

**PERFORMANCE OF ACTIVATED CARBON OBTAINED
FROM PINE WOOD AND DETERMINATION OF ITS
ADSORPTION CAPACITIES OF AMMONIA AND
GASOLINE VAPORS**

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This article is dedicated to Güneş Uçar on the occasion of his retirement as professor at the University of Istanbul

ABSTRACT

Two types of pine wood were used to produce activated carbon (AC) by chemical activation with phosphoric acid. The yield of the activated carbon (CAs) obtained by considering the following factors in the process: species, activation temperature and impregnation ratio ($R = \text{wood mass/dissolution mass}$), the variables of such factors being the species *Pinus*

pseudostrobus (Pp) and *Pinus leiophylla* (Pl), temperature values 400 and 500°C, and values of R (1:1, 1:2, 1:3), respectively, thus having 12 combinations.

The results indicate that the best performance was in the treatment with Pp 400°C and with R = 1:1. A surface area of 790 and 801 m²·g⁻¹ and a total pore volume of 0.312 and 0.316 cm³·g⁻¹ were obtained for the Pp 500-2 and Pl 500-2 carbons, respectively. Likewise, the adsorption capacity of ammonia and gasoline vapors of the carbon obtained was determined.

KEYWORDS: Activated carbon, chemical activation, yield, adsorption.

INTRODUCTION

The waste generated in the process of forest supply is excessive, among these remains are the branches, which generally remain in the forest, and can be used as a raw material for the production of activated carbon, which is a material widely used as adsorbent in its diverse applications due to its large surface area and porosity (Cukierman et al. 2000). There are two methods of obtaining activated charcoal: chemical activation and physical activation. Chemical activation involves contacting the precursor material with the activating agent prior to carbonization (Molina-Sabio et al. 1995, Toles et al. 1996). For chemical activation there are different activating agents among which are H₃PO₄, ZnCl₂, H₂SO₄, KOH, NaOH, H₃BO₃, (Márquez-Montesino et al. 2013) being the H₃PO₄ the most used due to its ease to be removed by washing with warm water (John Kennedy et al. 2004). In chemical activation, the activating agents cause carbonization of the precursor by dehydration, depolymerization, redistribution of biopolymers, and cross-linking of lignocellulosic materials (Jagtoyen and Derbyshire 1998). The properties of the chemically activated carbon are conditioned to different aspects such as the nature of the precursor, the activating agent and other variables such as impregnation ratio, residence time, activation temperature and heating ramps (Laine et al. 1989, Caturla et al. 1991, Ioannidou and Zabaniotou 2007).

The use of activated charcoal with H₃PO₄ as the precursor material for pine and other woods has been carried out previously (Jagtoyen and Derbyshire 1998, García-Lovera et al. 2008). However, the use of wood of branches to obtain CA has not been reported, this being of great importance since the content of cellulose, hemicellulose and lignin between the wood of branches and of the stem is not the same, on the other hand *Pinus pseudostrobus* and *P. leiophylla* are the most exploited species in the state of Michoacán, Mexico and thus generate large amounts of forest residues of these species. In the present work, from the two species mentioned, CA is produced by chemical activation with a solution of H₃PO₄. Furthermore, we study the influence of some variables of the activation process on the yield of the coals and their properties.

MATERIALS AND METHODS

Raw material and immediate analysis

The wood was obtained from the tree branches of *Pinus pseudostrobus* and *P. leiophylla* of the forest exploitation of the Indigenous Community of Nuevo San Juan Parangaricutiro, of Michoacan state, Mexico. The branches were cut into slices and allowed to air dry for two weeks, then the material was separated into wood and bark, chipped, ground and sieved. The study used wood flour with a 20-mesh particle size. An immediate physical chemical analysis was performed on the samples and the moisture content (ASTM 2004), ash content (ASTM 2007), percentage of volatiles (Prabir 2010) and percentage of fixed carbon (Cordero et al. 2001) was determined.

Preparation of activated carbon

For the preparation of the coals H_3PO_4 was used with 85% purity as the activating agent and a 30% aqueous solution was prepared. For the impregnation, four (R) ratios were applied: 1:1, 1:2 and 1:3. In each treatment the precursor material was impregnated with the activating agent, leaving it to stand for 24 hours in a glass desiccator. Carbonization of the samples was carried out in a Barnstead Thermolyne Model 21100 tubular oven, with a heating rate of $10^\circ C \cdot min^{-1}$ and an inert atmosphere of N_2 . Activation temperatures (maximum temperatures) were 400 and $500^\circ C$ and the residence time was 2 hours. The activated carbon was washed with hot distilled water to remove the acid, to a neutral pH. They were then left in an oven at $100^\circ C$ for 12 hours for drying.

Surface area and pore volume

The surface area of activated carbons with $R = 1:2$ and uncooked carbons obtained at $500^\circ C$ of *P. leiophylla* and *P. pseudostrobus* were determined, for which the BET method was used. For this purpose, a Micromeritics ASAP 2020 volumetric adsorption analyzer was used using nitrogen adsorption at 77 K temperature. Prior to the adsorption process, the samples were degassed for 24 hours at $150^\circ C$. The total pore volume (V_T) was estimated from the amount of N_2 adsorbed at the highest relative pressure. The volume of the micropores (V_{micro}) was calculated from the N_2 adsorption isotherms using the Dubinin-Radushkevich (D-R) equation (Gregg and Sing 1982, Dubinin and Kadlec 1987, Dubinin 1989).

$$W = W_0 \exp[-(A / \beta E_0)^2] \quad (1)$$

where: W - the volume of liquid adsorbate in the micropores at temperature (T) and relative pressure (p/p_0).
 W_0 - the volume of micropores or adsorption limit volume in $cm^3 \cdot g^{-1}$ or amount of substance ($mmol \cdot g^{-1}$).
 β - coefficient of similarity (for $N_2 = 0.35$).
 A - differential molar work or adsorption potential ($RT \ln 1/(p/p_0)$) ($mmol \cdot g^{-1}$)
 E_0 - characteristic energy (attraction energy exerted by the pore walls on the adsorbed particle)

The BET model was applied to the nitrogen adsorption isotherm to evaluate the surface area following the procedure detailed in Sing et al. (1985), Rouquerol et al. (1994) Masnadi et al. (2014).

The total pore volume (V_t) was estimated from the maximum adsorption in the N_2 adsorption isotherm, that is, from the amount of nitrogen adsorbed at relative pressure (p/p_0) to 0.98, the volume of micropores (V_{mic}) was calculated by the method t, by the following equation:

$$V_{mic} = Y (D) \quad (2)$$

where: Y - intercept,
 D - liquid adsorbate conversion factor ($N_2 = 0.0015468$).

The amount of nitrogen adsorbed at relative pressures near the unit corresponds to the total amount adsorbed by micropores and mesopores. Therefore, the volume of mesopores (V_{meso}) is obtained by subtracting the volume of micropores (V_{micro}) from the total adsorbed amount (V_T) (Molina-Sabio et al. 1995).

The average pore radius (r_m) was calculated from the following relation (Blanco Castro et al. 2000, El-Hendawy et al. 2001):

$$r_m = 2V_t / A_{BET} \quad (3)$$

Adsorption of gasoline and ammonia vapors

For this measurement, 0.1 g of each activated carbon in clock glass was placed in a glass desiccator, as well as a beaker containing 10 mL of ammonia for 24 hours. Subsequently the samples were weighed and the adsorbed ammonia was determined by difference between the mass of the sample of CA without adsorbed ammonia and after being in contact with the solution of ammonia. Then the same procedure was performed but using Pemex Premium Gasoline instead of ammonia.

RESULTS AND DISCUSSION

Immediate analysis of Wood

The results of the immediate chemical analysis for the flour of the two woods are shown in Tab. 1. The values obtained for caloric power are in accordance with those reported in the literature (Kieseler et al. 2013).

Tab. 1: Immediate chemical analysis of wood.

Species	Moisture content (%)	Volatile (%)	Ash (%)	Fixed carbon (%)
<i>Pinus pseudostrobus</i>	7.6	91.37	0.15	8.48
<i>Pinus leiophylla</i>	7.7	91.60	0.16	8.24

Performance of activated carbons

The yield obtained from the CAs is shown in Fig. 1 where a remarkable difference between the yield of activated carbons with $R = 1:1$ compared to the values of $R = 1:3$ is observed. The presence of H_3PO_4 accelerates the separation of oxygen and hydrogen at temperatures as low as $75^\circ C$ (Solum et al. 1995). It was observed that *P. pseudostrobus* had higher yield in general than *P. leiophylla*, this may be due to a higher lignin content in *P. pseudostrobus* (Pintor-Ibarra et al. 2017).

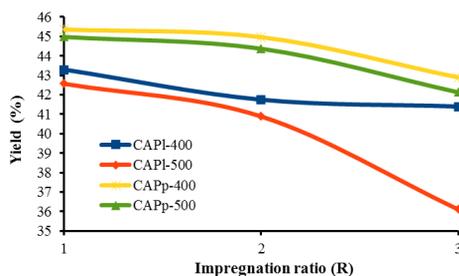


Fig. 1: Effect of R and comparison of the yields of the obtained carbons. For practical interpretation purposes the following is considered: CAPI = activated carbon of *Pinus leiophylla*, CAPp = activated carbon of *Pinus pseudostrobus*, 400 and 500 = activation temperature.

There is a tendency to decrease the yield of the CAs as R increases. Likewise, it was shown that at a higher temperature the yield is lower. Due to deeper dehydration of the precursor structure and increase in the porous structure during the activation process, the yield values of the activated carbons obtained decrease with the impregnation ratio and the activation temperature (Jagtøyen and Derbyshire 1998). It should be noted that the lower yields of the activated carbon CAPI with respect to the CAPP can be directly related by the lower fixed carbon, the higher volatile matter and ash, the latter acting as catalysts of the process cracking the carbon and conducting to the formation of liquids and gases.

Characterization of the activated carbons obtained

The types of physisorption isotherms for activated carbons CAPP500-2 and CAPI500-2 are shown in Fig. 2. Due to a very similar development of the porous structure of these two carbons, the isotherms are almost equal, classifying within group I in the IUPAC classification (Sing et al. 1985).

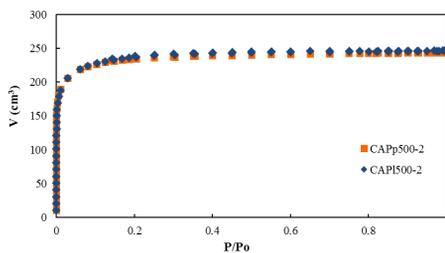


Fig. 2: Adsorption isotherm of activated carbons.

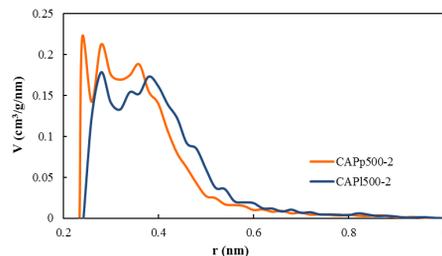


Fig. 3: MP-method. Distribution of micropore volume.

Fig. 3 shows a greater distribution of the volume of micropores in the CAPP500-2 towards larger sizes, which corresponds to the representations of Fig. 4 (distribution of the volume of mesopores) where it is shown that it is the carbon with the greatest amount of mesopores at low pore size values.

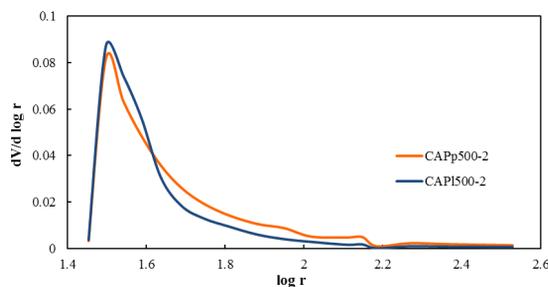


Fig. 4: Distribution of mesopores volume.

Figs. 5 and 6 shows a linearity for both coals, which implies greater compliance with the DR equation as a result of the micropores being filled from the smallest micropores to the largest micropores, from lowest to highest relative pressure, until the total filling to $P/P_o \approx 1$. The greater deviation of the linearity is the result of the adsorption in pores larger than the micropores and

the filling in multilayers, similar results are reported by Molina-Sabio et al. (1995), El-Hendawy et al. (2001) and Molina-Sabio et al. (2004).

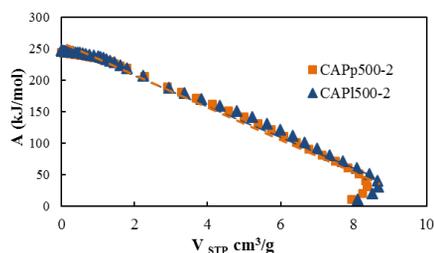


Fig. 5: Dubinin characteristic curve.

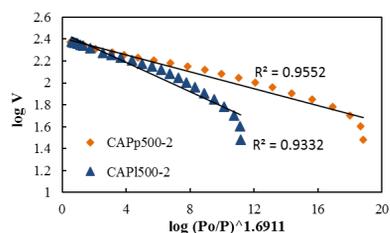


Fig. 6: Adsorption isotherms in Dubinin coordinates.

Nitrogen adsorption presents linear deviations at low values of relative pressures, due to diffusion problems of the adsorbate in narrow pores and deviations at high relative pressures, due to capillary condensation in the wide micropores and narrow mesopores.

The difference in BET surface area, micropores volume, mesopores, macropores and average pore radius compared to carbons activated at 500°C and with an impregnation ratio of 1:2 is shown in Tab. 2, where notes that the activated carbon of *P. leiophylla* showed greater development of surface area and total pore volume.

Tab. 2: BET surface area, pore volume and average radius of the activated carbons of *P. pseudosrobis* and *P. leiophylla* obtained at 500° C and with an impregnation ratio of 1:2.

Samples	HTT (°C)	R	ABET (m ² ·g ⁻¹)	V _t (cm ³ ·g ⁻¹)	V _{mic.} (cm ³ ·g ⁻¹)	V _{mes.} (cm ³ ·g ⁻¹)	rm (nm)	E _o (kJ·mol ⁻¹)
CAPp-500-2	500	2	790	0.37	0.349	0.021	0.48	3.55
CAPI-500-2	500	2	801	0.39	0.354	0.036	0.62	2.78

With the increase of the activation temperature, the BET surface area decreases and therefore the adsorption of N₂ at low relative pressure as a result of the combustion of the micropore walls, which give rise to the formation of larger pores, such as mesopores. These results can explain the behavior of adsorption of ammonia and gasoline vapors as shown below.

It can be concluded that the two species of wood with the same conditions of temperature and impregnation ratio during the activation process, led to activated carbons with similar volume values of micropores but different values of volumes of mesopores which can lead to a process of capillary condensation of ammonia and gasoline molecules in large micropores and mesopores.

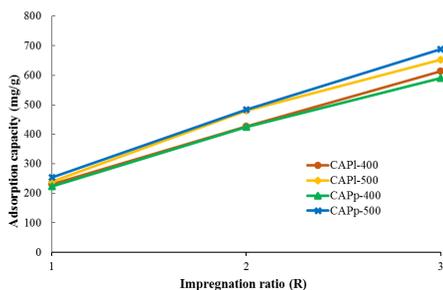
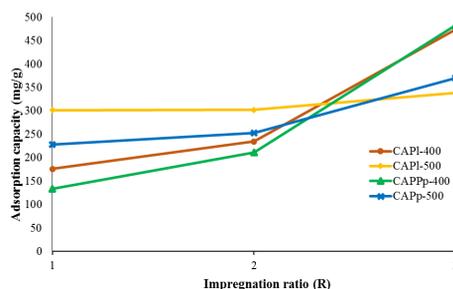
By increasing the volume of mesopores with the increase of the BET surface, a decrease in the characteristic energy is observed which is related to an increase in the size of the pores (decrease of the micropores) giving way to the increase of wider pores, that is to say, of the mesopores.

Adsorption of gasoline and ammonia vapors

Tab. 3 shows the adsorptive capacity per gram of the activated carbons obtained. With the increase of R, the tendency to increase the ammonia adsorption capacity of the obtained carbon is observed, because with the increase of the phosphoric acid the dehydration and phosphorolization of the cellulose is increased, increasing the reactions with the acid, increasing the porous surface area and the adsorption capacity (Diao et al. 2002, Romero-Anaya et al. 2011).

Tab. 3: Adsorption capacities of the coals for ammonia and gasoline vapors.

Carbon	Ammonia ($\text{mg}\cdot\text{g}^{-1}$)	Gasoline ($\text{mg}\cdot\text{g}^{-1}$)	Carbon	Ammonia ($\text{mg}\cdot\text{g}^{-1}$)	Gasoline ($\text{mg}\cdot\text{g}^{-1}$)
CAPi-400-1	231.35	176.45	CAPp-400-1	224.06	133.25
CAPi-400-2	425.90	234.65	CAPp-400-2	423.84	211.75
CAPi-400-3	613.18	473.48	CAPp-400-3	590.83	482.34
CAPi-500-1	240.11	301.72	CAPp-500-1	253.12	228.34
CAPi-500-2	479.34	302.31	CAPp-500-2	483.35	252.86
CAPi-500-3	652.21	338.23	CAPp-500-3	687.67	370.13

Fig. 7: Influence of the impregnation ratio (R) on the adsorption capacity of ammonia.Fig. 8: Influence of the impregnation ratio (R) on the adsorption capacity of gasoline vapors.

The increase in the functional groups on the surface of the coal could be related to the increase in the percentages of ammonia adsorption (Márquez-Montesino et al. 2013). The above is shown more clearly in Figs. 7 and 8.

CONCLUSIONS

With the coals obtained at 400°C and with $R = 1:1$ the highest yield was achieved. The CAPp500-2 and CAPi500-2 carbons did not develop a broadly mesoporous structure, which makes them carbon selective for molecules smaller than 2 nm. Possibly if we want to have a greater surface development, it may be convenient to use higher temperature and R values. The above is confirmed by the study of adsorption of ammonia and gasoline vapors, where an increase in adsorption is observed with the increase in the value of R . In this same sense, the CAs prepared at 500°C showed greater adsorption, both of ammonia as of gasoline vapors, than the CAs obtained at 400°C .

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