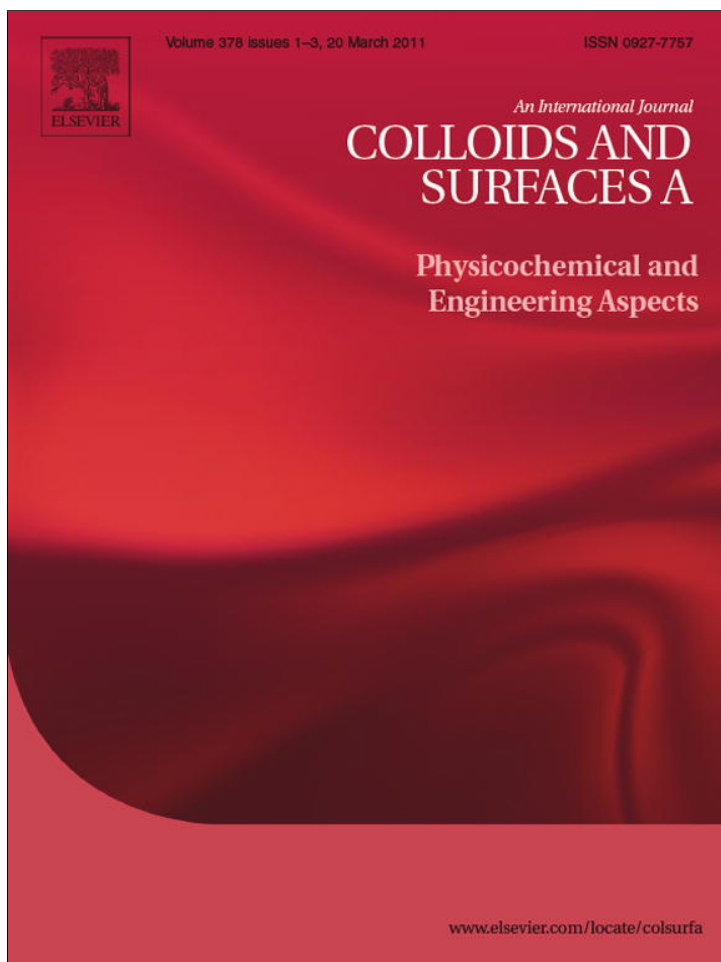


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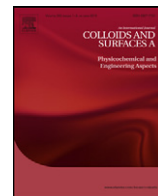
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Effect of the activating agent on physico-chemical and electrical properties of activated carbon cloths developed from a novel cellulosic precursor

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ABSTRACT

Different chemical reagents (phosphoric acid, boric acid, ammonium citrate, sodium dihydrogen phosphate, potassium dihydrogen phosphate, disodium hydrogen phosphate and trisodium phosphate) were employed to develop activated carbon cloths (ACC) by chemical activation of a lyocell precursor, in an attempt to explore their effect on main physico-chemical characteristics and electrical behaviour of the resulting ACC. The activating agent markedly influenced yield, elemental composition, and textural properties of the ACC. The ACC obtained with phosphoric and boric acids were essentially microporous, whereas those developed with the other reagents presented mesoporosity development. Phosphoric acid-derived samples showed the highest specific surface area (976 m²/g). The results also highlight the relevance of correcting the external surface adsorption in order to obtain reliable estimates of micropore volume. All the ACC were electrically conductive, their resistivity being also strongly dependent on the nature of the activating agent. The electrical resistivity of the ACC obtained with all the phosphorous compounds was successfully correlated with their C/H ratio and micropore volume.

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1. Introduction

In recent years, interest in activated carbon cloths (ACC) has significantly increased due to the growing number of new applications, as molecular sieves, catalysts or electrodes. ACC present technological advantages over more traditional powder or granular forms of activated carbons, including high adsorption capacity, uniform porosity as well as high rates of adsorption/desorption from the gas or liquid phase [1–3]. Besides, ACC are flexible and can be arranged in different stable configurations, and their contiguous nature is suited for electrical and electrochemical applications [4–7].

The versatility of ACC and the possibility of confining fibres in various physical forms have enabled the development of several

applications in different fields, such as electrodes, and in environmental and catalytic applications [8–25]. However, the cost of ACC has limited their utilization [26,27]. Therefore, ACC with more particular functions are essential for their practical development. Both pore structure and surface chemistry are key characters governing the properties of ACC [7,28]. These characteristics can often be tailored by a judicious selection of a precursor and a careful control of the experimental conditions of preparation of the ACC. Specifically for chemical activation, by selecting the activating agent it is possible to obtain adsorbents with a defined porous structure and to introduce different types of micro/mesoporosity [2,3,29].

Moreover, only a few studies have been devoted to examining the effect of physico-chemical characteristics of ACC on their electrical properties on a macroscopic scale [4]. They are relevant in order to extend applications of activated carbons and also in the context of industrial air treatment processes for *in situ* regeneration of the cloths saturated with organic vapours by Joule effect heating. As mentioned above, the properties of ACC are strongly dependent on the operating conditions of the manufacturing processes and on the intrinsic characteristics of the precursor used. Porous structure and surface chemistry modify electron mobility, leading to changes in ACC resistivity [4,6,30–33]. Particularly, for

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chemical activation, the choice of an impregnating agent has to be made with regard to the desired ACC electrical behaviour. To the best of our knowledge, this aspect has been almost unexplored, at least in the open literature.

Within this context, the present work investigates comparatively physico-chemical and electrical properties of ACC obtained from lyocell in the form of woven cloth, by chemical activation with different chemical reagents at pre-established experimental conditions. The influence of chemical reagent used in the impregnation stage on porosity development, textural characteristics, morphology, and electrical behaviour of ACC were investigated.

2. Experimental methods

2.1. Preparation of activated carbon cloths

A lyocell-based fabric, kindly provided by Santista Textile Group (Buenos Aires, Argentina), was used as precursor for the preparation of the ACC [34].

Lyocell strips previously weighed were soaked separately in solutions of 5 wt% concentration of different chemical reagents (phosphoric acid, boric acid, ammonium citrate, sodium dihydrogen phosphate, potassium dihydrogen phosphate, disodium hydrogen phosphate and trisodium phosphate, analytical grade) at 333 K overnight. The solution pH for each compound was 1.2, 3.6, 4.9, 4.1, 4.2, 9.3, and 12.4, respectively. Then, they were dried and weighed. The impregnated samples were thermally treated in a tubular stainless steel reactor of horizontal configuration externally heated by an electric furnace under a N₂ flow (100 mL/min). The activated carbon cloths were heated at 5 K/min up to 1137 K. Once the final temperature was attained, it was held for 1 h. The thermal program included an isothermal step at the initial cellulose decomposition temperature in order to improve mechanical resistance of the ACC. Afterwards, the resulting cloths were cooled under N₂ flow and rinsed thoroughly with distilled hot water until neutral pH. The products were then dried until constant weight. Yields were calculated from weight differences. For the sake of comparison, carbonized lyocell was prepared under the same experimental conditions without impregnation. Details of the experimental procedure have been reported earlier for ACC using other precursors [6,30,34].

2.2. Physico-chemical characterization of the activated carbon cloths

Porosity development of the ACC was evaluated from the adsorption isotherms of nitrogen at 77 K determined on a CE Instruments Sorptomatic 1990. The ACC were outgassed for 5 h at 423 K under vacuum prior to testing. The pressure sensitivity was 0.25% of reading. The BET model was employed to evaluate the apparent specific surface area from N₂ isotherms and total pore volumes (v_T) were estimated from the amount of N₂ adsorbed at the highest relative pressure, near unity. Nitrogen isotherms were also analyzed by the α_5 method using published standard data [35] in order to obtain the external surface area (A_S) and micropore volume (v_S). The Dubinin–Radushkevich (DR) equation was applied to calculate the DR micropore volume (v_0), characteristic energy (E_0), and the mean pore width (L_0). The density of N₂ was taken as 0.808 g cm⁻³ and the affinity coefficient as $\beta=0.34$. L_0 was estimated from the following relationship [36]:

$$L_0 = \frac{10.8}{E_0 - 11.4} \quad (1)$$

Surface morphology of the raw precursor and derived ACC were examined by scanning electron microscopy (SEM) with a Philips XL-30 microscope. Before the measurements, all the samples were

Table 1

Yield and elemental composition of the ACC obtained from the lyocell precursor by employing different activating agents.

Activating agent	Y (%)	C (%)	H (%)	N (%)	S (%)	O ^a (%)
Boric acid	27.6	85.7	0.7	0.1	0.1	13.4
Phosphoric acid	28.6	85.0	0.7	0.2	0.1	14.0
Ammonium citrate	23.3	82.2	0.6	0.8	0.2	16.1
Sodium dihydrogen phosphate	26.1	88.8	0.8	0.1	0.3	10.0
Potassium dihydrogen phosphate	21.0	89.5	0.7	0.2	0.3	9.1
Disodium hydrogen phosphate	16.1	76.1	1.0	0.1	0.4	22.4
Trisodium phosphate	9.1	66.2	1.1	0.1	0.2	32.4

^a Calculated by difference.

attached to mounting stubs, dried under vacuum, and sputter coated with Au–Pd [37].

Elemental carbon, hydrogen, sulfur and nitrogen content of the ACC were determined using a Carlo Erba EA 1108 elemental analyzer. The results reported are average values of triplicate determinations, where the standard error did not exceed 5%.

2.3. Electrical resistivity of the ACC

Macroscopic measurements of the electrical resistance of the ACC developed with different chemical reagents were carried out by applying the procedure previously described [6,30,33]. Electrical behaviour of the ACC developed with trisodium phosphate is not included because this sample was brittle.

Briefly, rectangular ACC samples were assembled between two copper electrodes. A standard DC method in a four probe configuration was employed. Measurements were carried out in ambient air and in the warp direction. The temperature at the centre of the samples' surface was measured with a copper-constantan thermocouple. ACC samples were dried during 48 h and the electrical measurements were performed. Different voltages were applied and the current and temperature were recorded. The electrical resistance of the ACC samples was calculated by Ohm's law. The electrical resistivity (ρ) was evaluated accounting for the dimensions of the ACC samples. To evaluate the electrical resistivity parameter (ρ_0) and thermal coefficient (α_0) of the ACC, a decreasing linear dependence of the resistivity on the temperature was applied to fit the experimental data (Eq. (2)):

$$\frac{\rho}{\rho_0} = 1 + \alpha_0(T - T_0) \quad (2)$$

where ρ is the electrical resistivity (Ωm) at the temperature of the activated carbon cloth surface (T), ρ_0 , the electrical resistivity (Ωm) at the reference temperature T_0 (273 K), and α_0 , the thermal coefficient (K^{-1}).

3. Results and discussion

3.1. Effect of the activating reagent on yield, elemental composition and textural properties of ACC

Table 1 shows yield (Y) and elemental composition for the ACC obtained with the different activating agents. The results in the table indicate that yield and elemental composition of the ACC were strongly dependent on the activating agent used at the impregnation stage. Phosphoric and boric acid-derived samples presented the highest yields, whereas the ACC developed with trisodium phosphate showed the lowest value. All the ACC, except those developed with trisodium phosphate, showed higher yield than the carbonized lyocell (12%) obtained at the same temperature without impregnation. The highest carbon content was obtained for the samples impregnated with sodium and potassium dihydrogen phosphate, whereas the ACC obtained with ammonium

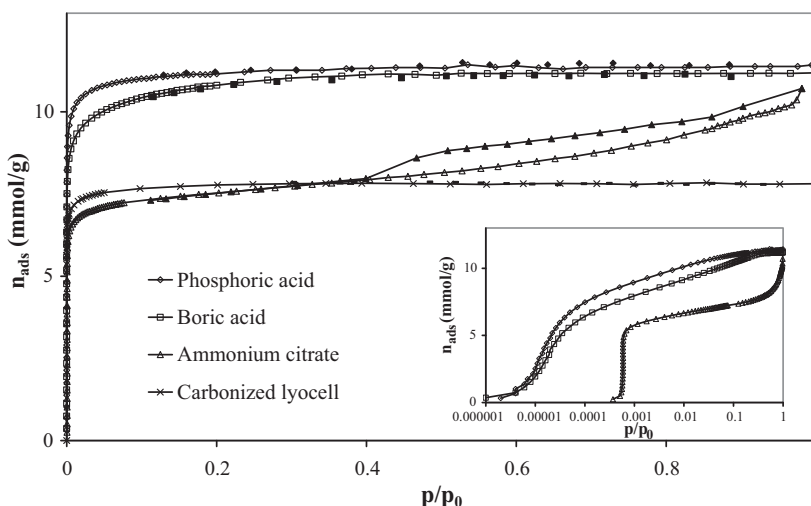


Fig. 1. N_2 adsorption/desorption isotherms at 77 K for the activated carbon cloths obtained using 5 wt% concentration solution of phosphoric acid, boric acid and ammonium citrate. Open points are the adsorption branch and closed points, the desorption branch of the isotherms.

citrate showed the highest nitrogen content. Moreover, hydrogen content increased and yield decreased as the phosphorous compound employed for impregnation was more basic. This could be attributed to a more intense reaction between the precursor and the activating agent during impregnation, leading to hydrolysis of the cellulose.

N_2 adsorption–desorption isotherms for the ACC obtained with the different activating agents and the carbonized lyocell are illustrated in Figs. 1 and 2. The insert in each figure shows the same isotherms represented on a semi-logarithmic scale, and allows examination of the adsorption behaviour of the ACC at very low pressures.

Variation in chemical reagent induced noticeable changes in the uptake of N_2 and in the shape of the isotherm. The ACC obtained with phosphoric and boric acids, as well as for the carbonized lyocell, presented Type-I isotherms, pointing to essentially microporous adsorbents. The isotherms were also reversible. However, the isotherm corresponding to the boric acid-derived ACC showed a more rounded knee, indicating an increase in the mean micropore width. The isotherms determined on the ACC obtained with the other phosphorous reagents (Fig. 2) and with ammonium citrate

(Fig. 1) presented Type I character at relative pressures below 0.4. It also could be seen hysteresis loops at higher pressures pointing to mesoporosity development, in turn depending on the activating agent. As inferred from the comparison of the ACC isotherms with the shape of the isotherm of the carbonized lyocell, the mesoporosity of most of the samples might be due to the impregnation with the different chemical reagents. The Type H4 hysteresis found for the ammonium citrate-derived sample is generally associated with adsorption/desorption in slit-shaped pores, while the Type H2 hysteresis observed for the sodium and potassium dihydrogen phosphate samples is related to the presence of irregular mesoporosity [38]. The isotherms determined on the disodium hydrogen phosphate and trisodium phosphate samples are much steeper at high pressures, do not appear to reach a well-defined plateau and the adsorption, at least on the latter, is also significantly greater. These differences indicate increasing contribution of wider mesopores as the basicity of the impregnating agent increased. The inserts in Figs. 1 and 2 show that only the ACC obtained with phosphoric and boric acids adsorbed an important amount of nitrogen at $p/p_0 < 10^{-4}$. It could be attributed to the presence of very narrow micropores.

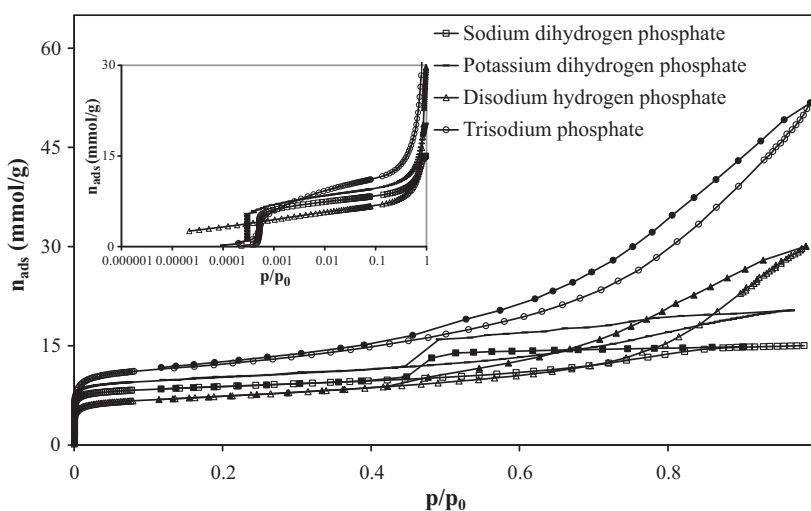


Fig. 2. N_2 adsorption/desorption isotherms at 77 K for the activated carbon cloths obtained using 5 wt% concentration solution of sodium dihydrogen phosphate, potassium dihydrogen phosphate, disodium hydrogen phosphate and trisodium phosphate. Open points are the adsorption branch and closed points, the desorption branch of the isotherms.

Table 2
Textural characteristics obtained by application of the BET, α_S and DR methods to N_2 (77 K) adsorption isotherms determined on ACC.

Activating agent	ν_T (cm ³ /g)	S_{BET} (m ² /g)	A_S (m ² /g)	ν_S (cm ³ /g)	ν_0 (cm ³ /g)	E_0 (kJ/mol)	L_0 (nm)
Boric acid	0.38	925	6	0.38	0.37	25.5	0.76
Phosphoric acid	0.39	976	6	0.39	0.39	27.8	0.66
Ammonium citrate	0.37	518	137	0.20	0.26	25.2	0.78
Sodium dihydrogen phosphate	0.52	634	214	0.22	0.30	22.6	0.96
Potassium dihydrogen phosphate	0.71	739	250	0.24	0.34	21.9	1.03
Disodium hydrogen phosphate	1.04	550	242	0.15	0.24	19.9	1.28
Trisodium phosphate	1.79	922	391	0.25	0.42	16.1	2.29

For all the samples, the DR plots corresponding to the isotherms were found to give good linearity over the relative pressure range between 10^{-4} and 10^{-2} ($R^2 > 0.99$, standard deviation ~ 0.001). Besides, experimental data were also fitted by using the Dubinin–Astakhov (DA) [38] equation. The results showed that DA equation led to similar values of micropore volume and characteristic energy to those calculated by using the DR equation. The α_S plots corresponding to the ACC developed with phosphoric and boric acids were linear for relative pressures above about 0.3, characteristic of microporous adsorbents. The ACC obtained with the other activating agents exhibited a linear region in the range of relative pressures between 0.04 and 0.07 ($R^2 > 0.996$, standard deviation ~ 0.002). The textural parameters obtained from the nitrogen isotherms by application of the BET, DR and α_S methods are summarized in Table 2. Some of the textural parameters and the C/H ratio are illustrated in Fig. 3, in order to facilitate their comparison.

The results listed in Table 2 indicate that the surface properties of the ACC were strongly dependent on the activating agent employed. Phosphoric acid-derived samples presented the highest apparent specific surface area (976 m²/g), whereas the ACC obtained with ammonium citrate showed the lowest A_{BET} value (518 m²/g). Samples prepared with boric and phosphoric acids presented similar ν_T , ν_S and ν_0 values and low A_S values, indicating strictly microporous samples. The ACC synthesized with the other activating agents had ν_T values significantly greater than the corresponding ν_S and ν_0 values due to the development of mesoporosity. This can also be inferred from the high L_0 and A_S values (Table 2). It can be observed from Fig. 3 that the ν_S and ν_0 values presented the same trend for the ACC developed with the different activating agents. Besides, for the ACC obtained with the phosphorous compounds, the pronounced increase in total pore volume is accompanied by a decrease in C/H ratio. It probably may be attributed to intensified reaction of cellulose in more basic media, leading to larger release of volatile matter and lower carbon content

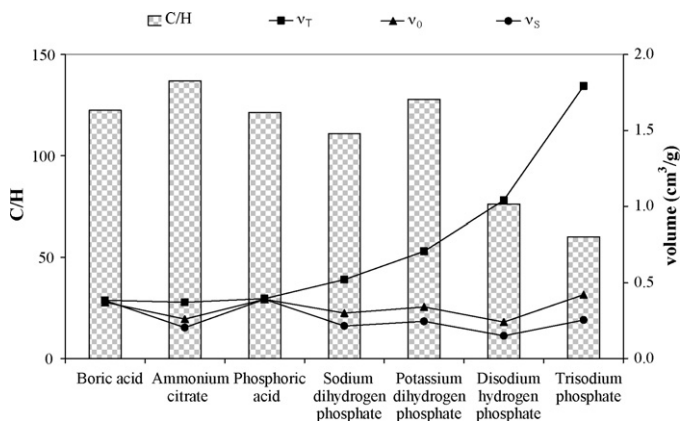


Fig. 3. Total pore volume, micropore volume calculated by means of the DR equation and α_S models and C/H ratio of the ACC developed from lyocell with different activating agents.

during carbonization and further washing steps. Accordingly, as the basic character of the phosphate reagents increases the interaction of the phosphate ions with the cellulose also increases, favouring an increment in total pore volume and promoting the development of mesopores.

Micropore volumes calculated by the α_S method were considerably lower than the values calculated by the DR equation for some ACC with mesopore development. This behaviour has also been observed for carbon aerogels [39] and it may be due to reversible capillary condensation that leads to overestimation of the external surface area and underestimation of the micropore volume ν_S . In turn, high A_S values of these ACC produce an overestimation of ν_0 and L_0 calculated by the DR equation. As shown in Fig. 4, a linear relationship ($R^2 > 0.99$) between differences in micropore values and external surface areas evaluated by the α_S method was found.

3.2. Effect of the impregnating agent on morphological characteristics of ACC

Fig. 5(a–c) shows comparatively SEM images of the surface of the ACC obtained with boric and phosphoric acids and disodium hydrogen phosphate (magnification 6000 \times). As can be observed in Fig. 5a and b, the surface of the boric acid-derived ACC was slightly less smooth than the surface of the ACC synthesized with phosphoric acid. Nevertheless, no hole or pore was visualized even at 6000 \times magnification for these samples. On the other hand, the ACC obtained with disodium hydrogen phosphate presented cavities on the surface corresponding to the development of meso- and macropores, in accordance with the shape of the nitrogen adsorption isotherms. The large development of meso- and macropores may be due to the spaces left by the compounds and chemical remaining after carbonization, which are eliminated during the subsequent washing [29].

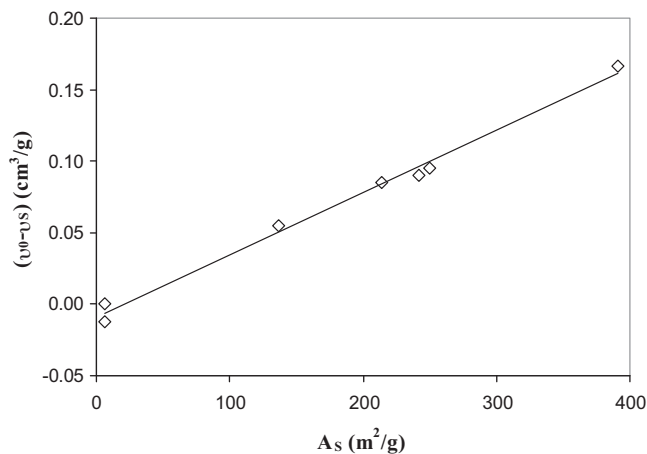


Fig. 4. Relationship between differences in micropore volumes and external surface areas evaluated by the α_S method for the ACC.

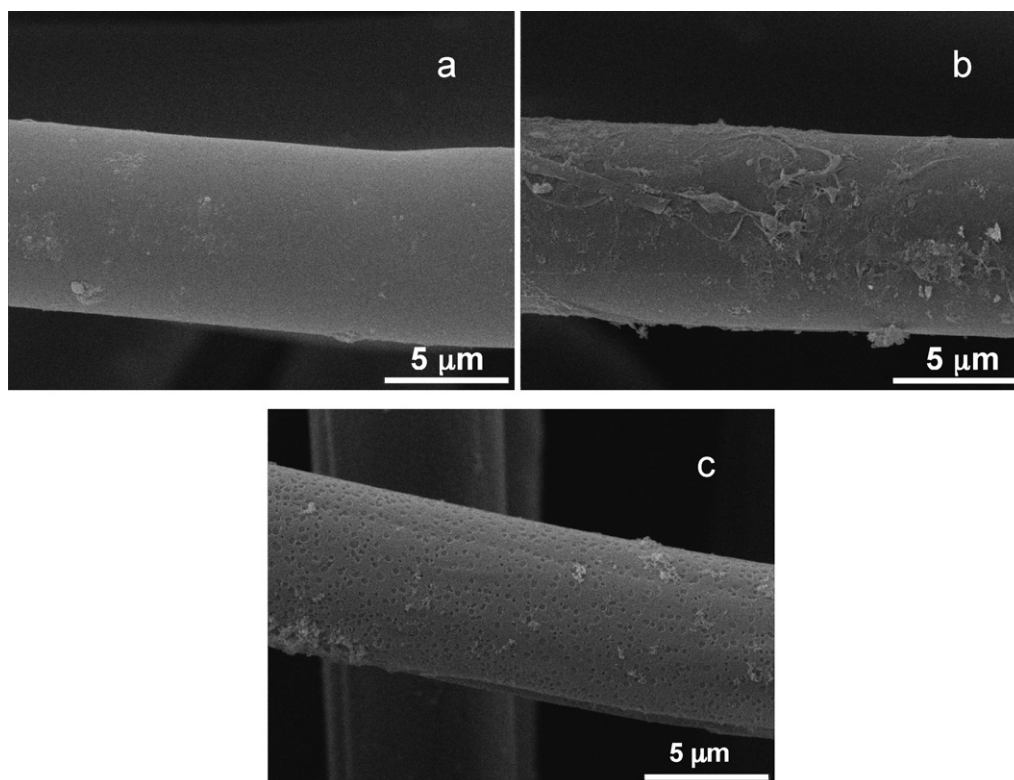


Fig. 5. Representative SEM micrographs of the ACC developed with (a) phosphoric acid, (b) boric acid and (c) disodium hydrogen phosphate. Magnification 6000×.

3.3. Influence of the activating agent on electrical behaviour of ACC

Fig. 6 shows values of the electrical resistivity (ρ) as a function of the central temperature measured at the surface of the ACC obtained with the different activating agents. The characteristic parameters, ρ_0 and α_0 , as obtained by linear regression analysis with high correlation coefficients ($R^2 \geq 0.99$), are reported in Table 3. All the examined ACC developed under the different experimental conditions were electrically conductive, following an ohmic behaviour.

The results in Fig. 6 show a linear decrease in the electrical resistivity with increasing temperature, which indicates that the ACC developed were semiconductor materials. Moreover, all the samples were heated by the Joule effect, reaching surface temper-

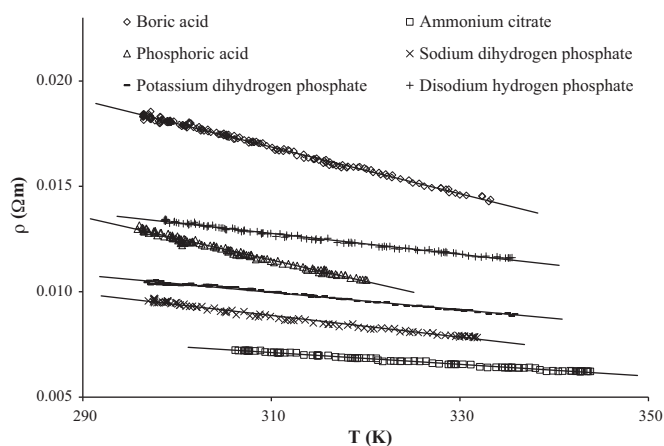


Fig. 6. Electrical resistivity as a function of the surface central temperature for the ACC obtained by using different activating agents.

atures up to 350 K. Accordingly, the ACC saturated with organic volatile solvents could be regenerated *in situ* and the compounds could be recovered. The results presented in Fig. 6 and Table 3 also indicate that the electrical properties of the ACC strongly depend on the inherent nature of the activating agent used as well as on the physico-chemical characteristics of the resulting ACC. The ACC developed with boric acid showed the lowest electrical conductivity, whereas the samples obtained with ammonium citrate presented the highest value. The thermal coefficients and resistivity values listed in Table 3 were within the range of those reported in previous works for commercial ACC [4,33].

Comparison of resistivity values (Table 3) for the developed ACC indicated that their electrical properties seem to depend on physico-chemical characteristics in a complex way. As discussed for other ACC samples [4,6,30], an increase in porosity could cause reduction of the real cross-section, leading to an increase of the electrical resistivity. However, porosity development might not be the only aspect that influenced the electrical behaviour. For instance, the ACC obtained with ammonium citrate and disodium hydrogen phosphate presented similar A_{BET} values, even though the corresponding resistivity values were different (Tables 2 and 3). Besides this, the activating agent markedly affected the pore size distribution of the developed ACC (Figs. 1 and 2). Larger pores could cause discontinuous jumps in the electrical flow leading to

Table 3
Electrical resistivity (ρ_0) and thermal coefficient (α_0) of the ACC obtained with different activating agents.

Activating agent	$\alpha_0 \times 10^3$ (K ⁻¹)	$\rho_0 \times 10^3$ (Ω m)	C/H
Boric acid	-5.3	21.0	122.4
Phosphoric acid	-6.3	16.4	121.4
Ammonium citrate	-3.4	8.1	138.0
Sodium dihydrogen phosphate	-5.1	10.9	111.0
Potassium dihydrogen phosphate	-3.6	11.5	127.8
Disodium hydrogen phosphate	-3.4	14.6	76.1

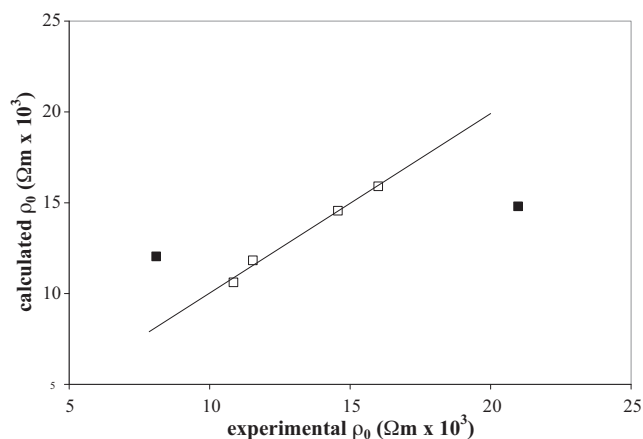


Fig. 7. Relationship between the calculated and experimental ρ_0 values of the developed ACC.

an increase in ACC resistivity. However, the ACC obtained with sodium dihydrogen phosphate and with disodium hydrogen phosphate presented similar ρ_0 although the latter showed higher L_0 and v_T values than those obtained with sodium dihydrogen phosphate (Tables 2 and 3). The electrical behaviour of the ACC might also depend on micropore volume, although the relationship is not straightforward. Fibre structure is related to structure aromatization and C/H ratio and it was another factor which influenced the resistivity value of the samples. Nevertheless, it can be seen from Table 3 that the ACC obtained with boric and phosphoric acids showed similar C/H ratios but different ρ_0 values.

According to the present results, electrical resistivity values of the ACC developed by using most of the activating agents were properly fitted to the following empirical equation [42]:

$$\rho_0 = 12.3 + 74.8v_0 - 0.21 \frac{C}{H} \quad (3)$$

where ρ_0 is the electrical resistivity ($\Omega\text{m} \times 10^3$) at the reference temperature T_0 (273 K), v_0 , the total micropore volume (cm^3/g), and C/H, the ratio of elemental carbon and hydrogen contents of the developed ACC. Fig. 7 shows a linear correlation between the experimental and calculated ρ_0 values of the ACC obtained with phosphorous compounds ($R^2 = 0.97$). Experimental and calculated ρ_0 values of the ACC developed with boric acid and ammonium citrate (black squares) were included in Fig. 7 for the sake of comparison.

As can be observed in Fig. 7, ρ_0 values of the ACC obtained by impregnating the lyocell precursor with the different phosphorous compounds were satisfactorily represented by Eq. (3). On the other hand, ρ_0 values corresponding to ACC synthesized by using boric acid and ammonium citrate could not be estimated by this equation. Differences in the electrical behaviour of the ACC could be attributed to modifications in surface chemistry and/or carbon substitution by heteroatoms in the pseudographitic structure which might cause changes in electron mobility, introduction or removal of electron traps, or changes in the conductivity activation energy [4,40,41].

4. Conclusions

The effect of different activating agents on yield, elemental composition, textural-morphological and electrical properties of lyocell-based ACC was comparatively examined. Results pointed out that the chemical reagent used at the impregnation stage markedly influenced the main characteristics of the ACC. Boric and phosphoric acids led to essentially microporous adsorbents, whereas ACC obtained with ammonium citrate and

the other phosphorous compounds presented development of meso/macroporosity. Moreover, the results highlight the relevance of correcting the external surface adsorption in order to obtain reliable estimates of micropore volume. The electrical behaviour was also significantly affected by the selected activating agent. The electrical resistivity of the ACC synthesized with all the phosphorous compounds was successfully correlated with their C/H ratio and total micropore volume. Overall, present results indicate that by a proper selection of the activating agent, properties of the ACC derived from this novel precursor may be conveniently tailored depending on the desired application.

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