

## REVIEW PAPER

# Sustainable Activated Carbon Production via Microwave for Wastewater Treatment: A Comparative Review

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## ABSTRACT

This is an era where the application of adsorption and usage of activated carbons (AC) are considered as mainstream water treatments. The upgrade of these materials may only be through its preparation methods, where most researchers have transitioned from using the conventional furnace methods to using microwave ovens. Derived from various precursors, ACs can be the key in developing numerous environmental applications. This paper reviews the development of production processes of AC from various precursors in the past decades by microwave heating. The importance of the applied methodology and how activating conditions play an influential role, such as carbonisation temperature, activation time, and impregnation ratio are also outlined in this review. From the review of AC production processes, ACs produced from various precursors by chemical method with microwave heating have shown to be the significant factor in developing ACs with relatively higher surface area compared to conventional heating ACs.

Keywords: Activated carbon, adsorption, microwave, optimum conditions

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## INTRODUCTION

Adsorption is one of the preferable treatments for wastewater due to its significant removal efficiency. The most popular and commonly used adsorption material is activated carbon (AC). It is classified as a graphite carbonaceous adsorbent with extraordinary features such as vast porous surface area, well-developed structure of its pores, low acid or base reactivity. ACs are produced from various raw materials such as coal, peat and agricultural wastes, through physical or chemical activation methods. Currently, commercial activated carbons are the most common adsorbent used in the market but their availability is limited due to the high cost resulting from the use of non-renewable and expensive starting materials (Alslaihi *et al.*, 2013).

In the past decade, adsorption by AC has shown high efficiency in the removal of water pollutants (Njoku *et al.*, 2014). Factors such as its quality, properties, and production cost of AC contribute to

its removal capability though these factors depend significantly on both raw materials and technique used. Generally, there are two methods of preparing ACs, physical or chemical activation. Physical activation involves carbonisation of the targeted raw material, followed by controlled gasification at high temperatures in a stream of oxidising gas (steam, CO<sub>2</sub>, air, or a mixture of these) without the use of any catalyst or activating agent. Meanwhile, chemical activation is achieved by impregnation of the precursor with a chemical. This procedure is followed by heat treatment at moderate temperature (usually ranging from 400-600 °C) in a one-step process with conventional heating or microwave heating.

The removal of carbon atoms and simultaneous production of wide range of pores (predominantly micropores) is the resulting reaction from the gasification process yielding porous AC (Yang *et al.*, 2010). On the other hand, activation *via* chemical process, a one-step activation involves pyrolysis and activation carried out simultaneously

in the presence of steam (Abioye & Ani, 2017). Comparing both methods, it shows that although chemical activation has many advantages such as low activation time, single step activation, low activation temperature, better porous structure and higher yields. Commercial manufacturers normally prefer physical activation process producing ACs. This is because the conventional method is widely practiced in the industry with proven results of its ability to produce ACs with well-developed microporous structure and desirable physical characteristics as well as the simplicity of the process (Yang *et al.*, 2010).

In recent studies, conventional heating method is the mainstream method for pyrolysis processes, in which the energy is produced by convective or conductive heating systems of electrical furnaces (EF) (Yuen & Hameed, 2009). Pyrolysis is a thermal process that heats and decomposes substances performed in an inert environment that can produce three types of products: solid char, liquid oil and incondensable gas (Shiung *et al.*, 2016; Mahari *et al.*, 2017). This common practice is time consuming for the thermal process to reach the desired temperature of activation. Another problem faced when preparing AC through thermal heating using EF is the surface heating of the furnace where uniform heating is difficult for different shapes and sizes of samples. This will create a thermal gradient from the hot surface of the char particle to its interior where inhibit effective removal of gaseous products, which will resulting in the decrease of the quality of the AC produced (Li *et al.*, 2007; Deng *et al.*, 2010).

Apart from the method of activation, various materials have been utilised as the precursor to produce ACs ranging from biomass to even agricultural wastes. Although, it has been widely known to be quite expensive for implementation, a major drawback for operators to be used as treatment for various types of wastewater, there have been extensive research to produce AC through alternative sources such as waste biomass notably agricultural wastes, because of its wide availability, renewability, cheap and require very little processing. Some examples of studies carried out using alternative material are acorn shells (Saka, 2018), coconut shells (Das *et al.*, 2015), banana peels (Keey *et al.*, 2018), orange peels (Xie *et al.*, 2014), rambutan peels (Njoku *et al.*, 2014)

and rice husks (Hegazi, 2013). This alternative route will likely reduce the capital and maintenance cost for implementation in wastewater treatments. Reusing these wastes as precursors may contribute to the reduction of the total composition of wastes thus, reducing the production cost of ACs. This is also an emphasis on the concept of 'cradle to cradle'.

In this paper, a comparative review of the advancement, of the production of AC using microwave technology from various precursors was conducted. The physical and chemical properties of AC and also evaluation of their applications wastewater treatment are also summarised.

## UNDERSTANDING THE CONCEPT OF MICROWAVE HEATING

The process of heating using microwave belongs to the group of electro-heating techniques that utilize specific parts of the electromagnetic spectrum (Metaxas, 1991). The heating of a dielectric material occurs through the conversion of electromagnetic energy into heat within the irradiated material. As stated by Haque (1999), there are several advantages of using the microwave method over conventional heating methods such as (1) non-contact heating; (2) energy transfer as compared to heat transfer in conventional methods; (3) rapid heating; (4) selective material heating; (5) volumetric heating; (6) quick start-up and stop; (7) heating from the interior of the material body; (8) higher levels of safety and automation; and, (9) can be transported from the source through a hollow nonmagnetic metal tube. Given this fact, most industrial applications of microwave heating are based on substances that contain polar molecules such as food processing, sterilization and pasteurization, different drying processes and etc (Menéndez *et al.*, 2010).

The basic concept of the conventional furnace is the production of heat through conduction, convection or radiation which is ineffective due to heat loss to the surroundings and heat transfer resistances causing damage to the reactor walls with continuous electrical heating. To avoid the temperature gradient that occurs slower heating is a proven solution but this results in longer heating

periods causing undesired or secondary reaction and is responsible for the production of low quality products due to further cracking of product. In contrast, microwave heating converts electromagnetic energy into heat energy within the particles by dipole rotation and ionic conduction (Ania *et al.*, 2005; Deng *et al.*, 2010; Salema & Ani, 2011).

The heating patterns comparing conventional and microwave heating is illustrated in Figure 1. The figure clearly demonstrates the heating pattern of a material. Microwave heating heats the material from within, which is not the same with the conventional treatment. The internal temperature of the sample treated with the conventional furnace is lower than at its surface because this heating method transports heat waves from the surface into the interior of the material. This differs in the case of microwave heating where radiation energy is dispersed within the sample more or less uniformly creating a higher heating rate (Zhang & Hayward, 2006). This method may create a significant temperature gradient where the inner side of the material is hotter than its surface. Thus, this proves the advantages of implementing microwave heating.

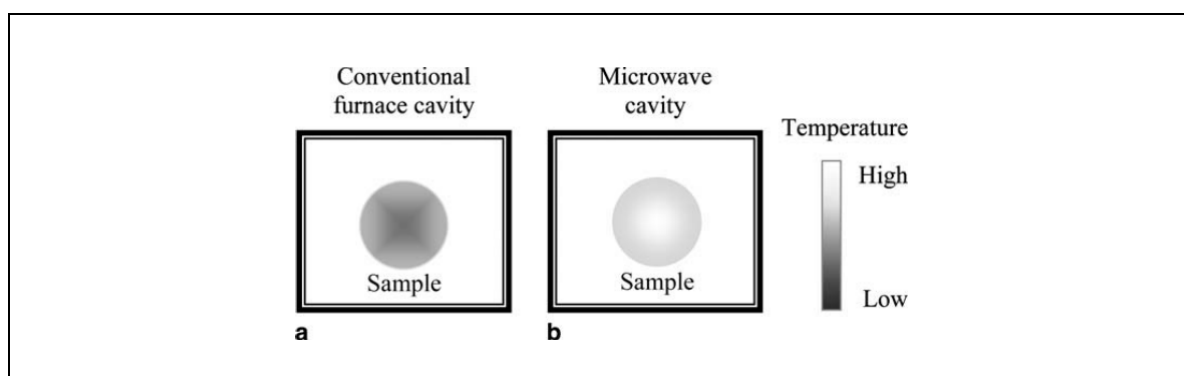
Another factor of microwave heating, the activated carbons present a sharp temperature rise and this high temperature treatment causes the contraction of carbon skeleton and changes in pore structure. However, the contraction of carbon skeleton has the same effects on micropores and mesopores, and the pores of different width are shrinking. So, microwave treatment has little effect on the average pore width. As seen in Table 1 and

2, the BET surface area developed in both physical and chemical treatments *via* microwave heating are similar or even higher compared to AC developed by conventional heating.

## ACTIVATED CARBON PREPARATION VIA MICROWAVE HEATING

Producing AC with excellent surface functionality and well-developed porosity can be prepared by thermal decomposition of carbonaceous materials such as coal, peat, lignite (Allen *et al.*, 1997) and various agricultural by-products in a furnace, conventional heating, or a microwave under controlled heat and atmosphere. As reported by Menéndez *et al.* (2010), carbon materials are generally very good absorbers of microwaves as they are easily heated by microwave radiation. Microwave heating transforms carbon materials, with the addition of their unique characteristics, giving rise to new carbons with upgraded properties.

As mentioned in previous section, conventional heating has long been in practice and remains the preferred method amongst commercial producers despite its disadvantages producing AC. Microwave heating has been reported in many works as a cost effective and efficient method for producing good quality of ACs. In order to produce quality ACs, the main application is significantly dependent on its porosity, includes its surface area, pore volume and internal porous structure. These adsorptive properties depend and may be controlled by the preparation and activation method as well as its raw material/precursor (Maldhure & Ekhe, 2011; Foo & Hameed, 2012).



**Figure 1.** The comparison of heating patterns of the conventional furnace and microwave (Zhang & Hayward, 2006)

**Table 1.** Recent studies for preparation of physical activation of AC *via* microwave

Material	Agent	S <sub>BET</sub> (cm <sup>3</sup> /g)	V <sub>tot</sub> (cm <sup>3</sup> /g)	AC Optimum Conditions for Production		AC Optimum Conditions for Treatment			Reference
				Microwave Power (W)	Irradiation Time (mins)	pH	Contact Time (mins)	Adsorbent Dosage (g)	
Apple waste	Steam	443	0.157	800	10	-	180	-	Ramdane <i>et al.</i> (2016)
	Steam	2079	1.212	900	5 – 7	-	-	-	
Coconut Shell	CO <sub>2</sub>	2288	1.299	900	5 – 7	-	-	-	Yang <i>et al.</i> (2010)
	Steam + CO <sub>2</sub>	2194	1.293	900	5 – 7	-	-	-	
Palm Shell	Steam	570.8	0.262	550	10	-	-	-	Yek <i>et al.</i> (2019)

**Table 2.** Recent studies for preparation of chemical activation of AC *via* microwave

Material	Agent	S <sub>BET</sub> (cm <sup>3</sup> /g)	AC Optimum Conditions for Production			AC Optimum Conditions for Treatment			Reference
			Microwave Power (W)	Irradiation Time (mins)	Impregnation Ratio	pH	Contact time (mins)	Dosage (g)	
Apple peel	H <sub>3</sub> PO <sub>4</sub>	1552	700	10	1.5	-	15	-	Zhang <i>et al.</i> (2014)
Apple pulp	H <sub>3</sub> PO <sub>4</sub>	1103	700	12.5	1.5	-	12.5	-	
Bamboo	H <sub>3</sub> PO <sub>4</sub>	1432	350	20	1.0	-	-	-	Liu <i>et al.</i> (2010)
Banana frond	KOH	847.66	600	4	4.5	8-9; 2-4	*9.33; 6.67	*3.5; 4.0	Foo <i>et al.</i> (2013)
Banana peel	KOH / NaOH	1038	700	10	1.0	6.8	10	0.25	Keey <i>et al.</i> (2018)
	KOH	729.33	680	10	1.6	-	120	0.10	
Cotton stalk	K <sub>2</sub> CO <sub>3</sub>	621.47	720	8	1.6	-	120	0.10	Deng <i>et al.</i> (2010)
	H <sub>3</sub> PO <sub>4</sub>	1370	640	10	0.5	12	210	-	
Date stones	K <sub>2</sub> CO <sub>3</sub>	1144	660	8	1.5	-	270	0.005	Abbas & Ahmed (2016)
Industrial waste lignin	ZnCl <sub>2</sub>	1164	600	4	1.0	6	500	1.0	Maldhure & Ekhe (2011)
Orange peel	KOH / NaOH	1350	550	5	1.0	-	-	3.0	Lam <i>et al.</i> (2017)
Pistachio nut shells	KOH	700.53	600	7	1.75	10-12	-	0.2	Foo & Hameed (2011a)
Peanut hull	H <sub>3</sub> PO <sub>4</sub>	952.60	500.70	9.8	3.33	-	-	0.04	Zhong <i>et al.</i> (2012)
Rambutan peel	KOH	971.54	600	12	1.0	2	2400	0.2	Njoku <i>et al.</i> (2014)
	HNO <sub>3</sub>	1719.32	800	15	1.5	< 5	90	1.0	
Rice husk	KOH	752	600	7	0.93	12	-	0.10	Foo & Hameed (2011b)
	K <sub>2</sub> CO <sub>3</sub>	1165	600	7	0.93	12	-	0.10	
Sugarcane bagasse	KOH	1620.69	600	5	1.25	4;6	60;150	5.0;5.0	Foo <i>et al.</i> (2013)

Ramdane *et al.* (2016) used apple waste to produce ACs by physical activation using microwave heating. In the presence of steam, the optimum conditions for this technique used 800 W of microwave power with the radiation time of 10 min where this yielded 39% of AC with BET surface area of 433 cm<sup>3</sup>/g. The AC was often used to treat cadmium, a heavy metal. The finding for the optimum condition for cadmium removal exhibited with initial ions rapidly adsorbed between 1 and 180 minutes which began to slow down after this period of time. Although the contact time of treatment was increased but, an insignificant amount of adsorption was recorded. This is because the metallic ions initially reach the boundary layer, followed by diffusion into the surface of the adsorbent and finally the ions diffuse into the pores of the adsorbent.

Yek *et al.* (2019) utilised physical activation on palm shell waste to produce ACs at the expense of 500 W for 9.95 min. The produced AC yielded 89%, developed BET surface area of 570.8 and capable of removing 77% of the treated methylene blue dye at optimum absorption conditions of 0.2 g dosage, 60 min of contact time and initial methylene blue concentration of 50 mg/L.

## REMOVAL MECHANISMS BY ADSORPTION

It is a common fact that the adsorption process depends on factors like specific surface area, heterogeneous adsorption sites, steric hindrance from bulk organic molecules, interactions between adsorbent and adsorbate, and significantly its surface charge.

### Dye Industry

Dyes adsorbed by metal oxides are determined by their negatively charged surfaces which are analysed by zeta potential method. ACs that have negative results indicate that the surface is negatively charged at natural pH. The process of adsorption depends on the force of dispersion and the electrostatic force of attraction between the adsorbate and adsorbent. If the surface of the adsorbent is negatively charged at  $\text{pH} > \text{pH}_{\text{zpc}}$ . Thus, it can be concluded that the adsorption of dye on the negatively charged-surface of AC occurs due to electrostatic attraction compared to that of dispersion forces (Anik & Cansizoglu, 2006; Xia *et al.*, 2015). The same mechanism concept can be applied for the removal of heavy metals by

adsorption. The negatively charged surfaces may attract heavy metals such as Cu<sup>2+</sup>, Pb<sup>2+</sup>, Cd<sup>2+</sup> and other positively charged ions onto the AC by adsorption.

Emami and Azizian (2014) utilised phosphoric acid by converting date sphate into AC. The optimum conditions of the AC produced was impregnation ratio of 3.0, microwave power level at 700 W and radiation time of 2 min. The AC was then analysed on its adsorption capacity to remove methyl orange and pore distribution. It was found that the AC was able to remove 98.9% of the targeted dye and it showed type I and II isotherms, indicating a combination of microporous-mesoporous structures of the AC. Another finding was the AC provides heterogeneous surface for adsorption of Me-Org. Another study involving the utilisation of orange peels was carried out by Lam *et al.* (2017). They produced ACs at optimum conditions of 500 W microwave power, radiation time of 5 min and IR of 1.0 yielding 87%, which was then used to treat Malachite Green dye. The removal efficiency of the AC was 56.9%.

### Organic Pollutants

Landfill leachate contains various types of pollutants, a combination of organic and non-organic pollutants. Furthermore, it is becoming more difficult to understand in terms of its adsorption mechanism due to the multi components species present. Ghani *et al.* (2017) concluded that adsorption on surface of AC where the functional groups improve the adsorption process through (1) ion-exchange capability and interactions with hydroxyl groups on the humic substances; (2) attraction of p-p with the molecules possessing more than two double and triple bonds, and polynuclear aromatics would be formed between the adsorbate and the adsorbent; and (3) through the formation complexes where the HS tend to chelate various metal ions present in the leachate and electrostatic interaction occur between negative charge surface and positive charge of the complexes.

### Heavy Metals

Saleem *et al.* (2019) studied the utilising olive stones for the adsorption of heavy metals, the mechanism involved in the process occurs by surface complexation, a widely known mechanism in adsorption systems of biomass. Another study by Fu *et al.* (2019) utilised KOH with rice husk *via* one

and two-step activations to produce ACs with the aim of adsorbing phenol. Their findings showed that it was not controlled by only one mechanism but rather a combination of both chemisorption and physisorption. This is also due to the nature of the phenol, an organic adsorbate, where its molecules can move through to the inner surface *via* liquid-film controlled diffusion, which can be concluded that the predominant behaviour of phenol adsorption onto the activated controlled *via* the chemisorption.

Yang *et al.* (2017) used straw which is abundant in China, to produce ACs *via* microwave-assisted technology. It was found that the optimum conditions for producing AC in this study was by mixing the straw with pyrophosphoric acid with an impregnation ratio of 1:2. It was then activated using microwave heating at a power of 400 W for 8 minutes. Although no BET surface area was reported in the research, they did carry out an adsorption test on its iodine number, 612.78 mg/g. Yao *et al.* (2016) studied the use of rice husk for the production of ACs by activating it using nitric acid with the aim of treating Pb(II) from wastewater.

#### **SUSTAINABLE PRODUCTION AND COST-EFFECTIVENESS OF MICROWAVE-ASSISTED AC**

Table 3 shows the process features and properties of AC produced from microwave-assisted technology with chemical activation compared with various ACs produced by other methods reported in the literature and commercial AC. The features and properties of AC obtained from the optimised parameters were carried out by Lam *et al.* (2017). These evaluations are essential in assessing the technical viability of the microwave-chemical activation approach, especially in scaling and optimising the design for operation to the commercial level.

#### **CONCLUSION**

Adsorption is not a widely implemented method of treating wastewater. Given its hype, attention and vast pollutant removal ability, it may be the core for treatment methods in near future. With that being said, microwave treatment for producing ACs have shown significant and tremendous abilities in terms of time, cost and quality

compared to conventional methods if produced in mass or commercially. Various methods of producing ACs by microwave heating has been discussed, through its optimised conditions and removal capability. However, whether or not adsorption by ACs can be efficient alone, the current implementation of ACs and problems regarding its regeneration process, may still be the question for industry players. The present review shows that alternative materials have equivalent or better adsorption capacity to commercial activated carbon which may reduce the overall cost of production. If the solid waste can be converted into low-cost adsorbents for the treatment of discharged wastewater, the cost of removal might decrease. AC production through microwave-assisted technology are of high quality with many beneficial features compared to conventional produced ACs:

- AC properties produced are dependent on the nature of the precursor, activation method, type of activating agent and its process conditions (temperature, retention time, and IR).
- Through the numerous studies reviewed, the production of ACs at its optimum conditions (microwave radiation power, microwave radiation time and IR) *via* chemical activation with microwave heating were 350–700 W, 5–15 min and 0.5–2, respectively.
- Microwave heating presents significant due to the different mechanisms involved during the heating process: (a) significant reduction in activation time scale which indicates lower energy consumption; (b) microwave heating also reduces the cycle time taken to produce ACs as it eliminates additional heating processes and use of reagents; (c) without doubt, the overall process increases in its effectiveness. This means that microwave heated ACs may be more economically competitive than *via* conventional techniques.

Lastly, a comparison between conventional heating and microwave heating processes *via* chemical activation with the same precursors revealed that impregnation with the microwave-assisted method led to development of higher surface areas than impregnation with conventional heating methods. The alternative adsorbents mentioned previously are found to be highly efficient for the removal of various pollutants from various wastewater industries. In addition to

**Table 3.** Cost of production and comparison between methods of AC production (Lam *et al.*, 2017)

	Activated Carbon (orange peels)	ACs from literature	Commercial ACs
Process Features			
Total activation time	25 mins (and less)	1 – 7 hours	≥ 5 hours
Temperature (°C)	460 – 490	450-900	1000-2000
Activation agent	Mixtures of KOH and NAOH	Single agents (i.e. KOH, NaOH, ZnCl <sub>2</sub> , K <sub>2</sub> CO <sub>3</sub> , CO <sub>2</sub> , steam)	Steam
Properties of Activated Carbon			
BET surface area (m <sup>2</sup> /g)	1350	900 – 1271	826
Total pore volume (cm <sup>3</sup> /g)	0.60	0.56	0.34
Production cost of AC (USD/kg)			
	Microwave assisted AC		0.20
	Zauba.com		1.17 – 2.14
	Alibaba.com		0.62 – 5.63
	MarketsandMarkets.com		1.73 – 2.26

benefiting the industries, the living organisms and surrounding environment are also benefited using microwave heating method. This evidence can conclude that microwave heating method has superior abilities and is the method to sustainably produce quality and economically competitive offer a lot of promising benefits for commercial purposes in the future.

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