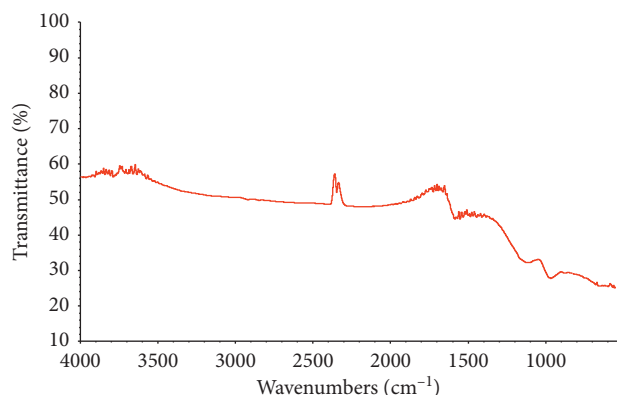

FIGURE 3: FTIR wavenumbers for commercial AC.

FIGURE 4: FTIR wavenumbers for synthesised AC (40% H_3PO_4 at 500°C).

$1025\,cm^{-1}$. Commercial AC had weak bands at 2900, 1000, 750, and $1550\,cm^{-1}$, which represented the presence of some small aliphatic hydrocarbon (alkanes), inorganic phosphate (PO_4^{3-}), aliphatic ester (–COO), and aliphatic carboxylic acid salt (–C(=O)O–) groups (Figure 3). The synthesised activated carbon with 40% H_3PO_4 at 500°C had small bands at 1550, 1200–1300, and $1000\,cm^{-1}$. This showed the presence of aliphatic carboxylic acid salt, inorganic nitrate (NO_3^-), and inorganic phosphate groups (Figure 4). The presents of aliphatic compounds suggest that these products are organic compounds that contain carbon-hydrogen bonds. These functional groups are capable of binding to other elements other than hydrogen. These include and are not limited to oxygen, sulphur, nitrogen, and chlorine [41]. Franca et al. [42] reported the presence of hydrogen, oxygen, and nitrogen atoms on the surface of the AC that can bind to other elements [3]. Mopoung et al. [40] noted the presence of O–H, C–H, C–O, C=O, –CO$_3$, and Si–H groups on the surface of AC produced from tamarind seeds.

4. Conclusion

The synthesised AC from Marula nutshells had characteristics that were not significantly different from commercial type AC. The carbon yield and total carbon values of samples activated using H_2SO_4 and H_3PO_4 were 32.2–93.2%, 26.9–95.8%, and 46–79%, 20.8–69.8%, respectively. The temperature of activation has a bearing on the yield of the activated carbon. The optimum temperature from Marula nutshells using activation agents was found to be 500°C. AC treated with 40% H_3PO_4 had the highest iodine value of 1075.7 mg/g. FTIR analysis indicated the presence of primary amine group (N–H), aliphatic hydrocarbons, and anhydride (CO–O–CO) groups in untreated Marula nutshell powder. Furthermore, aliphatic carboxylic acid salt, inorganic nitrate (NO_3^-), and inorganic phosphate groups were present in synthesised AC samples activated using 40% H_3PO_4 at 500°C. The carbonization process might have removed some functional groups in the raw material. A novel adsorbent was synthesised from Marula nutshells with the potential of being used in water treatment. There is a need to conduct further research on the potential of the synthesised AC from Marula nutshells on the removal of As, Pb, available chlorine, and reduction of bacterial load in contaminated water.

Data Availability

The data sets of this research are available and will be provided upon request.

Conflicts of Interest

The authors declare there are no conflicts of interest.

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