



# Texture and surface chemistry of activated carbons obtained from tyre wastes



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## ABSTRACT

Tyre wastes and their blends with coal and a bituminous waste material obtained from the benzol distillation column of the 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 CO<sub>2</sub> yielded mesoporous carbons with different pore size distributions and surface areas depending on the degree of burn-off. ACs with surface areas of 475 and 390 m<sup>2</sup>/g were obtained for the two tyre wastes. The inclusion of coal in the blend gave rise to surface areas of up to 1120 m<sup>2</sup>/g due to an increase in the microporosity. The time needed to obtain the desired degree of burn-off depended on the reactivity of the char. The coal-containing materials required the longest amount of time. The surface chemistry of the samples was studied by Infrared spectroscopy (FTIR) and X-Ray photoelectron spectroscopy (XPS). The principal oxygenated groups found were quinones, lactones and carboxylic acids.

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## 1. Introduction

The preparation of activated carbons has been the subject of extensive research study for many years [1–13] due to the diversity of their applications. Mainly though, activated carbons have been used as adsorbents and catalysts [14–19].

Although it is the porous structure of activated carbons that will determine their performance, the presence of surface groups containing heteroatoms will confer upon them different chemical properties that will contribute to determining their final application.

Activated carbons can be produced from a range of carbonaceous materials including coal or biomass (coconut shell, wood, coir pitch, cellulosic waste orange peel, sawdust, rice-husk, etc.) [20–25]. Waste tyres represent another valuable source of activated carbons because of their high carbon content [26]. This would also provide a way of recovering waste tyres, the generation of which is constantly increasing, causing numerous economic and environmental problems. Tyre material recycling is normally carried out by means of a shredding procedure that yields granulated rubber as its product and steel and reinforcing fibre in the form of fluff as sub-products. Rubber crumbs are applied in sports surfaces and as an additive for asphalt, etc. but so far no real use has been found for the fluff.

The pyrolysis of tyre wastes yields three products: gas with a high calorific value, pyrolytic oil that can be used as fuel and as a source of benzene, toluene, xylene (BTX), and limonene and thirdly char that can be used as fuel, adsorbent or carbon black [27–32]. In order to obtain products with a high percentage of carbon and low ash content,

co-pyrolysis with coal or bituminous wastes is a good option [26,33,34]. The co-processing of tyre crumbs with coal has also been studied as a way to improve coal liquefaction and hydro-pyrolysis [26,33,35,36]. However until now little work has been carried out on the co-pyrolysis of tyre wastes (reinforcing fibre and tyre crumbs) with coal or with a bituminous waste [34].

The objective of the present study is to investigate the activation of chars obtained from two tyre wastes and their blends with coal and a bituminous residue that have different porous textures and surface chemistry characteristics.

## 2. Materials and methods

### 2.1. Materials

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

The wastes used as raw materials for the production of the ACs were: tyre crumbs (TC) and reinforcing fibres (RF) derived from the grinding of 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 chars were produced in a rotary oven (5 °C/min up to 850 °C with a soaking time of 30 min) and in a nitrogen atmosphere as explained in a previous paper [26]. Physical activation was also carried out in the rotary oven at 850 °C with a flow of 250 ml/min of CO<sub>2</sub> for various periods of time in order to obtain different degrees of burn-off expressed on an ash free basis (B.O.) throughout the manuscript.

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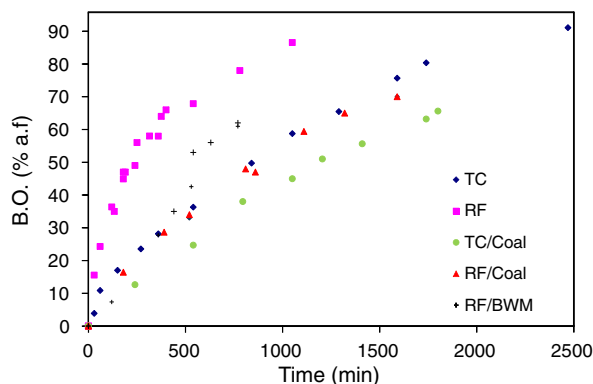


Fig. 1. Variation of carbon burn-off (B.O.) with time.

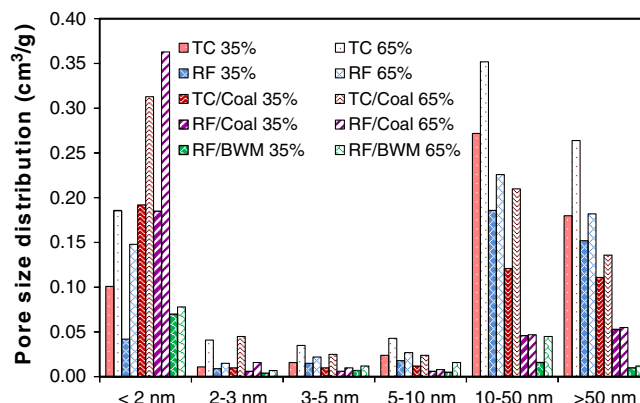


Fig. 3. Pore size distribution by Kelvin for ACs prepared at 35% and 65% B.O.

Table 1

Elemental analysis and ash content for various degrees of activation.

Sample	B.O.*	Cz (wt.% db)	C (wt.% db)	H (wt.% db)	N (wt.% db)	S (wt.% db)	O (wt.% db)
TC	33%	22.3	75.8	0.3	0.3	2.9	8.2
	47%	27.9	69.8	0.3	0.6	3.7	2.0
	70%	37.8	58.7	0.2	0.5	4.2	1.6
RF	36%	10.4	82.6	0.4	0.4	2.6	5.7
	47%	14.9	80.8	0.5	0.6	2.9	3.8
	56%	16.9	80.4	0.5	0.5	2.7	3.6
TC/Coal	66%	19.7	78.6	0.2	0.5	3.30	2.0
	38%	15.5	79.7	0.6	1.3	1.7	3.6
	51%	17.2	78.3	0.5	1.3	1.9	3.0
RF/Coal	66%	23.1	73.2	0.2	1.1	2.3	0.6
	34%	13.0	82.1	0.5	1.7	1.3	3.0
	47%	17.5	78.5	0.5	1.6	1.8	3.4
RF/BWM	59%	15.3	81.9	0.4	1.6	1.7	0.6
	65%	19.7	78.1	0.4	1.2	2.3	-
	35%	5.0	86.0	0.6	1.9	2.0	3.2
RF/BWM	56%	15.4	81.0	0.5	1.6	2.6	3.4
	61%	12.3	82.3	0.5	1.5	2.8	2.6

\*B.O.: burn-off expressed on an ash free basis.

The elemental analysis was carried out using a LECO CHN-2000 instrument for the C, H and N analysis, a LECO S-144 DR device for the sulphur analysis and a LECO VTF-900 instrument for direct oxygen determination.

## 2.2. Textural characterization

The textural properties of the ACs were studied from N<sub>2</sub> adsorption isotherms 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  $p/p_0 = 0.97$ . The micropore volume ( $V_{micro}$ ) 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 Kelvin condensation theory was employed to examine the mesopore volume distribution [37]. The samples (approximately 0.25 g) were degassed under vacuum at 200 °C for 12 h prior to N<sub>2</sub> 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 of <2 nm to micropores was employed.

## 2.3. Surface chemistry

Fourier transform infrared spectroscopy (FTIR) spectra were recorded on a Nicolet Magna-IR560 spectrometer equipped with a DTGS detector that operates at ambient temperature. Samples were prepared in the form of pellets using KBr as a matrix, in a ratio of 1 mg of sample to 600 mg of KBr. The pellets were dried at 120 °C for 24 h before analysis. The spectra were recorded from 4000 to 400 cm<sup>-1</sup> by 128 interferograms at a resolution of 4 cm<sup>-1</sup>.

X-Ray photoelectron spectroscopy (XPS) measurements were taken on a SPECS spectrometer equipped with a Phoibos 100 hemispherical analyser. The X-ray radiation source was a monochromatic Al K $\alpha$  (1486.74 eV) with a 100 W X-ray power and an anode voltage of 14.00 kV. The photo-excited electrons were analysed in constant pass energy mode, using pass energy of 50 eV for the survey spectra and 10 eV for the high resolution core level spectra. CasaXPS software was employed for data processing. The compositions in atomic percentage (at.%) were determined from the survey spectra on the basis of the integrated peak areas of the main XPS peaks of the different elements (C(1s), N(1s), O(1s), S(2p) and Si(2p)) and their respective sensitivity factors.

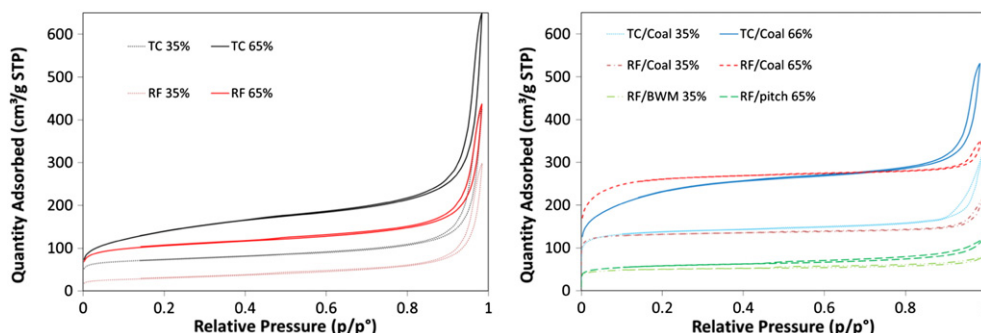


Fig. 2. Adsorption isotherms of nitrogen at 77 K for ACs prepared at 35% and 65% B.O.

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