

Materials science communication

## Influence of thermal treatment conditions on porosity development and mechanical properties of activated carbon cloths from a novel nanofibre-made fabric

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## ARTICLE INFO

## Article history:

Received 18 July 2008

Received in revised form 17 December 2008

Accepted 19 March 2009

## Keywords:

Microporous materials

Adsorption

Microstructure

Surface properties

## ABSTRACT

Activated carbon cloths (ACC) were prepared from Tencel<sup>®</sup>, a novel regenerated cellulose nanofibre fabric, by phosphoric acid activation in an inert atmosphere using different thermal treatment conditions. The effect of the final thermal treatment temperature (663–963 °C) and temperature programme (temperature of isothermal step and heating rate) on yield, elemental composition, porosity development and mechanical strength of the resulting ACC were examined. Nitrogen isotherms at –196 °C, carbon dioxide isotherms at 0 °C, breaking loads and Youngs modulus values were determined for all the samples. Adsorption data were fitted to different models (BET, Dubinin–Radushkevich,  $\alpha_S$ ) to evaluate textural parameters of the ACC. The samples were essentially microporous and presented good physical appearance. Higher treatment temperatures resulted in higher BET surface areas and total pore volumes, also leading to larger micropore widths. The ACC prepared at 963 °C showed maximum BET surface area (1705 m<sup>2</sup> g<sup>-1</sup>) and total pore volume (0.67 cm<sup>3</sup> g<sup>-1</sup>). On the other hand the best mechanical strength was observed at 864 °C after a lower temperature isothermal step. Overall, the results indicated a stronger dependence of surface area, total pore volume, microporosity development and mechanical strength on the final treatment temperature than on the temperature programme.

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## 1. Introduction

The field of potential applications of activated carbon cloths (ACC) has been increasingly investigated in the last years because they offer several technological advantages in comparison with traditional forms of this widely used porous material. These include faster pore diffusion and adsorption kinetics, higher efficiency, larger capacity for adsorption due to higher surface area, and low pressure drops in flow units. In addition, ACC provide a continuous carbon form which is ideally suited for electrical and electrochemical applications [1–4]. Owing to their advantages, either untreated or modified ACC have been investigated as catalyst support [5–8], electrode materials [9–12], for abatement of gaseous and liquid pollutants, as well as for gas storage and separation, H<sub>2</sub> recovery and purification, and sour gas sweetening [13–20].

As also occurs with granular and powder forms, chemical activation leads to ACC with high adsorption capacity in higher yields and

at relatively lower temperature than physical activation [2,21–23]. This comparatively modern form of activated carbon is generally made from natural and synthetic polymeric precursors such as cellulose, regenerated cellulose (mainly, viscose rayon), polyacrylonitrile (PAN) and phenolic fabrics [24–28]. The precursor material selected together with processing parameters play an important role in determining the physico-chemical, mechanical, and electrical properties of the ACCs [29,30]. Accordingly, exploration of novel textiles as starting materials for ACC development is relevant.

Tencel<sup>®</sup> is the brand name for a recently improved fabric from a fibre generically called lyocell. Lyocell is a natural man-made fibre, produced in an environmentally friendly process from wood pulp, that has become popular in clothing. As a result of the controlled and regular structure of its nanofibrils, new functional properties can be introduced to Tencel<sup>®</sup> fabrics. Compared to cotton and viscose fibres, higher strength (specially when wet) and higher modulus are two of the major physical benefits of lyocell fibres. Although regenerated cellulosic textiles such as ACC precursors have been successfully employed, to the best of our knowledge, Tencel<sup>®</sup> has not previously been investigated for this purpose [31].

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**Table 1**  
Elemental composition of Tencel® and yield and elemental composition of the ACC obtained under different experimental conditions.

Sample	Yield (%)	N (%)	C (%)	H (%)	O <sup>a</sup> (%)
Tencel®	–	0.2	42.4	5.9	51.5
ACC-1A	19.8	0.0	88.0	0.4	11.5
ACC-2A	26.9	0.1	85.9	0.6	13.4
ACC-3A	34.4	0.1	70.5	0.8	28.7
ACC-4A	38.5	0.0	66.4	1.0	32.6
ACC-2B	27.2	0.0	83.9	0.5	15.6
ACC-2C	26.4	0.0	80.7	0.6	18.7
ACC-2D	27.6	0.1	84.5	0.7	14.7
ACC-2E	26.2	0.0	80.6	0.5	18.9

<sup>a</sup> Calculated by difference.

The aim of the present work was to evaluate the feasibility of using Tencel® fabric as a precursor for the manufacture of ACC by phosphoric acid activation. The influence of thermal treatment strategy on yield, porosity development and mechanical properties was investigated.

## 2. Experimental

### 2.1. Synthesis of activated carbon cloths

Tencel® fabric, kindly provided by Santista Textile Group (Buenos Aires, Argentina), was used as precursor for the preparation of the ACC. The fabric had a specific mass of 200 g m<sup>-2</sup> and warp and weft of 39 and 24 yarn cm<sup>-1</sup>, respectively. The elemental composition of the precursor is given in Table 1.

The samples previously weighed were impregnated with H<sub>3</sub>PO<sub>4</sub> acid (analytical grade) solutions of 10 wt% concentration at 60 °C overnight. Impregnation ratio, calculated as mass of phosphoric acid/mass of dried precursor, was 0.21 for all the samples. The impregnated samples were thermally treated in a tubular stainless steel reactor of horizontal configuration under a N<sub>2</sub> flow (100 mL min<sup>-1</sup>), externally heated by an electric furnace. They were heated at 5 °C min<sup>-1</sup> up to different final temperatures (HTT) between 660 and 970 °C. Once the desired final temperature was attained, it was held for 1 h. In order to improve mechanical resistance of the ACC, an isothermal step of 40 min was included in the thermal programme. The temperature of the isothermal step was varied in the range 170–270 °C for the ACC prepared at 864 °C. The range of temperatures for the isothermal step was selected from measurements by thermogravimetric analysis using the precursor previously impregnated with a 10 wt% phosphoric acid solution [32]. For the sake of comparison, ACC were also prepared at HTT of 864 °C with a heating rate of 15 °C min<sup>-1</sup>. Afterwards, the resulting cloths were cooled under N<sub>2</sub> flow and rinsed thoroughly with distilled hot water until the wash water had a neutral pH. They were dried until constant weight. Yields were calculated from weight differences. Experimental conditions employed and designations of the samples are summarized in Table 2.

### 2.2. 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 and from the adsorption isotherms of carbon dioxide at 273 K determined using a classical manually operated manometric apparatus. N<sub>2</sub> isotherms were analyzed by the  $\alpha_s$  method using published standard data in order to obtain the external surface area ( $A_s$ ) and micropore volume ( $v_s$ ). They were also analyzed by means of the Dubinin–Radushkevich (DR) equation in order to obtain the DR micropore volume ( $v_0$ ), characteristic energy ( $E_0$ ) and the mean pore width ( $L_0$ ). The latter was estimated from the relationship [33]:

$$L_0 = \frac{10.8}{E_0 - 11.4}$$

**Table 2**  
Experimental conditions employed for the preparation of the regenerated cellulose-derived ACC.

Sample	HTT (°C)	Isothermal step (°C)	Rate (°C min <sup>-1</sup> )
ACC-1A	963	270	5
ACC-2A	864	270	5
ACC-3A	763	270	5
ACC-4A	663	270	5
ACC-2B	864	220	5
ACC-2C	864	170	5
ACC-2D	864	None	5
ACC-2E	864	270	15

The BET equation was applied to evaluate the specific surface area, whereas total pore volumes ( $v_T$ ) were estimated from the amount of N<sub>2</sub> adsorbed at the highest relative pressure, near unity. The DR equation was employed to fit the CO<sub>2</sub> isotherms. The vapor pressure and density of CO<sub>2</sub> were taken as 26.126 bar and 1.023 g cm<sup>-3</sup>, respectively. The value used for the CO<sub>2</sub> affinity coefficient was  $\beta = 0.35$ . The density of N<sub>2</sub> was taken as 0.808 g cm<sup>-3</sup> and the affinity coefficient as  $\beta = 0.34$ .

Elemental composition of the precursor and the derived ACC was determined using a Carlo Erba EA 1108 elemental analyzer. The results presented are average values of triplicate determinations, where the standard error did not exceed 5%.

Sample breaking strength ( $\sigma$ , MPa) and Young's modulus ( $E$ , MPa) measurements were performed by using an Instron 3345 tensile strength tester at room temperature. The dimensions of the samples were 20 mm × 5 mm.

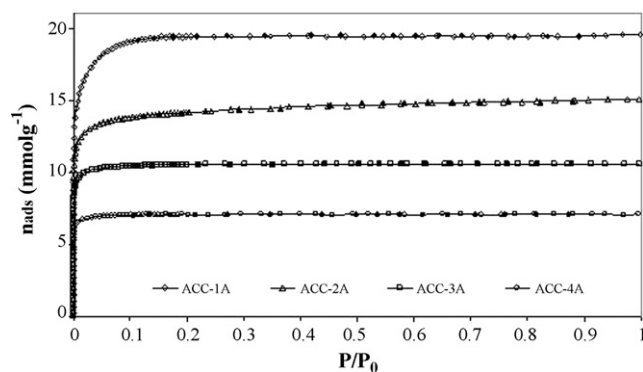
## 3. Results and discussion

Table 1 reports yield and elemental composition of the ACC. The results indicate that increasing the HTT induced a progressive decrease in ACC yield. However, all of the values are similar to others previously reported for predominantly cellulosic precursors. For all the samples the amount of N present is almost negligible. Likewise, the amounts of H and O are much less than those found in the precursor, whereas the contents of C are higher. With increase in HTT, and corresponding decrease in yield, the H and O contents decrease significantly whereas the C content increases. These changes in yield and elemental composition may be attributed to a larger release of volatiles accompanied by enrichment in carbon content due to an aromatization process [29] which is favored at higher temperatures. In the case of samples prepared at HTT of 864 °C, but using different temperature programmes, almost negligible variation in product yield and elemental composition are observed.

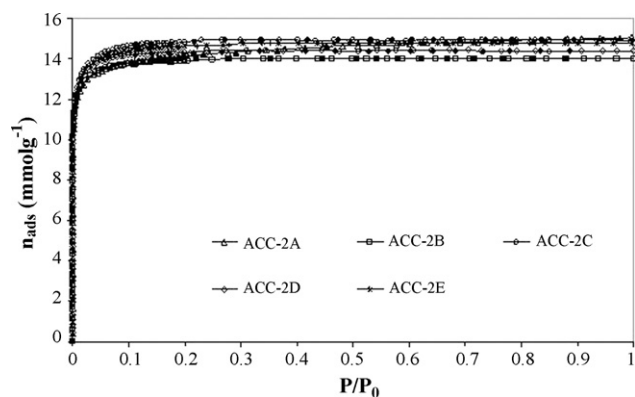
N<sub>2</sub> adsorption–desorption isotherms for the ACC obtained under different thermal treatment conditions are illustrated in Figs. 1 and 2. The amounts of N<sub>2</sub> adsorbed per sample mass unit ( $n_{ads}$ ) are represented as a function of the relative pressure ( $P/P_0$ ), where  $P$  is the equilibrium pressure, and  $P_0$ , the saturation pressure of the adsorbate at –196 °C.

All the isotherms in Figs. 1 and 2 present a plateau over wide ranges of the relative pressure. They are characteristic of Type I isotherms according to the IUPAC classification, pointing to essentially microporous materials [29]. It can be seen that only sample ACC-2A presented a slight slope in the multilayer region. The isotherms were also reversible.

Fig. 1 shows that the N<sub>2</sub> adsorption capacity of the ACC increased substantially with increase in the final treatment temperature used for their preparation. Moreover, the knee of the isotherms became more rounded with increase in HTT indicating an increase in the mean pore width [1]. Comparison of the results in Figs. 1 and 2 also points to a stronger dependence of porosity development on the



**Fig. 1.** N<sub>2</sub> adsorption/desorption isotherms for the activated carbon cloths obtained at different final thermal treatment temperatures and a 10 wt% acid concentration solution. Open points are the adsorption branch and closed points the desorption branch of the isotherms.



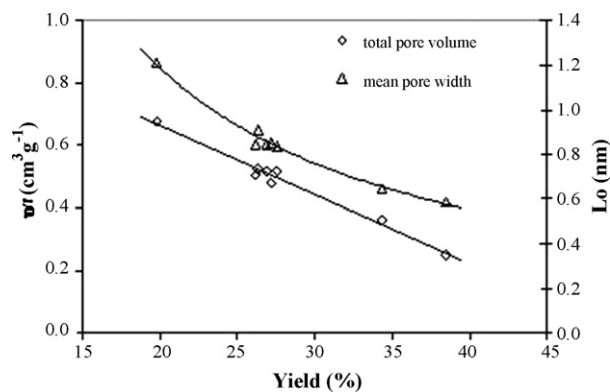
**Fig. 2.** N<sub>2</sub> adsorption/desorption isotherms for the activated carbon cloths obtained using different temperature programs at HTT 864 °C and a 10 wt% acid concentration solution. Open points are the adsorption branch and closed points the desorption branch of the isotherms.

final treatment temperature than on the temperature programme. Higher temperatures could accelerate dehydration and depolymerization reactions, leading to increased aromatization as well as to incorporation of the acid into the precursor's structure, thus favoring the formation of new pores, or pore widening (increase in pore width) and pore deepening (increase in pore depth) of pre-existing pores [2,12]. Accordingly, increasing the temperature promotes porous matrix development, leading to the enhancement of the volume of N<sub>2</sub> adsorbed on the ACC at a given relative pressure.

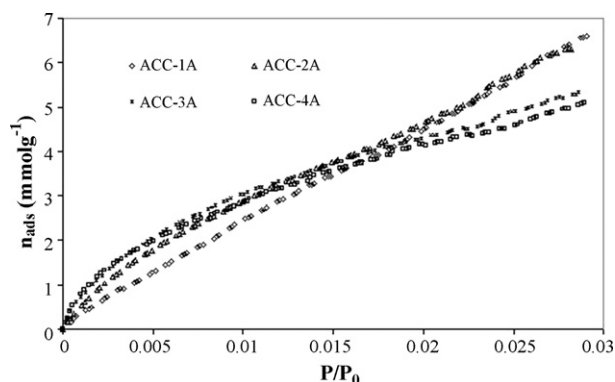
Textural parameters obtained from the nitrogen isotherms by application of the BET, DR and  $\alpha_S$  methods are summarized in Table 3. In all cases, the DR plots corresponding to the isotherms were found to give good linearity over the relative pressure range between  $10^{-5}$  and  $2 \times 10^{-2}$ , and the  $\alpha_S$  plots were linear from relative pressures above about 0.3. The derived parameters given in Table 3 confirm that increasing the temperature resulted in an increase in total pore volume and apparent surface area. Maximum values of  $0.67 \text{ cm}^3 \text{ g}^{-1}$  and  $1705 \text{ m}^2 \text{ g}^{-1}$ , respectively, were attained at the highest HTT of 963 °C. On the other hand, the external surface areas,  $A_S$ , of all the samples were found to be very low ( $<11 \text{ m}^2 \text{ g}^{-1}$ ), indicating that no significant mesoporosity was developed. This is confirmed by the good agreement between the total pore volumes and the micropore volumes calculated by both DR and  $\alpha_S$  methods.

For all samples, the corresponding values of  $\nu_S$  and  $\nu_0$  are in close agreement, which is indicative of the presence of fairly narrow micropores only. This is confirmed by the values of  $L_0$ , given in column 7 of Table 3, which are all  $<1.1 \text{ nm}$ . These values also show that there is an increase in mean micropore width as the HTT is increased.

The increase in both pore volume and pore width with increasing HTT or decreasing yield is, in qualitative terms, expected. The quantitative relationship is shown in Fig. 3. This figure shows, on the one hand, that there is no significant difference between the samples prepared at 864 °C, but with different temperature programmes.



**Fig. 3.** Variation of total pore volume and mean pore size with yield.



**Fig. 4.** CO<sub>2</sub> adsorption isotherms for the activated carbon cloths obtained at different final thermal treatment temperatures.

On the other hand, the figure also shows that there is a linear relationship between pore volume and yield, but that the mean pore size increases more rapidly at low yield/higher HTT. That is, there are changes in the pore size distribution as the HTT is increased.

Additional information on changes in pore size distribution can be obtained by comparing the N<sub>2</sub> adsorption isotherms with CO<sub>2</sub> adsorption isotherms determined on corresponding samples. In Fig. 4 the CO<sub>2</sub> isotherms obtained for the ACC prepared using different final treatment temperatures are shown. The corresponding DR plots gave very good linearity and the textural parameters calculated from the DR plots for all of the samples are listed in the last 2 columns of Table 3.

For the highest yield sample, prepared at the lowest temperature,  $\nu_0(\text{CO}_2)$  is significantly higher than  $\nu_0(\text{N}_2)$  indicating the presence of very narrow microporosity and, possibly, constrictions at the micropore entrances. The differences are due to the fact that adsorption of nitrogen at  $-196 \text{ °C}$  may be kinetically restricted in the ultra-microporosity ( $<0.7 \text{ nm}$  diameter) range [24]. The critical dimension of the CO<sub>2</sub> molecule is smaller than that of N<sub>2</sub> and,

**Table 3**

Textural characteristics obtained by application of the  $\alpha_S$ , DR and BET methods to N<sub>2</sub> and CO<sub>2</sub> adsorption isotherms determined on activated carbon cloths.

Sample	N <sub>2</sub> at 77 K				CO <sub>2</sub> at 273 K			
	$\nu_T$ (cm <sup>3</sup> g <sup>-1</sup> )	$A_{\text{BET}}$ (m <sup>2</sup> g <sup>-1</sup> )	$A_S$ (m <sup>2</sup> g <sup>-1</sup> )	$\nu_S$ (cm <sup>3</sup> g <sup>-1</sup> )	$\nu_0$ (cm <sup>3</sup> g <sup>-1</sup> )	$L_0$ (nm)	$\nu_0$ (cm <sup>3</sup> g <sup>-1</sup> )	$L_0$ (nm)
ACC-1A	0.67	1705	1.6	0.67	0.64	1.09	0.41	0.84
ACC-2A	0.52	1229	10.8	0.51	0.48	0.84	0.45	0.75
ACC-3A	0.37	927	0.6	0.37	0.37	0.65	0.39	0.64
ACC-4A	0.25	625	2.1	0.24	0.25	0.59	0.33	0.59
ACC-2B	0.49	1225	1.5	0.49	0.49	0.85	0.45	0.72
ACC-2C	0.52	1305	0.3	0.52	0.51	0.91	0.43	0.70
ACC-2D	0.52	1266	0.6	0.50	0.49	0.84	0.42	0.68
ACC-2E	0.51	1284	6.3	0.51	0.50	0.85	0.44	0.70

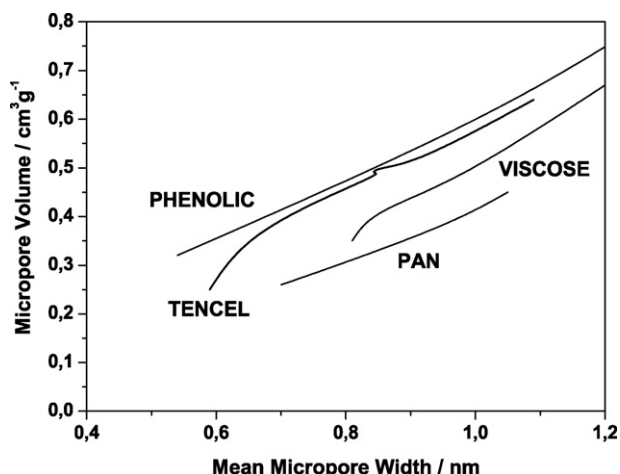


Fig. 5. Relationship between micropore volume and mean micropore width for different activated carbon fibres [1,34,36].

furthermore, the adsorption temperature for CO<sub>2</sub> is higher, which results in a larger kinetic energy. The CO<sub>2</sub> molecules are therefore able to enter more easily into very narrow micropores [35]. For the lowest yield sample, on the other hand,  $v_0(\text{CO}_2)$  is significantly lower than  $v_0(\text{N}_2)$  indicating the presence of wider microporosity [35]. The lower values of  $L_0(\text{CO}_2)$  in comparison with  $L_0(\text{N}_2)$  for the samples prepared at the two highest temperatures is also an indication of the presence of some wider microporosity in these samples.

For the adsorption of small molecules, such as VOCs or phenolic pollutants, for example, the adsorbent needs to have narrow micropores, in which the adsorptive–adsorbent interaction energy is maximised, as well as a high micropore volume, in order to maximise the adsorption capacity. Hence, a convenient way to compare the performance of different adsorbents is to plot the micropore volume as a function of the micropore width. Such a comparison, of ACC prepared from Tencel® fabrics with some other common types of activated carbon fibre and cloth, is presented in Fig. 5. It can be seen that the ACC from Tencel® have excellent properties. For a given narrow pore width they have higher micropore volumes than viscose or PAN and, over the approximate range 0.7–1.1 nm, give a micropore volume/width relationship very similar to phenolic fibres.

Characteristic mechanical parameters of the ACC are listed in Table 4. It can be seen that ACCs were stronger in the weft direction, showing higher breaking strength values. The trend could be attributed to mechanical stresses in the warp direction generated during the precursor's manufacture. The results in Table 4 also indicate that an increase in the final treatment temperature up to 864 °C enhances the ACC strength, but that further increase in temperature to 963 °C results in the sample which has the lowest values of both

Table 4

Breaking load,  $\sigma$ , and Young's modulus,  $E$ , in the weft and warp directions of the activated carbon cloths produced.

Sample	Weft		Warp	
	$\sigma$ (MPa)	$E$ (MPa)	$\sigma$ (MPa)	$E$ (MPa)
ACC-1A	0.28	25.4	0.20	8.5
ACC-2A	0.47	73.8	0.43	16.9
ACC-3A	0.40	68.8	0.26	16.7
ACC-4A	0.33	57.3	0.24	11.4
ACC-2B	0.41	68.9	0.37	15.5
ACC-2C	0.47	60.0	0.35	17.6
ACC-2D	0.29	43.7	0.27	17.7
ACC-2E	0.33	43.8	0.32	11.2

breaking strength and modulus in both the warp and weft directions. For low activation degree, it is likely that the increase in HTT led to better mechanical properties due to the predominant development of more ordered structures. In contrast, the deterioration in mechanical strength for the highest degree of activation could be due to the presence of higher and wider porosity, as seen on Fig. 3 for example, which considerably reduces the real cross-section of the fibres [29]. With regard to the effect of the temperature programme, an isothermal step and a slower heating rate produced more resistant ACCs. These conditions seem to induce a slower evolution of volatile matter, resulting in a more ordered structure and higher breaking load [24]. However, the temperature of the isothermal step did not exert a strong influence on the ACC strength in the range of temperatures studied.

#### 4. Conclusions

Activated carbon cloths with well-developed micropore structure and good mechanical strength were achieved using Tencel® fabric as a precursor. ACC with predominance of fairly narrow micropores and good physical appearance were obtained using different strategies for the thermal treatment stage. The textural characteristics of the derived ACC were found to be mainly dependent on the final treatment temperature with the precise thermal programme having little effect. A maximum BET surface area of 1705 m<sup>2</sup> g<sup>-1</sup> and total pore volume of 0.67 cm<sup>3</sup> g<sup>-1</sup> were reached at HTT 963 °C. For the ACC obtained at lower temperatures, there was a development of very narrow microporosity and constrictions in the entrance of the micropores. On the other hand, the highest values of breaking strength and modulus were achieved at 864 °C and it was found that an isothermal step at lower temperature was beneficial.

Overall, the present results indicate that Tencel® fabric represents a suitable starting material for manufacture of good quality ACC through phosphoric acid activation. The results obtained suggest that the ACC obtained should be promising materials for applications where narrow micropore size coupled with comparatively high micropore volume are required.

#### Acknowledgements

Financial support from Consejo Nacional de Investigaciones Científicas y Técnicas (CONICET), Universidad de Buenos Aires (UBA), and Agencia Nacional de Promoción Científica y Tecnológica (ANPCYT–FONCYT) from Argentina is gratefully acknowledged. The authors also express their thanks to the EUROPEAID Co-operation Office–Alfa Programme (Project ALFA II 0412 FA FI).

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