



A review on Activated Carbon Prepared from Agricultural Waste using Conventional and Microwave Activation

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Abstract

In the recent years the research on the activated carbon preparation from agro-waste and byproducts have been increased due to their potency for agro-waste elimination. This paper presents a literature review on the synthesis of activated carbon from agro-waste using microwave irradiation method for heating. The applicable approach is highlighted, as well as the effects of activation conditions including carbonization temperature, retention period, and impregnation ratio. The review reveals that the agricultural wastes heated using a chemical process and microwave energy can produce activated carbon with a surface area that is significantly higher than that using the conventional heating method.

Keywords: *Agricultural waste, microwave heating, conventional heating, activated carbon.*

1. Introduction

Activated carbon is a type of amorphous carbon, which physically or chemically treated to produce large internal surface area and pore volume therefore, it regarded as an excellent adsorbent for different pollutants [1-5]. The specification of the large surface area of the produced activated carbon that may be reaching 3000 m²/gm and the high degree of surface reactivity are the main reason to consider using activated carbon for liquid inorganic and organic or gaseous pollutants elimination [6-10]. Various materials and methods were used for activated carbon products such as chemical activation by impregnating the raw material with phosphoric acid, nitric acid, sulfuric acid, potassium hydroxide, sodium hydroxide, zinc chloride and physical activation with CO₂ or steam followed by carbonization via conventional heating with temperature range from 400 to 800 °C [11, 12] or

carbonization via microwave heating [13-15]. In conventional heating, the heat generated at the surface of the treated material and gradually transferred to their interior parts that lead to caloric transport difficulties. Whereas the microwave irradiation technique is currently applied in different fields for dielectric materials heating due to its ability to heat materials internally and volumetrically in a short time therefore it reduces energy consumption [16-20]. The removal of pollutants by adsorption using activated carbon is considered the best method as compared with adsorption, evaporation, ion exchange, precipitation, and reverse osmosis methods [21, 22]. The ion exchange method required high operating costs and it was applied for color, heavy metals, nitrate, COD and heavy metals [15-20]. Coal, and wood as carbonaceous materials are expensive and still widely used for large-scale production of activated carbon [21]. Therefore, the development of low-cost and available raw

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materials to produce activated carbon for water, and wastewater purification. Agricultural waste such as banana stalk [6], alhaji [1], Albizia lebbeck and Melia azedarach seed shells [5], date palm fronds [4], apricot seed [2], sawdust [23], cotton stalk [16], tobacco stems [18], olive cakes and olive stones [21, 22]. olive-waste cakes [24], date stones precursor [25], rice husks precursor [26], almond shells precursor [27], corn cobs precursor [28], waste tea precursor [29], cherry stones precursor [30], rice bran precursor [31], durian shells precursor [32], herb residues precursor [33], coffee husks precursor [33], spent grains precursor [34], giant reeds precursor [35] and Giant Reed precursor [36] have been studied for gas and water pollutants treatments. The present article reports literature review on the AC preparation from different types of agricultural waste using chemical activation with conventional and microwave heating.

2. Production of Activated Carbon

Activated carbon involves different steps including raw materials pretreatment, raw material impregnation with chemical activators followed by carbonization and activators removing, below the production steps of activated carbon with their information.

2.1 Raw material pretreatment

The raw materials are preliminarily treated including cleaning, drying, crushing, and sieving to the desired particle size before the activated carbon production. The raw material particle size is a very important factor for handling, such as chemical impregnation or mixing; likewise, its influence on the properties of the activated carbon produced. Several researchers use agriculture shells or stones as raw materials without crushing, but the crushed and sieved raw materials around 1-2 mm enhanced the handling and materials activation [11]. While other researchers investigated the impact of the activated carbon's particle size on the adsorption of the bilirubin bound to albumin [37]. Particle sizes in the ranges of 63-100 m, 100-250 m, and 250-500 m were all examined. The outcomes showed that the bilirubin adsorption capacity rose with increasing adsorbent surface area and fell with increasing particle size. The maximum adsorption was obtained with the activated carbon of 1413 m²/g and the particle size of 82 μm. Likewise, [37] studied the apricot stones particle size effect on the

activated carbon preparation. Three ranges of particle sizes were investigated: 0.85-1.7 mm, 1.7-3.35 mm, and 3.35-4.00 mm. The obtained results show that the minimum particle size enhanced the surface area of the activated carbon produced and consequently, increases the adsorption.

2.2 Activation processes

For the manufacture of activated carbon, there are two activation processes: Physical and chemical activation. Physical activation involves the carbonization of the raw material up to 700 °C under an inert atmosphere than the carbonized material is treated under oxidizing atmosphere using air, CO₂, steam, or their mixture. Whereas, in the chemical activation, the raw material is treated with acid, base, or metal salts and then carbonized with temperature range from 400 to 600 °C [38].

2.2.1 Chemical activation with conventional heating

Different chemical compounds including sodium hydroxide or phosphoric acid, potassium hydroxide, or zinc chloride were used for chemical activation. The Depending on the chemical agent used and the type of raw material, the raw material must be impregnated in liquid or solid activation agents for up to 24 hours. the chemical impregnation improves the surface porosity of the produced activated carbon via carbonization due to dehydration, agent evaporation and degradation. The produced activated carbon is washed by distilled water or diluted base or dilute acid to remove residual chemicals from the activated carbon. The main advantages of chemical activation are to enhanced activated carbon yield with high porosity; low energy consumed for carbonization. Whereas the disadvantages of the chemical activation are high cost of the chemical activation as well as further washing with distilled or deionized water to remove the residual chemical agents. Haimour and Emeish [39] have studied the effect of the temperature, chemical impregnation ratio, and impregnation time on the date stone activated carbon yield and the capacity of iodine adsorption. Two chemical agents H₃PO₄ and ZnCl₂ for impregnation under similar operating conditions (impregnation ratio, activation time, carbonization temperature. The result showed that the yield and iodine adsorption capacity for activated carbon produced using ZnCl₂ as an impregnation agent was better than those obtained from the impregnation with H₃PO₄. Lua and Yang

[40] have studied the temperature, chemical impregnation ratio, and impregnation time parameters effects on the properties of the activated carbon produced from pistachio nut shells. $ZnCl_2$ impregnation agent was used for activation nitrogen and vacuum conditions, respectively. The results showed that the activated carbon produced under vacuum conditions has better quality compared to that produced under a nitrogen atmosphere. As well the results showed that the product surface area increased with impregnation ratio, impregnation time, and carbonization temperature to a certain limit. Several researchers have indicated that the surface area and pore volume of the activated carbon prepared under vacuum conditions was slightly enhanced compared to that produced under a nitrogen atmosphere [40, 41]. [42] have investigated the effect of separately dual impregnation for cherry stones in $ZnCl_2$ with 3:1 impregnation ratio and 2 h with heating temperature range from 400 °C to 800 °C under nitrogen gas atmosphere on the activated carbon surface area. Their results showed that the surface area decreased from 1472 m^2/g to 966 m^2/g with temperature increasing from 400 °C to 800 °C. [43] have investigated olive seeds' activated carbon surface area produced from a mixture of KOH with char at a ratio of 4:1 and carbonization at different temperatures (800-900 °C) for different time intervals of 1, 2, 3, and 4 h. Their results showed that the activated carbon surface area increased as the temperature and retention time increased a maximum surface area of 3000 m^2/g was obtained at maximum conditions. [44] have conducted an experimental study for the adsorption of Cu from aqueous solution by rice husk activated carbon using the central composite methodology to analyze the activation temperature, time, and $ZnCl_2$ to char impregnation ratio. Two quadratic models were generated for the correlation of activated carbon yield and Cu removal efficiency as responses with the

preparation variables. The results showed that most significant variable was the activation temperature as compared to the other variables and the maximum surface area of the activated carbon was obtained with an activation time of 1.71 hr, and temperature of 500 °C. Waste tea-based activated carbon was prepared by [45] using potassium acetate for activation. The effects of impregnation ratio (0.3-0.5), activation temperature (500-800) °C, and activation time (60-150) min were studied using RSM technique for preparation conditions optimization. Three responses were selected; the percentage of yield, methylene blue, and acid blue 29 removals for investigation. The results showed that the optimal preparation conditions for maximum surface area with good responses obtained were 800 °C for activation temperature, 1.4 for impregnation ratio, and 120 min for activation time which demonstrated good adsorption capacities. The waste tea was studied also by [46] for activated carbon preparation using K_2CO_3 for activation. The effects of impregnation ratio (0.5:1-1:1), activation temperature (400-900) °C, and activation time (0.5:1-2:1) hr on the activated carbon surface area were studied. The results show that the activation temperature and the impregnation ratio have the main effect on the pore volume and surface area of the activated carbon produced. In addition, guava seeds, wheat straws, lignite, biomass waste and biomass tremella were investigated to prepare activated carbon using KOH and NaOH with different impregnation ratio and different carbonization temperatures under nitrogen [47-51]. A summary of different agricultural byproducts as raw materials for activated carbon preparation with different activating agents and conditions with their results respect to surface area obtained are illustrated in Table 1.

Table 1,
Literature summary of chemical activation with conventional heating.

Material	Chemical	Mass ratio	Activation conditions	Atmosphere	Surface area m ² /g	References
Alhagi	KOH	1:1	600 C, 1 hr	N ₂	641.6	[1]
Date Stone	H ₃ PO ₄	0.4:1	800 C, 1 hr	N ₂	495	[52]
pistachio-nut shell	ZnCl ₂	0.75:1	400 C, 1 hr	Vacuum N ₂	1647.16	[40]
		1.5:1	500 C, 2 hr		2527	
Sawdust	ZnCl ₂	1:1	30 Kpa	Vacuum N ₂	1079.21	[41]
Cherry stones	ZnCl ₂	3:1	400 C, 2 hr	N ₂	1472	[42]
			800 C, 2 hr		992	
Olive-seed waste	K ₂ CO ₃	---	900 C, 4 hr	N ₂	3049	[43]
Rice Husk	CO ₂	---	400 C, 0.5 hr	N ₂	370.75	[44]
Waste tea	CH ₃ CO ₂ K	1.4:1	800 C, 2 hr	N ₂	854.3	[45]
Waste tea	K ₂ CO ₃	1:1	900 C, 2 hr	N ₂	1722	[46]
Guava seeds	NaOH	3:1	800 C, 1 hr	N ₂	2573.6	[47]
Wheat straws	NaOH	4:1	650 C, 2 hr	N ₂	1067	[48]
Lignite	KOH	4:1	800 C, 1 hr	N ₂	3036	[49]
Biomass waste	KOH	4:1	850 C, 1 hr	N ₂	421.4	[50]
Biomass tremella	KOH	5:1	800 C, 3 hr	N ₂	3760	[51]

2.2.2 Chemical activation with microwave heating

Carbonaceous source materials are considered to produce good absorbers; these properties explain the wide usage of microwave heating techniques for activated carbon preparation over the last years as compared to conventional heating. Therefore, these techniques have been applied to activated carbon preparation by many researchers in the last five years. Cotton stalk precursor was selected with microwave-assisted and chemical activation to prepare activated carbon was studied by [53]. The effects of ZnCl₂ impregnation ratio, microwave power, and microwave radiation time on the yield-activated carbon produced with methylene blue adsorption capacities were investigated. The results showed that the optimum values of activated carbon preparation conditions were 1.6 g/g, 560 watts, and 9 min for impregnation ratio, microwave power, and radiation time, respectively. Likewise, [54] has used cotton stalks precursor with phosphoric acid for chemical activation and microwave heating to optimize the operating conditions for activated carbon preparation. The effects of radiation power, radiation time, H₃PO₄ concentration by volume, and impregnation time, respectively. In addition, apricot seed precursor was used with microwave, and 45% w/w pyrophosphoric chemical activation to prepare activated carbon was studied by [55]. The apricot seed was impregnated in 45% w/w of H₃PO₄ at 80 °C (IR of 1: 3) via impregnation time of 4 hr, with

a microwave power of 700 watts and radiation time of 20 min. A set of experiments was conducted to investigate the effect of adsorption time, pH, and diclofenac concentration with activated carbon dose on removal efficiency using the central composite method. The results showed that the maximum adsorption efficiency was obtained with the optimum values of adsorption time = 150 min, pH = 3.25, diclofenac concentration = 30 mg/L, and adsorbent dose = 0.267g. Isotherm and pseudo-kinetic models were applied for experimental data which implies monolayer adsorption for isotherm with chemisorption for kinetics models. Tobacco stems precursor with K₂CO₃ as a chemical activation agent via microwave heating with their effects on the surface area of the activated area produced was investigated by [18]. The range of microwave power (80-700) watts, radiation time (5-60) min, and impregnation ratio (1-2) on the yield and the adsorption capacities of methylene blue dye were studied. The results demonstrated that the optimum values of 700 W, 30 min, and 1.5:1 for microwave power, radiation time, and impregnation ratio, respectively produced 517.5 mg/g for yield, and 1834 mg/g for adsorption capacity. Waste tea precursor via phosphoric acid activation with conventional and microwave heating for activated carbon preparation was studied by [56]. The waste tea was carbonized from 250 °C to 700 °C under N₂ atmosphere under a nitrogen atmosphere with different impregnation ratios from 1:1 to 3:1. The waste tea precursor with phosphoric acid was heated in microwave oven prior heating in muffle furnace. The results showed

that the maximum surface area of activated carbon sample treated with microwave and conventional heating was 1157 m²/g. Whereas the maximum surface area of activated carbon sample treated conventional heating was 928.8 m²/g. This implies that samples treated with microwave heating prior conventional heating was reasonably influenced on the micropore structure and surface area. Bamboo precursor was used with microwave and H₃PO₄ chemical activation to prepare activated carbon was studied by [57]. The effects of impregnation ratio, microwave power, and microwave radiation time on the yield and surface area of the activated carbon produced were investigated. The results demonstrated that the optimum values of 350 W, 20 min, and 1:1 for microwave power, radiation time, and impregnation ratio, respectively produce 1432 mg/g for surface area, and 48% of activated carbon yield, as well as the results, showed that the microwave-assisted activation demonstrated faster and higher activated carbon yield compared with the conventional heating. Jatropha hull precursor via steam and CO₂ activation with conventional and microwave heating for activated carbon preparation was studied by [58]. The results showed that the activation under steam with conventional and microwave heating offered approximately same yield and porous nature. Whereas the values surface area and pore volume were doubled with steam activation with microwave treatment as compared to CO₂ activation with microwave and conventional heating. investigated coconut shells for activated carbon manufacture [59] utilizing steam, CO₂, and

a CO₂-steam mixture with microwave radiation. The activation method was explored in two steps, with carbonization under N₂ atmosphere at 1000 °C with conventional heating, followed by activation with microwave heating at 900 °C. d. The results showed that the activated carbon produced offered surface area was exceed 2000 m²/g irrespective to the activation, and the activated carbon produced with CO₂ activation offered highest surface area surface compared steam, CO₂, and CO₂-steam mixture. The results also revealed that the microwave-assisted activation demonstrated faster activated carbon produced compared with the conventional heating. Based on the above literature review the most efficient of operating parameters ranges of the activation time, activation temperature, and impregnation ratio for activated carbon preparation from agricultural waste using chemical activation with conventional heating techniques were obtained with 1-3 hr of activation time, 400-800 °C of activation temperature, and 0.5-2 of impregnation ratio. Whereas the activated carbon prepared from agricultural waste using chemical activation with microwave heating were obtained with 5-15 min of activation time, 350-700 watt of microwave power, and 0.5-2 impregnation ratio. A summary of different agricultural byproducts as raw materials for activated carbon preparation with different activating agents and microwave conditions with their results respect to surface area obtained are illustrated in Table 2.

Table 2,
Literature summery of chemical activation with Microwave heating

Material	Chemical	Mass ratio	Activation conditions	Atmosphere	Surface area m ² /g	References
Cotton stalk	ZnCl ₂	1.6:1	560 W, 9 min	N ₂	794.84	[53]
Cotton stalk	H ₃ PO ₄	0.4:1	400 W, 9 min	N ₂	652.82	[54]
Apricot seeds	H ₃ PO ₄	3:1	700 W, 20 min	N ₂	758. 649	[59]
Waste tea	H ₃ PO ₄	3:1	350 C, 10 min	N ₂	758. 649	[56]
Bamboo	H ₃ PO ₄	3:1	350 W, 20 min	N ₂	1432	[57]
Jatropha hull	Steam and CO ₂	----	900 C, 19 min	N ₂	1778.5	[58]
Coconut shells	Steam and CO ₂	----	700 W, 15 min	N ₂	2000	[60]

According to the above literature review, a comparison between the conventional and microwave heating for activated carbon

preparation based on activation time (carbonization), heating mode, gas consuming, and experiments set-up size are given in Table 3.

**Table 3,
Summary of the conventional and microwave heating for activated carbon preparation.**

Parameters	Conventional heating	Microwave heating
Activation time	<ul style="list-style-type: none"> It takes several hours to reach the desired specification which mean extra operating cost. 	<ul style="list-style-type: none"> It takes several minutes to reach the desired specification
Heating mode	<ul style="list-style-type: none"> External (surface) heating The temperature gradient transmitted from the hot outer surface of the material to its interior may lead to its damage The conventional heating induced a quick rise in temperature of the surface leading to irregular temperature distribution led to negatively effect on the quality of the product. 	<ul style="list-style-type: none"> Internal heating The temperature gradient transmitted from the hot interior particles of the material to its outer surface is done rapidly and so highly effective material produced The microwave heating induced a quick rise in temperature of the particles, leading to uniform temperature distribution, and energy savings.
Gas consuming	<ul style="list-style-type: none"> Highly gas consumed due to long time of activation 	<ul style="list-style-type: none"> Low gas consumed due to short time of activation
Set-up size	<ul style="list-style-type: none"> Large equipment (Furnace) 	<ul style="list-style-type: none"> Small equipment (Furnace)

3. Conclusion

- In recent years, attention has turned to microwave technology instead of traditional heating in the preparation of activated carbon from agricultural waste and products as an interesting alternative approach in providing many advantages, including:
 - Reducing the time required for production, which means lower energy consumption required for activated carbon preparation.
 - Reducing the number of steps required for production, consequently reducing the number of reagents and equipment required for activated carbon preparation.
 - (iii) Microwave-assisted production technique is more efficient and more economically competitive than that obtained through conventional techniques.
- The comparison of activated carbon produced via microwave heating and conventional heating techniques for the same raw materials revealed that microwaves with chemical activation enhanced the surface areas more than those obtained by conventional heating with chemical activation.
- The activated carbon properties depend on the raw material type, activation technique, activation agent type, and process conditions including activation temperature, activation time, and impregnation ratio (IR).

- The efficiency operating parameters ranges of the activation time, activation temperature, and impregnation ratio for activated carbon prepared from agricultural waste using chemical activation with conventional heating techniques were obtained with 1-3 hr of activation time, 400-800 °C of activation temperature, and 0.5-2 of impregnation ratio. Whereas the activated carbon prepared from agricultural waste using chemical activation with microwave heating was obtained with 5-15 min of activation time, 350-700 watt of microwave power, and 0.5-2 impregnation ratio.

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Conflict of Interest Statement

All authors declare that they have no conflicts of interest to disclose

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الكربون المنشط المحضر من المخلفات الزراعية باستخدام التنشيط التقليدي والميكروويف

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الخلاصة

في السنوات السابقة، تمت زيادة عدد البحوث حول تحضير الكربون المنشط من المخلفات الزراعية والمنتجات الثانوية لهذه المنتجات وذلك لغرض التخلص من المخلفات الزراعية بشكل مفيد. هذا البحث يقدم مراجعة الى الأدبيات حول تحضير الكربون المنشط من النفايات الزراعية، باستخدام طريقة التسخين الأمواج الميكروية، وتسليط الضوء على تطبيق هذه الطريقة في تحضير الكربون المنشط من خلال بيان تأثير ظروف التنشيط بما في ذلك درجة حرارة الكربنة، وفترة التنشيط، ونسبة الغمر. وقد كشفت عملية المراجعة أن استخدام المخلفات الزراعية في تحضير الكربون المنشط بواسطة المعاملة الكيميائية وطاقة الميكروويف للتسخين يمكن أن تنتج الكربون المنشط بمساحة سطحية أعلى بكثير من تلك التي تستخدم طريقة التسخين التقليدية للإنتاج.