Physicochemical Processing of Date Fruit Pit and Mesocarp into Activated Carbon and Syrup

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ABSTRACT

Activated carbon was derived from date palm (*Phoenix dactylifera*) seeds using orthophosphoric acid (H_3PO_4) as the activating agent. The activation process occurred at 400 °C with an impregnation ratio of 4:1 and a residence time of 120 minutes. Physicochemical analysis of the resulting date-seed activated carbon (DSAC) showed a pH of 6.5, a moisture content of 15.69%, a yield of 66.67%, and a bulk density of 0.14 g/cm³. Textural properties were analyzed using nitrogen adsorption (BET), SEM, and FT-IR techniques. The material exhibited a BET surface area of 1044.11 m²/g, an average pore size of 6.52 nm, and a pore diameter of 2.12 nm, confirming its mesoporous structure. These findings indicate that date seeds are a suitable and abundant precursor for activated carbon production, providing a low-cost and effective alternative to commercial activated carbon (CAC). The date mesocarp was also processed to produce date syrup. Proximate analysis of the syrup revealed 10.12% moisture, 2.6% protein, 1% fat, 2.14% ash, and 84.11% carbohydrate. Total sugar, glucose, and soluble solids content were 21.36%, 14.28%, and 62%, respectively. The high simple-sugar content suggests that date syrup is a promising natural alternative to conventional sugar syrup.

Keywords: Activated carbon, date fruit, pore structure, FT-IR, SEM, proximate analysis.

INTRODUCTION

Activated carbon is a carbonaceous material with amorphous development, high internal surface area and a genuine degree of porosity. It has a microcrystalline and non-graphitic type of carbon [1]. Activated carbon is widely used in various environmental applications. It is a porous carbonaceous material with continually expanding applications in water treatment, wastewater treatment, air purification, and even desalination [2]. Due to its unique characteristics, activated carbon can serve not only as an adsorbent for water and gases but also as a catalyst or co-catalyst for the removal of pollutants from gases, liquids, and the recovery of chemicals [3,4]

Activated carbon may contain micropores, mesopores, and macropores in its structure, which play critical roles in determining the properties of activated carbon and its adsorption performance [5] Any carbonaceous or lignocellulosic material can serve as a precursor for activated carbon. Recently, many researchers have used agricultural wastes as precursors for activated carbon for four main reasons: they are renewable, economical, readily accessible, and environmentally friendly. They can also provide an additional income source for agro-based industries [1].

Agricultural waste precursors can be derived from many parts of plants: roots, stems, bark, flowers, leaves, fruit peels, husks, shells, and stones. These wastes can be classified into woody and non-woody resources. The sample used in this study is obtained from date stone (i.e., date seed). In principle, activated carbon can be produced by two methods: physical activation (a two-step process involving carbonization of raw material under inert atmosphere followed by activation with steam/air/CO_2) [6]. and chemical activation (a one-step process where the raw material is impregnated with activating agent such as H_3PO_4, $ZnCl_2$, KOH or NaOH and then carbonized under nitrogen). Therefore, production of activated carbon involves two important steps: carbonization and activation [5].

Date palm (*Phoenix dactylifera L.*) is a fruit-bearing tree . Its fruits and seeds (also called pits) are comprised of dietary and therapeutic possibilities. The date fruit has not been fully exploited as a functional ingredient in various health-promoting food products. Date fruits are rich in nutrients such as amino acids, vitamins, minerals, dietary fibre, and phenolics [7]. Date palm shown in Figure 1 is an organic product-bearing tree with a ton of prospects. Its organic products, seeds, and by-products, are comprised of dietary and restorative possibilities.

Figure 1: Date Fruit [7]

There is a need for sustainable waste management strategies and the development of value-added products from biomass resources. Date fruit (*Phoenix dactylifera*) processing produces large quantities of pits and mesocarp residues, which are often discarded despite their considerable physicochemical potential. Date pits are rich in carbonaceous materials and possess structural features suitable for conversion into activated carbon, a widely applied adsorbent in water treatment, pollution control, and industrial separation processes [5,10]. Concurrently, the mesocarp of date fruits contains high concentrations of fermentable sugars and nutrients that can be processed into date syrup, a natural sweetener with nutritional and commercial importance [9,11,12].

Previous studies have largely examined the utilization of either date pits for activated carbon production or the mesocarp for syrup extraction independently. However, integrated valorization of both components remains limited, which is essential for maximizing resource efficiency, minimizing waste, and promoting sustainable biomass utilization. This study addresses this gap by investigating the physicochemical transformation of both the pit and mesocarp of date fruit into activated carbon and syrup, respectively. and by evaluating the properties of the resulting products.

Plate 1: Date Seed [10] Plate 2: Date Syrup [8]

Therefore, this study aims to investigate the physicochemical transformation of date fruit (*Phoenix dactylifera*) pit and mesocarp into activated carbon and date syrup, respectively, and to assess the physicochemical properties of the derived products.

The specific objectives of this study are to; synthesize activated carbon from date fruit pits using chemical activation and carbonization techniques, and characterize the physicochemical properties of the produced activated carbon, extract date syrup from the mesocarp of date fruits using suitable processing methods, and determine the physicochemical characteristics of the extracted date syrup.

MATERIALS AND METHODS

Materials

The raw material used for the preparation of activated carbon. was obtained from Tanke Local Government, Ilorin South, Kwara State, Nigeria The raw material used was Date seed and mesocarp from Date fruit. The following materials were used: Standard sieve (212 µm), electric pyrolizer (Local generic model, China) , pH meter (Hanna Instrument, China), desiccator (Pyrex ,USA), hot air oven (Labconco,USA), and electric weighing balance (Shimadzu, Japan). All reagents used in this research work were of analytical grade without further purification. The reagents used include: Citric acid, Orthophosphoric acid, NaOH, and HCl.

Preparation of Carbonized Date Seed

The mesocarp was preserved after the Date fruit was deseeded. The seed was collected and rinsed with distilled water. The seeds were dried in an oven at 110 °C for 16 hours before being crushed and ground into powder. The powdered sample was sieved with a standard mesh (212 µm), and 30 g of crushed date seed was used as a precursor, mixed with prepared 2 M of H_3PO_4 at a 1:4 impregnation ratio in accordance with the literature [10]. The mixture was dried in an oven at 110 °C until it was completely dry and crisp. The dried mixture was carbonized in a pyrolizer at a temperature of 400 °C. The sample was placed in a crucible and placed inside the furnace, and was heated at 400 °C in a Nitrogen controlled atmosphere by passing N_2 through the furnace. The carbonized sample was cooled to room temperature, and the samples were washed repeatedly with distilled water and then dried in an oven at 110 °C to ensure complete dryness [13].

Physicochemical Characterization of Date Seed

Carbonized Date seed samples were cooled to room temperature in a desiccator prior to analysis. All measurements were performed in triplicate and reported as mean values. Carbonization temperature was recorded directly from the programmable muffle furnace at the maximum set point. The pH was determined by suspending 1.0 g of sample in 20 mL distilled water, agitating for 30 min, allowing equilibration, and measuring with a calibrated digital pH meter. Moisture content was assessed by oven-drying ~2 g of sample at 105 °C to constant weight and calculating weight loss. Bulk density was measured by gently filling a graduated cylinder with a known mass of sample without compaction and computing the mass-to-volume ratio (g/cm³). Percentage yield was calculated from the mass balance before and after carbonization. All procedures followed standard AOAC and ASTM guidelines.

pH

The pH measures the degree of alkalinity or acidity of the adsorbent. This depends on the preparation, inorganic mater, and chemically active oxygen group on its surface as well as the kind of treatment to which the adsorbent was subjected to. After preparation of the sample the DSAC was acidic at a pH of 5.3, it was then washed by 0.5 M NaOH to bring up the pH to a more neutral level of 6.5 when the pH test was carried out again. Because effect of pH is an important factor that affects adsorption.

Moisture Content

Moisture content is used to test for the presence of liquid especially water. The moisture content of activated carbon is use to define and express its properties in relation to the net weight of the carbon. The value obtained for the DSAC is 15.69% which is less than the one obtain by [10]. This value is also within the acceptable range of moisture content for an activated carbon. It has been observed that high moisture content decreases the adsorption capacity especially for organic dyes [15].

Preparation of Date Syrup

The deseeded Dates were weighed, sliced, and soaked in 4 L of water overnight. A paste was made from the soaked dates by blending. To make a slurry, the paste was placed in a mesh cloth with 4 L of water added, and the liquid was filtered out. The obtained liquid was then transferred into a pot and boiled. It was continually stirred to avoid burning until it reached a thick, viscous consistency. While stirring, 2 ml of 0.1 M citric acid was added as a preservative to prevent the syrup from molding due to bacteria or fungi. Afterwards, the syrup is allowed to and stored in a cool, closed airtight bottle for further analysis [14].

Proximate Analysis of the Date Syrup

Date syrup samples were homogenized prior to analysis. All determinations were conducted in triplicate following standard AOAC procedures, and results were expressed on a wet weight basis.

Moisture content was determined by oven drying approximately 5 g of sample at 105 °C to constant weight [22]. Ash content was measured by dry ashing in a muffle furnace at 550 °C for 4–6 h. Crude protein was determined using the Kjeldahl method, with total nitrogen multiplied by a factor of 6.25 [22]. Crude fat was determined by Soxhlet extraction using petroleum ether (40–60 °C), while crude fibre was determined by sequential digestion of defatted samples with 1.25% sulfuric acid followed by 1.25% sodium hydroxide. Total carbohydrate content was calculated by difference [22,25,29].

The pH of the syrup was measured at room temperature using a calibrated digital pH meter with direct immersion of the electrode into the sample. Total soluble solids (TSS) were determined using a digital refractometer and expressed as °Brix.[29].

Total sugar content was determined by the phenol–sulfuric acid method. Absorbance was measured spectrophotometrically at 490 nm, and sugar concentration was calculated using a glucose calibration curve. Glucose content was determined using the dinitrosalicylic acid (DNS) method, with absorbance measured at 540 nm and results expressed as percentage glucose [26, 29].

Fourier Transmission Infrared Spectroscopy (FT-IR)

The IR-spectrum is used to show the functional groups present in the activated carbon, thereby indicating the surface activation of carbon in the activated carbon. The equipment used in the characterization of the DSAC is a Fourier Transformed Infrared spectroscopy (FTIR - 630, Cary) and it was used to determine the functional groups of the DSAC.

Brunauer Emmett Teller (BET) Method

BET method is used for the determination of the surface area of solid materials. The textural properties of the activated carbon produced from the date seed were deduced from nitrogen adsorption at 77 K using a Quantachrome Novawin, which was degassed at 250 °C for 3 h. The Barett, Joyner and Hallonda (BJH) method was employed to obtain the pore size distribution of the sample.

RESULTS AND DISCUSSION

The physicochemical parameters of carbonized samples are presented in Table 1.

Yield of Products

The percentage yield obtained from 30 g of the powdered date seed was 66.67% which is more than half the sample used. This result indicates a positive and good yield of the sample DSAC.

Bulk Density

Bulk density is the measure of how dense and compact an activated carbon is. The bulk density of the DSAC is 0.14 g/cm³.

pH

The pH measures the degree of alkalinity or acidity of the adsorbent. This depends on the preparation, inorganic material, and chemically active oxygen group on its surface, as well as the kind of treatment to which the adsorbent was subjected. After preparation of the sample the DSAC was acidic at a pH of 5.3; it was then washed with 0.5 M NaOH to bring up the pH to a more neutral level of 6.5, when the pH test was carried out again. Because effect of pH is an important factor that affects adsorption.

Moisture Content

Moisture content is used to test for the presence of liquid especially water. The moisture content of activated carbon is use to define and express its properties in relation to the net weight of the carbon. The value obtained for the DSAC is 15.69% which is less than the one obtain by Priya et al [10]. This value is also within the acceptable range of moisture content for an activated carbon. It has been observed that high moisture content decreases the adsorption capacity especially for organic dyes [15].

Table 1: Physicochemical Data of the Activated Carbon

Physicochemical Test	DSAC	Reference value	Reference
Temperature (^oC)	400	400-500	[16]
pH	6.5	6.7	[17]
Moisture Content (%)	15.69	7.99	[17]
Bulk Density (g/cm^3)	0.14	0.36	[17]
% Yield (%)	66.67	60-10%	[16]

The result of BET analysis is shown in Figure 2.

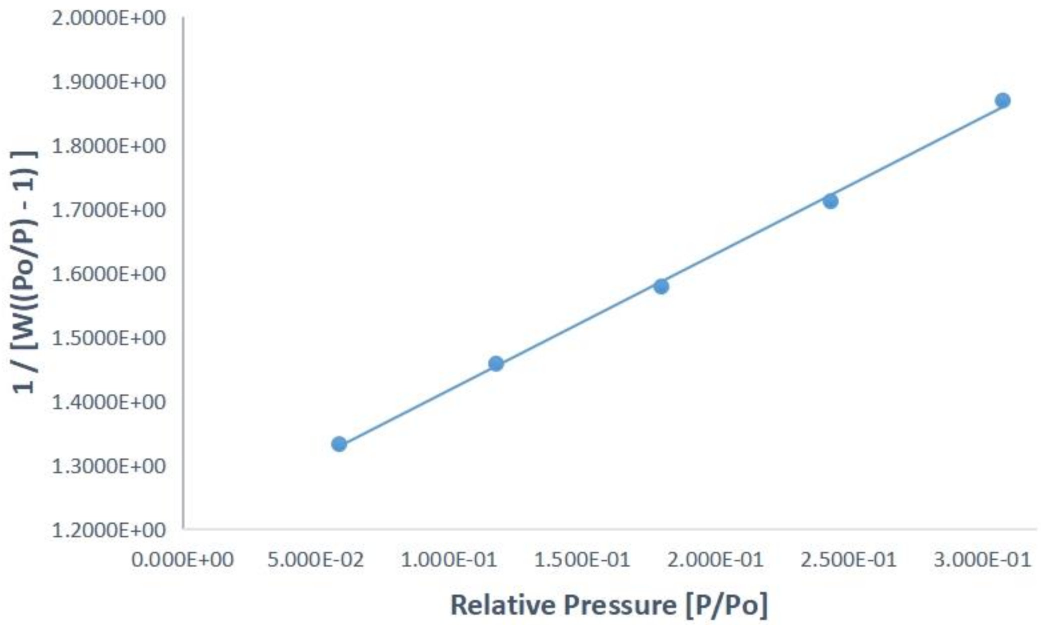


Fig. 2: Graphical Representation of BET

Table 2 shows the results of measurement of surface area from different methods.

Table 2: Surface Area Data of Produced DSAC

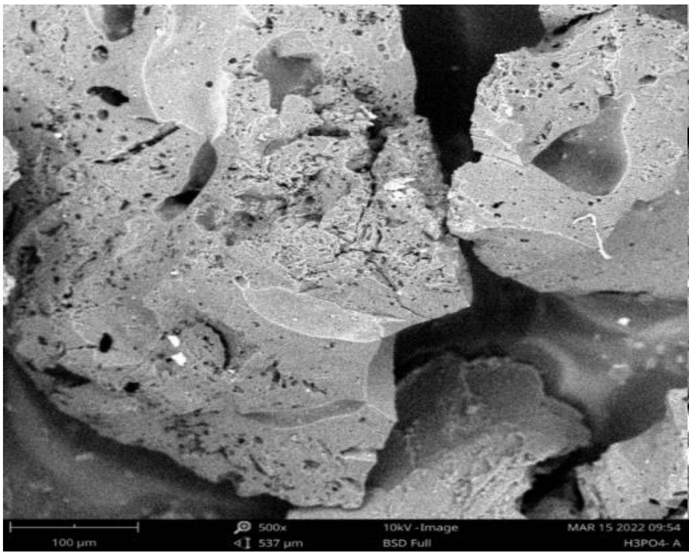
Parameter	Value	Reference Value	Reference
Multipoint BET surface area	1044.113 m^2/g	980 m^2/g	[18]
Single point surface area	573 m^2/g		
Langmuir surface area	24485.619 m^2/g		
BJH method cumulative adsorption surface area	1080 m^2/g		
t – method external surface area	1044 m^2/g		

From Table 2. it could be seen that the sample DSAC has a high surface area at 1044.113 m^2/g which is more than the one obtained by Suresh *et al.* [18] backing the BET line[ar graph, indicating that the DSAC sample is a suitable adsorbent for adsorption although this alone is not enough evidence of its application for adsorption.

Scanning Electron Microscopy (SEM)

The SEM image shows the porosity of the activated carbon. The SEM was performed to examine the physical structure change of the DSAC samples using SEM model Phenom ProX

(PhenomWorld Eindhoven, the Netherlands). Figures 3(a-d) illustrates the surface morphology using different magnifications (2000x, 1000x, 500x, and 4000x), The different pore sizes and shapes can be observed from the image. The rough surface morphology with uniform pore distribution and high porosity could be ascribed to the presence of various carbon-based functional groups in DSAC. The enhancement in porosity, especially with the formation of new micro pores due to acid activation of the DSAC by H_3PO_4 also enlarged the existing micropores to mesopores. The DSAC can be identified as mesopores because the average pore is < 50 nm.



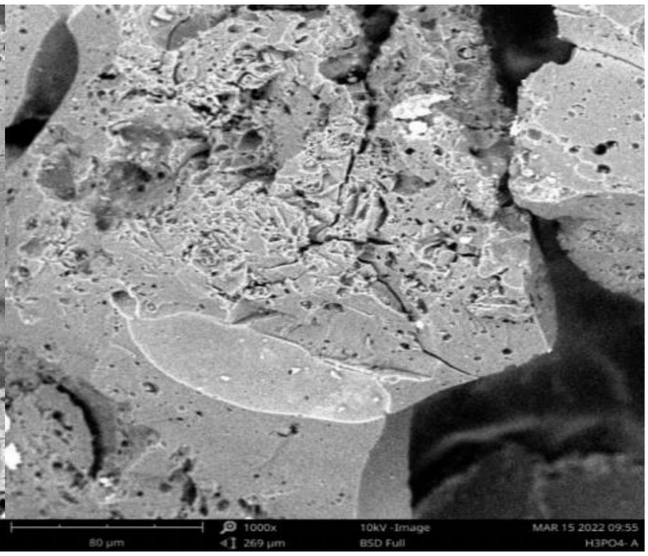
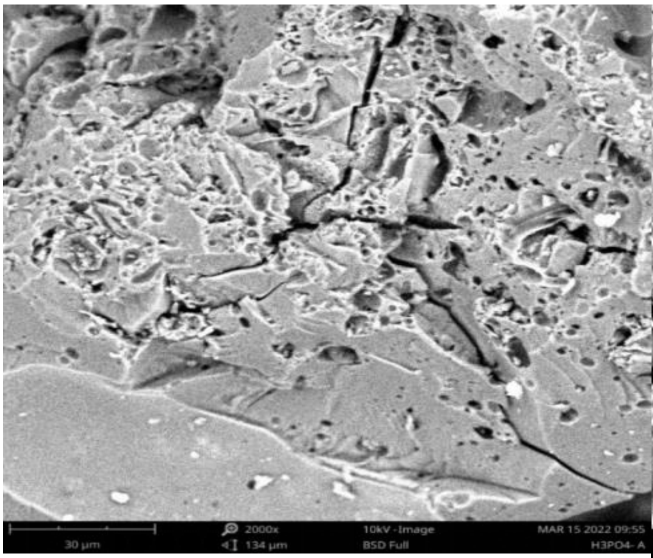


Fig. 3 (a): 500x

Fig. 3 (b): 1000x



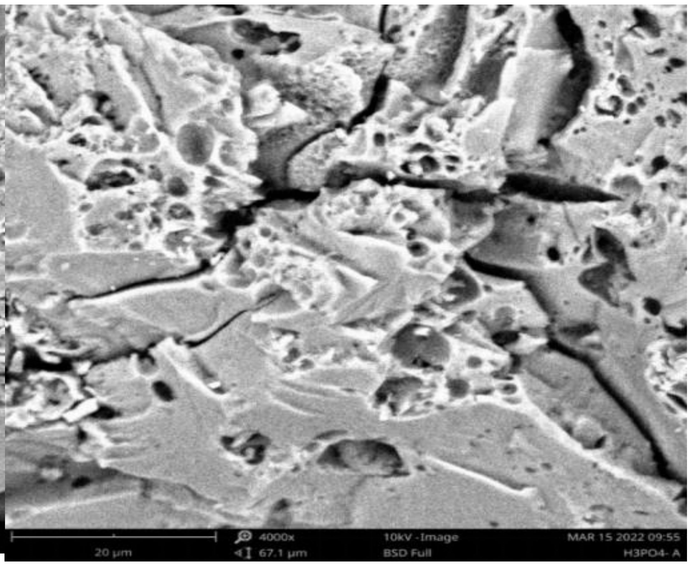


Fig. 3 (c): 2000x

Fig. 3 (d): 4000x

Fig. 3(a, b, c, d): SEM analysis of DSAC of different magnification

Pore Size Distribution

The pore size distribution is used to describe the internal structures and adsorption capacities of activated carbons [19]. Tables 3 and 4 give the results of the pore widths and diameter, from this we can deduce that the DSAC can be identified as a mesopore since the pore width is 6.523 nm which is less than (< 50 nm) and a pore diameter of 2.118 nm, which is less than 2.91 nm obtained in literature [20].

Table 3: Pore Size

Parameters	Value	Reference value	Reference
BJH method adsorption pore diameter (Mode Dv/(d))	2.118 nm	2.91 nm	[15] [14]
DR method micropore Pore width	6.523 nm		
HK method pore Diameter (Mode)	1.847 nm		

Table 4: Pore Volume of Produced Date Seed Activated Carbon

Parameter	Value	Reference Value	Reference
BJH method cumulative adsorption pore volume	0.5327 cm^3/g	1.26 cm^3/g	[15] [14]
HK method micropore volume	0.1460 cm^3/g		

Fourier Transmission Infrared Spectroscopy (FT-IR)

The IR-spectrum is used to show the functional groups present in the activated carbon, thereby indicating the surface activation of carbon in the activated carbon. The equipment used in the characterization of the DSAC is a Fourier Transformed Infrared spectroscopy (FTIR - 630, Cary) and it was used to determine the functional groups of the DSAC.

From the spectrum in Figure 4, the following adsorption band was shown: 3753 cm^{-1} and 3652 cm^{-1} representing a very weak O – H stretch, 2754 cm^{-1} the C-H stretch of an aldehyde group, 2374 cm^{-1} and 2344 cm^{-1} at O=C=O stretching. The peak with a medium intensity of 2109 cm^{-1} represent the C≡C of a stretching alkyne compound, 1982 cm^{-1} represent a C-H bending, 1830 cm^{-1} and 1751 cm^{-1} C=O stretching (s), 1643cm^{-1} C=C stretching, 1058cm^{-1} at S=O stretching, 972 cm^{-1} at C=C bending (s), 864 cm^{-1} and 738 cm^{-1} at C=C bending (m) and 708 cm^{-1} represent a C-H bend.

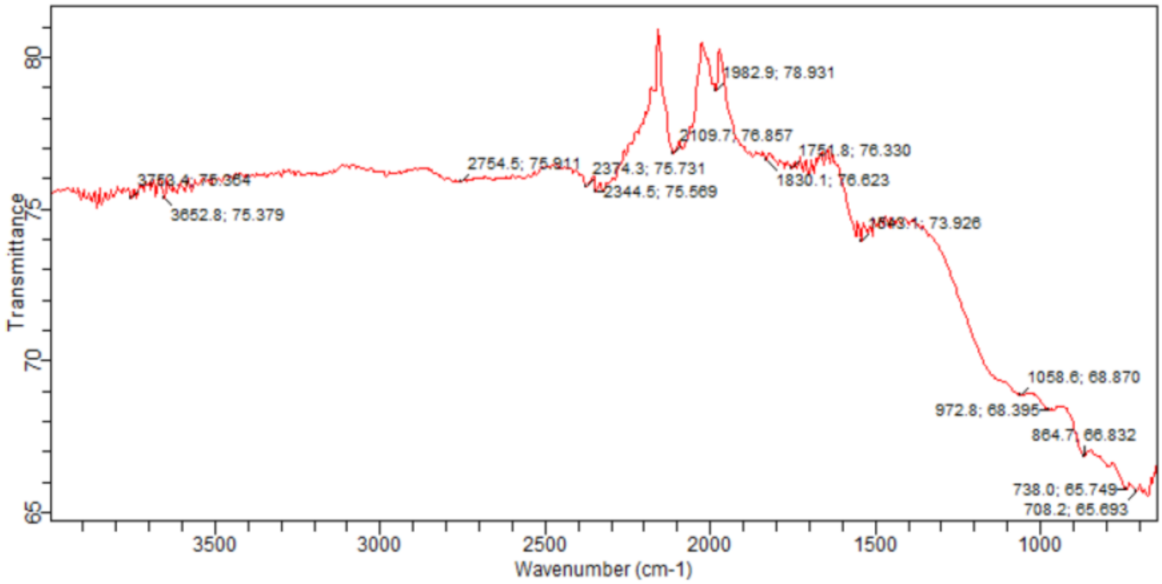


Fig. 4: FTIR Spectrum of Activated Carbon

From Fig. 4, there is a very weak presence of O-H band at 3753 cm^{-1} and 3652 cm-1 and a weak broad band of 2374 cm^{-1} and 2344 cm^{-1}that corresponds to a carbon-oxygen compound (CO_2 likely to be present). The peak with medium intensity of 2109 cm^{-1} that represent the alkyne compound, it represents the presence of a very strong C-C bond which aids in the adsorption process of the activated carbon. The intensity of the following peak from 1830 cm^{-1} downwards kept decreasing gradually. The peak at about 700cm^{-1} may be due to polycyclic and C-H bending vibration of benzene rings and these functional groups can have negative or positive charge depending on the pH of the solution [21].

Proximate Analysis of the Date Syrup

In Table 5, the moisture content of date syrup was 10.21%, which is lower than that reported by Hassan & Ganbi [22]. This can be due to the method employed during the preparation of the date syrup. The protein content was 2.63% lower than the 2.87% obtained by Hassan & Ganbi [22]. The fat content of the date syrup sample was 1%/ which falls within the range of 0.94 – 1.04% as reported by Al-Farsi *et al*. [14]. The ash content (2.14%) was just a little below the 2.93% obtained by Hassan & Ganbi [22], which is an indication of high mineral content. Carbohydrates were the major component at 84.11%, which is similar to other results [14, 23, 24]. The obtained pH value of the date syrup is 6.5.

Table 5: Proximate Analysis of Date Syrup

Component	Date Syrup	Reference Value	Reference
Moisture	10.12%	13.69%	[22]
Protein	2.63%	2.87%	[22]
Fat	1%	0.95-1.04%	[22]
Ash	2.14%	2.93%	[25]
Carbohydrate	84.11%	85.90-86.84%	[26]
pH	6.5	4.83-4.91	

Sugar

The sugar content of date syrup is shown in Table 6.

Table 6: Physicochemical Composition of Syrup

Component	Parameter	Reference Value	Reference
Total sugar	21.36 %	81.88 %	[29]
Glucose	14.28 %	34.50 %	[26]
Soluble Solids	62 %	69.5 – 80.2 %	[28]

The extracted date syrup has 62% of total solids and 21.36% of total sugar which is less than the result reported by Elsharnouby & Aleid [25]. According to the data in Table 5, there was 14.28% glucose present as against the average range of 17.6-26.1g per 100 g found in the date fruit [27]. The value obtained for the glucose level is less than the result in a literature report [26]. This showed that during the manufacture of the syrup, 50% of the glucose and fructose were removed. The presence of simple sugars in the Date syrup could be an advantage if used as an ingredient in baked products [27].

CONCLUSION

Date seeds were de-pitted, dried, pulverised, and chemically activated with 2 M H_3PO_4 prior to carbonisation. Process parameters—impregnation ratio, activation temperature, residence time, and pH, were critical in determining pore development and adsorption potential.

Characterisation confirmed successful conversion into activated carbon. BET analysis revealed a high surface area (1 044.113 m²/g), mesoporous pore diameter (6.523 nm), and total pore volume of 0.5327 cm^3/g. FT-IR spectra identified functional groups consistent with effective adsorption behaviour.

The mesocarp-derived date syrup showed favourable proximate composition, dominated by carbohydrates (84.11%) with low moisture (10.12%), moderate protein (2.63%), fat (1%), and ash (2.14%). These results align with literature identifying date syrup as a nutritious, natural sweetener.

Collectively, the findings indicate that date seed is a suitable, low-cost precursor for activated carbon production. Although adsorption testing was not conducted, the physicochemical properties strongly support its viability as an adsorbent. The utilisation of both seed and mesocarp minimises waste and demonstrates a waste-to-wealth approach that yields valuable products: activated carbon and nutrient-rich syrup.

Given the rising cost and declining sustainability of conventional activated carbon precursors, this study demonstrates that date seed offers an efficient, economical, and renewable alternative. The produced activated carbon exhibited favourable textural and structural properties—high surface area, mesoporosity, and distinct functional groups—confirming its potential for adsorption applications despite the absence of performance testing.

The parallel production of date syrup from the fruit mesocarp further enhances the resource utilisation efficiency, yielding a nutritious natural sweetener with added economic value.

Overall, the comprehensive valorisation of date fruit supports circular-economy principles by converting agricultural biomass into high-value products, reducing environmental waste, and offering sustainable pathways for activated carbon production and natural sweetener development.

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