High Value Activated Carbons from Waste Polystyrene Foams

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Abstract

Activated carbons (AC) with excellent textural properties have been obtained for the first time from waste polystyrene foam (PF), without any previous treatment, following a simple and conventional two-step procedure (formation of char followed by chemical activation). Even considering that the PF is not a graphitizable material, the best AC produced from this precursor has a very high BET surface area larger than $2700 \text{ m}^2 \text{ g}^{-1}$ and a pore volume of $1.2 \text{ cm}^3 \text{ g}^{-1}$, with a significant contribution of small mesopores. As a consequence, this AC reveals a surprising capacity to adsorption of relatively large molecules and a high specific capacitance when applied as a supercapacitor electrode. The maximum amount of adsorbed methylene blue obtained by batch equilibrium experiments are greater than 1 g g^{-1} . In the context of the technical difficulties and low economic return of the reuse and recycling of waste PF, this work offers a strategic destination for this environmentally unfriendly residue.

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1. Introduction

Activated carbons are commonly used products that have been produced from a large number of different precursors, which may be mineral, biological or synthetic [1-3]. Increasing concern about sustainability in our society is encouraging the production of re-usable materials obtained not from natural resources but from waste [4, 5]. Millions of tons of plastic waste are discarded each year, most of which is either incinerated or dumped in landfills [6]. Many efforts have been made by researchers to use these wastes as feedstock for the production of value-added products, such as fuels, high added-value carbons (e.g. carbon nanotubes) or large surface area carbons [6-9]. In recent years there has been considerable research in the area of activated carbon production from plastic wastes, as a result of which products with large surface areas and pore volumes have been obtained [6]. A survey was recently published on the use of various plastics to produce activated carbons with special focus on the use of PET [6, 7].

Polystyrene (PS) is an abundant plastic material (with annual production estimated at about 2.95 million tons) that generates a very appreciable volume of waste [10]. PS is produced in a variety of solid and foams and it is used in several applications. As an example, the expanded polystyrene foam (EPF) is frequently employed as packaging for different goods [11]. Unfortunately, this material is difficult to recycle and their reuse or recycling has little business interest due to low economic return [11, 12].

Different forms of polystyrene have been used to produce activated carbons, not as precursor material but as sacrificial hard template to obtain hollow spherical sludge carbon [13], or as a thin film over magnesium oxide to obtain a porous carbon film [14], or a copyrolysis agent together with biomass [15] or as a catalyst support [16]. It is

important to note that to date no attempts to directly convert this material into activated carbon have been reported. Wang et al. used polystyrene-based macro-reticular ion-exchange resin spheres, which is a copolymer of polystyrene and divinylbenzene functionalized with sulfuric acid, as precursor [17, 18]. During the pyrolysis/carbonization, the C-C linking bonds between the monomers break releasing small molecules and aromatic hydrocarbons which are distilled from the system. The use of copolymers or structure modifications (acidic pre-oxidation) strengthens the link between the monomers favoring the aromatization reactions and the formation of char [19]. Otherwise, only liquid and gas products are obtained in the polystyrene pyrolysis [20]. In this study, this problem is solved by using a simple two-step conventional process where first char is produced from waste polystyrene foams (PF) under Ar pressure to maintain the volatile compounds in the system until their total aromatization. Then, the char obtained is chemically activated to yield ACs with a very large surface area and high adsorption capacity. Finally, the produced ACs were characterized by several techniques and their adsorption capacity was measured using methylene blue (MB) as model molecule. Also, the electrochemical performance of these materials was tested for their application as electrodes in supercapacitors.

2. Experimental

Initially, waste EPF without any previous treatment was used as precursor for the production of char. 10 g of EPF that was first dissolved in acetone and then dried by means of a hot air gun, was introduced into a vertical furnace and pyrolyzed at a relatively low temperature (530 °C), at a heating rate of 10 °C min⁻¹ and under Ar at a pressure of 10 bar for 5 h. Acetone was only used to reduce the polymer volume, as air bubbles are removed from the expanded polymer. Thereby, the amount of PF carbonized per batch is increased.

In a second step the char thus produced (PF-Char) was subjected to chemical activation using anhydrous KOH as activation agent, with a KOH to PF-Char ratio of 4:1. Three activated carbons (ACs) were prepared by applying different activation temperatures (600, 700 and 800°C) in a horizontal furnace for 1 h. After this process, the products were washed in a 10% HCl solution and then in distilled water in a Soxhlet apparatus until a pH 7 was obtained.

The textural characteristics of the carbon materials were obtained from their N₂ isotherms at 77 K, which were carried out in a Micromeritics ASAP 2420. The C, N and H content were quantified from the combustion (at 1050 °C) of 1 mg of the corresponding sample by means of a LECO-CHNS-932 microanalyzer. The total oxygen content of the samples was determined directly in a LECO-TF-900 furnace coupled to the previous equipment. The electrical conductivity was measured on the shaped discs prepared by mixing 90 wt% of the carbon material and 10 wt% of polytetrafluoroethylene using a four-point probe system (Keithley Instruments, Model 238 High Current Source Measure Unit, USA). SEM images were obtained using a field emission gun scanning electron microscope (QUANTAN FEG 650, FEI) operating at 5 kV. TEM observations were performed on a JEOL 2000 EX-II instrument operating at 160 keV. Suspensions of ACs were deposited on standard holey carbon copper grids using the drop cast method and loaded into the microscope.

MB adsorption tests were performed by introducing 20 mL of MB solution (2.0 M) into tubes which contained 15 mg of each AC. The tubes were shaken and placed vertically in a thermostatically controlled bath in the absence of light at 298.15 K for 24 h. The amount of adsorbed MB was directly calculated from the difference between the initial and final concentrations in the supernatant. These concentrations were determined

by absorption measurements (663 nm) using a Shimadzu spectrometer UV-2550 (Japan).

The electrochemical performance of the ACs was studied on pellet type electrodes prepared by pressing a mixture of AC (90 wt.%) and PVDF (10 wt.%). The mass of each electrode was about 30 mg. Supercapacitors were built with glass fiber separators and gold current collectors, using a "T-type" Swagelok[®] cell. To record the potentials of the electrodes, a Ag/AgCl/3.5M KCl reference electrode was used as a third-end connection and the tests were performed in 1 M H₂SO₄. The electrochemical measurements tests were carried out at ambient temperature in a BioLogic VMP Multichannel Potentiostat/Galvanostat.

3. Results and discussion

The N₂-adsorption/desorption isotherms obtained for AC600, AC700 and AC800 are presented in Fig. 1(A). All the ACs show type 1 isotherms (IUPAC system [21]), characteristic of microporous materials. The highest temperature (800 °C) favored the formation of a material with a wider pore size distribution (PSD), as indicated by the wide knee observed in the isotherm. The PSD calculated by QSDFT corroborates these results (Fig. 1(B)), as it can be seen that the AC obtained at 800 °C has pore diameters within the range of supermicropores and even a small proportion of narrow mesopores.

Fig. 1(A) and 1(B)

The main textural parameters of the ACs are summarized in Table 1. The BET surface areas are in all cases larger than 2000 m² g⁻¹ and increase with the activation temperature to over 2700 m² g⁻¹ in the activated carbon obtained at 800 °C. A similar trend was observed for the pore volume. Sample AC800 shows the largest pore volume

($1.2 \text{ cm}^3 \text{ g}^{-1}$) with a very significant contribution of mesoporosity ($0.27 \text{ cm}^3 \text{ g}^{-1}$). The mesopore contribution to the pore volume is 2.5 higher than in the AC600 sample.

Table 1

From the mesopore volume contribution (Table 1) and the DFT curves, that in the case of AC800 show a significant contribution of supermicropores, it can be inferred that this carbon would be especially suitable for the adsorption of large molecules, such MB used here as model molecule. The maximum amounts adsorbed by AC600, AC700 and AC800 were 270, 536 and 1042 mg g^{-1} respectively. The AC obtained at 800°C evidenced an excellent adsorption performance with an outstanding amount of MB adsorbed, one of the highest, in fact, ever published in the literature (Table 2) [23-33].

Table 2

Other non-carbon materials are known for their good adsorption properties. Metal organic frameworks (MOF's) reach adsorption values for MB close to the results presented above [31]. Yet, compared to the ACs production, MOFs synthesis is usually more expensive and time consuming. In addition, Zeolites and Mobil Composition of Matters (MCM) adsorption capacity of MB do not reach the values found in this study [32, 33].

Increasing the activation temperature also affects the chemical composition of the carbon materials and, therefore, their surface chemistry. Table 3 shows the elemental compositions and electrical resistivity for the different ACs studied. It is worth noting that the functional group content of the activated carbons decreases drastically with an increase of 200°C in the activation temperature. At 600°C the oxygen content remains rather high (13.2%) since the oxidation of the carbon material progress slowly and some intermediate oxidation species still remain in the carbon. Higher temperatures favor the decomposition of these groups (which are eliminated in the form of CO or CO_2)

resulting in a decrease in the oxygen content and in a further development of the porosity.

Table 3

The samples were further characterized by means of SEM and TEM. Images corresponding to sample AC800 are shown in Fig. 2.

Fig. 2(A) and 2(B)

SEM studies indicate the presence of large particle ($< 200 \mu\text{m}$) and a good population of smaller particles, all of which have smooth surfaces. TEM studies evidence the typical highly disordered structure of non-graphitizable carbons, with small individual layers that are twisted, bent and randomly arranged, resulting in the high surface area measured by nitrogen adsorption. Nevertheless, although TEM does not show any significant changes in the structural arrangement of these samples, it is known that activated carbons obtained at higher temperature present a more organized structure, as has been demonstrated by Ruiz et al. [34], probably due to the loss of oxygen and a low-range structural rearrangement. This is reflected in the electrical resistivity (ρ), which is also an important property for materials to be used in energy storage devices. For the samples under study it varies from more than $1000 \Omega \text{ cm}$ for AC600 to just $5.5 \Omega \text{ cm}$ for AC800 (Table 3).

To exemplify the potential use of these ACs in energy storage, symmetrical supercapacitors were constructed with these materials derived from PF. The best result is obtained with AC800 due to its highest S_{BET} and low resistivity. The specific capacitance achieved by this electrode exceeds 250 F g^{-1} , values similar to those obtained with the best electrodes based on mesophase-derived activated carbon [34]. The electrochemical properties for all cells constructed in this work are summarized in the supporting information (Fig. S1 and Table S1).

The excellent adsorption results obtained for MB envisages a good adsorption for other dyes and pharmaceutical contaminant molecules that resembles the MB molecule size and structure [35]. Also, adsorption/storage of gases such as methane and CO₂ has a great dependence on the adsorbents microporosity volume, suggesting another possible application for these materials [36, 37]. Furthermore, our process does not differ considerably from the conventional process that is already used in industry for the production of ACs with high surface areas, that is, pyrolysis of the precursor followed by chemical activation using KOH [38]. In comparison to the process described in this paper, the major difference is that pressure is used during pyrolysis. Therefore, although PF is a non-graphitizable material, ACs synthesized from this precursor presents outstanding physicochemical properties, with equal or even better results than most of the graphitizable precursors offering an interesting destination for waste PF [4, 24, 39, 40].

4. Conclusion

In conclusion, activated carbons with very remarkable properties have been obtained for the first time from waste-expanded polystyrene foam, offering a new alternative for the recycling of this environmentally unfriendly residue. The surface area of the activated carbon produced from PF at 800 °C exceeded 2700 m² g⁻¹ and the relatively wide microporosity/small mesopososity showed a great ability to adsorb organic molecules like MB (1042 mg g⁻¹). The best material activated at 800 °C still presents low resistivity and is strategic as supercapacitor electrodes, reaching specific capacitance values of 250 F g⁻¹. Finally, with these results we hope to leverage the attention of specialists and encourage the use of waste PF to produce ACs on large

scales in place of other precursors with less environmental appeal. This could also be a possible and simple solution to add value to the recycling of this material.

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Table 1. Textural parameters for the ACs obtained at different activation temperatures.

Material	S_{BET} ($\text{m}^2 \text{g}^{-1}$)	V_{total} ($\text{cm}^3 \text{g}^{-1}$)	V_{micro} ($\text{cm}^3 \text{g}^{-1}$)	V_{meso} ($\text{cm}^3 \text{g}^{-1}$)
AC600	2109	0.88	0.77	0.11
AC700	2315	0.96	0.84	0.12
AC800	2712	1.2	0.93	0.27

S_{BET} , BET apparent surface area; V_{micro} , volume of micropores; V_{total} , Total volume of pores; V_{meso} , volume of mesopores

Table 2. Previously reported adsorption capacities of various adsorbents for methylene blue.

Adsorbent	Adsorption capacity (mg g^{-1})	Ref.
Graphene oxide	243.9	[24]
Carbon nanotubes	99.8	[26]
Activated carbon	435	[27]

Mesophase pitch-derived AC	950	[23]
Pyrophyllite	70.4	[28]
Calcium alginate/MWCNTs	606.1	[29]
Diatomite	156.6	[30]
MOF [NH ₂ -MIL-125(Ti)]	862	[31]
Zeolite (NZVI/SuZSM)	87,0	[32]
AC800	1042.0	This work

Table 3. Elemental composition and electrical resistivity for the ACs obtained at different activation temperatures.

Material	C (wt%)	H (wt%)	N (wt%)	O (wt%)	ρ (Ω cm)
AC600	86.65	0.76	0.14	13.18	1045.09
AC700	90.83	0.43	0.13	8.35	63.64
AC800	94.36	0.22	0.17	3.83	5.52

Figure captions:

Fig. 1. (A) N₂ adsorption-desorption isotherm at 77 K up to 1 bar for the AC materials. (B) Pore size distribution by applying Quenched Solid Density Functional Theory analysis to the N₂ adsorption-desorption isotherm (QSDFT) for the three materials.

Fig. 2. SEM (A) and TEM (B) images of AC800.