Use of Thermoplastic Polyurethane Elastomers in the Preparation of Fabric/Activated Carbon Composites

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ABSTRACT: A new application of thermoplastic polyurethane elastomers (TPUs), as composite binder in the preparation of fabric/activated carbon composites, is investigated. Different amounts of TPU in the impregnating solutions, different fabrics, and different activated carbons (ACs) were used to prepare the fabric/carbon composite samples. The adsorption properties of the resulting samples were tested, and the results obtained show the suitability of the composite preparation method proposed in this article. Moreover, the adsorption properties of the composite samples prepared are independent of the fabric used and dependent of the AC properties and the TPU amount used in the preparation. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 118: 3509–3517, 2010

Key words: thermoplastic polyurethane; activated carbon; composite; adsorption; adsorbent fabrics

INTRODUCTION

The widely known importance of thermoplastic polyurethane elastomers (TPUs) in many industrial fields has resulted in a marked increase of the research on this subject. There are studies dealing with different topics, from fundamentals of structure[1–3] to applications in such varied fields as automotive or footwear industry,[4] biomedical materials,[5] elastomers,[6] or adhesives.[7,8]

In this article, we investigate a new route to prepare fabric/activated carbon composites using a thermoplastic polyurethane as composite binder. The polymer choice was due, on one hand, to its good adhesion and mechanical properties and, on the other, to its versatile and controllable properties, depending on its composition and synthesis conditions.[9–11]

Activated carbon (AC) is the collective name for a group of porous materials consisting mostly of carbon, exhibiting appreciable apparent surface area and micropore volume (MPV).[12–14] They are solids with a wide variety of pore size distributions (PSD) and micropore size distributions (MPSD). The interest in carbon-based adsorbent materials is in constant increase due to their many applications, such as in environmental protection, energy and gas storage, protective clothes, filters in clean rooms, gas separation and purification or adsorption in solution.[12–20]

One of the main advantages of the carbonaceous sorbents is that they can present different morphologies (powder, granular, fiber, etc.), sizes (nanoscale, i.e., nanotubes and nanofibers; microscale, i.e., microfibers; macroscale, i.e., fibers), and forms (monoliths, pellets, spheres, etc.).[20–22] Moreover, for many applications (e.g., automotive interior filtration, respiratory masks, laser printers, air conditioning systems, vacuum cleaners, stoma products, wound pads), their use as AC fabrics and felts is interesting, specially due to their flexibility and wide range of shapes, besides their high adsorption capacity.[23–26] However, their high cost has encouraged the research on preparation and characterisation of adsorbent-based materials which, keeping the above mentioned qualities, could be prepared from granular ACs supported on fabrics or other adequate materials.

In this sense, many efforts have been made (most of them patented) to obtain different fabric/AC composites avoiding the use of AC fabrics. For example, Liu et al.[27] used a sol–gel and dip coating method to immobilize activated carbon fiber (ACF) on terylene (PET). Kim et al.,[28] used low melting point resins to coat a nonwoven fabric with AC powders. Wang[29] bonded AC particles together via solid hot-melt adhesive. Finally, Jones et al. used Lathylen 500 (ethylene terpolymer) or a polyacrylate to bind AC to a nonwoven fabric.[30]

In previous works, and with the aim of obtaining adsorbent materials supported on fabrics or polymers, polyurethane foams or fabrics have been used as supports of zeolites or ACs (in form of pellets or in powder).[31–35] Nevertheless, though the results obtained with zeolites or AC pellets were acceptable,

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they reveal the need to improve the preparation method of the adsorbents.

This context lead us to investigate new preparation methods for fabric/AC adsorbent materials, using thermoplastic polyurethane as composite binder. The results showed in this article, obtained with different fabric/AC composites (prepared with various activated carbons, supporting fabrics, and polymer content) allow us to confirm the interest of the use of TPU as a binder for the preparation of fabric/AC composites.

EXPERIMENTAL

Experimental techniques

Textural characterization of the raw and composite materials was accomplished by N_2 and CO_2 adsorption isotherms at -196 and $0°C$, respectively, using an automatic adsorption system (Autosorb-6, Quantachrome). In the isotherm plots, the results are expressed in standard temperature and pressure conditions. The samples were outgassed at $25°C$ (to avoid TPU melting) under vacuum for 4 h. Specific BET surface area (S_{BET}) was calculated applying the BET equation to N_2 adsorption data:

$$\frac{P/P_0}{n\left(1 - \frac{P}{P_0}\right)} = \frac{1}{n_m C} + \frac{C-1}{n_m}\frac{P}{P_0}$$

In this equation n_m is the monolayer adsorption capacity (mmol/g), n is the amount of gas adsorbed (mmol/g), P is the pressure, P_0 is the adsorptive saturation pressure at the adsorption temperature, and C is a parameter related to the heat of adsorption. Plotting $\frac{P/P_0}{n\left(1-\frac{P}{P_0}\right)}$ vs P/P_0 n_m and C can be obtained from the slope and intercept.

The adsorbent specific surface area (S_{BET}) can be obtained from n_m using this equation:

$$S_{BET} = n_m N_a a_m 10^{-21}$$

where S_{BET} is the solid specific surface area (m^2/g), n_m is the monolayer adsorption capacity (mmol/g), N_a is Avogadro number, in molecules/mol and a_m is the surface occupied by an adsorbed molecule, in nm^2 (0.162 nm^2 for nitrogen).

Total micropore volume was calculated from N_2 adsorption data at $-196°C$ [V_{DR} N_2] and narrow micropore volume was calculated from CO_2 adsorption data at $0°C$ [V_{DR} CO_2], using in both cases the Dubinin–Radushkevich equation (DR).[36,37]

The Dubinin-Radushkevich equation used for such calculations is the following:

$$\log W = \log W_0 - D \log^2(P_0/P)$$

where $D = 2.303$ K $(RT/\beta)^2$

In this equation W is the volume adsorbed (as a liquid) at P pressure, W_0 is the micropore volume, K is a constant dependent on the pore structure, and β is the affinity coefficient.

A plot of $\log W$ against $\log^2 (P_0/P)$ will be a straight line having an intercept equal to $\log W_0$, being W_0 the total micropore volume (cm^3/g). From its slope, the value of D is obtained.

In addition, mesopores percentage of each sample was calculated using the equation:

mesopores percentage =
$$\{[(V_{N_2})_{0.99} - (V_{N_2})_{0.20}]/(V_{N_2})_{0.99}\} \times 100$$

$(V_{N_2})_{0.99}$: micropore volume, calculated from N_2 adsorption data ($-196°C$), at relative pressure 0.99.

$(V_{N2})_{0.20}$: micropore volume, calculated from N_2 adsorption data ($-196°C$), at relative pressure 0.20.

Morphological characterization of the raw and composite materials was accomplished with a JEOL SEM JSM 840 microscope. The samples were metallised (Au) with a BALZERS SCD 004 metalliser to improve their conductivity and obtain high resolution images.

Materials

TPUs were prepared from diphenylmethane-4,4'-diisocyanate (MDI, 98% purity; Aldrich Chemicals, Gillingham, Dorset, UK), poly(ε-caprolactone) ($M_n = 3000$ g/mol) as macroglycol (Solvay Interox, Cheshire, UK), and 1,4-butanediol (chain extender, 99% purity; Aldrich). Moisture in the macroglycol was removed by heating overnight at $70°C$ under 0.7 MPa pressure. The NCO content of the prepolymer was obtained by titration with n-butylamine (ASTM D 2572-80).

Three commercial activated carbons were used, including ACW (SA-30 from Measd Westvaco Corporation, Richmond, VA), ACM2, and ACM3 (Maxsorb 2000 and Maxsorb 3000, respectively, from Kansai Coke and chemicals, Hyogo, Japan). Their different textural characteristics, deduced from their adsorption isotherms (N_2 at $-196°C$ and CO_2 at $0°C$), are shown in Figure 1 and Table I.

Three commercial fabrics were used to prepare the fabric/AC composites, F1 and F2 based on polyester and F3 based on cellulose fibers. Their different textural characteristics, deduced from their adsorption isotherms (N_2 at $-196°C$ and CO_2 at $0°C$), are shown in Figure 2 and Table I. As expected, their adsorption capacities are quite low (even negligible) compared to those of the activated carbons.
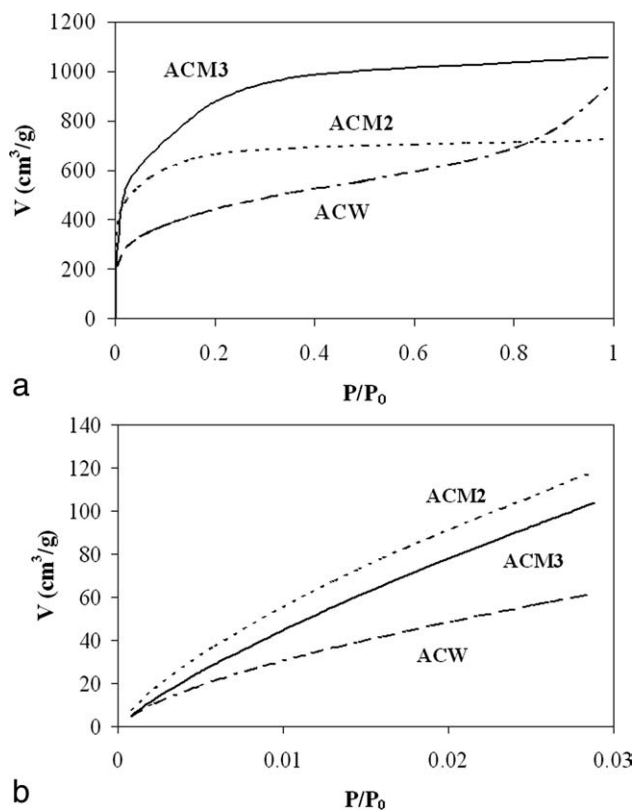
Figure 1 (a) Nitrogen ($-196°C$) and (b) CO_2 ($0°C$) adsorption isotherms of the activated carbons used in this study (ACW, ACM2, and ACM3).

Figure 2 (a) Nitrogen ($-196°C$) and (b) CO_2 ($0°C$) adsorption isotherms of the fabrics used in this study (F1, F2, and F3).

Preparation of TPUs and adhesive solutions

TPUs were prepared according to the prepolymer method.[9,10] The prepolymer was obtained by reacting the macroglycol with MDI (diphenylmethane-4,4'-diisocyanate, 98% purity; Aldrich Chemicals) in excess (NCO groups/OH groups = 1.2), under stirring at 65°C during 70 min. To complete the polymerization, the prepolymer was further reacted (in a vacuum oven, 12 h at 80°C, after mixing) with stoichiometric amounts (NCO remaining groups = OH/COOH groups) of 1,4-butanediol. Some properties of the TPUs obtained are shown in Table II. Adhesive solutions were prepared by solving the TPU in 2-butanone using a laboratory mixer.
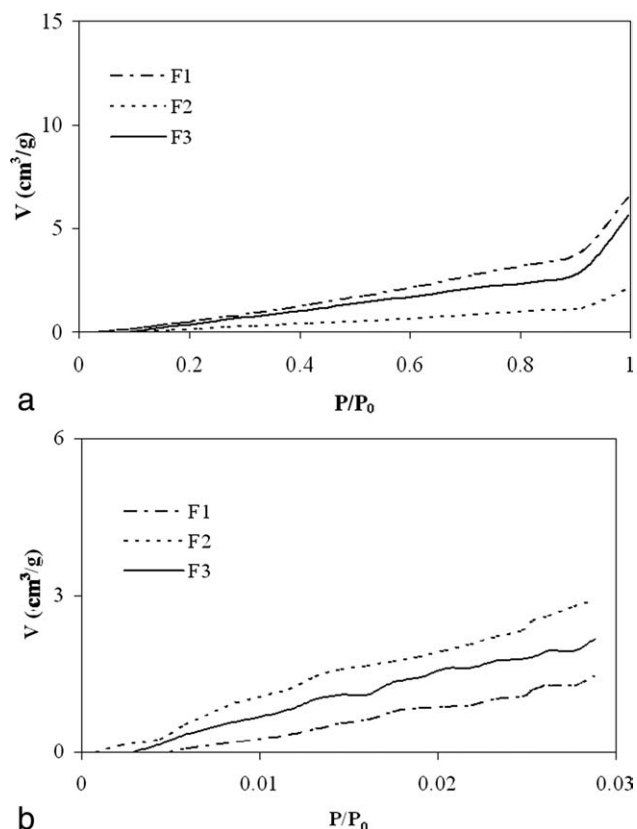
Composite preparation: deposition of the activated carbon on the fabric

Pieces of fabric (4 cm^2) were soaked in different TPU/2-butanone solutions and then dried at room temperature until constant weight. The obtained samples were heated at 120°C in a laboratory oven for 20 min, allowing the TPU to recover its original tack to fix the sprinkled AC on the fabrics surface. Next they were shacked to remove the possible remaining unfixed AC. In all cases, the final amount of AC in the different samples was about 19 wt %. Photographs of the original fabrics and resulting composites prepared using 5 wt % of TPU are shown in Figure 3.

TABLE I
Textural Characteristics of the Activated Carbons and Fabrics Used in this Study

Nomenclature	S_{BET} (m^2/g)	V_{DR} N_2 (cm^3/g)	V_{DR} CO_2 (cm^3/g)	Mesopores (%)
ACW	1534	0.70	0.32	52.5
ACM2	2421	1.07	0.64	8.5
ACM3	3040	1.46	0.61	17.6
F1	1.30	0.002	0.03	–
F2	0.04	0.004	0.02	–
F3	0.26	0.005	0.02	–

TABLE II
Some Properties of the TPU Prepared

$\overline{M}_w$ (g/mol)	T_g (°C)	T_m (°C)	ΔH_m (J/g)
87700	−41.1	49.3	46.4

$\overline{M}_w$, average molecular weight.
T_g, glass transition temperature.
T_m, melting temperature.
ΔH_m, melting enthalpy.

The textural characteristics of the prepared fabric/AC composites, deduced from N_2 at −196°C and CO_2 at 0°C, are shown in Figure 4 and Table III. Additionally, a commercial adsorbent fabric (FR, provided by Freudenberg & Co, Wheinheim, Germany) is used in the present study for comparison purpose. Its adsorption isotherms (N_2 at −196°C and CO_2 at 0°C) and its porous texture characterisation are also compiled in Figure 4 and Table III, respectively.

RESULTS AND DISCUSSION

All the studies detailed below were performed with, at least, three different samples of each raw or composite material, showing a good reproducibility.

Porosity of the precursors used in the preparation of the composites

Figure 1(a,b) includes, respectively, the nitrogen and carbon dioxide adsorption isotherms for the activated carbons studied. The porous texture characterisation for these materials is included in Table I. These figures and data show that ACM2 and ACM3 are essentially microporous activated carbons, being ACM3 a material with a more developed porosity, wider micropore size distribution and larger mean micropore size than ACM2. Sample ACW shows both microporosity and mesoporosity contribution, as denoted by the slope of the isotherm "plateau" and the calculated mesopores percentage.
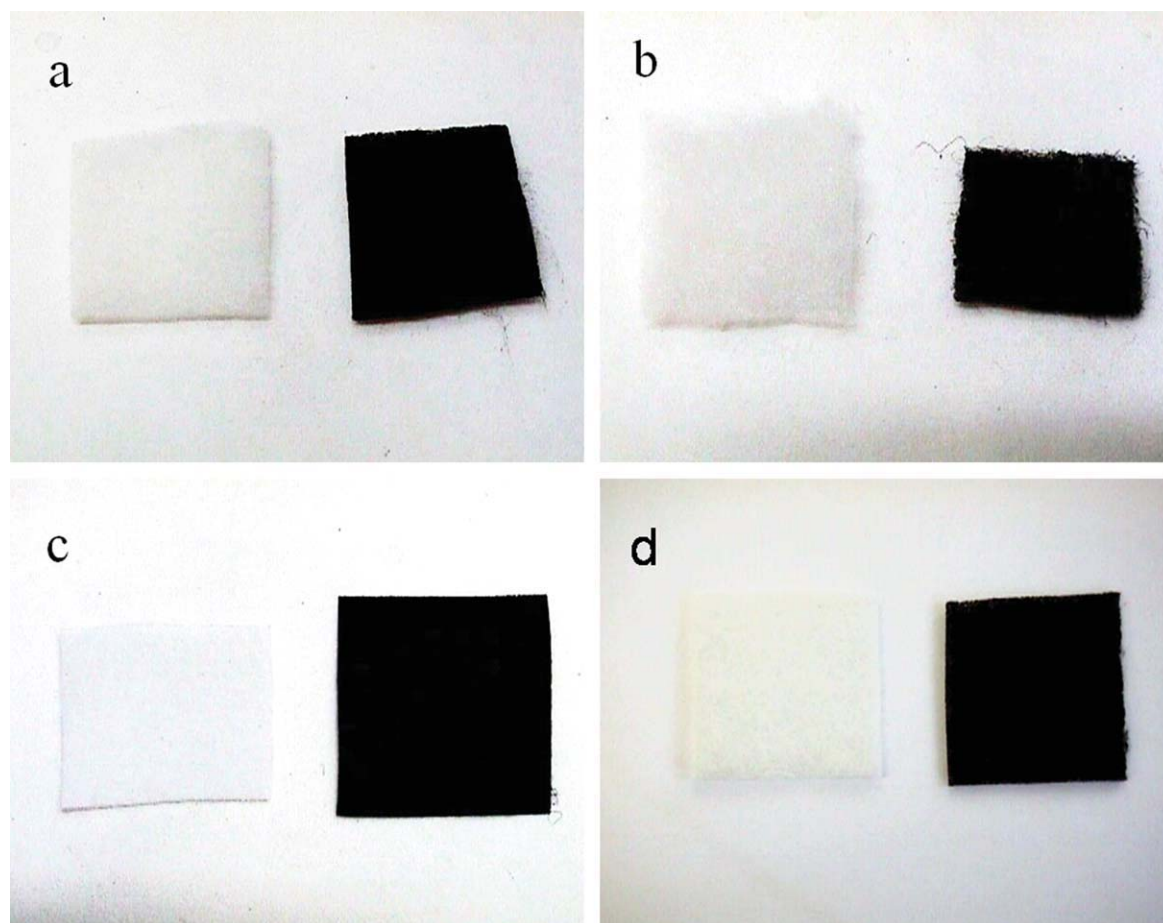

Figure 3 Photographs of the original fabrics and the fabric/AC composites (ACW with 5 wt % of TPU) for (a) F1, (b) F2, (c) F3, and (d) the original commercial fabric and fabric/AC composite FR. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
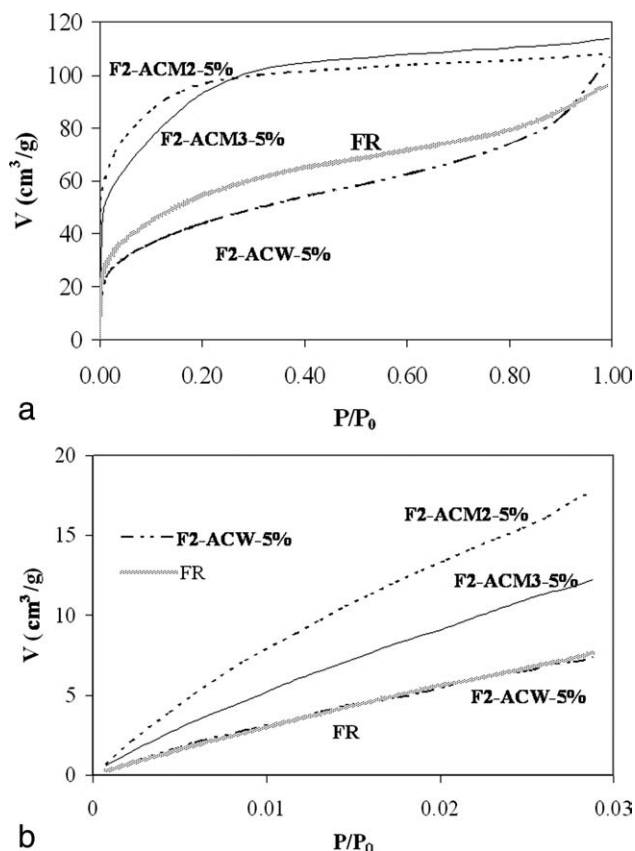
Figure 4 (a) Nitrogen ($-196°C$) and (b) CO_2 ($0°C$) adsorption isotherms of the fabric-AC composites prepared with different AC (5 wt % TPU and F2 as fabric). The commercial fabric/AC composite FR is included for comparison purpose.

The porous texture characterisation of the fabrics used in the preparation of the composites is compiled in Table I. These data, calculated from the fabrics nitrogen and carbon dioxide adsorption isotherms [Figs. 2(a,b), respectively] show that they present negligible porosity.

Composite materials

Figure 3 shows the photographs of the original fabrics and fabric/AC composites (ACW with 5 wt % of TPU) for (a) F1, (b) F2, (c) F3, and (d) the original commercial fabric and fabric/AC composite FR. It evidences that the use of TPU to prepare fabric/AC composites, not reported before, seems to be appropriate. Moreover, these composites show suitable properties to be easily handled and good stability of the AC on their surface, as shown when pieces of composite were magnetically stirred in water during 24 h, without any AC loss. In general, it can be seen that the texture, flexibility, and general appearance are quite similar to the commercial fabric/AC com-

posite (FR), also included in Figure 3 for comparison purpose.

To analyze the effect of the preparation conditions on the final properties of the fabric/AC composites, three variables have been studied: the weight percentage of TPU, the type of AC, and the fabric used. Though all of them have been considered, to simplify the discussion the results are presented keeping always constant two of the variables while the third one is analyzed.

Effect of the TPU percentage introduced in the fabric/AC composites. As mentioned above in this section, and as an example of the study carried out, the fabric (F2) and the type of activated carbon used (ACW) will be kept constant, introducing different amounts of TPU as composite binder.

Concerning the effect of the TPU amount used, it should be noted that with the increase in the TPU weight percentage, the stiffness and the weight of the samples considerably increase. The stiffness increase was deduced by macroscopic observation and explained as follows: while the original fabrics were quite soft and flexible, after the application of the TPU solution (and subsequent solvent evaporation), a thin film of TPU (with higher stiffness than the original fabric) covered the sample, bringing it an average stiffness higher than the original one. However, while a stiffness increase could be interesting from a mechanical point of view, the weight increase dramatically reduces the adsorption properties of the samples (as shown in Fig. 5) expressed per sample weight, and hence, the TPU amount used had to be limited.

TABLE III
Textural Characteristics of the Fabric/AC Samples Prepared with Different Fabrics (ACW, 5% TPU in the Adhesive Solution), Different TPU Amounts (F2/ACW) and Different AC (5% TPU in the Adhesive Solution and F2 as Fabric) Referred to the Total Sample Weight. (The Commercial Fabric/AC Composite FR is Included for Comparison Purpose)

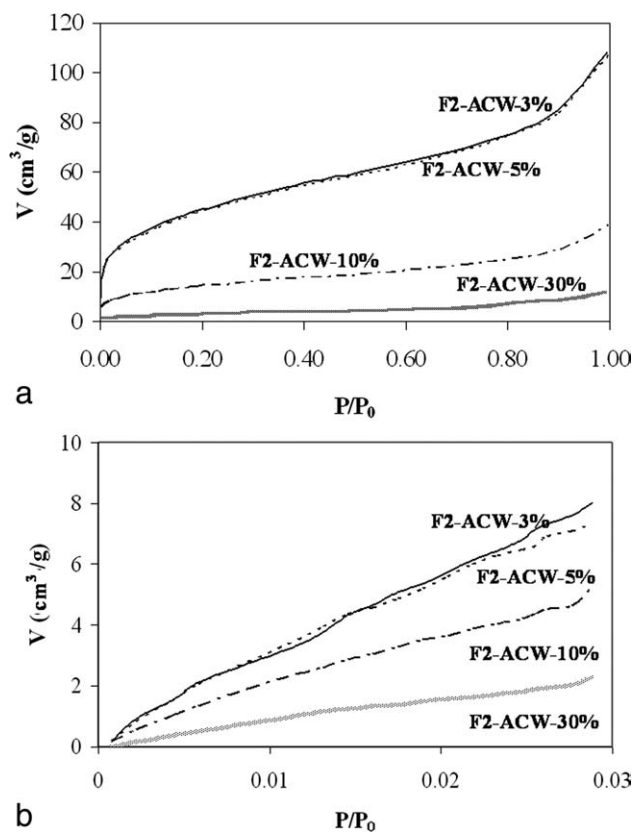| Sample	S_{BET} (m^2/g)	V_{DR} N_2 (cm^3/g)	V_{DR} CO_2 (cm^3/g)
F1-ACW-5%	190	0.09	0.06
F2-ACW-5%	159	0.07	0.04
F3-ACW-5%	163	0.07	0.05
FR	192	0.09	0.05
F2-ACW-3%	162	0.07	0.05
F2-ACW-5%	159	0.07	0.04
F2-ACW-10%	42	0.02	0.02
F2-ACW-30%	9	0.00	0.01
F2-ACW-5%	159	0.07	0.04
F2-ACM2–5%	340	0.16	0.10
F2-ACM3–5%	345	0.16	0.07

Figure 5 (a) Nitrogen (-196°C) and (b) CO_2 (0°C) adsorption isotherms for the fabric/AC (F2/ACW) composites prepared with different TPU amounts.

Thus, four different TPU concentrations (3, 5, 10, and 30 wt % of TPU in 2-butanone) were used to fix the activated carbon on the fabrics. The four resulting fabric/AC composites were named F2-ACW-3%, F2-ACW-5%, F2-ACW-10%, and F2-ACW-30%, respectively. The influence of the amount of TPU in the adsorption properties of the fabric-AC composites is studied in Figure 5 and Table III.

It can be observed that, the higher the amount of TPU in the sample, the larger the loss in its adsorption properties, which may be due to the progressive blocking of the activated carbon porosity as the amount of TPU in the mixture increases. From Table III and Figure 5, it is noticed that such decrease in the adsorption properties affects both the nitrogen and carbon dioxide adsorption results.

Considering both the adsorption properties of the resulting composites and its ease to be handled (see Fig. 5 and Table III) the most suitable TPU concentration in the impregnating solution would be 5 wt %.

Influence of the adsorption properties of the carbon used to prepare the samples. The importance of the AC used for the composite preparation is analyzed next. The fabric/AC composites were prepared using F2

as fabric, 5 wt % TPU in the impregnating solution and three commercial activated carbons (ACW, ACM2, and ACM3), which gave the samples F2-ACW-5%, F2-ACM2–5%, and F2-ACM3–5%, respectively. The properties and isotherms of these composites are shown in Figure 4 and Table III.

Comparing the porosities of these composites with those of the activated carbons used in their preparation (see Table I), it can be observed that, after its deposition on the fabric, every AC suffers an important decrease in its porosity (as expected from the results shown above), due to a partial porosity blocking with the addition of the TPU solution during the preparation of the composites. Similar results are obtained with the other types of fabrics studied.

It can also be concluded that the adsorption properties of ACM3 and ACW are more affected by the preparation process than those of ACM2, which may be due to the higher mesoporosity of the first two compared to the last one (Table I). In this case, the TPU in solution added would mainly cover the mesopores rather than the micropores, giving the obtained results.

The comparison of the three composites prepared in the present study (Fig. 4) shows that those prepared with ACM2 and ACM3 (F2-ACM2–5% and F2-ACM3–5%) have higher adsorption capacity than the one prepared with ACW (F2-ACW-5%), due to the different activated carbons used in the preparation. This figure also includes the results for the commercial composite FR, which contains 23 wt % of AC. Comparison between the composites prepared in the present study and the commercial one (FR) shows that the composites prepared with ACM2 and ACM3 present larger porosity than the commercial one.

The results obtained highlight, for a given amount of TPU (e.g., 5 wt %) and for a given amount of AC (e.g., 19 wt %) that playing with the activated carbon used, the adsorption capacity of the composite can be improved.

The nature of the fabric used to prepare the samples. Keeping constant the AC (ACW), the use of three fabrics (F1, F2, and F3) in the preparation of the composites was compared. The amount of TPU used in the impregnating solution was 5 wt %, as established from the study discussed above. The nomenclature of the three composites prepared is F1-ACW-5%, F2-ACW-5%, and F3-ACW-5%.

Figure 6 and Table III show that the three fabrics used give quite similar results, since the adsorption isotherms (N_2 and CO_2), S_{BET} and V_{DR} (N_2 and CO_2) show small differences.

The fact that all the samples show almost the same adsorption properties regardless their different nature (polyester or cellulose fabrics) indicates
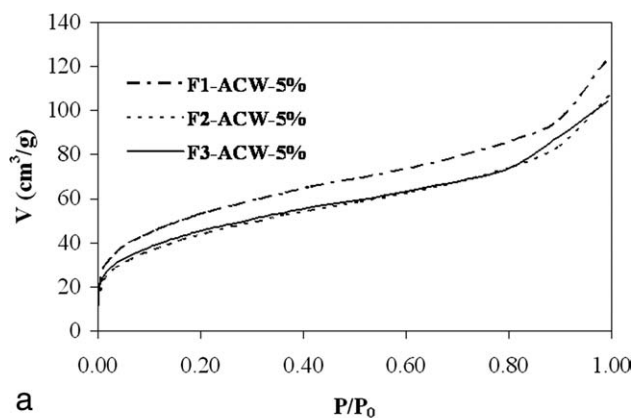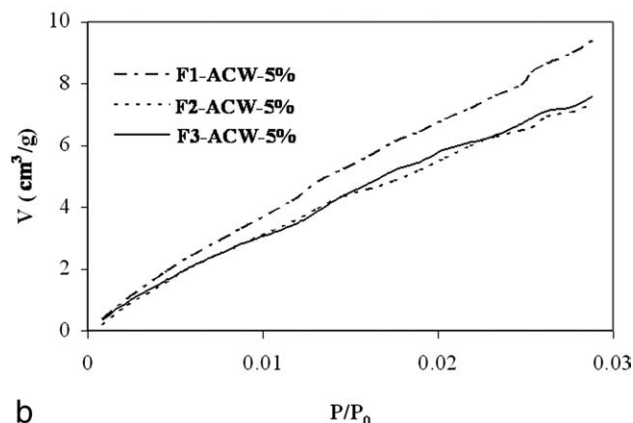
Figure 6 (a) Nitrogen ($-196°C$) and (b) CO_2 ($0°C$) adsorption isotherms of the fabric/AC composites prepared with different fabrics at 5 wt % TPU in the adhesive solution.

that this is not the main factor determining the behavior of the adsorbent materials, but the properties of the original AC, as shown in the previous section.

Study of the fabric/AC composite materials morphology

As a final step, a microscopic study of the precursors and the composite materials, using Scanning Electron Microscopy (SEM), has been included to attempt to understand the morphology of the composites prepared.

Figure 7 shows the surface morphology of ACW, ACM2, and ACM3 at 500 magnifications. As it can be observed, there are no significant differences between the three ACs.

Regarding the surface morphology of the fabric/ AC composites, it is shown in Figure 8 at two different magnifications: $50\times$, for a general view of the fabric/AC composites, and $500\times$, for a closer and more detailed view. The commercial composite FR is also included for comparison purpose. Regardless the considered magnification, no significant differences are observed among the

composites prepared in the laboratory. FR shows a different morphology, probably due to a different preparation method. It must be remarked that the laboratory prepared composite materials show much better fabric coverage than FR. As any information regarding the fabric, the AC or the preparation method of this commercial composite material is available, our comments can only be restricted to the observation of these SEM results.
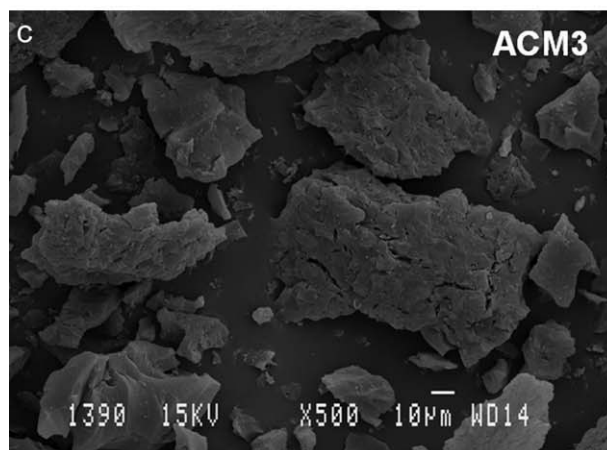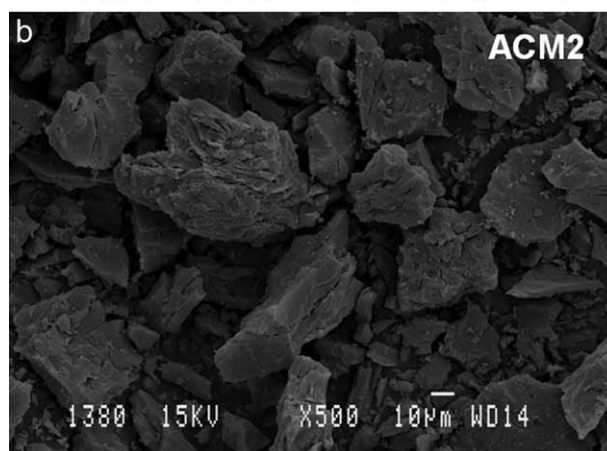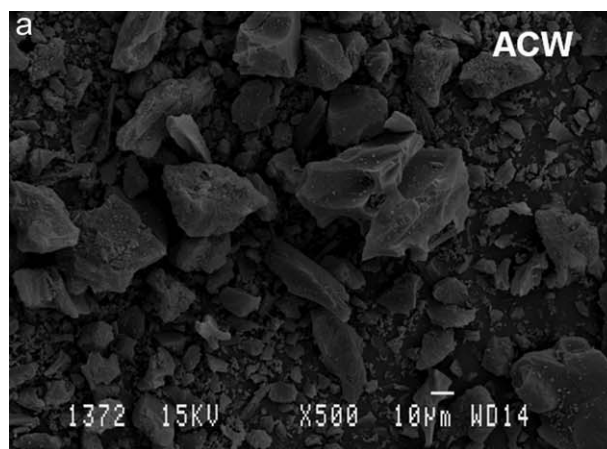

Figure 7 SEM image obtained from (a) ACW, (b) ACM2, and (c) ACM3, at 500 magnifications.
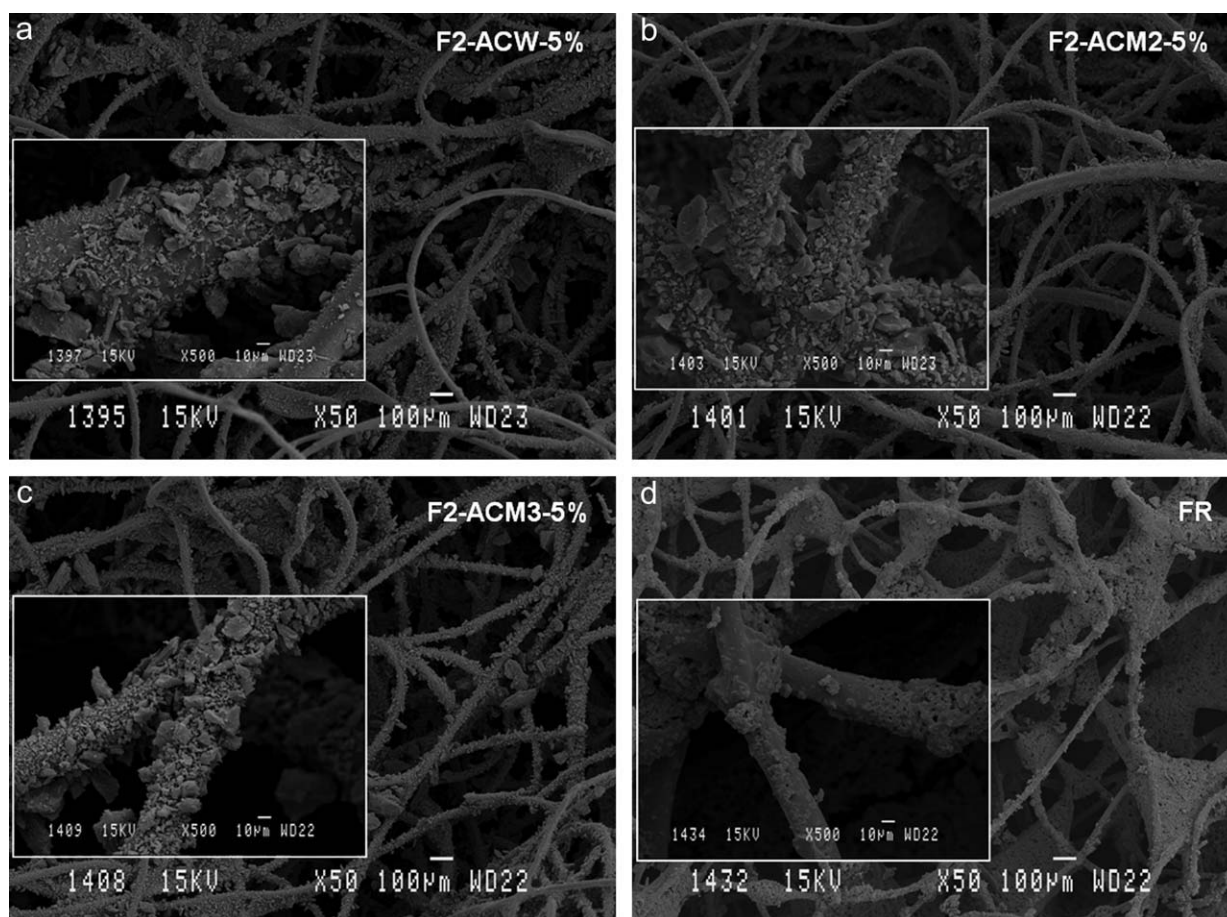
Figure 8 General view of the composite (a) F2-ACW-5%, (b) F2-ACM2-5%, (c) F2-ACM3-5%, and (d) commercial composite FR, obtained with SEM at 50 and 500 magnifications.

CONCLUSIONS

From these results, it may be concluded that the use of TPU to prepare fabric/AC adsorbent composites is suitable. The amount of TPU used in the impregnating solution strongly affects the adsorption properties of the composites prepared. Thus, the higher the TPU concentration in the solution used, the lower the porosity in the composite. The AC adsorption properties loss, as a consequence of the composites preparation, is smaller than in other results found in bibliography,[33] showing a strong dependence on the properties of the AC used. Thus, the higher the mesoporosity of the AC used, the larger the loss in the composites adsorption properties. Finally, selecting the porosity of the activated carbon, it is possible to prepare fabric/AC composites with higher adsorption capacity than the selected commercial sample, chosen due to its well known good performance.

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