



REMOVAL OF MICRO POLLUTANTS FROM AQUEOUS SOLUTION USING ACTIVATED CARBONS FROM PET WASTE

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ABSTRACT

The aim of this study was the production of activated carbons (ACs) starting from urban plastics waste that is the post-consumer bottles made of polyethylene terephthalate (PET) as raw material, the characterization and the investigation of their efficiency in the removal of micro-pollutants such as chloro- and nitro-phenolic compounds from an aqueous solution. Plastics waste were carbonized at 600°C in an inert N₂ atmosphere and the resulting char was subjected to the physical and physico-chemical activation at 850°C. The ACs prepared by this method were characterized in terms of structure, pore texture, microstructure and surface functional groups. Finally, the ACs prepared were successfully tested as adsorbent in the removal of 4-chlorophenol and 4-nitrophenol from an aqueous solution. The influence of the activation method on the performance of the so-prepared ACs was investigated and discussed. On the basis of the results obtained the greatest performance in the removal performance of 4-chlorophenol and 4-nitrophenol was observed with the sample obtained from physico-chemical activation. It was thus suggested that the physico-chemical activation leads to the formation of chemical bond at the surface of the adsorbent so prepared, showing that the impregnation of char with KOH before physical activation enhance the formation of functional groups necessary for chemisorption.

Keywords: Urban plastics waste, activated carbon, adsorption, phenolic compounds, wastewater.

INTRODUCTION

World increasing population and industrialization have resulted in a large increase in public demand of consumer goods. The industrial and agricultural activities carried out with a view of meeting this demand involve the use of many different chemicals which are subsequently released to various extents in the effluents from such industries. As these wastes are by and large discharged through aquatic route, they are detected as water pollutants and over the years, this has grown to a widespread contamination of surface and ground waters by various organic and inorganic compounds. In particular, huge quantities of phenolic, chloro and nitro aromatic compounds used as raw materials in many industrial processes are disposed as waste by petrochemical, pharmaceutical, agribusiness, polymer, textile and paper industries. There is consequently a growing public concern about this environmental pollution which has led to the instatement by global organizations such as the World Health

Organization (WHO) of stringent worldwide regulations to be abided with by industry (Tonni *et al.*, 2006).

In order to comply with the stringent contaminant limit set forth by environmental protection institutions, industrialists have been resorting to various technologies developed over the years to remove pollutants from water such as chemical precipitation, electro flotation, ion exchange, reverse osmosis and adsorption on activated carbon (CPC). Among these, adsorption on activated carbon has become commonplace as compared to other physico-chemical techniques owing to its simplicity, low price, ease to scale-up and its high efficiency in removing low substance concentrations even at part per million levels (Goyal, 2005). Most importantly, activated carbon has been widely used in water treatments because of its high specific surface area, well-developed micropores, chemical stability and durability (Goyal, 2005).

The economic advantage of the use of activated carbons in water treatments based on adsorption processes can be enhanced by exploring new synthetic routes or using non

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valuable by-products, residues or post-consumer waste materials as precursors for their preparation. In addition, the utilization of by-products, residues or post-consumer waste materials as precursors of carbon adsorbents appears to be a valuable strategy to deal with the problem of urban wastes disposal and recycling (Arenillas *et al.*, 2005). In recent years a lot of research has been reported on the use of miscellaneous residues as starting materials in the preparation of activated carbons for the adsorption of micro pollutants (Laszlo, 1997), including waste materials of agricultural origin (Boumazza, 2012), agricultural waste (Bharathi, 2013), agricultural by-products (Ahmedna *et al.*, 1997), olive mill solid residue (Nameri *et al.*, 2000), cork waste (Carvalho *et al.*, 2003), waste apricot (Önal, 2006), olive-seed waste residue (Stavropoulos, 2005), coir pith (Namasivayam, 2002), oil palm shell (PoedjiLoekitowati Hariani, 2013), corn cobs (Leelavathy, 2015), grapefruit peel (Huang *et al.*, 2014), coconut shell waste (Kurniawan *et al.*, 2011), biogas residue (Xiangzheng Yuan *et al.*, 2011), waste newspaper (Kiyoshi Okada *et al.*, 2003), waste tyres (Mui, 2004), city wastes (Tamon, 1998). From among the potential residues candidates, Polyethylene terephthalate (PET) captures a great attention (Miroslaw Marzec *et al.*, 1999). Researchers in the developing countries are working hard to reduce environmental pollution caused by non-biodegradable plastics waste (i.e. post-consumer products like plastics bottles, plastic bags, plastic floor tiles, window and door frames in the construction industry), through their transformation to a biodegradable materials such as activated carbon, well known as useful material for the wastewater treatment. Although the effectiveness of the application of activated carbon made of plastics materials for the treatment of contaminated water have been demonstrated, up to date, just few works are reported in the literature (Parra *et al.*, 2004; Kartelet *et al.*, 2006; Ania *et al.*, 2007). Another study, Parra *et al.* (2004) prepared activated carbons from post-consumer waste using physical activation at 400°C in a nitrogen atmosphere followed by activation of the char with carbon dioxide at 925°C. The obtained AC had a composite porous structure. Two years later Kartel *et al.* (2006) obtained from the same precursor an AC prepared by physical activation with steam, precursor material which was at first treated with sulfuric acid and the resulting AC had a surface area of 1030 m²/g and an effective pore size of 1.8 nm. The preparation of low-cost adsorbents from waste materials presents several advantages, mainly with regard to economic and environmental aspects. The transformation of plastic wastes to activated carbon (AC), appears as a means to solve the pollution problem caused by plastic wastes. Therefore, the transformation of plastic wastes to activated carbon is considered as one of the concrete and immediate contribution to the alleviation of environmental pollution.

The present study focused on the preparation, the characterization and the evaluation of the adsorption properties of ACs obtained from post-consumer plastics and prepared using both physical and physico-chemical activation methods. The adsorption properties of the ACs obtained were studied using the Langmuir and Freundlich isotherms.

MATERIALS AND METHODS

Preparation of activated carbon

The polyethylene terephthalate (PET) polymer plastics from post-consumer soft-drink bottles used as the raw material were collected from the municipal waste. They were cut down to desirable particle sizes and dried under sunlight for 05hrs before carbonization process. 20 g of the polymer plastics were loaded in the furnace and heated from room temperature (18°C) with a temperature increment rate of 10°C/min up to a final temperature of 600°C. The heating process was stabilized with nitrogen at a flow rate of about 100 mL.min⁻¹. The residence time of the samples at the final stabilized temperature of 600°C was 03 hours. After the heat treatment, the nitrogen flow was stopped and the char obtained was cooled down to the room temperature. Two samples of char (5g of each carbonized sample) were subjected to physical activation with steam of nitrogen at the rate of 0,13mL/min by increasing the temperature up to 850°C at a heating rate of 10°C.min⁻¹, with a residence time of 2 and 3 hours respectively. The sample of char (5g) was also used for physico-chemical activation, the carbonized samples were mixed with the potassium hydroxyl (KOH) in the ratio 1:2 (char/KOH) and dried in the oven overnight at 110°C and then heated in the same condition as described above for the physical method. The resulting activated carbons were grinded and sieved to a particle size < 150 µm. The activated carbon obtained by the physico-chemical activation method was firstly washed with distilled water then dried in the oven overnight at 110°C before it was grinded and sieved into particles of size < 150 µm. The activated carbons obtained by physical activation during 2 and 3h are referred as **PET1** and **PET2**, respectively, in relation to the raw material PET, whereas the activated carbon produced by physico-chemical activation is referred as **PETA**.

Characterization

The textural characteristics of the activated carbon were obtained using a chemisorption and physisorption surface area analyzer (Micromeritics TriStar 3000). The activated carbons were out-gassed in vacuum at 353K and 5µm Hg during 6h prior to measurement. The surface areas of activated carbons were estimated by BET (Brunauer–Emmett–Teller) model with the assumption of the adsorbed nitrogen molecule having a cross sectional area of 0.162 nm², while the total pore volume was determined at the relative pressures of 0.01–0.30. The micro pore

volume and external surface areas were obtained from the t-plot method. The mesopore volume was calculated by subtracting the micropore volume from the total pore volume and the average pore width could be roughly calculated from the BET surface area and the total pore volume. The adsorptive property of the activated carbon in liquid phase was determined by iodine and methylene blue adsorption capacities. The surface functional groups of the obtained samples were determined by Fourier transform infrared (FTIR) analysis using FT-IR (SPECTRUM ONE brand). The wave number was varied between 4000 and 450 cm^{-1} . The micro-structural morphology was investigated using the Transmission electron microscopy (TEM) (A JEM 100s, an FEI Tecnai G2 Spirit and an FEI Tecnai F20 X-Twin at 200 kV FEG with an Oxford EDS system). For TEM analysis, all samples were ultrasonically suspended in the methanol and a drop of the suspension was transferred to a copper grid and allowed to dry before the TEM analysis. XRD was also carried out to study of crystalline structure of the carbon using the diffractometer Bruker D8 X-ray FOCUS. Raman spectroscopy is often used to reveal fine structural information of the carbon material. In this work Raman spectra were obtained with a T6400 Raman Spectrometer (JOBIN/YVON).

Batch Equilibrium Experiments

The adsorption equilibrium studies were performed using isotherm technique. 10 mg of the adsorbent were mixed with 100 mL of each aqueous solution (4-chlorophenol and 2-nitrophenol) with initial concentration in the range of (11-72 mg/L). Each solution was shaken vigorously at the rate of 200 rpm at room temperature for 4 hours. At the end of the adsorption process, the solutions were filtered and the equilibrium concentrations were determined by spectrophotometric analysis. Q_e the quantity of phenolic compounds adsorbed per gram of adsorbent was determined using the following formula:

$$Q_e = (C_0 - C_e)V/m \dots\dots\dots(1)$$

where, C_0 and C_e are respectively the initial and equilibrium concentration (mg/L), of phenolic compounds in solution; V the volume (L), and m is the weight (g) of the adsorbent.

RESULTS AND DISCUSSION

The polyethylene terephthalate (PET) from post-consumer soft drink bottle was used as the raw material

for the production of an adsorbent. After the carbonization process, the samples were first subjected to physical activation with two different residence time and then different burn-off which lead to the ACs, PET1 and PET2. Another sample PETA was prepared by the physico-chemical activation process. The burn-off and the yield of activated carbons are listed in Table 1. It can be seen that the activated carbons obtained from post-consumer plastics give a low yield; this is directly due to the structure of raw material. The raw material is constituted of volatile compounds which escape during the carbonization, the departure of volatile compounds leaves pores, which are developed during activation process (Harry Marsh, 2006) The burn-off shows that the sample PET1 and PETA are micro and mesoporous (burn-off between 50-70%), for PET2 the burn off is less than 50% mainly microporous. This can be explained by the higher degradation of the material by steam water during the activation process.

Characterization of activated carbon

The characterization is an important tool that helps to understand the properties of carbon that may affect the removal of micro pollutants in aqueous solution. The Figure 1 shows typical N_2 adsorption-desorption isotherms of the activated carbons obtained from both physical activation and physico-chemical activation. All the samples exhibited the development of both micropores and mesopores. We observed an inflection at low relative pressure $P/P^0 > 0.1$ and at high relative pressure of $P/P^0 > 0.9$ where extents of adsorption rise very rapidly. The differences in the shape of the three isotherms are not very significant at relative pressure $P/P^0 < 0.9$. The samples presented greater adsorption capacities at low pressures, indicating the presence of a more developed micropores structure. The sample obtained from physico-chemical activation PETA exhibited a higher quantity of mesopores (63.45%), they allow the rapid filling of micropores before the beginning of filling by capillary condensation in mesopore. According to the IUPAC classification (Goyal, 2005), these activated carbons can be described under type II isotherm with type H3 hysteresis, which is associated with a narrow pore size distribution of microporous material. For all the samples the pronounced intensity of hysteresis implies the presence of a more extensively developed pore network wherein the fraction of larger pores trapped within the network of smaller pores is much higher

Table 1. Characteristics of different ACs obtained and their adsorptions test capacities.

Activated carbons	Activation time (hours)	Yields (%)		Burn-off (%)	$Q_{(ads)MB}$ (mol/g)	$Q_{(ads)I_2}$ (mol/g)
		Carbonization	Activation			
PET1	2	15	65,46	61.4	272.8	1110,4
PET2	3	15	61.4	38.6	386.0	1237,3
PETA	2	15	33,33	67.6	276 .0	1181.8

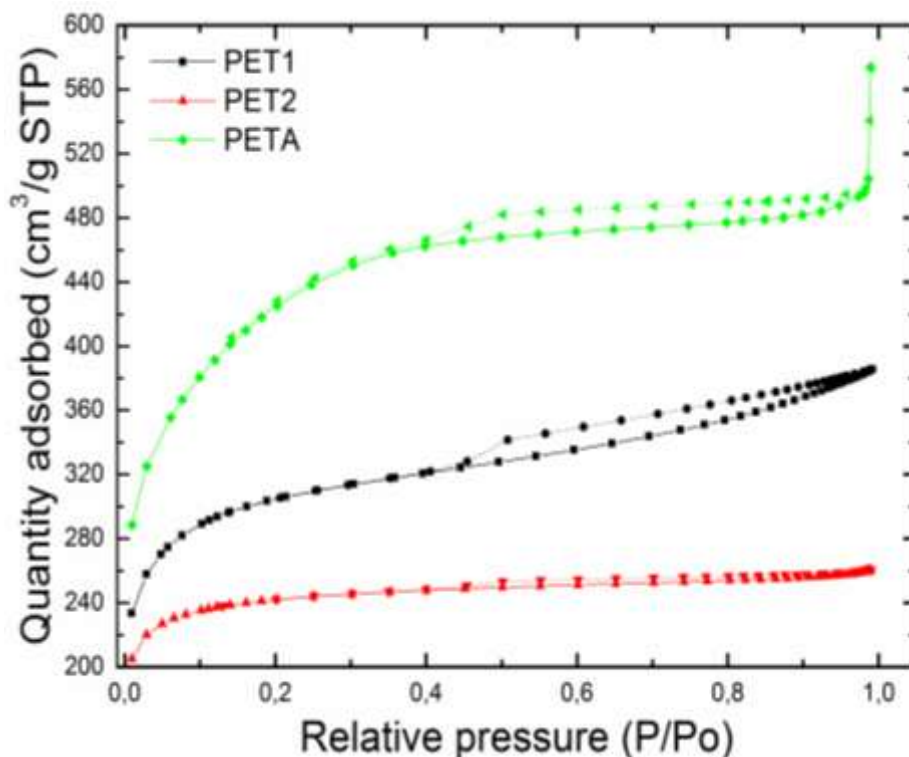


Fig. 1. Adsorption-desorption N_2 isotherm of ACs prepared by physical activation at 800°C under H_2O flow and by physico-chemical activation at 850°C using KOH under H_2O flow.

Table 2 shows that sample prepared by physico-chemical activation PETA presents higher surface area ($1412.9 \text{ m}^2/\text{g}$) compared to those obtained by physical activation. This means that impregnation of carbonation material with KOH increases considerably the formation of pore network into the carbon. But in the depth analysis, the percentage of micropores in sample PET2 is higher, this means that the increase of the residence time during the activation process opens the external pores and gives access to more micropores, this is shown well by the transmission electron microscopy (TEM) images (Fig. 2). The average pore diameters were between 1.9 nm and 2.4 nm, indicating its microporous character. It appeared that all the samples of activated carbons were predominantly microporous.

The FTIR is mainly used for the study of the surface chemical functional groups on activated carbon. The carbon materials are black materials thus they absorb the radiations almost in the visible spectrum and the peaks obtained in the FTIR are commonly a sum of interactions of different types of groups. The Figure 3 shows the FTIR spectra of carbons prepared by physical and physico-chemical activation with nitrogen atmosphere and by impregnation with KOH, respectively. The samples show the band around 3437 cm^{-1} assigned to the O-H stretching mode of hydroxyl groups and adsorbed water. The spectra

show that for carbon obtained by impregnation with KOH the degree of oxidation enhances absorption bands at 1563 cm^{-1} , 1082 cm^{-1} and even at 3437 cm^{-1} . The 1563 cm^{-1} band observed only on PETA spectrum is attributed to aromatic structures, also at unconjugated carbonyl groups obtained after acetylating of C-C with KOH. The 1082 cm^{-1} can be attributed to C-O vibrational stretch.

Raman spectra of PETA sample (Fig. 4) obtained from physico-chemical activation show a line at 1350 cm^{-1} in addition to the line near 1600 cm^{-1} . It has been shown that the peak at 1600 cm^{-1} named G-band (G = graphite) corresponds to an E_{2g} mode of hexagonal graphite and is related to the vibration of sp^2 hybridized carbon networks in a graphite layer. The D-band (D = disorder) at about 1343 cm^{-1} is due to the breathing modes of six-atom rings and requires a defect for its activation. The presence of defects improves the performance of carbon materials because of the high anisotropy of the mechanical strength or the electrical conductivity between the in-plane and out-of-plane direction.

The diffractograms of sample PETA is shown in Figure 5. The XRD spectra of AC sample exhibits an intense peak at $2\theta = 36.27^\circ$. This peak is assigned to the turbostratic structure of the carbon.

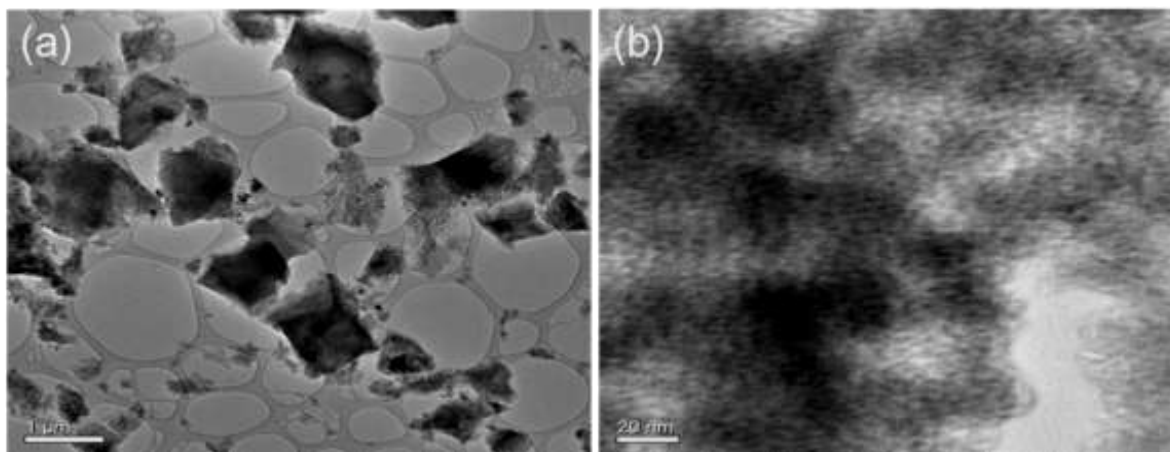


Fig. 2. Higher and low magnification TEM image of AC prepared by physical activation PET2: (a) at 1 μm (b) at 20 nm.

Table 2. Porous parameters of the activated carbons.

Samples	$^a S_{\text{BET}}$ (m^2/g)	$^b S_{\text{Langmuir}}$ (m^2/g)	$^c S_{\text{mic}}$ (m^2/g)	$^d S_{\text{ext}}$ (m^2/g)	Pores sizes (nm)				Pores Volumes (cm^3/g)
					BET _{ads}	BJH _{des}	BJH _{ads}	V_{mic}	V_{cum}
PET1	1063.5	1293.934	753.9	309.6	2.238	4.190	3.874	0.335	0.595
PET2	849.2	1041.815	703.268	145.891	1.8929	3.095	3.331	0.3104	0.4018
PETA	1412.9	1566.70	516.714	896.144	22.084	36.253	37.252	0.2634	0.4553

^aBET surface ; ^bLangmuir surface ; ^cmicropore surface ; ^dExternal surface

SEM analysis of the activated carbons

The Scanning electron microscopy (SEM) technique was used to observe the morphology of the surface of the activated carbon obtained from plastics. The Figure 6 shows the cavities of different sizes and different shapes on the external surface of carbons.

Application of prepared activated carbon to liquid phase adsorption

Adsorption isotherms

The equilibrium adsorption isotherms are usually used to determine the relationship between liquid phase and the adsorbents. The Figure 7 shows adsorption isotherms of 4-chlorophenol and 2-nitrophenol on the three samples of activated carbon PET1, PET2 and PETA. The shape of the 4-chlorophenol and 2-nitrophenol adsorption isotherms appears to be type I characteristic of a medium containing mainly micropores. The isotherms show rapid adsorption at the beginning for all the samples, the number of adsorption sites available reduces when the concentration of the phenolic compound increases, indicating that there is a strong adsorption between the active sites of the activated carbon and the molecules of

phenolic compounds. The amount adsorbed steadily increases and the adsorption isotherms display a plateau indicating the formation of a complete monolayer. It can be seen that the highest uptake was obtained for PETA, which is normal because PETA has larger surface area with an important quantity of mesopores which act like gateway for micropores. The higher adsorption of phenolic compounds on PETA can also be attributed to the higher quantities of functional groups on the surface, mainly carbonyls functions which may establish the hydrogen bonds with OH function of phenolic compounds.

The experimental data were simulated with the Langmuir ($1/Q_e = 1/q_m k C_e + 1/Q_m$) and Freundlich ($\ln Q_e = \ln K_F + \ln C_e$) models (Figs. 8 and 9), where Q_e (mg/g) is the adsorption density at the equilibrium, C_e is the equilibrium concentration of adsorbate in solution (mg/L), Q_{max} is the maximum adsorption capacity corresponding to complete monolayer coverage, K_L is the Langmuir constant related to energy of adsorption (L/mg), K_F is the Freundlich constant, $1/n$ is the heterogeneity factor which is related to the capacity and intensity of the adsorption.

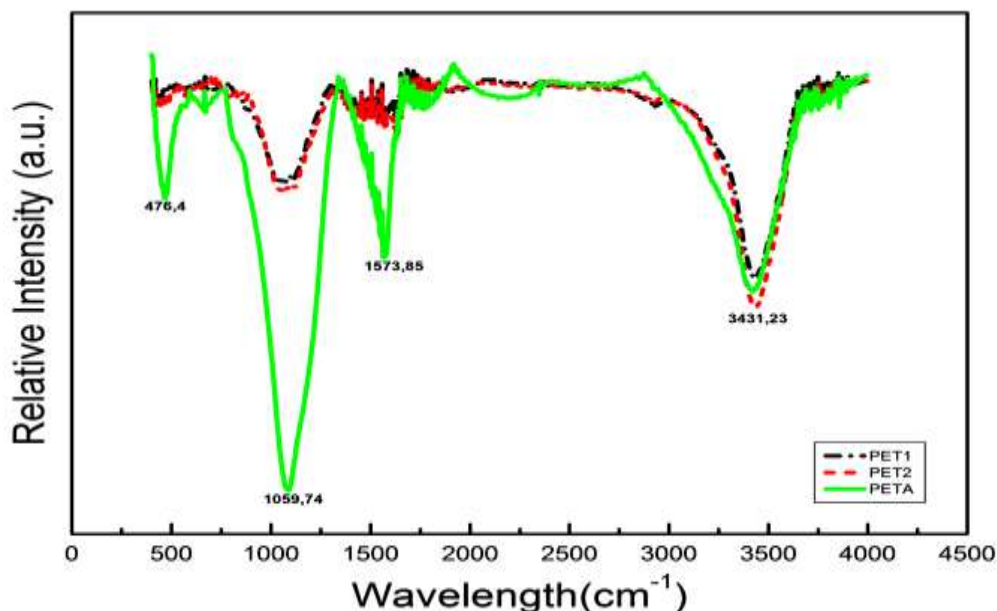


Fig. 3. FTIR spectra of three Activated Carbons obtained by physical activation and physico-chemical activation.

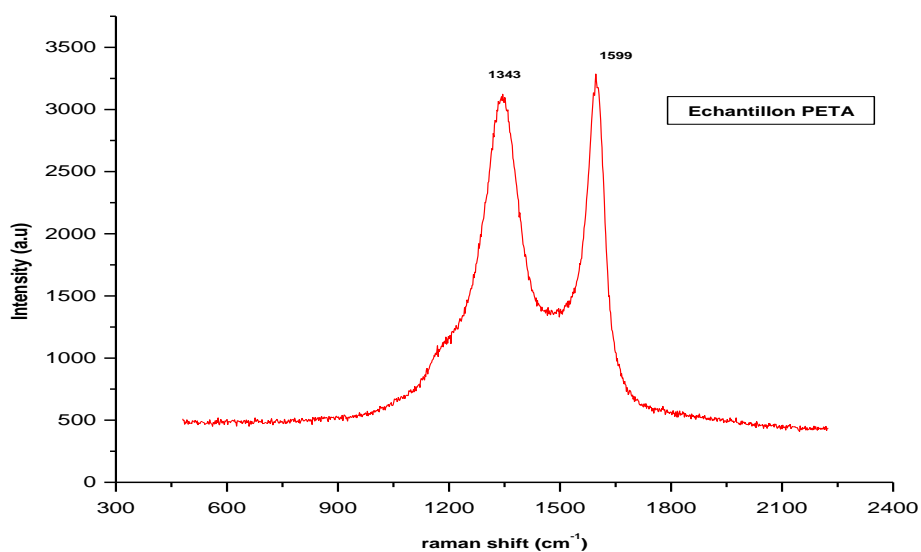


Fig.4. The Raman spectrum of PETA sample prepared by physico-chemical activation showing disordered sp^2 bonded graphite.

The Table 3 provides a compilation of the main parameters obtained from the fittings to both equations, along with the correlation coefficients. The constant of Langmuir, K_L measures the intensity of adsorption. In fact, the higher the K_L value the stronger the affinity between the phenolic compounds and the activated carbon. Thus, it comes out from results that PET1 has stronger affinity for 4-Chlorophenol adsorption than PET2 and PETA. In contrast the sample PETA prepared by physico-chemical activation has stronger affinity for

adsorption of 2-nitrophenol than PET2 and PET1. Despite the values obtained for constants K_L , the values of the monolayer adsorption capacity (Q_m) of PET1 is larger than PET2 and PETA for 2-nitrophenol adsorption, as already noticed for values of K_L , this values of Q_m is in contrast larger for PETA than PET2 and PET1 for 4-chlorophenol adsorption. This can be explained by the fact that the sample PETA has a great amount of micropores and the existent of mesopores act only as the gateway of micropores (the functional group at the

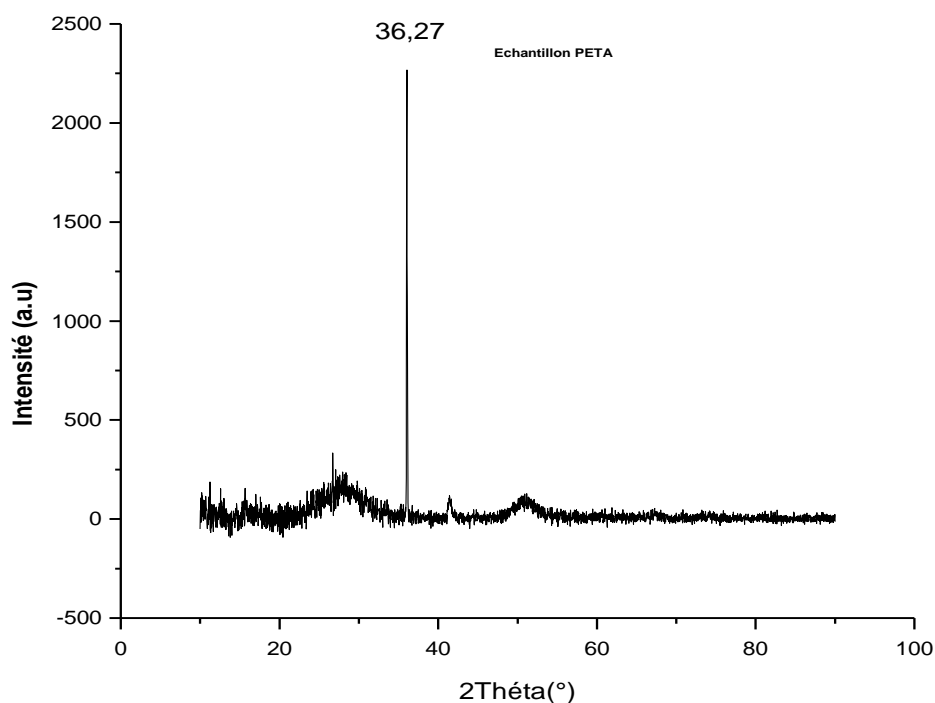


Fig. 5. X-ray diffractogram for activated carbon obtained by physico-chemical activation (PETA).

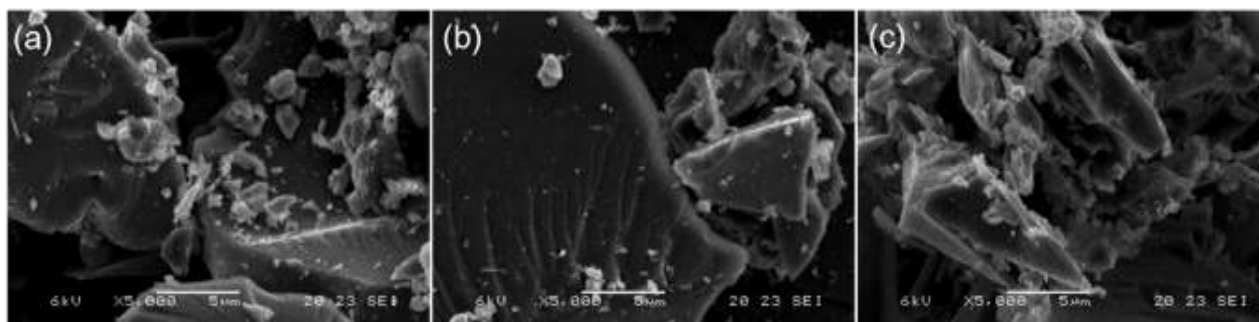


Fig. 6. SEM images of the three Activated Carbons: (a) PET1, (b) PET2 and (c) PETA.

surface of activated carbon). In addition, the linear correlation coefficients R^2 for this model are all higher than 0.96 making the Langmuir model suitable to explain the adsorption of micropollutants onto activated carbon prepared from post-consumer plastics.

The values of the Freundlich constants n lie between 1 and 10 and vary in the interval 1.107 - 4.098 showing that the phenolic compounds adsorption on the activated carbon is favourable. However, the Freundlich model has values of correlation coefficient lower than 0.96, which restricts the utility of the model to describe the phenolic compounds adsorption on these samples.

CONCLUSION

The adsorption capacities of three activated carbons obtained from post-consumer plastics using both physical and physico-chemical activation for the removal of phenolic compounds were investigated. The characterization of the materials revealed that the adsorption of phenolic compounds is favorable and depends of the structure of adsorbent. It was observed that there are good interactions between the adsorbate and the adsorbent. The adsorption capacity was higher for the sample PETA prepared by physico-chemical activation compared to others obtained by physical activation, due to higher quantity of hydroxyl and carboxylic functional

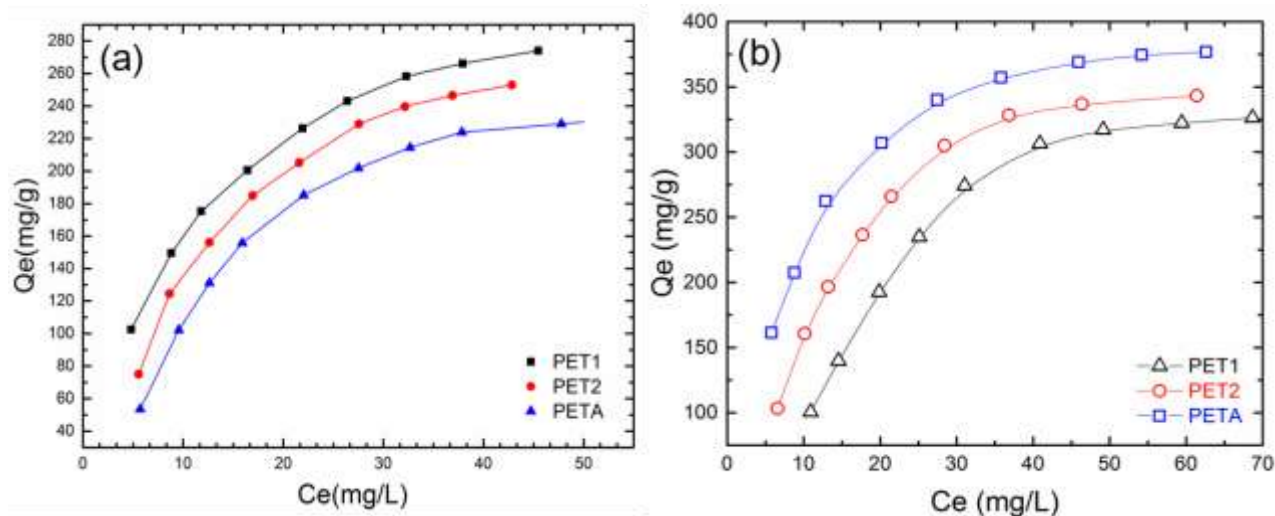


Fig. 7. Adsorption isotherms of (a) 4-chlorophenol and 2-nitrophenol on the three ACs samples from PET wastes.

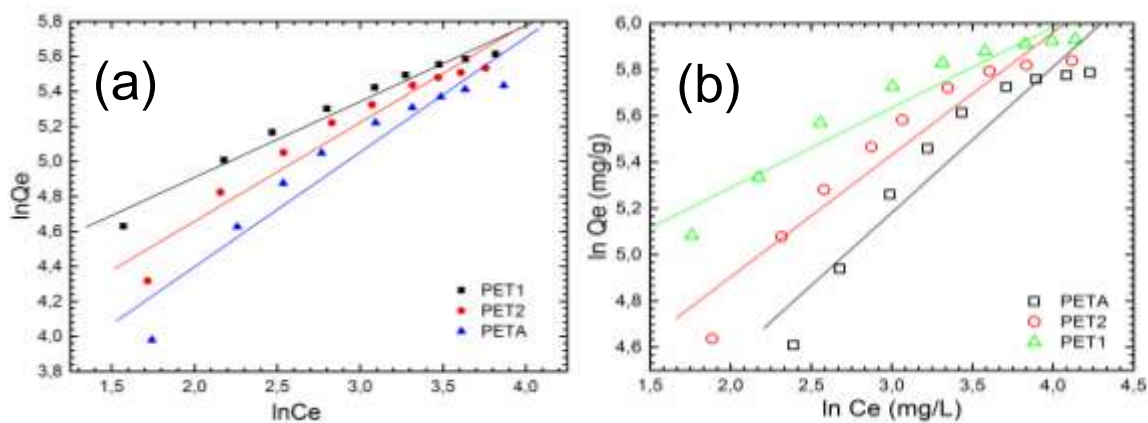


Fig. 8. Linear transforms of Langmuir isotherm for (a) 4-chlorophenol and 2-nitrophenol on the three samples of AC from post-consumer plastics.

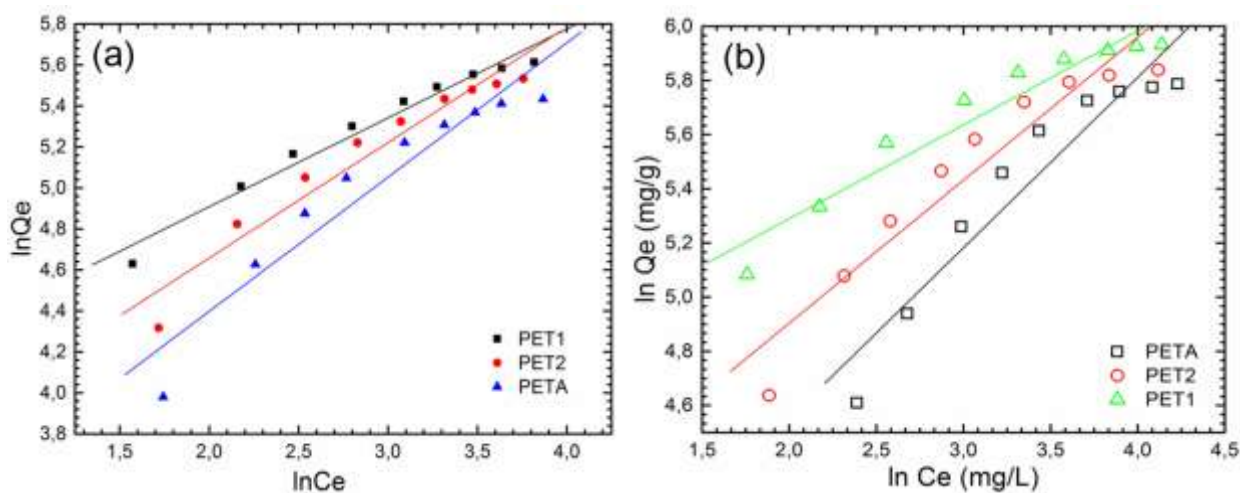


Fig. 9. Linear transforms of Freundlich isotherm of (a) 4-chlorophenol and 2-nitrophenol on the three samples of AC from post-consumer plastics.

Table 3. Fitting parameters of the equilibrium adsorption isotherms to the Langmuir and Freundlich models.

Adsorbates	Adsorbents	Langmuir			Freundlich		
		K_L	Q_m	R^2	K_F	$1/n$	R^2
2-nitrophenol	PET1	0.01361	826.446	0.983	26.936	0.6296	0.952
	PET2	0.03288	609.756	0.989	46.894	0.5280	0.953
	PETA	0.09491	458.716	0.998	99.066	0.3467	0.943
4-chlorophenol	PET1	0.08756	344.828	0.9998	56.818	0.4339	0.985
	PET2	0.03828	450.451	0.988	34.174	0.5632	0.970
	PETA	0.01648	666.667	0.981	21.891	0.6555	0.956

groups on the surface that interact with adsorbate. Therefore, the ACs prepared are mainly microporous with a greater adsorption capacity thus they can be used as low-cost adsorbents and as an alternative for environmental pollution remediation.

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