

Thermal Treatments and Activation Procedures Used in the Preparation of Activated Carbons

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1. Introduction

The preparation of activated carbons (ACs) generally comprises two steps, the first is the carbonization of a raw material or precursor and the second is the carbon activation. The carbonization consists of a thermal decomposition of raw materials, eliminating non-carbon species and producing a fixed carbon mass with a rudimentary pore structure (very small and closed pores are created during this step). On the other hand, the purpose of activation is to enlarge the diameters of the small pores and to create new pores and it can be carried out by chemical or physical means. During chemical activation, carbonization and activation are accomplished in a single step by carrying out thermal decomposition of the raw material impregnated with certain chemical agents such as H_3PO_4 , H_2SO_4 , HNO_3 , NaOH, KOH and $ZnCl_2$ (Hu et al., 2001; Mohamed et al., 2010). Physical or thermal activation uses an oxidizing gas (CO_2 , steam, air, etc.) for the activation of carbons after carbonization, in the temperature range from 800 to 1100 °C. The carbonization can be carried out using tubular furnaces, reactors, muffle furnace and, more recently, in glass reactor placed in a modified microwave oven (Foo & Hameed, 2011; Tongpoothorn et al., 2011; Vargas et al., 2010).

Nowadays, the raw materials more used in the preparation of carbons are of lignocellulosic origin. Wood and coconut shells are the major precursors and responsible for the world production of more than 300, 000 tons/year of ACs (Mourão et al., 2011). However, the precursor selection depends of their availability, cost and purity, but the manufacturing process and the application of the product are also important considerations (Yavuz et al., 2010). Figure 1 shows the number of publications studied in this chapter, related with the preparation of activated carbons from lignocellulosic materials in last two decades. A clear trend can be observed: the number of works increased in the years from 2000 to 2010. The obtained carbons were mainly employed in the removal of water pollutants.

In the present chapter the principal methods used in the preparation of activated carbons from lignocellulosic materials by chemical and physical procedures are discussed. An analysis of the experimental conditions used in the synthesis of ACs has been made attending to the carbon specific surface area. Also the advantages and disadvantages of each method are discussed.

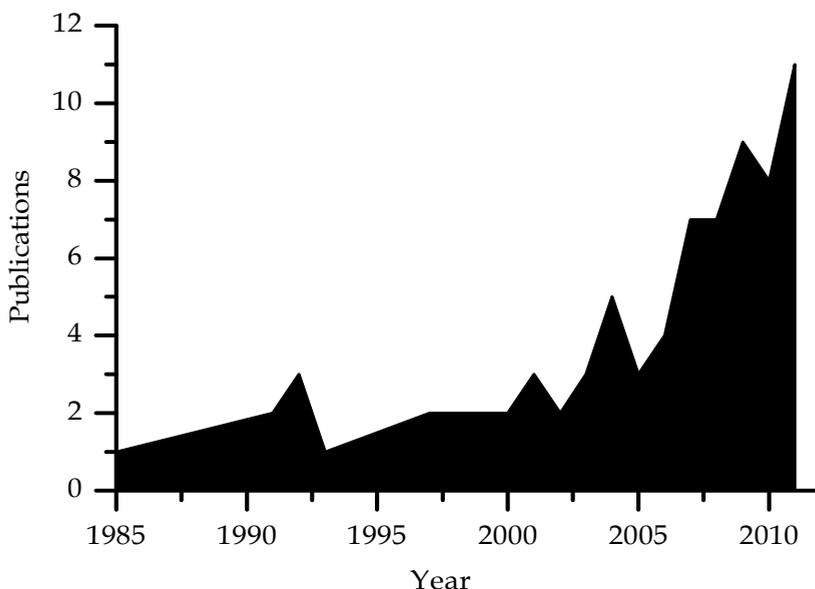


Figure 1. Number of publications related with the preparation of activated carbons from lignocellulosic precursors in the last two decades

2. Preparation of activated carbons

The preparation of ACs from lignocellulosic materials involved two processes, the carbonization and the activation, which can be performed in one or two steps depending on the activation method (physical or chemical, respectively). Specifically, when the carbonization is carried out in an inert atmosphere the process is called *pyrolysis*. According to the literature, the pyrolysis of lignocellulosic materials as coconut shells, olive stones, walnut shells, etc., gives rise to three phases: the char, oils (tars) and gases. The relative amount of each phase is a function of parameters such as temperature of pyrolysis, nitrogen flow rate and heating rate. For example, slow heating rates promote high yields of the carbon residue while flash pyrolysis is recommended for high liquid (oil) ratios (Mohamed et al., 2010).

During the pyrolysis of lignocellulosic precursors, a rudimentary porosity is obtained on the char fraction as a consequence of the release of most of the non-carbon elements such as hydrogen, oxygen and nitrogen in form of gases and tars, leaving a rigid carbon skeleton formed by aromatic structures.

There are two conventional methods for activating carbons: physical (or thermal) and chemical activation. During the chemical activation, the precursor is first impregnated or physically mixed with a chemical compound, generally a dehydrating agent. The impregnated carbon or the mixture is then heated in an inert atmosphere (Moreno-Castilla et al., 2001). On the other hand, during a physical activation process the lignocellulosic

precursor is carbonized under an inert atmosphere, and the resulting carbon is subjected to a partial and controlled gasification at high temperature (Rodríguez-Reinoso & Molina-Sabio, 1992).

In the following sections the principal characteristics of the procedures used in the preparation of activated carbons from lignocellulosic precursors by physical and chemical methods are described.

2.1 Chemical activation

The carbonization step and the activation step simultaneously progress in the chemical activation (Hayashi et al., 2002a). In this case, the lignocellulosic precursor is treated primarily with a chemical agent, such as H_3PO_4 , H_2SO_4 , HNO_3 , $NaOH$, KOH or $ZnCl_2$ by impregnation or physical mixture and the resulting precursor is carbonized at temperatures between 400 and 800 °C under a controlled atmosphere. The function of the dehydrating agents is to inhibit the formation of tar and other undesired products during the carbonization process. Also, the pore size distribution and surface area are determined by the ratio between the mass of the chemical agent and the raw material. Besides, activation time, carbonization temperature and heating rate are important preparation variables for obtaining ACs with specific characteristics (Mohamed et al., 2010). The effects of all these parameters in the textural characteristics of ACs employing different activating agents are discussed in the following sections.

2.1.1 Phosphoric acid (H_3PO_4)

In the last 20 years, the activation of lignocellulosic materials with H_3PO_4 has become an increasingly used method for the large-scale manufacture of ACs because the use of this reagent has some environmental advantages such as ease of recovery, low energy cost and high carbon yield. H_3PO_4 plays two roles during the preparation of ACs: i) H_3PO_4 acts as an acid catalyst to promote bond cleavage, hydrolysis, dehydration and condensation, accompanied by cross-linking reactions between phosphoric acid and biopolymers; ii) H_3PO_4 may function as a template because the volume occupied by phosphoric acid in the interior of the activated precursor is coincident with the micropore volume of the activated carbon obtained (Zuo et al., 2009).

The chemical and physical properties of ACs obtained by chemical activation with H_3PO_4 are affected by the experimental conditions of preparation such as acid concentration, time of activation, impregnation ratio, carbonization temperature and heating rate. Also some recent works have shown that the atmosphere used in the carbonization process has an obvious effect on the physicochemical properties of ACs (Zuo et al., 2009). Table 1 collects some experimental conditions used in the preparation of activated carbons from lignocellulosic materials using N_2 as activation atmosphere.

Precursor	H ₃ PO ₄ (%)	Impregnation ratio	Activation temperature (°C)	Heating rate (°C min ⁻¹)	Reference
Avocado kernel seed	85	6	800	5	Elizalde-González et al. (2007)
China fir	50	4.6	475	5	Zuo et al. (2009)
Coconut Fibers	30	4	900	20	Phan et al. (2006)
Fruit stones	60	1.02	800	-	Puziy et al. (2005)
Jackfruit peel waste	85	4	550	-	Prahas et al. (2008)
Jute	30	4	900	20	Phan et al. (2006)
Licorice residues	89	1.5	400	2.5	Kaghazchi et al. (2010)
Oil palm shell	85	0.09	450	5	Arami-Niya et al. (2011)
Olive Stone	50	2	400	5	Yavuz et al. (2010)
Olive waste	75	2.4	500	10	Moreno-Castilla et al. (2001)
Pecan shell	50	-	450	-	Ahmedna et al. (2000)
Pine Wood	85	1.5	400	-	Hared et al. (2007)
Pistachio-nut shells	89	0.5	400	5	Kaghazchi et al. (2010)
Sea-buckthorn stones	85	0.5	550	10	Mohammadi et al. (2010)
Stem of date palm	85	5	600	10	Jibril et al. (2008)
Tea plant	85	3	350	-	Yagmur et al. (2008)

Table 1. Experimental conditions of activated carbons obtained by chemical activation with H₃PO₄ using different lignocellulosic precursors

In most of the cited papers, the concentration of acid is greater than 50% (w/w) and the activation temperature for 75 % of these studies is between 350 and 600 °C (see Table 1). Figure 2 shows the specific surface area calculated by the Brunauer, Emmett and Teller method (S_{BET}) of the ACs prepared in the contributions collected in Table 1. Carbons obtained with the highest phosphoric impregnation ratio (China Fir and avocado kernel seeds) are the materials with the largest S_{BET} (1785 and 1802 m² g⁻¹). Additionally, the carbon obtained from Oil palm shell and activated using a rather low impregnation ratio (0.09) was one of the materials with a lower specific surface area (356 m² g⁻¹).

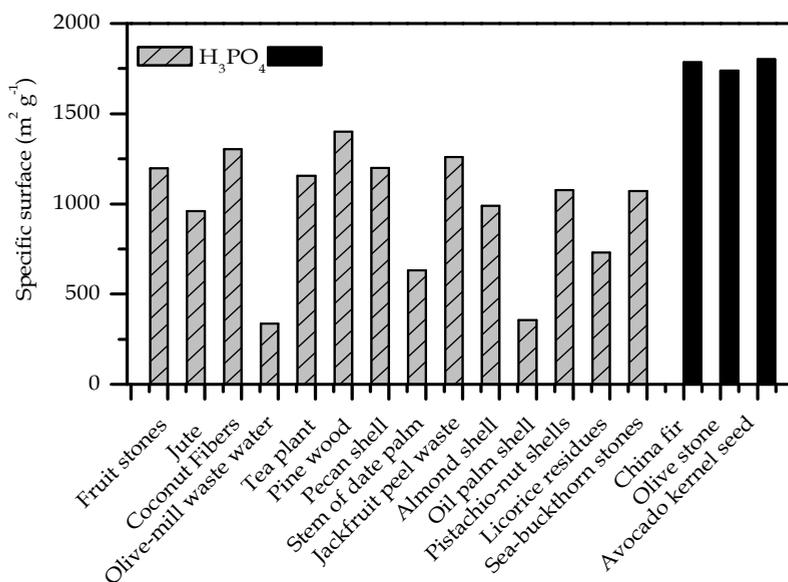


Figure 2. Specific surface area of activated carbons obtained by chemical activation of lignocellulosic materials with H_3PO_4 (black bars: ACs with greater S_{BET})

2.1.2 Zinc Chloride ($ZnCl_2$)

Chemical activation of lignocellulosic materials with $ZnCl_2$ leads to the production of activated carbons with good yield a well-developed porosity in only one step. Impregnation with $ZnCl_2$ first results in degradation of the material and, on carbonization, produces dehydration that results in charring and aromatization of the carbon skeleton and creation of the pore structure (Caturla et al., 1991). In this case, the precursor is impregnated with a concentrated $ZnCl_2$ solution during a given contact time, followed by evaporation of the solution and, finally, the precursor is carbonized in an inert atmosphere and thoroughly washed to extract the excess of $ZnCl_2$. The amount of $ZnCl_2$ incorporated in the precursor and the temperature of heat treatment are the two variables with a direct incidence in the development of the porosity. Table 2 shows the experimental conditions used in the preparation of ACs by chemical activation with $ZnCl_2$ using N_2 as activation atmosphere.

The specific surface areas of the carbons reported in the papers of Table 2 are shown in Figure 3. Carbons obtained using the highest impregnation ratios (2 and 2.5) and an activation temperature of 800 °C are the materials with the largest S_{BET} (Caturla et al., 1991; Hu et al., 2001). The carbon obtained from coconut shells reaches an S_{BET} value of 2400 m² g⁻¹, whereas for the carbon prepared from peach stones the S_{BET} was 2000 m² g⁻¹. Other carbons prepared from coconut shells using an impregnation ratio of 1 and an activation temperature of 500 °C show lower specific surface areas (1200 m² g⁻¹). In any case, all the carbons prepared by chemical activation with $ZnCl_2$ attain S_{BET} greater than 750 m² g⁻¹ (Azevedo et al., 2007). The principal disadvantage of this activation is the environmental risks related to zinc compounds.

Precursor	Impregnation ratio (IR)	Activation temperature (°C)	Heating rate (°C min ⁻¹)	Reference
Almond shells	2	600	-	Torregrosa & Martín (1991)
Cherry stones	3	500	10	Olivares-Marín et al. (2006)
Coconut shells	1	500	4	Azevedo et al. (2007)
Coconut shells*	2	800	10	Hu et al. (2001)
Coffee residue	1	600	10	Boudrahem et al. (2009)
Coir Pith	1	700	-	Namasivayam & Kadirvelu (1997)
Hypnea valentiae	-	800	10	Aravindhan et al. (2009)
Licorice residues	1	500	2.5	Kaghazchi et al. (2010)
Oil palm shell	-	500	5	Arami-Niya et al. (2011)
Oil palm shells	-	500	10	Arami-Niya et al. (2010)
Peach stones	2.5	800	-	Caturla et al. (1991)
Pistachio-nut shells	1.5	500	5	Kaghazchi et al. (2010)
Sargassum longifolium	-	800	10	Aravindhan et al. (2009)
Sea-buckthorn stones	0.5	550	-	Mohammadi et al. (2010)
Walnut shells	2	450	5	Yang & Qiu (2010)

Table 2. Experimental conditions of activated carbons obtained by chemical activation with ZnCl₂ using different lignocellulosic precursors

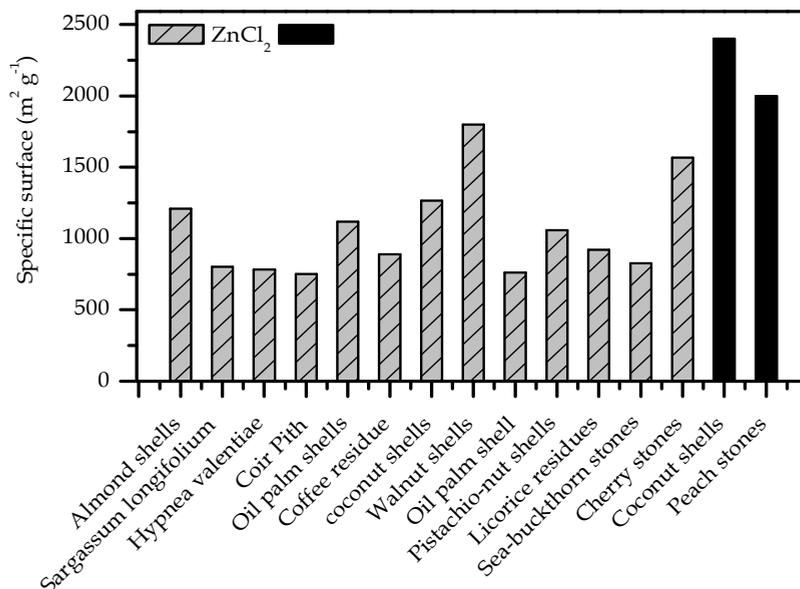


Figure 3. Specific surface area of activated carbons obtained by chemical activation of lignocellulosic materials with ZnCl₂ (black bars: ACs with greater S_{BET})

2.1.3 Alkalis

Alkaline hydroxides (KOH, NaOH) and carbonates (K₂CO₃, Na₂CO₃) have been used as activation reagents in the preparation of activated carbons with high specific surface. In general terms, chemical activation by KOH and NaOH consists in a solid-solid or solid-liquid reaction involving the hydroxide reduction and carbon oxidation to generate porosity (Adinata et al., 2007). The activation with KOH was first reported in the late 1970s by AMOCO Corporation; since then many studies have been devoted to the preparation of ACs by chemical activation with KOH (Lua & Yang, 2004). In this context, two procedures have been used. The carbon precursor can be mixed with powder of KOH or impregnated with a concentrated solution of KOH and then the solid mixture or impregnated precursor is thermally treated under nitrogen (Bagheri & Abedi, 2009; Moreno-Castilla et al., 2001). Alternatively, the preparation of ACs by alkaline activation is made in two steps, in which the precursor is first pyrolyzed and the obtained carbon is activated with a solution of KOH (Bagheri & Abedi, 2009) or with pellets of KOH and finally thermally treated again. The activation step can be conducted in a glass reactor placed in a modified micro wave oven with a frequency of 2.45 GHz (Foo & Hameed, 2011).

Sodium hydroxide has been also shown to be more interesting activation agent due to the possibility of reducing chemical activation costs and environmental load when compared with KOH activation (Tongpoothorn et al., 2011). The activation procedure with NaOH is similar to KOH (Tseng, 2007; Vargas et al., 2011).

In general, the preparation of ACs by chemical activation with KOH and NaOH allows to obtain carbons with high specific surface areas ($>1000 \text{ m}^2 \text{ g}^{-1}$). However, KOH and NaOH are corrosive and deleterious chemicals (Hayashi et al., 2002a). For this reason, recent studies have proposed the preparation of activated carbons by chemical activation with K_2CO_3 in one step, in which the lignocellulosic materials is impregnated with a K_2CO_3 solution and finally the impregnated precursor is thermally treated. K_2CO_3 is a not deleterious reagent and it is broadly used for food additives (Hayashi et al., 2002a).

Table 3 summarizes the experimental conditions used in the preparation of ACs from lignocellulosic materials by chemical activation with NaOH, KOH and K_2CO_3 . Carbons obtained by activation with NaOH are the materials showing higher S_{BET} (see Figure 4), for example, the carbon obtained from flamboyant exhibiting a S_{BET} near to $2500 \text{ m}^2 \text{ g}^{-1}$. Also, the activation with K_2CO_3 renders carbons with a competitive S_{BET} (between 1200 and $1800 \text{ m}^2 \text{ g}^{-1}$) compared with those obtained by activation with KOH or NaOH.

Other interesting observation is that the specific surface areas of two ACs obtained from pistachio nut shells activated with KOH and treated in two different thermal configurations (a conventional electric oven and a modified microwave oven), were very similar (700 and $796 \text{ m}^2 \text{ g}^{-1}$), thus suggesting that the two methods (conventional and non-conventional) are effective for the preparation of ACs.

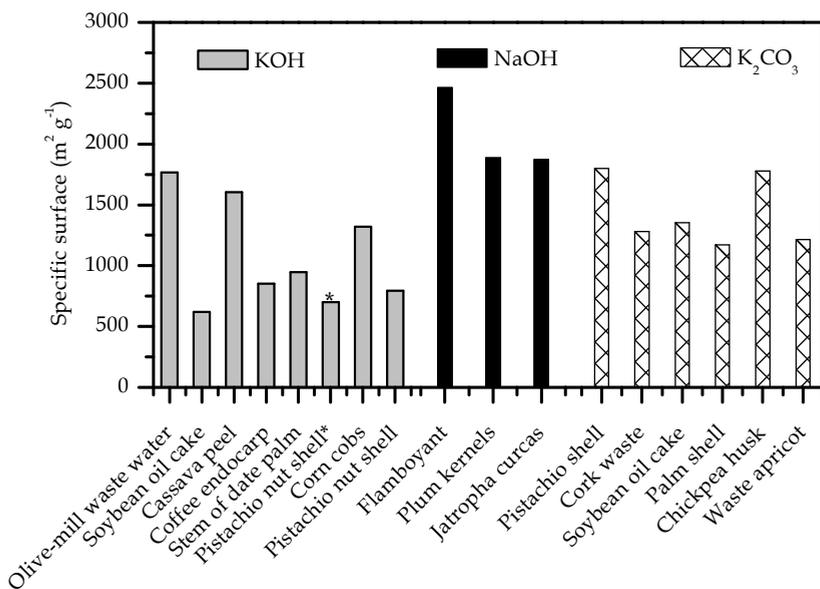


Figure 4. Specific surface area of activated carbons obtained by chemical activation of lignocellulosic materials with KOH, NaOH and K_2CO_3

Precursor	Activating state	IR	Carbonization temperature (°C)	Heating rate (°C min ⁻¹)	Reference
KOH					
Cassava peel	-	2.5	750	10	Sudaryanto et al. (2006)
Coffee endocarp	Powder	1:2	850	5	Nabais et al. (2008)
Corn cobs	Solution	2	550	10	Bagheri & Abedi (2009)
Olive-mill waste water	Solution	2	800	10	Moreno-Castilla et al. (2001)
Pistachio nut shell	Pellets	0.5	300	10	Lua & Yang (2004)
Pistachio nut shell*	Solution	1.75	Frequency of 2.45 GHz.	600 W	Foo & Hameed (2011)
Soybean oil cake	Solution	0.95	800	5	Tay et al. (2009)
Stem of date palm	Solution	3	600	50	Jibril et al. (20089)
NaOH					
Flamboyant	Pellets	3	700	20	Vargas et al. (2011)
Jatropha curcas	Solution	4	400	-	Tongpoorthorn et al. (2011)
Plum kernels	Solution	4	780	-	Tseng (2007)
K₂CO₃					
Chickpea husk	Solution	-	800	10	Hayashi et al. (2002b)
Cork waste	Solution	3	800	10	Carvalho et al. (2004)
Palm shell	Solution	2	800	10	Adinata et al. (2007)
Pistachio shell	Solution	-	800	10	Hayashi et al. (2002a)
Soybean oil cake	Solution	1	800	5	Tay et al. (2009)
Waste apricot	Solution	1	900	10	Erdoğan et al. (2005)

Table 3. Experimental conditions of activated carbons obtained by chemical activation with NaOH and KOH using different lignocellulosic precursors

2.2 Physical or thermal activation

In a physical activation process, the lignocellulosic precursor is carbonized under an inert atmosphere, and the resulting carbon is subjected to a partial and controlled gasification at high temperature with steam, carbon dioxide, air or a mixture of these (Rodriguez-Reinoso & Molina-Sabio, 1992). Steam and CO₂ are the two activating gases more used in the physical activation of carbons. According to the literature, steam or CO₂ react with the carbon structures to produce CO, CO₂, H₂ or CH₄. The degree of activation is normally referred to as "burn-off" and it is defined as the weight difference between the carbon and the activated carbon divided by the weight of the original carbon on dry basis according with the following equation,

$$\text{Burn off} = \frac{W_0 - W_1}{W_0} \times 100\% \quad (1)$$

where W_0 is the weight of the original carbon and W_1 refers to the mass of the activated carbon. The use of CO₂ during the activation process of a carbon material develops narrow micropores, while steam widens the initial micropores of the carbon. At high degrees of burn-off, steam generates activated carbons with larger meso and macropore volumes than those prepared by CO₂. Consequently, CO₂ creates activated carbons with larger micropore volumes and narrower micropore size distributions than those activated by steam (Mohamed et al., 2010)

Tables 4 and 5 show the experimental conditions used in the preparation of activated carbons from lignocellulosic materials by physical activation with CO₂, steam and steam-N₂ admixtures. Normally, in these experiments the lignocellulosic precursor is carbonized in an inert atmosphere (N₂) at temperatures ranging from 400 to 950 °C to produce carbons with rudimentary pore structures. These carbons are then activated with the selected gasification agent at temperatures around 800-1000 °C to produce the final activated carbons.

Some additional studies combine the thermal or physical activation with chemical activation (also known as physicochemical activation, Table 6). Normally, physicochemical activation is performed by changing the activation atmosphere of the chemical activation by a gasification atmosphere (i.e., steam) at higher temperatures. In other cases, the chemical activation is carried out directly under the presence of a gasifying agent. The combination of both types of carbon activation renders ACs with textural and chemical properties which are different from those obtained by any of the activations alone. For example, steam reduces the occurrence of heteroatoms into the carbon structures. Also, combination of oxidizing reagents in the liquid phase (i.e., nitric or sulfuric acids) with gasification agents improves the development of porosity on the final carbons.

Figure 5 shows the specific surface area of activated carbons obtained by physical and physicochemical activation according with the experimental conditions cited in Tables 4, 5 and 6. In general, the ACs obtained by physical activation with CO₂ show a higher specific surface area than those obtained by activation with steam. Additionally, the ACs obtained by physical activation with CO₂ using high heating rates (20 °C min⁻¹) are the adsorbents showing lower S_{BET} (Corn-cob, Bagasse bottom ash and Sawdust fly ash).

Precursor	Carbonization Conditions			Activation conditions			Flow (cm ³ min ⁻¹)	Reference
	Temperature (°C)	Heating rate (°C min ⁻¹)	Atm.	Temperature (°C)	Heating rate (°C min ⁻¹)	Atm.		
Almond shell	400	10	N ₂	800	10	CO ₂	85	Mourão et al. (2011)
Almond shell	400	10	N ₂	800	10	CO ₂	85	Nabais et al. (2011)
Bagasse bottom ash	500	20	N ₂	800	20	CO ₂	200	Aworn et al. (2008)
Coconut Fibers	950	-	N ₂	950	-	CO ₂	250	Phan et al. (20069)
Coffee endocarp	700	5	N ₂	800	5	CO ₂	85	Nabais et al. (2008)
Corn cob	500	20	N ₂	800	20	CO ₂	200	Aworn et al. (2008)
Eucalyptus kraft lignin	350	10	N ₂	800	10	CO ₂	150	Rodriguez-Mirasol et al. (19939)
Jute	950	-	N ₂	950	-	CO ₂	250	Phan et al. (2006)
Macadamia nut-shell	500	20	N ₂	800	20	CO ₂	200	Aworn et al. (2008)
Oil-palm-shell	600	10	N ₂	900	10	CO ₂	-	Lua et al. (2006)
Olive-mill waste	350	-	Air	850	-	CO ₂	300	Moreno-Castilla et al. (2001)
Pistachio-nut shells	500	10	N ₂	900	10	CO ₂	100	Lua et al. (2004)
Pistachio-nut shells	500	10	N ₂	800	10	CO ₂	100	Yang & Lua (2003)
Sawdust fly ash	500	20	N ₂	800	20	CO ₂	200	Aworn et al. (2008)
Vine shoots	400	10	N ₂	800	10	CO ₂	85	Mourão et al. (2011)

Table 4. Experimental conditions of activated carbons obtained from different lignocellulosic precursors by physical activation with CO₂

Precursor	Carbonization Conditions			Activation conditions		
	Temperature (°C)	Heating rate (°C min ⁻¹)	Atm.	Temperature (°C)	Heating rate (°C min ⁻¹)	Atm.
Almond shell	600	-	N ₂	850	-	Steam -N ₂
Almond tree pruning	600	-	N ₂	850	-	Steam -N ₂
Date stones	700	10	N ₂	700	10	Steam
M. oleiferu seed	-	-	-	800	20	Steam
Olive stone	600	-	N ₂	850	-	Steam -N ₂
Walnut shell	600	-	N ₂	850	-	Steam -N ₂

Table 5. Experimental conditions of activated carbons obtained from various lignocellulosic precursors by

Precursor	Activating agent	Temperature (°C)	Heating rate (°C min ⁻¹)	Atm.	Flow (cm ³ min ⁻¹)
Date stones	H ₃ PO ₄	600	-	Steam	-
Date stones	HNO ₃	600	-	Steam	-
Olive stones	CaCl ₂	800	-	CO ₂	100
Sugarcane bagasse	H ₂ SO ₄	160	10	Air	2000

Table 6. Experimental conditions of activated carbons obtained from different lignocellulosic precursors

Disadvantages

- ✓ Corrosiveness of the process
- ✓ Requires a washing stage
- ✓ Inorganic impurities
- ✓ More expensive

3.2 Physical method

Advantages

- ✓ Avoids the incorporation of impurities coming from the activating agent
- ✓ The process is not corrosive
- ✓ A washing stage is not required
- ✓ Cheaper

Disadvantages

- ✓ The activated carbons are obtained in two steps
- ✓ Higher temperatures of activation (800-1000 °C)
- ✓ Poorer control of the porosity

4. Conclusions

Attending to the works considered in this chapter, chemical activation is the most used method for the preparation of ACs (~60 %) from lignocellulosic precursors. Physical activation methods is used in 28% of the studies and a low quantity of studies combine both methods (i.e., physicochemical) to produce ACs. H_3PO_4 and $ZnCl_2$ are the two more employed activating agents in the impregnation of lignocellulosic materials (30% and 24 %, respectively), whereas alkaline reagents such as KOH, NaOH and K_2CO_3 have been considered because ACs with high specific surface can be obtained (1500-2500 $m^2 g^{-1}$). Physical activation of lignocellulosic precursors normally renders carbons with lower specific surface area. However, when compared with chemical activation, this method is not corrosive and does not require a washing step.

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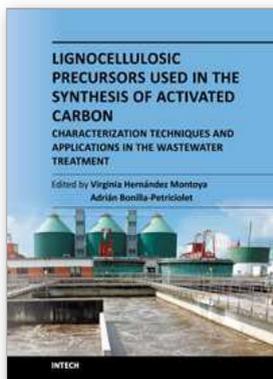
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Lignocellulosic Precursors Used in the Synthesis of Activated Carbon - Characterization Techniques and Applications in the Wastewater Treatment

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The present book discusses the principal lignocellulosic precursors used in the elaboration of activated carbons in different countries such as Asia, America, Europe and Africa; the different methods and experimental conditions employed in the synthesis of activated carbons, including one analysis of the principal stages of the preparation such as carbonization and activation (i.e., chemical or physical activation). Also, the recent and more specialized techniques used in the characterization of activated carbons are discussed in this book. For example, the techniques employed to determine textural parameters (mercury porosimetry and gas adsorption isotherms at 77 K) and different spectroscopies to determine chemical functionality (Raman, FT-IR, etc.) and other X-Ray techniques. Additionally, an overview of the application of activated carbons obtained from lignocellulosic precursors for wastewater treatment. Specifically, the analysis and discussion are focused on the advantages and capabilities of activated carbons for the removal of relevant toxic compounds and pollutants from water such as heavy metals, dyes, phenol, etc. Finally, the use of pyrolysis method for the valorization of two Mexican typical agricultural wastes (orange peel and pecan nut shell) for energy and carbon production is considered in this book.

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