



Review on preparation and characterization of activated carbon from low cost waste materials



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Abstract

The selection of activating agents for the thermochemical creation of high-grade activated carbon (AC) from agricultural residues and wastes, like feedstock, requires innovative techniques. Overcoming energy losses, and using the best methods to minimize secondary contamination and increase absorptivity, are critical. The present review focuses on the study of the synthesis and characteristics of low cost activated carbon. Various investigators focus on physico-chemical characterization of AC prepared from waste and low-cost materials using TGA, FTIR, XRD, and SEM techniques. Physical properties like bulk volatile matter content, moisture content, density, surface area, ash content, porosity, apparent density, acid porosity, iodine number, fixed carbon, soluble matter, and pH were studied and analysed in detail by various researchers. Here, we also review the importance and impact of activating agents like H₃PO₄, KOH, CaCl₂, ZnCl₂, and H₂SO₄ on agricultural waste and discuss some of the procedures for the preparation of AC. These activating agents are used by various researchers for activation of carbon prepared from waste and performed at various conditions. The review also covers the adsorption isotherms that were developed by various researchers in their work.

Keywords: Activated carbon, activation methods, agricultural waste, biomass, adsorption, adsorption isotherms, kinetic model.

1. Introduction

Activated Carbon (AC) is a carbonaceous solid derived from biomass or coal by thermochemical or thermal methods [1–4]. These materials have become an important research area due to their excellent properties, including high porosity, nontoxicity, large specific areas, thermal stability and rapid adsorption, which has led to their broad utilization. as adsorbent materials for a wide range of applications [5–7]. Some common applications for AC are the purification of drinking water, gas phase adsorption in air pollution, liquid phase adsorption, and wastewater treatment. Until now, AC has been relatively expensive to produce, so that the capability to synthesize a relatively cheap and commercially available AC for

large-scale applications seems to be an interesting challenge. This aim can be approached through the utilization of economical and widely available raw materials [4,8]. A cheap source of raw precursors for AC is agricultural wastes with low inorganic contents and high carbon contents. Presently, AC is often used in the removal of polluted dyes from industrial wastewater.

It is well known that industrial effluents formed from various industrial and human actions can cause high levels of environmental pollution as these wastes are highly colored and contain large amounts of toxic organic species [9,10]. According to published reports, more than 7×10^5 tons per year of wastewater containing dyes and pigments is formed worldwide

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[11,12]. The disposal of this colored water into the surrounding environment results in the contamination by these polluted materials of the soil and/or water. In terms of industrial wastewaters, an important class of polluted materials comprises dyes and pigments. This material can affect the environment for both toxicological and environmental reasons [13–17]. Nevertheless, these dyes are not biodegradable and are costly. Generally speaking, chemical and physical methods involved in the removal of the dye are more effective than bioremediation, but they are very expensive and cannot be used for large scale operations. As a result, adsorption methods are more attractive for dye removal [18]. AC can be used as a good adsorbent in both granular and powder form for a wide range of adsorbates. Consequently, many investigators have been focused on the development of methods to produce AC from low-cost raw including palm ash, sugarcane, soy meal hull, fly ash, rice straw, saw dust, orange peel, rice husk, and bagasse [4,19,20]. In this review, we will summarise the synthesis and properties of AC. We will also consider several applications of AC in the removal of dyes from aqueous solutions.

Synthesis and characteristics of AC

Ekpete and Horsfall [21] studied the preparation and characterization of AC derived from fluted pumpkin stem waste. Bulk density, porosity, iodine number and pH was characterized in AC produced from fluted pumpkin stem waste and compared to a commercially available AC. They found a significant difference in porosity, moisture, iodine number, pH, ash content, lactones, carboxylic acid content and active sites content in pumpkin stem waste-derived AC vs commercial AC [21]. In characterization studies conducted by Yakout and Sharat [22], the influence of activating agent concentration on the surface chemistry and pore structure of AC prepared by phosphoric acid activation of olive stones [22,23].

Preparation and characterisation of AC from biomass material

Fałtynowicz et al. [24] characterized AC prepared from giant knotweed *Reynoutria sachalinensis*, a major component of biomass. Giant knotweed pellets of 10 mm diameter used as raw material to produce AC were obtained from a plantation in Russia (see Figure 1A). In their work, the authors produced AC using several different activation methods: physicochemical activation by steam and CO₂, and chemical activation using solid potassium hydroxide.

According to their results, the characteristics of giant knotweed pellets were similar to those of other previously studied biomass materials [24,25]. Thermogravimetric analysis showed the analysed pellets thermally decomposed in three main phases as shown in Figure 1B.

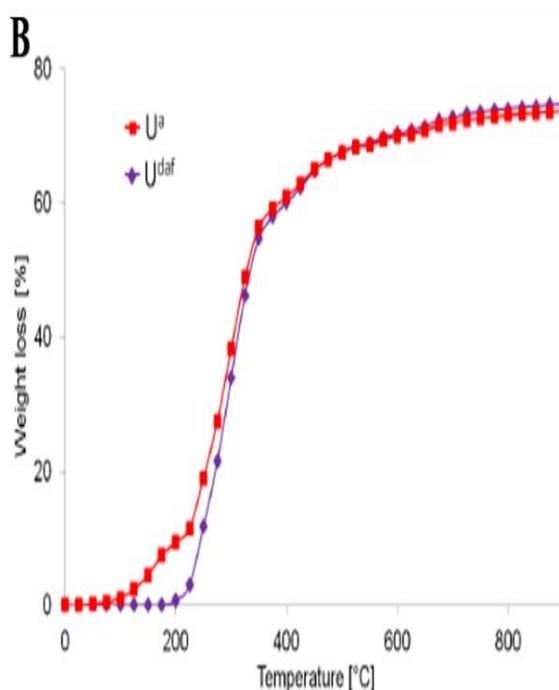


Fig. 1. (A) Giant knotweed pellets used as a raw material for AC preparation. (B) Thermogravimetric analysis of giant knotweed pellets in analytical state (U^a) and dry ash-free state (U^{daf}) [24].

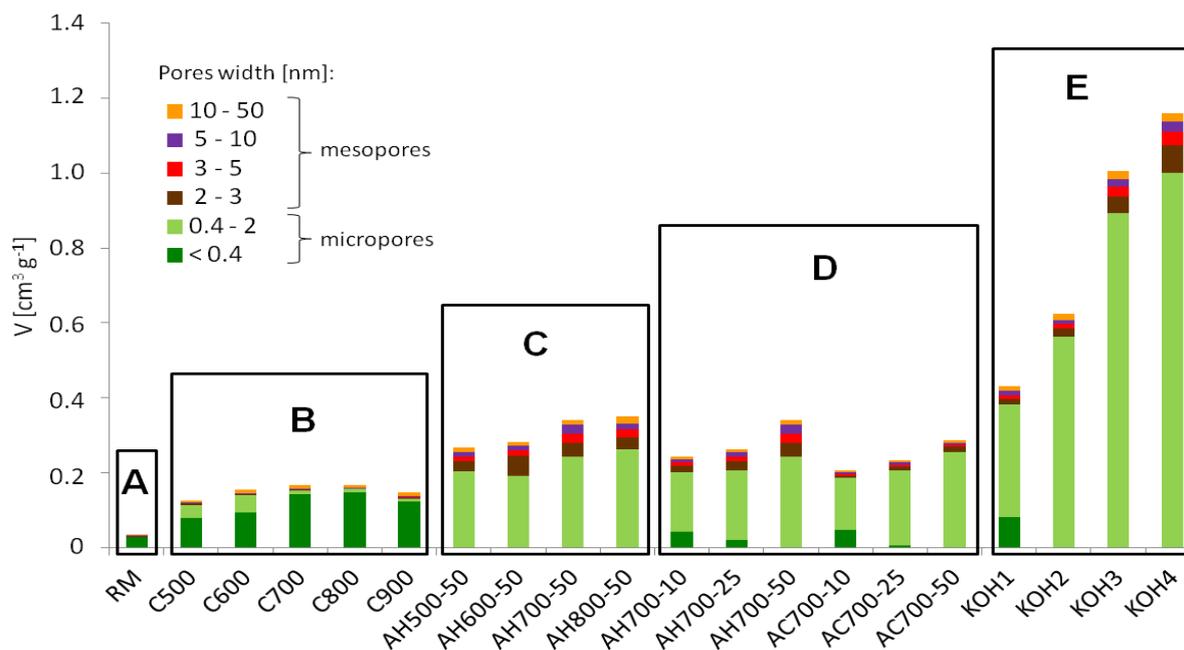


Fig. 2. Distribution of micro- and mesoporous volume according to pore size in raw material (A), chars (B) and AC got by steam activation at various temperatures (C), steam and CO_2 activation to various burn-off (D) and chemical (potassium hydroxide) activation (E) [24].

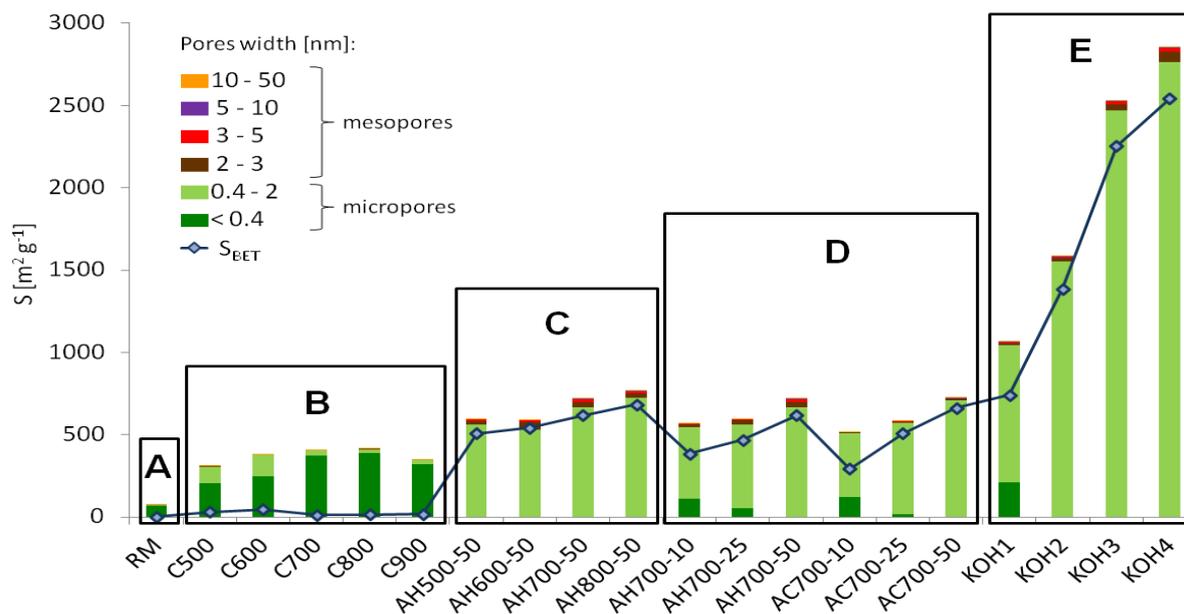


Fig. 3. Distribution of micro- and mesoporous surface area according to pore size and BET surface area in raw material (A), chars (B) and AC produced through steam activation at various temperatures (C), steam and CO_2 activation to various burn-offs, (D) and chemical (potassium hydroxide) activation (E) [24].

Sorption measurements discovered that the giant knotweed biomass did not have a developed porous texture. The volume of micro- and mesoporous (V) did not exceed $0.034 \text{ cm}^3 \text{ g}^{-1}$ (see Figure 2). The porous structure of the raw material consisted mainly of submicropores (pores of width below 0.4 nm) which constituted 92% of a total micro- and mesopores

surface area (S). Consequently, S ($77 \text{ m}^2 \text{ g}^{-1}$), the surface area of meso- and micropores (including submicropores), was significantly more than BET surface area (S_{BET}) ($2 \text{ m}^2 \text{ g}^{-1}$) (see Figure 3). The latter was calculated from an adsorption isotherm of benzene [24]. The authors have shown that the AC derived from steam activation was micro- and

mesoporous, with 20–30% mesopores contribution to the total adsorption pores volume. Meanwhile, AC derived from CO₂ and potassium hydroxide activation were notably microporous, with ~90% proportion of micropores in the total adsorption pores volume. Physicochemical activation of chars resulted in sorbents with BET surface area no more than 700 m² g⁻¹ [24].

utilization in producing AC. This is done by milling (Figure 4a) [26], a high-energy operation [27]. Particle size is one of the essential factors in the thermochemical route; physical condition of biomass notwithstanding, the technique affects activation rate and other production factors [28]. Osman et al. [29] have reported that the preparation of AC usually includes two major steps: carbonization or pyrolysis of the precursor, and activation. Carbonization produces

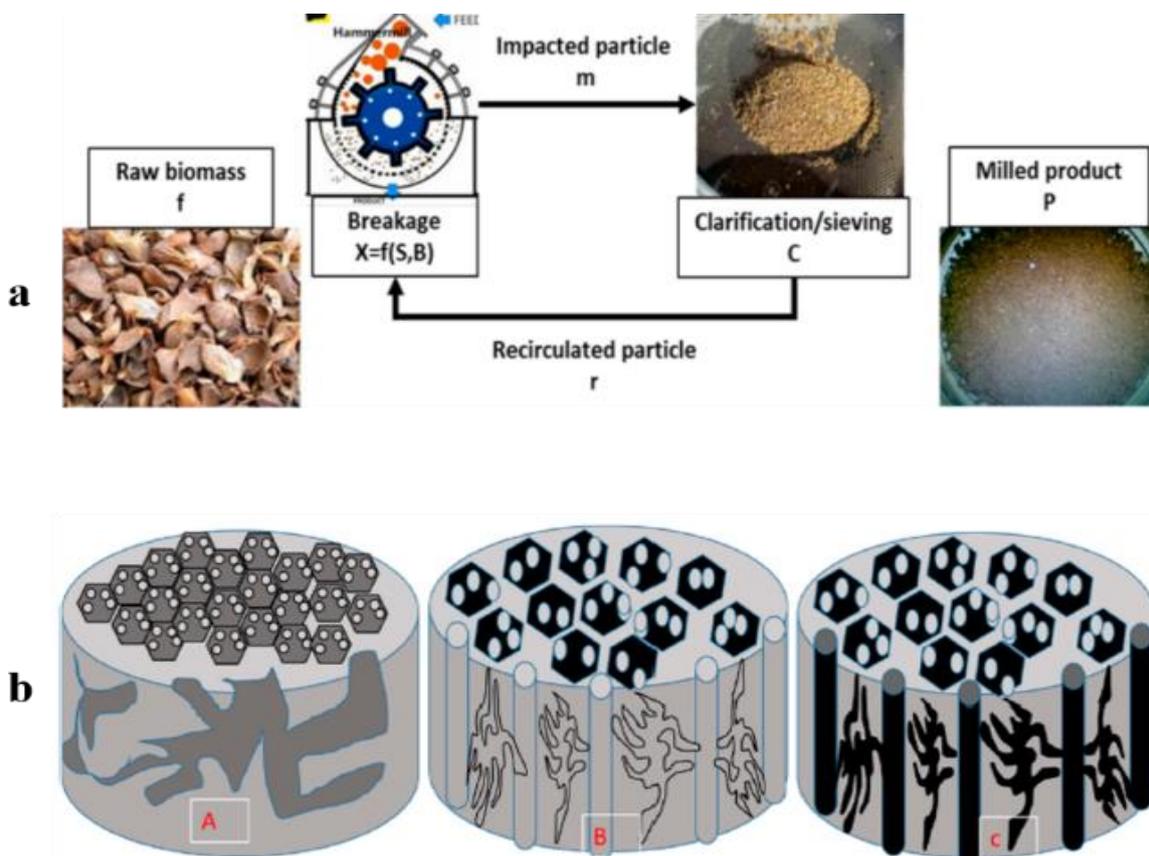


Fig. 4. (a) Hammer milling process for biomass. (b) Activation temperature's influence on AC pore development and morphology A: biomass structure; B: partially developed char structure; C: well-defined porous AC structure [2].

Gil et al. [26] have reported that some biomass wastes, for example oil palm residue, have a high ratio of tramp material, including soil, whereas oil palm processing waste contains a high amount of oil. Therefore, methods used for washing can vary significantly. The need for a large amount of water raises the production cost and hot water may be essential to eliminate oil present in the biomass precursor. These authors have described that oil palm treating waste is mostly made up of large particles and lumps; hence, size reduction is essential for its

a stable structure, with elementary and partially-developed pores [29], which must be enlarged properly by activation by physical activation (Figure 4b) or physicochemical activation [30]. Figure 4b shows the pore development of empty fruit bunch AC, symbolized with A, B and C, following thermal treatment at 350, 500 and 600 °C, respectively [31]. According to Pathak et al [32], pore development is better defined with increased temperature, and modification and treatment methods for raw biomass (see Figure 4b) [32].

Table 1. Analytical characterization and composition of agricultural waste biomass [2].

Agricultural Waste	Proximate Analysis (% w/w)			Ultimate Analysis (% w/w)					Lignocellulosic Composition (% w/w)			Ref.
	Moisture	Ash	Volatiles	C	H	N	S	O	Cellulose	Hemicellulose	Lignin	
Almond stone	11.05	0.76	77.32	48.76	7.52	0.48	0.56	43.6	21.70	27.70	36.10	[40]
Bamboo	15.30	1.76	70.12	34.40	4.61	0.22	0.07	-	26	15	21	[41]
Banana peel	11.56	9.28	88.02	35.65	6.19	1.60	20.7	45.9	-	-	-	[42]
Cassava peel	14	4.50	59.40	59.31	9.78	2.06	0.11	28.7	37.90	23.90	7.50	[43]
Coconut shell	8.21	0.80	77.82	49.62	7.31	0.22	0.10	42.7	14.00	32.00	46.0	[44]
Cotton stalks	6.00	6.30	70.50	43.60	5.80	0.80	0.00	49.8	80-95	5-20	-	[45]
Durian shell	11.27	4.84	-	39.30	5.90	1.00	0.06	53.7	60.45	13.09	15.45	[46]
Grape stalk	8.86	3.15	96.80	34.40	0.43	1.11	0.08	63.9	-	-	-	[47]
EFB	15.01	4.48	82.98	43.89	5.33	0.52	0.10	54.3	42.00	18.90	11.70	[48]
Oil Palm MF	11.10	7.90	84.03	42.20	5.21	2.21	0.14	42.3	42.00	22.00	14.00	[48]
PKS	7.96	1.10	72.47	50.01	6.90	1.90	0.03	41	20.80	22.70	50.70	[46]
Olive stone	10.40	1.40	74.40	44.80	6.00	0.10	0.01	49.0	30.80	17.10	32.60	[49]
Peanut shell	7.98	12.80	79.10	41.52	7.43	2.12	0.60	27.9	-	-	-	[50]
Rice husk	6.34	16.70	67.50	36.52	4.82	0.86	-	41.1	30.42	28.03	36.02	[51]
Sugarcane BG	8.61	4.05	86.02	47.30	6.20	0.27	-	44.1	42.16	36.00	19.30	[51]
Walnut shell	8.73	1.27	77.42	49.30	5.82	44.4	0	-	40.10	20.70	18.20	[49]
Waste tea	5.80	4.29	-	52.72	6.34	2.61	0.18	38.1	17.50	41.30	41.20	[52]

C: carbon, H: hydrogen, N: nitrogen, S: sulphur, O: oxygen, EFB: empty fruit bunch, MF: mesocarp fibre, PKS: palm kernel shell, BG: bagasse.

Martí-Rosselló et al. [33] have described that agricultural waste can be chemically and thermally treated to produce a wide range of prized products, for example bio-solids, bio-gases, bio-oils and biofuels [33]. It can also be considered an energy storage medium [34]. For such wastes, it is necessary to allow for the wide variation in chemical contents. Table 1 shows the proximate, ultimate and lignocellulose contents of such materials through biochemical analysis. The three major structural components: cellulose, lignin and hemicelluloses [35] are formed in varying amounts, depending on whether they are formed by thermochemical [36] or biochemical conversion methods, and this affects the properties of the AC. Rashidi et al. [37] have also shown that agricultural waste is renewable, abundant and available at low cost [37]. Its use is potentially environmentally friendly [38]. Nevertheless, there are some caveats; Abbas and Ahmed [39] determined that leaves are not good raw materials for AC, because of their low carbon content, high volume to weight ratio

and ash content. It is essential to understand the characteristics of any material used, because research has also displayed that in chemical activation, the activating agent digests amorphous lignin better than it does the biomass cellulose [2,39].

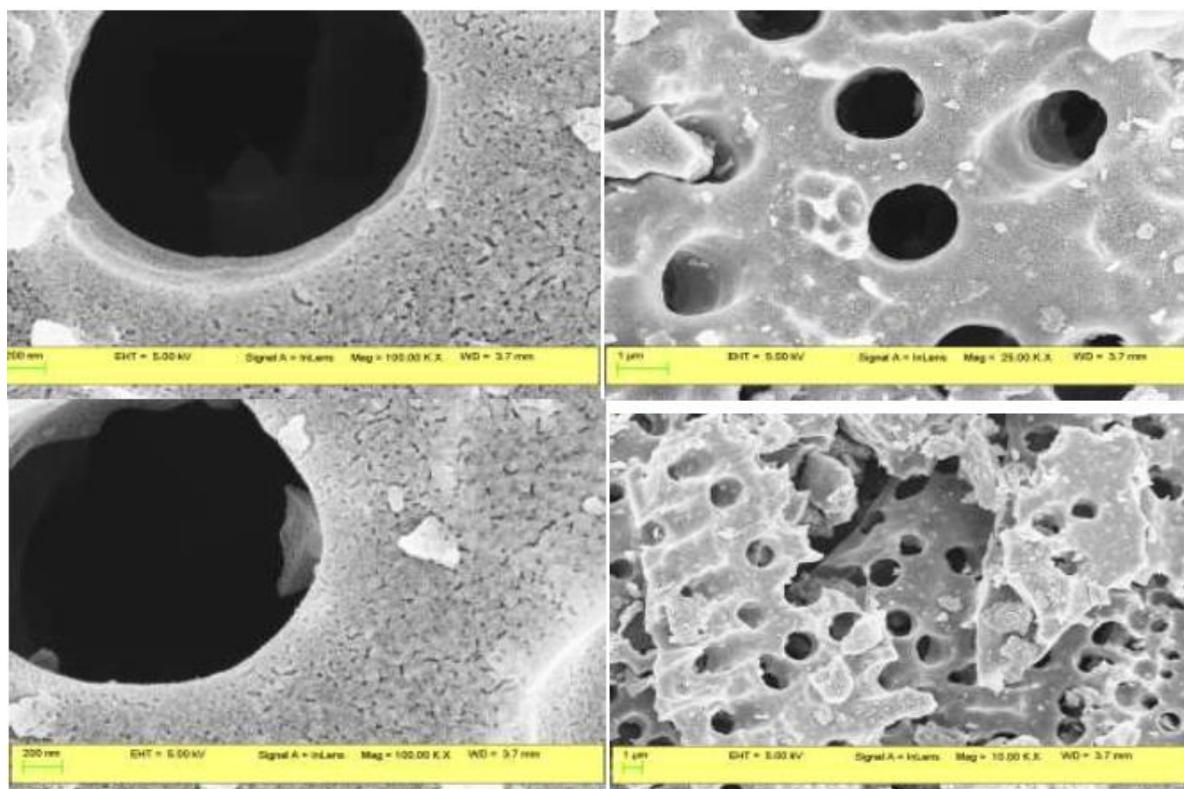
Gawande and Kaware [23] have researched the characterization of AC produced from coconut shell by chemical activation using various activating agents including H_3PO_4 , $CaCl_2$, KOH , H_2SO_4 and $ZnCl_2$, as well as through thermal activation. The authors found that AC which was activated by H_2SO_4 gives better results (see Table 2). The yields of the AC produced by chemical activation were found to be higher than untreated carbon [23]. In this study, various parameters of the prepared AC were characterized by determining various parameters, including pH, volatile matter content, moisture content, ash content, iodine number, fixed carbon, Scanning Electronic Microscope (SEM), BET surface area. Their results are included in Table 3. SEM has been extensively used to study AC surface morphology.

Table 2. Removal of Sulphur [23].

Activating agent	Concentration of adsorbent									
	2 g		4 g		6 g		8 g		10 g	
	Conc in ppm	% sulphur removal	Conc in ppm	% sulphur removal	Conc in ppm	% sulphur removal	Conc in ppm	% sulphur removal	Conc in ppm	% sulphur removal
CaCl ₂	220	34.23	220	34.23	210	37.21	260	22.27	260	22.27
ZnCl ₂	268	19.88	260	22.27	250	25.26	250	25.26	210	37.21
KOH	268	19.88	200	40.20	220	34.23	210	37.21	220	34.23
H ₃ PO ₄	220	34.23	260	22.27	200	40.20	190	43.19	200	40.20
H ₂ SO ₄	220	34.23	200	40.20	190	43.19	150	55.15	140	58.14
Thermally activated	268	19.88	268	22.27	260	22.27	250	25.26	230	31.24

The AC sample was analyzed in an SEM to visualize the porous structure. The SEM images of the H₂SO₄-impregnated AC are shown in Figure 5. From this study, researchers found that the AC prepared from coconut shell can be used for the desulphurization of diesel [23].

In another study, Wilson et al. [53] examined select metal adsorption by AC prepared from peanut shells. These authors produced AC using steam activation, followed by air oxidation of peanut shells. In comparing metal ion binding of three reference ACs to ACs produced using steam-activation and air oxidized peanut shells, they observed similar adsorption

Fig. 5. SEM images of H₂SO₄ impregnated coconut shell AC [23].

properties similar to those achieved using the best commercial, Coal-based ACs [53]. Gumus and Okpeku [54] have characterized AC they produced from snail shell waste (*Helix pomatia*) using CaCl_2 and ZnCl_2 and temperatures ranging from 500°C to 800°C . Their results indicated demonstrated the impact of temperature on pore volume, porosity and ash content [54]. A number of other studies involve preparation and characterization of AC produced using various other plant products. Roozbeh Hosein et al. [55] have characterized AC prepared from apple waste (e.g., apple peels and pulp) using microwave-assisted H_3PO_4 activation [55], while Hassan and Ashfaq [56] have studied the production and characterization of AC from Saudi Arabian dates trees' fronds wastes as a raw material. These authors used H_3PO_4 for activation and studied the effects of different concentrations of phosphoric acid on properties of the resulting AC [56].

Table 3. Characterization of AC [23].

Sr No	Properties	Value
i.	pH	6.9
ii.	% Moisture content	0.5
iii.	% Ash content	1.88
iv.	Volatile matter content	18.86
v.	Iodine number	942 mg/g
vi.	BET surface area	435.1 m^2/g
vii.	Bulk density	0.590 g/cc

Preparation and characterization of AC prepared from Iraqi palm

Athab [57] has studied the preparation and characterization of AC prepared from Iraqi Palm fiber (PFAC) by zinc chloride activation (Figure 6). PFAC was used as low cost adsorbent material for removal of Brilliant Cresyl Blue dye (BCB) from aqueous media using batch adsorption. She investigated the influence of various parameters, including pH, amount of adsorbent, and contact time. PFAC showed excellent removal efficiency (98%) of Brilliant Cresyl Blue. The author concluded that the optimum adsorption conditions were an adsorbent dose of 0.15 g in 100 mL of solution, with 20 min contact time and a pH of 6.5. Athab [57] examined surface morphological changes of PFAC samples using SEM, observing good bonding between fiber and matrix. Figure 7A, B, C and D show SEM images at different

magnifications of palm fibers modified with zinc chloride. Athab [57] also compared the adsorption isotherms empirically derived from her work to the Langmuir and Freundlich models (Figure 7E and F). Her empirically derived isotherms of adsorption fit the

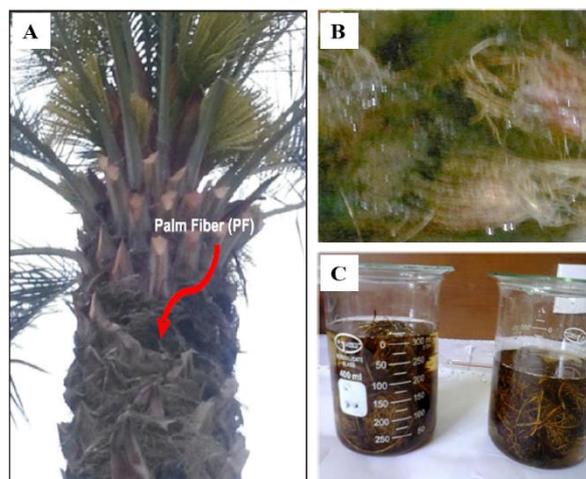


Fig. 6. (A) Photographic image of Iraqi Khestawy palm. (B and C) A bundle of palm fiber before and after washing [57].

Langmuir equation more closely than the Freundlich equation, with R^2 values of 0.97 and 0.915, respectively [57]. Hussein et al. [4] have also investigated the preparation and characterization of AC prepared from Iraqi khestawy date palm through physiochemical activation using H_3PO_4 , the same activating agent used in their work with Saudi Arabian date palms. They synthesized three types of AC from three different parts of the same Iraqi khestawy date palm: palm fronds (AC1), date palm seeds (AC2), and palm fibre (AC3) (Figure 8A). These materials were examined using SEM (see Figure 8C) and Fourier transform infrared spectroscopy (FTIR) (see Figure 8B). They also examined the adsorption activity of the synthesized AC samples by observing the removal of both reactive yellow dye 145 (RY145) and Bismarck brown G (BBG) (see Figure 9C). Both adsorption kinetics and removal percentage of these dyes were studied and compared based on various reaction parameters and adsorption conditions. The effects of contact time and dosage on the removal of BBG from their aqueous solutions for AC1 (synthesized from palm fronds) are shown in Figure 9A. They also investigated the closeness of fit of their empirically derived adsorption isotherms with both the Freundlich and Langmuir equations, as well as adsorption capacity, percent humidity, and the point zero charges of the synthesized samples (PZCs) (see Figure 9D) [4].

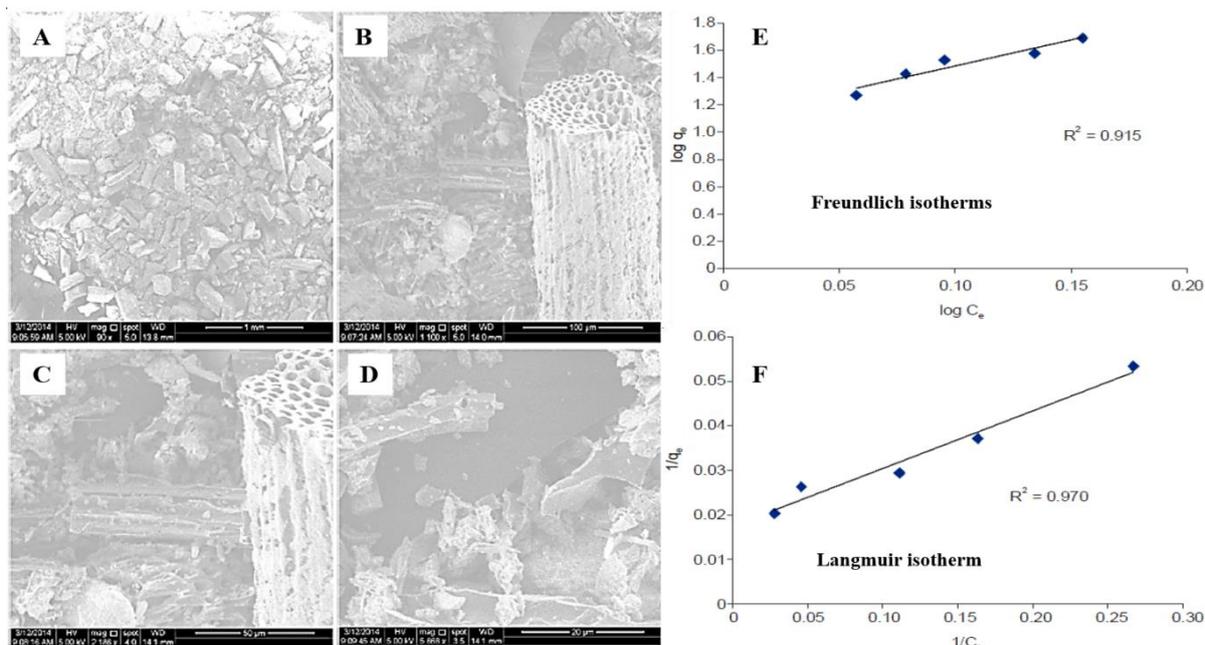


Fig. 7. SEM micrographs of carbon samples from Iraqi Palm fiber: 200 mesh (A, B, C and D at different magnifications). (E) The linear Freundlich adsorption isotherms for brilliant cresyl blue dye adsorption by the activated Iraqi palm fiber carbon. (F) The linear Langmuir adsorption isotherms for brilliant cresyl blue dye adsorption by the activated Iraqi palm fiber carbon [57].

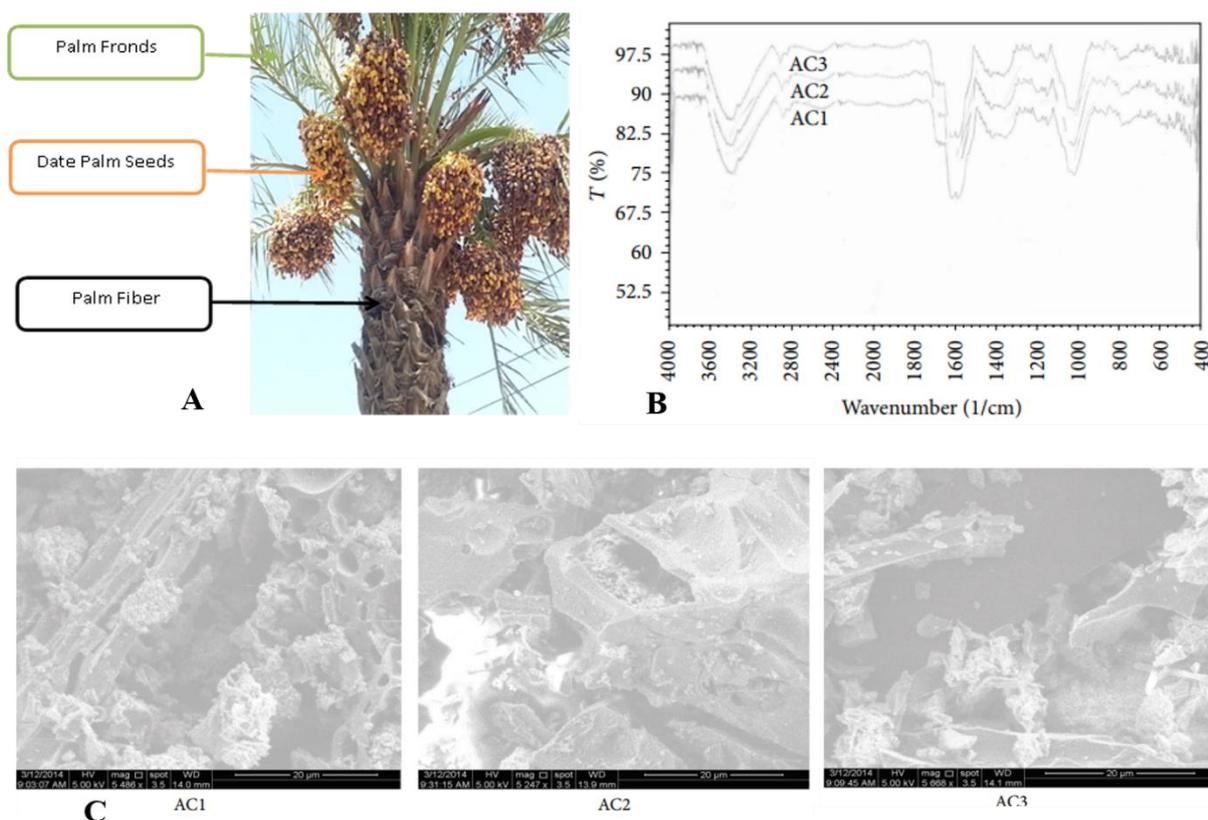


Fig. 8. (A) Diagrammatic representation of Iraqi Khestawy date palm structure. (B) FTIR spectra for AC1, AC2, and AC3 samples synthesized from Iraqi Khestawy date palm. (C) SEM images for the AC1, AC2, and AC3 samples synthesized from Iraqi Khestawy date palm [4].

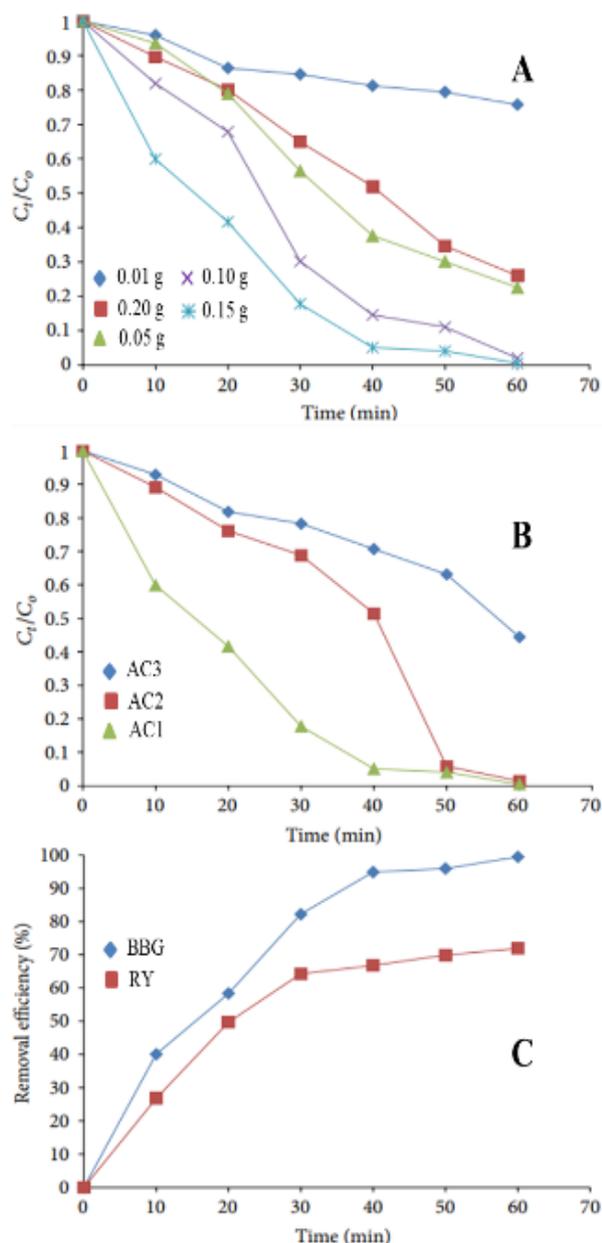


Fig. 9. (A) The effect of dosage and contact time of AC1 on the removal of BBG dye. (B) Comparison between the efficiency of AC1, AC2, and AC3 on the removal of BBG. (C) Comparison between removal efficiency of BBG and RY145 dyes on AC1. (D, Table 1) Langmuir and Freundlich isotherm constants, (D, Table 2) adsorption capacity for the synthesized AC by MB adsorption, (D, Table 3) the percentage of moisture content for AC samples and (D, Table 4) PZC for AC1, AC2, and AC3 samples synthesized from Iraqi Khestawy date palm [4].

They demonstrated the most effective removal of BBG from aqueous solutions was achieved using AC derived from palm fronds, followed by AC derived from palm seeds. AC derived from palm fibre were the least efficient in removing BBG from aqueous solutions (see Figure 9B) [4]. Taken as a whole, their results highlighted the utility of preparing AC from widely available raw agricultural materials in Iraq

D TABLE 1: Langmuir and Freundlich isotherm constants.

Isotherms	Constants/correlation coefficients	Values
Langmuir	R^2	0.9508
	q_m	65.7894
	K_L	0.49673
Freundlich	R^2	0.6407
	K_F	21.9280
	N	2.3798

TABLE 2: Adsorption capacity for the synthesized AC by MB adsorption.

Type of AC	Uptake capacity of AC (mg/g)
AC1	199.8
AC2	199.4
AC3	198.8

TABLE 3: The percentage of moisture content for AC samples.

Type of AC	Humidity%
AC1	48
AC2	46
AC3	45

TABLE 4: PZC for AC1, AC2, and AC3 samples synthesized from IKDP.

Type of AC	pH of AC
AC1	8.25 ± 0.10
AC2	8.62 ± 0.12
AC3	8.45 ± 0.15

such as date palms for use as an efficient adsorbent for a wide range of waste materials. Moreover, this study indicated that palm fronds of the Iraqi Khestawy date palm are well-suited for production of AC for use in BBG removal from aqueous solutions, in comparison to other parts of the Iraqi Khestawy date palm such as date palm seeds or palm fibre [4].

Halbus et al. [58] have studied the adsorption isotherm and kinetics of disperse blue dye on AC prepared from Iraqi date seeds (referred to throughout the remainder of this review as Zahdi date seeds, or ZDS). These authors also investigated the impact of contact time, pH and different doses on adsorption isotherms and kinetics. Additionally, they compared their equilibrium adsorption data for ZDS-derived AC (ZDSAC) using the Langmuir and Freundlich equations. Their results showed that the best fit was achieved with the Langmuir isotherm equation. ZDSAC have been found to have high efficiency for removal of dispersed blue dye from wastewater. In their work, the authors demonstrated significantly higher dispersed blue dye removal efficiencies using ZDSAC than commercially available AC (66.47% vs 54.33%), respectively. The researchers also studied adsorption kinetics using pseudo-first order, pseudo-second order, and intraparticle diffusion models, finding that the adsorption kinetics was more accurately represented by a pseudo-second order model (see Figure 10) [58].

khestawy (AC1) and zahdy (AC2) date palm seeds using H_3PO_4 as an activator. Specific physical properties studied in this work included adsorption capacity, humidity, and ash content. These material also characterized these materials using FTIR. According to their results, AC2 (from zahdy date palm seeds) was more effective than AC1 (Iraqi khestawy date palm seeds) in dye removal under the same adsorption conditions. Their findings provide an important contribution to the exploration of using raw agricultural waste – especially from Iraqi palm seeds, to produce adsorbent materials for a wide variety of industrial and environmental applications [59].

Al-Sharifly et al. [15] characterized the removal efficiency of AC prepared from hazelnut shells (HS) for reactive red 2 dye (RR2) in aqueous solutions. They studied the comparative adsorption behavior of RR2 onto the porous AC at varying temperatures, contact times, pH and adsorbent doses. Their results indicated increased RR2 adsorption with increased temperature. They also studied variation of entropy, enthalpy, and Gibbs Free Energy (ΔS° , ΔH° , ΔG°) of RR2 dye adsorption at varying temperatures. Their

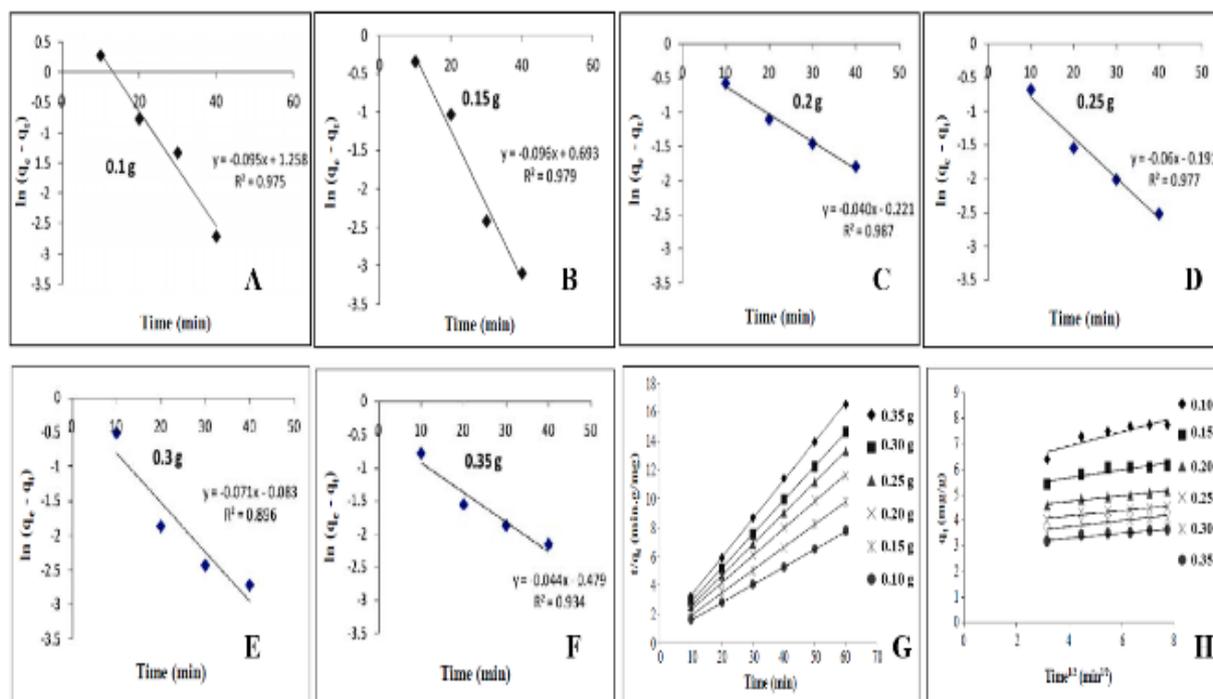


Fig. 10. Pseudo-first order kinetic model (A-F), pseudo-second order kinetic model (G), and intraparticle diffusion model (H) for the adsorption of disperse blue dye onto ZDSAC [58].

In another study, Halbus et al. [59] compared reactive yellow dye 145 (RYD145) removal from aqueous solutions using AC derived both from Iraqi

date indicate that the process of adsorption RR2 dye adsorption from aqueous solutions is a spontaneous

and endothermic process and that adsorption increases with increased temperature [15].

Other researchers have studied yellow-145 dye adsorption using AC derived from Iraqi zahdi date palm seeds chemically activated using $ZnCl_2$, KOH and H_3PO_4 [60–62]. Their results indicated highest adsorption of reactive yellow-145 dye using AC activated with KOH, followed by AC activated using H_3PO_4 and AC activated with $ZnCl_2$, respectively. AC not treated with any activation agent demonstrated the lowest adsorption efficiency. They also examined the ash content, the external of surface area and the humidity of the AC. The authors observed large external surface areas, which makes them good adsorbent materials, and low ash but relatively high moisture content in the ACs they produced [62].

2. Conclusions

ACs can be used as an efficient adsorbent for a wide variety of pollutants and can be easily prepared from a number of types of agricultural waste and low-cost materials. The chemical activation approach for AC creation is widely applied due to its ability to produce effective, high porosity AC with a wide range of activating agents under a myriad of processing conditions, although adsorption efficiency varies significantly with production parameters and activating agent used. Common chemical activating agents include KOH, H_2SO_4 , NaOH, $ZnCl_2$, H_3PO_4 , and $CaCl_2$. Each activating agent has a unique influence on the characteristics and applications of the resultant AC. Nevertheless, because of the challenges of AC production from agricultural waste and biomass, there remains a need to improve techniques to achieve industrial-scale formation of AC. Many researchers reported that physically activated AC demonstrates better adsorbent behaviour than chemically activated AC. AC synthesized from oil palm empty fruit bunches and rice husks can reduce the cost of production because of their abundance as an excess agricultural waste in our environment. Using these agricultural wastes can also solve disposal problems by converting them to valuable materials useful in treating other wastes. The large body of published reports summarized in this review confirms that AC prepared from such agricultural wastes can be effectively used as an adsorbent for removing a wide variety of hazardous compounds from wastewaters and industrial waste effluents.

3. Conflicts of interest

“There are no conflicts to declare”.

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