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ADSORPTION OF ORGANIC COMPOUNDS FROM SOLUTION BY ACTIVATED CARBONS PRODUCED FROM WASTE TYRE RUBBER

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ABSTRACT

Activated carbons produced from waste tyre rubber have shown exceptional characteristics for the adsorption of organic species from solution. Adsorption capacities were found to be dependent primarily on the degree of activation and the molecular size of the adsorbate compound. For the purpose of this work, a series of activated carbons were produced by steam activation of waste tyre rubber at 925°C over a period of 80–640 min. The resulting carbons were investigated for their porosity, using nitrogen gas adsorption, and for their aqueous adsorption characteristics, using phenol, methylene blue, and textile dyes Turquoise H-A and Red H-E2B. Two widely used commercial adsorbents were also tested for comparative purposes. Aqueous adsorption data were modeled to the Langmuir equation in order to determine the adsorption

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capacities (X_m) and affinity parameters (b) associated with each sample. Rubber-derived carbons proved superior to the commercial adsorbents for the removal of medium and large molecular weight compounds from solution, which was attributed to their extensive total micropore volume and external surface area.

Key Words: Activated carbon; Adsorption; Dyes; Pyrolysis; Tyres

INTRODUCTION

The United Kingdom produces around 400,000 t/year (1) of used tyres and over 2,500,000 t/year are produced in the US (2). The generation of this residue represents a major environmental problem, which still remains to be solved. Owing to their resistance to degradation and mechanical characteristics, landfilled tyres are susceptible to being displaced by more dense materials, which results in the disruption of compacted landfill sites. Buried or dumped tyres also represent a serious fire hazard. Fires in tyre deposits have been reported to generate high levels of pollution to the atmosphere, soil, and water (3,4).

Various recycling alternatives are being adopted, the most successful being retreading, incineration with energy recovery, and granulation (1,2). Despite the environmental problems associated with this activity, incineration, either in purposely built power stations or in cement kilns, is becoming the main disposal route for this waste stream. Furthermore, it has been argued that incineration may not maximize the potential economic value of waste tyres (1,2,5).

Pyrolysis is currently receiving renewed attention as a commercial alternative to tyre disposal and incineration. The process involves heating the tyres to temperatures around 700°C under inert atmospheric conditions using batch processes or rotary kilns. This results in the decomposition of the rubber polymer and the formation of a hydrocarbon oil, a high calorific gas and a carbonized char (1,6). The oil has been suggested for use as a fuel or as a source of refined chemicals, while the pyrolysis gas can be recycled on-site to self-fuel the process (7–10). The use of the pyrolytic char for the production of activated carbon has been investigated by a number of authors (11–16). These carbons have been reported to exhibit extensive pore structures and BET surface areas up to 1000 m^2/g.

Rubber-derived carbons have low mechanical strength and are generated mainly in powdered form, which makes them best suited for use in liquid phase applications. The presence of inorganic impurities in the rubber feed, particularly zinc and sulphur, has created concerns about their use for the treatment of potable waters. However, recent studies have shown that the availability of these species in neutral pH conditions is very limited (17). Although rubber derived carbons may still not be acceptable in applications with high purity requirements, they are very well suited for other commercial uses such as the treatment of industrial or municipal effluents (18).

Little work has been produced on the aqueous adsorption characteristics of activated carbons produced from waste tyre rubber. Previous studies reported in the literature have shown that tyre rubber-derived carbons exhibited high adsorption capacities for the removal of phenolic compounds from solution (19,20). However, these results are based on the analysis of a small number of carbon samples (two at the most) which exhibited BET surface areas significantly inferior to those reported by other authors (up to $350\,\mathrm{m}^2/\mathrm{g}$).

This paper provides a systematic investigation of the adsorption characteristics exhibited by activated carbons produced from waste tyre rubber. The results can be used to determine the potential use of these carbons in industrial applications that require the removal of a range of impurities from solution. Rubber-derived carbons produced to different degrees of burn off were investigated for their porosity and surface characteristics using nitrogen gas adsorption. Subsequently, the carbons were investigated for their aqueous adsorption characteristics using four organic species of different molecular size: phenol, methylene blue, and textile dyes Procion Turquoise H-A and Procion Red H-E2B. Experimental data were modeled to the Langmuir equation to determine the adsorption capacities (X_m) and affinity parameters (b) associated with each sample. Adsorption and porosity results were compared with those obtained from two widely used commercial powdered active carbons (PAC).

EXPERIMENTAL

Tyre Rubber and Commercial Activated Carbons

The tyre rubber employed in this work was supplied by Duralay Ltd (Haslingden, UK) as a powder of particle size <0.42 mm. This material is generated as a by-product of the tyre shredding process. The characteristics of the tyre carbons were tested against two widely used commercial powdered carbons: Chemviron GW (Chemviron Carbon UK Ltd, Oldham, UK) and Hydrodarco-C (Norit UK Ltd, Glasgow, UK). The former is described by the manufacturers as a multipurpose activated carbon, suitable for the removal of both low and high molecular weight compounds from potable water and waste water. The latter is commercialized for the treatment of industrial and municipal effluents containing large molecular weight compounds.

Production of Rubber-Derived Activated Carbons

The tyre rubber was pyrolysed and steam activated in one step using a Carbolite HTR 11/150 laboratory scale rotary furnace described elsewhere (13). For the production of each sample, 200 g of rubber were heated at 5°C/min to 925°C. A flowing (500 mL/min) nitrogen atmosphere was used at temperatures below 700°C when the pyrolysis of the tyre rubber occurred. At temperatures above 700°C, the inert atmosphere was replaced by a 80/20 (v/v) mixture (500 mL/min total flow rate) of steam/nitrogen. To achieve these conditions, the nitrogen flow rate was reduced (100 mL/min) and distilled water (2.99 mL/min, equivalent to 400 mL/min at 1 atm and 20°C) was injected into the reaction vessel by means of a calibrated peristaltic pump. Activation at 925°C was conducted over 80–640 min to produce samples of different degrees of carbon burn off. When activation was completed, the peristaltic pump automatically switched off and the furnace was allowed to cool down under a continuous purge of nitrogen gas. Prior to analysis, the powdered carbons were ground and sieved to a particle size $<150\,\mu m$. The pyrolytic char produced at 700°C is referred to in this work as P_700. Individual steam activated carbons are identified as SP_925 followed by their activation time (in minutes).

Characterisation of Rubber Chars by Nitrogen Gas Adsorption

Carbon samples were characterized for their surface area and pore size distribution using continuous volumetric nitrogen gas adsorption at liquid nitrogen temperature. The analyses were conducted using a Coulter Omnisorp 100 automatic adsorption analyzer (Beckman Coulter Ltd, High Wycombe, UK). The samples were outgassed at 175°C for 6–8 hr to a vacuum of between 10^{-5}–10^{-6} Torr. Total surface areas were determined by application of the BET equation (21) to the adsorption data. The BET equation was applied in the p/p^0 range 0.015–0.15 and generated correlation coefficients $r^2 > 0.9999$. External surface areas (surface areas not associated with micropores) were determined by application of the *t*-plot model typically in the range 7–9 A (22). Correlation coefficients in this case were always $r^2 > 0.9995$. The Dubinin–Astakhov (DA) model was employed to characterize the microporosity of the tyre rubber carbons (23). The DA equation was applied in the pressure range p/p^0 0.0005–0.1 and generated correlation coefficients $r^2 > 0.9995$.

Determination of Aqueous Adsorption Characteristics

Procion Red H-E2B and Procion Turquoise H-A adsorption isotherms were produced by contacting 100 mg of carbon sample with 100 mL of dye solution of concentration 100–1000 mg/L. In the case of phenol and methylene blue, different masses of each carbon sample (25–600 mg) were contacted with 100 mL of phenol (235 mg/L) or methylene blue (561 mg/L) solution. All the carbons tested in this work were of an alkaline nature. In order to reduce the effect of pH conditions on the adsorption determinations, stock solutions contained 500 mg/L of sodium bicarbonate buffer and were adjusted to pH 6.8 ± 0.2 by addition of concentrated hydrochloric acid.

Carbon mixtures were shaken end-over-end for 24 hr in a temperature-controlled room at 20°C. Solutions were subsequently filtered through Whatman WCN 0.45 μm membranes (textile dyes) or Whatman No. 1 (phenol and methylene blue). Residual concentrations were determined using a Perkin-Elmer Lambda 3 UV-VIS spectrophotometer (Perkin Elmer Ltd, Beaconsfield, UK) at 265 nm (phenol), 661 nm (methylene blue), 622 nm (Procion Turquoise), and 510 nm (Procion Red). Adsorption results were modeled to the Langmuir equation (24) to determine adsorption capacities (X_{m}) and affinity values (b).

RESULTS AND DISCUSSION

Product Yields

For the production of each carbon sample, the tyre rubber (200 g at a time) was initially heated to 700°C under a continuous purge of nitrogen gas. Previous work had demonstrated that these conditions were sufficient to effect the complete pyrolysis of the tyre rubber (13) while the use of inert atmospheric conditions (nitrogen) ensured that no oxidation (activation) of the carbonized char occurred at this stage. Volatile species generated during this stage were removed from the furnace and condensed at room temperature in a glass trap. The product yields generated were determined as follows: 41 wt% of char, 53 wt% oil, and 6 wt% gas (by difference).

The pyrolytic chars were activated in steam at a temperature of 925°C using different reaction times between 80–640 min. The results in Table 1 show that activation resulted in a gradual reduction of the carbon mass which followed a linear relationship with the reaction time. Under the conditions described, the reaction rate was calculated at 4.32 g/hr and produced a correlation coefficient (r^2) of 0.993. Table 1 also illustrates the degree of activation associated with each carbon sample which represents the mass (wt%) of pyrolytic char oxidized during the activation process.

Table 1. Carbon Yields and Porosity Characteristics Exhibited by Activated Carbons Produced from Tyre Rubber

Sample Identifier	Degree of Activation (wt% Burn-Off)[a]	Carbon Yield (wt%)[b]	BET Surface Area (m^2/g)	External Surface Area (m^2/g)	DA Microporosity: Volume (mL/g)[c]	DA '*A*' (nm)[d]	DA '*n*'[e]
P_700	0	41	82	72	0.042	1.090	1.39
SP_925_80	12.5	35	283	92	0.122	0.682	2.49
SP_925_240	30.1	29	658	124	0.296	0.788	2.39
SP_925_400	43.9	23	859	154	0.443	0.884	1.56
SP_925_480	47.8	20	928	180	0.491	0.913	1.24
SP_925_640	63.2	15	1071	306	0.554	0.926	1.21
Chemviron GW	n/a	n/a	788	39	0.319	0.587	3.06
Hydrodarco-C	n/a	n/a	545	199	0.250	0.764	1.59

[a] Relative to mass of pyrolytic char.
[b] Relative to the initial mass of tyre rubber.
[c] DA = Dubinin–Astakhov.
[d] A = average half width of micropore.
[e] n = exponential value.

Porosity and Surface Characteristics

The aqueous adsorption characteristics exhibited by an activated carbon are largely predetermined by its surface area and porosity characteristics (18,24). For this reason, the activated carbons tested in this work were investigated for their pore structure using nitrogen gas adsorption. The results in Table 1 show that the P_700 is essentially a mesoporous material of limited BET surface area (82 m^2/g) and micropore volume (0.004 mL/g). Most of the surface area in this material is external (72 m^2/g).

Steam activation resulted in a continuous development of the BET surface area and micropore volume up to 1071 m^2/g and 0.554 mL/g, respectively. These values are significantly greater than those exhibited by the two commercial adsorbents investigated in this work. External surface areas also increased significantly with the degree of activation to reach values up to 306 m^2/g. This increase was particularly rapid at high degrees of activation, which was associated with the transformation of large micropores into mesopores due to the burn out of pore walls.

Results obtained by application of the DA equation show that the rubber carbons developed a narrow microporosity during the initial stages of the activation process. As illustrated in Table 1, the pores in the micropore range were progressively widened as evidenced by a gradual increase in the average micropore half width (A) and a reduction in the exponential values (n). One of the commercial adsorbents tested in this work, Chemviron GW, presented a significantly narrower microporosity than rubber-derived carbons of comparable BET surface area. The other one, Norit Hydrodarco-C, commercialized for the treatment of waste-water, was significantly less microporous but exhibited a large external surface area.

Aqueous Adsorption Characteristics of Rubber-Derived Activated Carbons

Narrow pores provide activated carbons with an extensive surface area where adsorption processes may take place. However, size limitations may prevent the access of large compounds into pores of small dimensions due to molecular sieve effects. In this respect, it has been reported that adsorption of organic compounds from solution is significantly reduced when the pore width is less than 1.5–2.0 times the critical diameter of the adsorbate molecule (25).

The adsorbate compounds employed in this work cover a wide range of molecular sizes, which makes them useful to investigate adsorption in pores of different dimensions. Phenol (94 Da) is preferentially adsorbed in small and medium sized micropores while methylene blue (394 Da) is mainly adsorbed in medium and large micropores (25). Owing to their larger molecular dimensions, adsorption textile dyes Procion Red H-E2B (1300 Da) and Procion Turquoise H-A (1231 Da) is expected to take place preferentially in larger micropores and small mesopores (26).

Figures 1–4 show the adsorption isotherms for phenol, methylene blue, Procion Red H-E2B, and Procion Turquoise H-A exhibited by the P_700, the tyre rubber activated carbons (SP_925) and the commercial adsorbents Chemviron GW (GW) and Hydrodarco-C (Hyd-C). Langmuir parameters for phenol and methylene blue are given in Table 2 and those for the textile dyes are presented in Table 3.

Rubber-derived P_700 presented a very limited adsorption capacity for small molecular sized phenol (6.8 mg/g), which was mainly attributable to its poorly developed microporosity. Results in Table 2 show that phenol adsorption increased rapidly during the initial stages of the activation process to reach capacity values in excess of 100 mg/g. This observation was associated with the formation of an extensive microporosity. However, activation in excess of 30–40 wt% (carbon burn-off) produced a less significant increment in the adsorption capacity for this compound. During this stage it is believed that the increase in the total micropore volume is counteracted by an enlargement in the average micropore width.

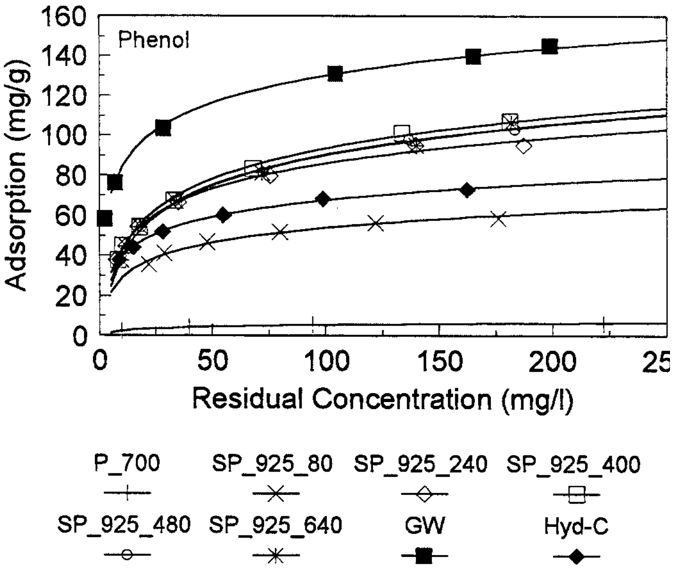

Figure 1. Phenol adsorption isotherms exhibited by rubber-derived and commercial activated carbons.

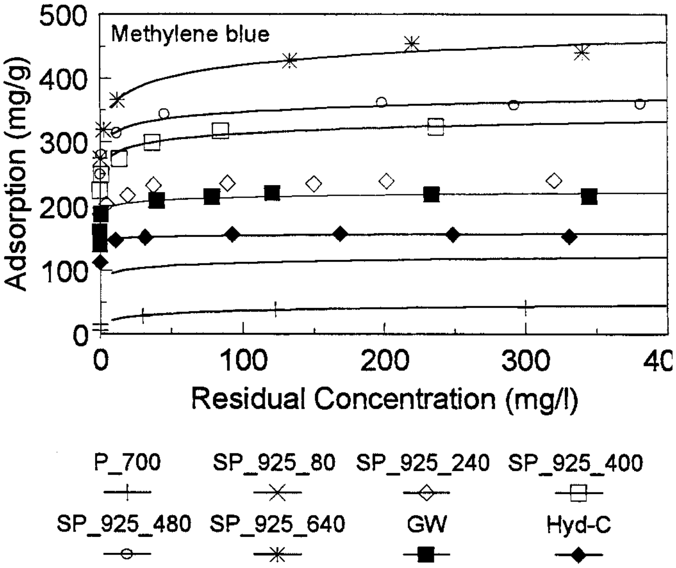

Figure 2. Methylene blue adsorption isotherms exhibited by rubber-derived and commercial activated carbons.

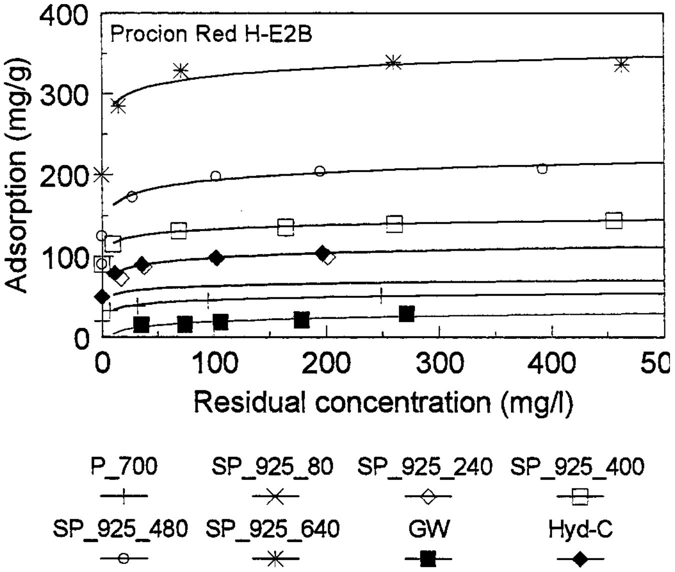

Figure 3. Procion Red H-E2B adsorption isotherms exhibited by rubber-derived and commercial activated carbons.

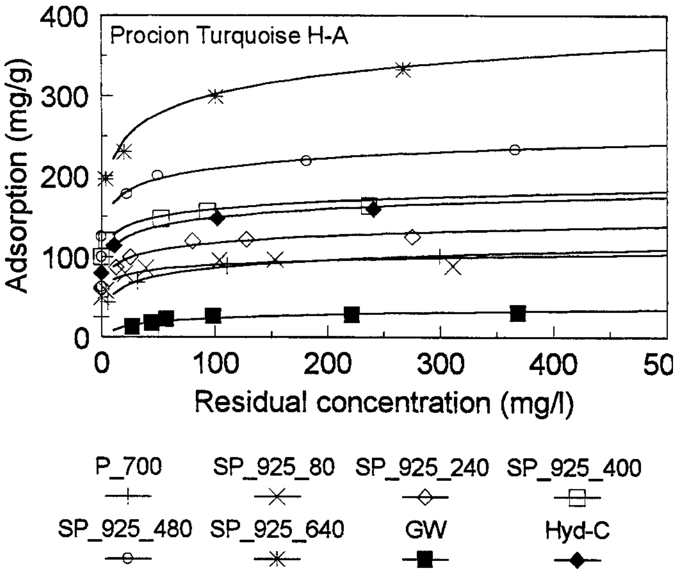

Figure 4. Procion Turquoise H-A adsorption isotherms exhibited by rubber-derived and commercial activated carbons.

Table 2. Langmuir Adsorption Parameters for the Adsorption of Phenol and Methylene Blue by Rubber-Derived Carbons

	Phenol			Methylene Blue		
	X_m(mg/g)	b	r^2	X_m (mg/g)	b	r^2
P_700	6.8	0.05	0.99	49.0	0.05	0.97
SP_925_80	64.2	0.06	0.99	125.0	0.60	0.90
SP_925_240	92.6	0.09	0.97	236.4	0.52	0.87
SP_925_400	105.3	0.07	0.98	324.7	0.37	0.99
SP_925_480	106.0	0.06	0.99	359.7	0.57	0.99
SP_925_640	108.7	0.06	0.98	448.4	0.40	0.96
Chemviron GW	130.7	0.27	0.92	215.1	0.67	0.90
Hydrodarco-C	71.2	0.12	0.96	156.7	1.39	0.94

Table 3. Langmuir Adsorption Parameters for the Adsorption of Textile Dyes by Rubber-Derived Carbons

	Procion Turquoise H-A			Procion Red H-E2B		
	X_m (mg/g)	b	r^2	X_m (mg/g)	b	r^2
P_700	92.3	0.16	0.96	46.9	0.32	0.85
SP_925_80	93.0	0.41	0.96	67.1	0.20	0.98
SP_925_240	127.2	0.16	0.99	104.2	0.14	0.99
SP_925_400	166.9	0.52	0.88	138.9	0.48	0.92
SP_925_480	232.0	0.14	0.97	211.0	0.17	0.99
SP_925_640	335.5	0.41	0.86	333.3	0.35	0.99
Chemviron GW	37.6	0.02	0.97	36.1	0.10	0.97
Hydrodarco-C	166.7	0.19	0.90	102.0	0.29	0.95

Despite its comparatively lower micropore volume and BET surface area, commercial adsorbent Chemviron GW exhibited a greater phenol adsorption capacity (130.7 mg/g) than any of the rubber-derived carbons (up to 108.7 mg/g). This observation may be attributed to the narrower microporosity exhibited by the commercial adsorbent, as described in Table 1. However, other aspects not investigated in this work, such as surface chemistry, may also have some influence in these results. The other commercial adsorbent, Norit Hydrodarco-C, which presented a more pronounced mesoporous character, was outperformed even by less activated rubber-derived carbons in terms of phenol adsorption capacity.

The P_700 presented a greater adsorption capacity for methylene blue with 49 mg/g. However, this value was largely exceeded by the more microporous commercial adsorbents. Results in Table 2 show that the adsorption capacity for this compound increased continuously with the degree of activation. It is believed that the increase in the total micropore volume and the progressive enlargement in the average micropore width both contribute positively to the adsorption of this compound. Highly activated rubber carbons exhibited methylene blue adsorption capacities up to 448 mg/g, which is twice as much as the best performing commercial adsorbent.

Results in Table 3 illustrate that Chemviron GW exhibited a very limited adsorption capacity for textile dyes Procion Turquoise H-A and Procion Red H-E2B. This behavior was largely attributed to molecular sieve effects. As discussed above, the porosity in this commercial adsorbent is mainly concentrated in the narrow micropore range and remains inaccessible to large molecular size species. Despite its reduced BET surface area and total micropore volume, Norit Hydrodarco-C was significantly more effective than Chemviron GW in the adsorption of both textile dyes.

The P_700 adsorbed 47 mg/g of Procion Red H-E2B and 92 mg/g of Procion Turquoise H-A. These values are significantly greater than those exhibited by Chemviron GW, but around 40–50% lower than those produced by the more mesoporous Norit Hydrodarco-C. Results in Table 3 show a marked increase in the adsorption capacities for both textile dyes as a result of steam activation. This increase was more pronounced at high degrees of activation, which was associated with the formation of larger micropores and the development of a more extensive mesoporosity. Highly activated rubber carbons adsorbed up to 335.5 mg/g of Procion Turquoise and 333.3 mg/g of Procion Red. These values are between two and three times greater than those exhibited by the best performing commercial adsorbent tested in this work.

CONCLUSIONS

Activated carbons produced from tyre rubber exhibited exceptional characteristics for the adsorption of organic compounds from solution, particularly those of medium and large molecular size. Such characteristics make these carbons very suitable for the treatment of effluents such as those generated by the dyeing textile industry. The porosity and adsorption characteristics of these carbons were found to be largely dependent on the degree of activation to which they had been produced.

Owing to its poorly developed micropore structure, pyrolytic char produced from tyre rubber presented a very limited adsorption capacity for small molecular size compounds, such as phenol. This char presented greater adsorption capacities for larger molecular size compounds than some commercial adsorbents, which was associated with its well developed external surface area.

Steam activation resulted in a significant development in the carbon porosity, which was reflected in greater capacities for the adsorption of organic compounds from solution. However, different patterns were observed depending on the molecular size of the adsorbate compound. Phenol adsorption increased rapidly, particularly during the initial stages of activation, to reach values over 100 mg/g. This behavior was mainly attributed to the development of narrow micropores. Methylene blue adsorption capacity increased almost linearly with the degree of activation to reach values up to 448 mg/g, which suggests that adsorption of this compound takes place preferentially in medium and large micropores. Owing to molecular sieve effects, textile dyes Procion Red H-E2B and Procion Turquoise H-A are preferentially adsorbed in very large micropores or small mesopores. Highly activated rubber-derived carbons exhibited adsorption capacities for these dyes in excess of 330 mg/g. These values are significantly greater than those exhibited by commercial activated carbons tested in this work.

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