



Properties and performance of mesoporous activated carbons from scrap tyres, bituminous wastes and coal



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HIGHLIGHTS

- Activated carbons were prepared from tyre wastes and its blends with coal and a bituminous waste.
- The carbons were useful for the adsorption of large molecules.
- Their surface characteristics of ACs make them suitable for the adsorption of anionic dyes.
- Congo red dye was successfully adsorbed on tyre waste-derived activated carbons.

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ABSTRACT

Tyre wastes and their blends with coal and a bituminous waste material obtained from the benzol distillation column of a by-product section of a coking plant were employed as a precursor for the production of activated carbons (ACs). Pyrolysis up to 850 °C followed by physical activation with CO₂ produced mesoporous carbons with different pore size distributions and surface areas. The surface chemistry of the samples was studied by measuring the point of zero charge (pH_{pzc}) and by temperature programmed desorption (TPD). The activated carbons obtained contained higher amounts of basic functional groups. Their textural and surface chemistry characteristics make them highly suitable for adsorbing anionic dyes of large molecular size, such as Congo red. The adsorption kinetics was found to conform closely to the pseudo-second-order kinetic model. To determine the adsorption mechanism, the kinetic data were also analyzed using the Weber and Morris intraparticle diffusion model and the Boyd model to distinguish between the pore and film diffusion steps. The equilibrium isotherms were of the Langmuir isotherm type. The efficiency of the low-cost ACs prepared for the removal of Congo red dye was similar to that reported in the literature for coal-based ACs and greater than that of other low-cost ACs.

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1. Introduction

Effluents from the textile, plastic, food, cosmetics or paper industries contain dyes that need to be removed to avoid water contamination. The chemicals present in the waste range from organic to polymers or inorganic compounds. In addition even very low concentrations of dyes are visible and therefore need to be eliminated. Dyes are non-biodegradable or photo-degradable. Moreover the decolouration of textile dye effluents does not occur when treated aerobically by municipal sewerage systems, and some are known to be carcinogenic and mutagenic. These industries consume an enormous amount of water that needs to be treated, posing a serious economic and environmental problem.

Although activated carbons may be used to treat these effluents, the cost of such adsorbents is high. Hence there is a need to find low-cost sorbents from wastes [1–4]. The wastes used so far to prepare what are often referred to as “unconventional” adsorbents include coir pitch [5], fly ashes [6,7], orange peel [8], sawdust [9–11], palm shell [12], rice husk [9,13,14], etc.

The porous structure of activated carbons is the principal physical characteristic that will determine their performance as adsorbents. In addition the presence of surface groups containing heteroatoms will confer different chemical properties that will decide their final application. The polarity, solubility and molecular size of the adsorbate and the pH of the solution are other important factors that need to be taken into account when assessing the effectiveness of an adsorbent [15]. There are many structural varieties of dyes that can be classified as either cationic, nonionic or anionic. This is one of the reasons why it is important to study the surface chemistry of the activated carbons used to eliminate them.

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In addition the economic and environmental problems associated with the generation of scrap tyres (End-of-Life-Tyres, ELTs) are increasing due to the enormous generation of such wastes. Pyrolysis is considered as an optimal and environmentally friendly method for the thermo-chemical conversion of wastes such as tyres. Three products can be obtained from pyrolysis: gas with a high calorific value, pyrolytic oil that can be used as fuel and as a source of benzene, toluene, xylene (BTX) or limonene and char that can be used as fuel, adsorbent or carbon black [16–21]. In order to obtain products with a high percentage of carbon and a low ash content, co-pyrolysis with coal or bituminous wastes is a good option [22–24]. The co-processing of tyre crumbs with coal has been studied as a way to improve coal liquefaction and hydro-pyrolysis [22,23,25,26]. However until now little work has been carried out on the co-pyrolysis of tyre wastes (reinforcing fiber and tyre crumbs) with coal or with a bituminous waste [24].

The objective of the present study is to investigate the mechanism of the adsorption of Congo red dye by activated carbons that present different porous textures and surface chemistry characteristics and were prepared from blends of two tyre wastes with coal and a bituminous residue.

2. Materials and methods

2.1. Materials

Three components are derived from the grinding of scrap tyres: tyre crumbs, reinforcing fiber and steel. In the present study tyre crumbs and reinforcing fiber were used to prepare five tyre waste-based activated carbons (ACs).

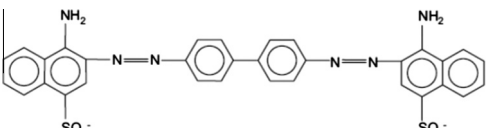
The activated carbons were obtained by means of pyrolysis in a rotary oven as explained in a previous paper [23] followed by physical activation carried out in the rotary oven at 850 °C with a flow of 250 ml/min of CO₂ to a burn-off of 65 ± 5%. The wastes used as raw materials for the production of the ACs were: Tyre crumbs (TC) and reinforcing fibers (RF) derived from grinding End-of-Life-Tyres (ELTs), 1:1 blends of TC and RF with a low rank coal, and 1:1 blends of RF with a bituminous waste material (BWM).

The activated carbons (ACs) were milled and sieved to <0.100 mm for the elemental and proximate analyses and to between 0.5 and 0.1 mm for the adsorption analysis.

Ash content was determined following the ISO1171 standard procedure. The elemental analysis was carried out using a LECO CHN-2000 instrument for C, H and N analysis, a LECO S-144 DR device for sulfur analysis and a LECO VTF-900 instrument for direct oxygen determination.

The Congo red anionic acid dye (CR) is commonly used in the textile industry to confer a red color. The physical properties of this dye are listed in Table 1.

Table 1
Physical properties of Congo red.

Molecular weight (g/mol)	650.7
Width (nm)	2.62
Depth (nm)	0.74
Thickness (nm)	0.43
pK _a	5.5
Molecular structure:	
	

2.2. Textural characterization

The textural properties of the ACs were studied by means of N₂ adsorption at 77 K on a Micromeritics ASAP 2420 apparatus. The software package provided with the equipment was used to determine the BET surface area (S_{BET}) and the total pore volume (V_T) at $p/p_0 = 0.96$. The micropore volume (V_{DR,N_2}) was determined by applying the Dubinin–Radushkevich (D–R) equation to the lower relative pressure zone of the isotherm. The mesopore volume (V_{meso}) was calculated by subtracting the micropore volume from the total pore volume (V_T). The average width of the micropores (L_{0,N_2}) of the ACs was calculated following the procedure of Stoeckli [27]. The Kelvin condensation theory was employed to examine the mesopore volume distribution [28]. The mesopore fraction was expressed as the ratio of the mesopore volume to the total pore volume (V_{meso}/V_T).

The samples (approximately 0.25 g) were degasified under vacuum at 200 °C for 12 h prior to N₂ adsorption to eliminate any moisture and condensed volatiles. The IUPAC pore size classification that assigns a size of 2–50 nm to mesopores and a size <2 nm to micropores was used.

2.3. Surface chemistry

The pH at which the sorbent surface charge has a zero value, is referred to as the point of zero charge (pH_{pzc}). At this pH, the charge of the positive surface sites is equal to that of the negative ones.

The point of zero charge determines the surface charge of the sorbent at a given pH and this information reveals the possible electrostatic interactions between the sorbent and chemical species [29]. The pH_{pzc} of the ACs was determined according to the procedure described by Moreno-Castilla et al. [30].

TPD analyses were carried out in an Autochem II apparatus (Micromeritics). The samples were heated at a constant heating rate of 10 °C/min up to 1000 °C under a He flow of 50 cm³/min. The desorbed gases were monitored using an Omnistar (Pfeiffer Vacuum) mass spectrometer.

2.4. Adsorption test

In order to determine the equilibrium time, adsorption experiments were carried out by shaking 50 mg of adsorbent mixed with 100 cm³ of dye solution in a concentration of 50 mg/dm³ at 25 °C in an orbital shaker.

The concentration of the dye was measured using a U-2800A, Hitachi UV–Vis spectrophotometer at a wavelength 497 nm. A digital pH meter (Mettler Toledo) with a glass electrode was employed to measure the pH before and after contact.

Lagergren pseudo-first-order and pseudo-second order models and the intraparticle diffusion model were applied to describe the CR adsorption process.

The Lagergren pseudo-first order model can be described by Eq. (1), and pseudo-second order model by Eq. (2).

$$\frac{dq}{dt} = k_1(q_e - q) \quad (1)$$

$$\frac{dq}{dt} = k_2(q_e - q)^2 \quad (2)$$

Upon integration, Eqs. (1) and (2) may be written as:

$$q = q_e(1 - e^{-k_1 t}) \Rightarrow \log(q_{e,exp} - q_t) = \log q_e - (k_1 t)/2.303 \quad (3)$$

$$q = \frac{k_2 q_e^2 t}{1 + k_2 q_e t} \Rightarrow t/q_t = 1/(k_2 q_e^2) + t/q_e \quad (4)$$

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