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Mechanisms Involved In the Removal of Phenolic Compounds from Aqueous Solution Using Activated Carbons Based Palm Kernels Shells

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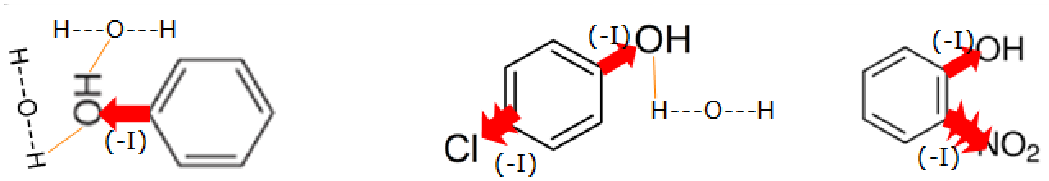
ABSTRACT

The batch removal of phenol (Ph), 4-chlorophenol (4CPh) and 2-nitrophenol (2NPh) was investigated onto two physico-chemically prepared activated carbons namely ACPKS-KOH and ACPKS-H_3PO_4, in order to determine their adsorption efficiency. The mechanisms involved known as the π - π interactions, the acceptor-donor complex formation together with the solubility and the hydrophobicity were thoroughly elucidated on the removal of Ph, 4CPh and 2NPh. The acidic functional groups present on the activated carbons reduced significantly the π - π interactions together with the establishment of hydrogen bonds which inhibited the adsorption of pollutants. The basic functional groups favourize the formation of acceptor-donor complex thus increasing the percentage removal. Both Langmuir and Freundlich isotherms models were used to describe the adsorption equilibrium. The maximum adsorption capacities obtained from Langmuir equation were found to be 79.36 mg g^{-1}; 113.63 mg g^{-1} and 238.09 mg g^{-1} for Ph, 4CPh and 2NPh respectively onto ACPKS-KOH, while a significant decrease was observed onto ACPKS-H_3PO_4, where it was found to be 44.84 mg g^{-1}; 46.08 mg g^{-1} and 84.03 m g^{-1} for Ph, 4CPh and 2NPh respectively. Therefore, these adsorbents prepared are a promising route for the removal of phenolic compounds from wastewaters.

Graphical Abstract/Highlights of Abstract:



Hydrogen Bonds: (-OH) group of phenol (Ph) and 4-chlorophenol (4CPh) easily establishes hydrogen bonds with water molecules whereas (-OH) group of 2-nitrophenol (2NPh) doesn't establish hydrogen bonds.

Inductive effect: (-OH), (-Cl) and (-NO_2) have inductive attractive effects I(-) hence the electronic density of aromatic ring is reduced significantly, thus increase their acid character and acceptor of elector.

Acceptor-donor complexes formation: 2NPh (more acidic) forms more complexes with oxygen functions of activated carbons: thus the more adsorbed specie. Ph and 4CPh (less acidic) form fewer complexes with oxygen functions of activated carbons: thus the less adsorbed species.
Basic functional groups of activated carbons act as electron donor to the aromatic ring.

Keywords: Palm kernel shells; Activated carbons; Phenolic compounds; Acceptor-donor complexes.

INTRODUCTION

The phenolic compounds are released into water as wastes generated from industrial activities like petrochemical, pharmaceutical, wood preserving and used plastics [1, 2]. Other sources of phenolic compounds are owing to environmental degradation of complex molecules [2]. For example, many synthetic pesticides are based on phenolic compounds, which are a group of priority toxic pollutants listed by the US EPA in the Clean Water Act [2,3] and by the European Union Decision in view of the risk factor for human health and environment due to their high toxicity and low biodegradability. Their presence in drinking water is severely restricted by the international sanitary organizations because they are suspected to be very harmful to organisms even at low concentrations due to their toxicity and carcinogenicity properties [4, 5]. In order to follow the norms, drinking water producers must search new ways of removing the phenolic compounds from water before distribution for consumption. To achieve this goal, several processes such as electrochemical oxidation, chemical coagulation, membrane retention, photocatalytic degradation and adsorption are used [5-9]. The adsorption process is the most popular technique because of its efficiency and its simplicity, thus it has attracted worldwide concern for its economical and environmental perspectives. For the others processes mentioned above, principally the first cited, it is rarely used due to its inconvenience like optimization difficulties, by-product pesticide production and the membrane technology which is rather costly [5]. Unlike other techniques are costly from the standpoint of implementation, they have the merit of describing well with clear mechanisms the phenomena involved during their removal operation [2,10] which is not the case with the adsorption on activated carbons**.** As far as our knowledge is concerned, it's clearly demonstrated that the adsorption is preferred from other techniques on removing phenolic compounds from aqueous solution, but studies are rarely focused on the explanation of the mechanisms involved during the adsorption processes of phenolic compounds on activated carbons. Most authors emphasize on the influence of the solubility of the molecules, the hydrophobicity, the influence of substituents and the pH of the solution, which is right but not sufficient to explain the mechanism involved in this process. Classically, the activated carbons are prepared by two methods known as physical and chemical activation. However, there are also studies which combined the two classical methods called physiochemical activation [5, 10, 11]. In this present work, the emphasis was made on the physicochemical activation because it introduces different functional groups which subsequently play an important role on the formation of chemical bonds between the adsorbate and adsorbent, especially in the adsorption of organic compounds [5, 11, 12]. Additionally, the physiochemical activation used in this present work differed from others works because the chemical impregnation was carried out on chars and gasified with steam, while in others works the impregnation is carried out directly on precursors before sending for thermal treatment. The reasons of impregnating the chars are recommended because: It increases chance of carbon atoms to react directly with chemicals by promoting a better formation of cross-linked structures; it decreases the complex reactions between the sample and impurities and it lowers the formation of viscous slurry [5]. In the scientific literature, palm kernel shells and palm kernel shells have been converted into high quality activated carbon through KOH-steam and H_3PO_4-steam activations [11, 13]. The combination of two activation methods cited above promotes several types of functional groups. These functional groups are classified in two major sets. The acidic groups consisting mainly of hydroxyl, carboxylic and carbonyl groups while the basic groups, are composed of pyron and chromene [14]. Amongst the various precursors of activated carbons preparation, the palm kernel shells have been proven to be suitable precursor because of their hardness, high carbon and low ash content thus they have been successfully converted into well-developed activated carbons for the removal of phenolic compounds [4, 12, 15, 16]. Although, the activated carbons prepared from palm kernel shells for the removal of phenolic compounds have been found in the scientific literature, the mechanism involved have not been thoroughly investigated. Emphasis was not given to the π - π interactions, the acceptor-donor complex, the hydrophobicity, the hydrophilicity and the solubility. The groups attached to the aromatic ring have different inductive and mesomeric effects, which of course interact with the functional groups present on the activated carbons and consequently affect the adsorption capacities. Therefore, the aim of this work is to investigate the mechanism involved in the adsorption process of the phenol, 4-chlorophenol and 2-nitrophenol in aqueous solution.

MATERIALS AND METHODS

Material and reagents used: The Palm kernel shells collected in the locality of Bongo-Cameroon were washed with distilled water and dried in an oven at 105°C until constant weight. The dried materials were grinded and sieved to a particle size 2.0 mm and stored at room temperature. Phenol, 4-chlorophenol and 2-nitrophenol used in this work were obtained in laboratory grade (Merck, Germany) and were used without any further purification.

Activated carbons preparation: The palm kernel shells were carbonized in the CARBOLITE 1200C Tube Furnaces, KEISON Products at 800°C for1h under nitrogen flow rate of 100 mL min^{-1} and maintained until the cooling of the furnace. An exact amount of the carbonized material was weighed and put in two beakers containing saturated solutions of KOH and H_3PO_4 at the ratio of 1/1 and 2/1 respectively. Each beaker was mixed for two hours at 60°C and then was dried at 120°C overnight for complete water evaporation. The furnace's reactor was loaded with 10 g of each impregnated sample and was raised at the pre-determined activation temperatures of 630°C and 770°C for sample impregnated with KOH and H_3PO_4 respectively. It was held at these temperatures for a residence time of two hours under steam activation of 0.1 mL min^{-1}. After cooling to the ambient temperature, the sample impregnated with KOH was washed with 0.1N HCl and with distilled water for several times, whereas the sample impregnated with H_3PO_4 was only washed with distilled water. The washing process was done until the absence of chloride and phosphate ions in the washing water rejection to attain a pH ranged between 6 –7 [16]. The two samples of activated carbons named ACPKS-KOH and ACPKS-H_3PO_4 were crushed into powder form and kept for further tests.

Surface chemistry: Fourier transformed infrared (FTIR) spectrum of the samples was recorded by Fourier Transform Infrared (Vertex 70 DTGS) spectrophotometer. The FTIR spectrum ranged from 4000 to 400 cm^{-1} by making a KBr pellet with PKS, ACPKS-H_3PO_4 and ACPKS-KOH.

Titration of functional groups: The Boehm titration method was used to quantify the surface functional groups on ACPKS-H_3PO_4 and ACPKS-KOH. An exact amount of each sample activated carbon (0.5 g) was placed in 25mL of NaOH, Na_2CO_3, $NaHCO_3$ and HCl of 0.2M respectively. The sealed vials were shaken at room temperature for 24 h. Then each solution was filtered and the excess base or acid was back-titrated with HCl or NaOH both of 0.1M.

Batch adsorption experiments: Ph, 4CPh and 2NPh stock solutions of 1000mg L^{-1} were prepared a 1000mL conical flask. An exact amount (0.5g) of each adsorbent was added into 25 mL of adsorbate solution of known initial concentrations (50-500mg L^{-1}) at pH ranged (4.5 and 5.5). The conical flasks were covered and placed on a shaker at a speed of 125 rpm at 25°C. At the equilibrium time, the samples were filtered using a 0.2 μm Millipore filter and analysis done on a double beam UV-visible spectrophotometer (DR5000) at different wavelengths of 270 nm, 225 nm and 210 nm for Ph, 4CPh and

2NPh respectively. Prior to analysis a technical calibration curve of each aqueous solution was obtained. Each experiment was done in triplicate in the same conditions and the average values were used in the calculation of the quantity adsorbed at equilibrium concentrations, Q_e(mg g^{-1}) and the percentage removal, %R (%) giving by the following equations.

$$Q_e = \frac{(C_o - C_e)}{m} \times V \qquad ;$$

$$\%R = \frac{(C_o - C_t)}{C_o} \times 100$$

Where C_0, C_e and C_t (mg L^{-1}) are initial, equilibrium and concentration at time, t, respectively (L) is the volume of solution and m (g) is the mass of adsorbent.

Adsorption isotherms: The Langmuir isotherm was used in order to describe the equilibrium of gas molecules onto the metal surface [17]. The linear form of Langmuir equation is expressed as the following equation:

$$\frac{C_e}{Q_e} = \frac{1}{K_L Q_m} + \frac{C_e}{Q_m}$$

Where, K_L is Langmuir isotherm constant (L/mg). Q_m (mg/g) is the maximum adsorption capacity at monolayer. Q_e (mg/g) is the amount of adsorbate adsorbed at equilibrium. C_e (mg L^{-1}) is the equilibrium adsorbate concentration.

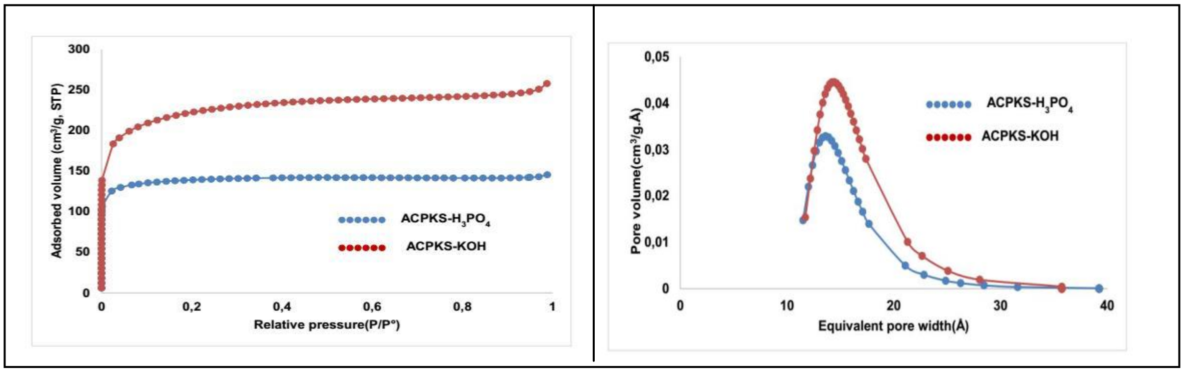
In 1906, Freundlich presented his isotherm as an empirical model [18]. This model can be applied to non-ideal sorption on heterogeneous surfaces as well as multilayer sorption. Linearizing the Freundlich équation is :

$$\mathrm{Log}Q_e = \mathrm{Log}K_F + \frac{1}{n}\mathrm{Log}C_e$$

Where, K_F (mg g^{-1})(L mg^{-1})$^{1/n}$, Freundlich isotherm constant; 1/n, the heterogeneity factor, dimensionless. C_e (mg L^{-1}), the equilibria adsorbate concentration.

RESULTS AND DISCUSSION

Characterization of activated carbons and titration of functional groups: The two activated carbons all exhibit the typical type I isotherm. The major uptake occurs at a relatively low pressure, indicating the formation of highly microporous materials with a narrow pore size distribution [19]. We noticed a high contribution of micropores volume and a less contribution of mesopores volume on both samples. The nitrogen adsorption-desorption isotherms and the pore size distributions of the two activated carbons are shown in figure 1 below.

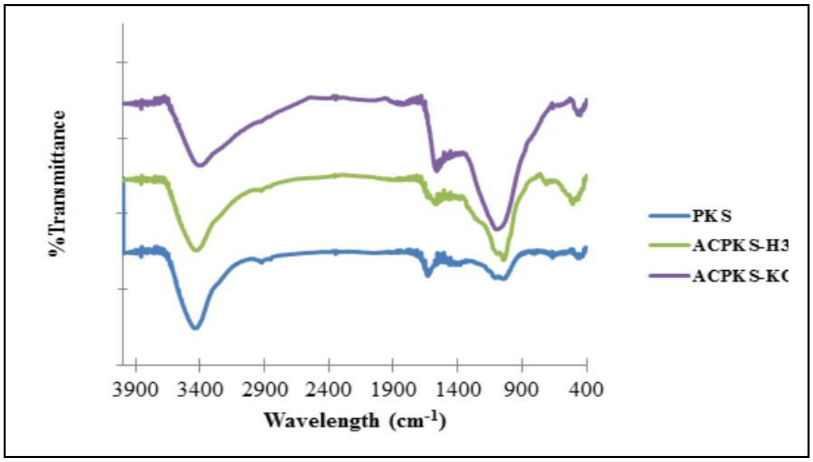


The results of the Boehm titration and of pore analysis are summarized in table 1. The ACPKS-H_3PO_4 is predominated by acidic groups whereas ACPKS-KOH is by the basic functional groups. It can be noticed the absence of carboxylic groups on ACPKS-KOH, while the phenolic and lactonic groups are in equal amount. This difference in functional groups can be attributed to the chemical agent of impregnation, because KOH impregnation promotes basic functional groups while H_3PO_4 impregnation leads to acidic functional groups which make the carbon surface hydrophilic and polar in character [20].

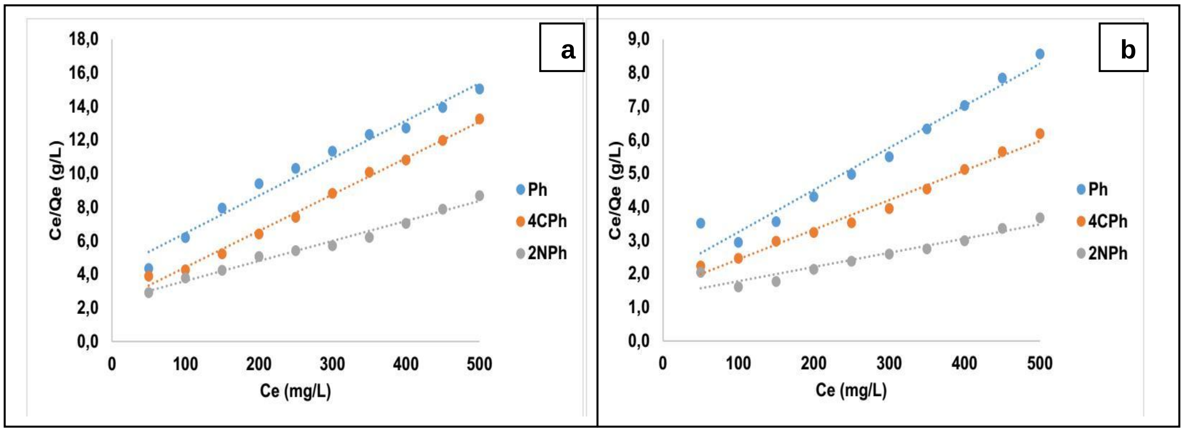
Table 1. Porosity and groups characteristics on ACPKS-H_3PO_4 and ACPKS-KOH

Samples	S_{BET} (m^2/g)	Vtotal (cm^3/g)	Vmicropores (cm^3/g)	Vmesopores (cm^3/g)
ACPKS-H_3PO_4	538.89	0.23	0.21	0.02
ACPKS-KOH	838.03	0.39	0.33	0.06
Samples	basicity(mmol/g)	Carboxylic(mmol/g)	Phenolic(mmol/g)	Lactone(mmol/g)
ACPKS-H_3PO_4	0.07	0.25	0.09	0.11
ACPKS-KOH	0.17	0.00	0.10	0.10

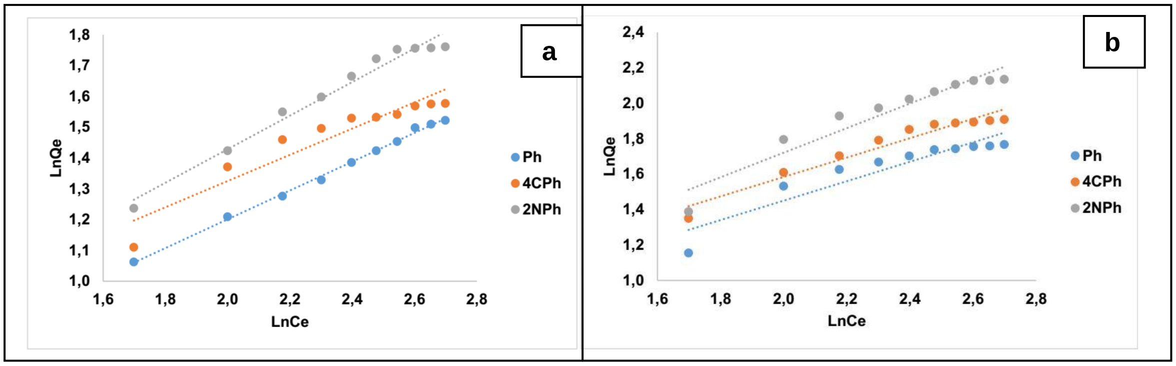
FTIR Analysis: The FT-IR spectroscopy of PKS, ACPKS-H_3PO_4 and ACPKS-KOH are shown in Figure 2. On the spectra of PKS, it was found bands at 1639 and 1058 cm^{-1} are the characteristic adsorption peaks of cellulose, and hemicellulose. In addition, bands at 1754, 1519 and 2129 cm^{-1} are characteristic of the aromatic skeleton of lignin macromolecules [21].The band at1639 cm^{-1}can be attributed to C=O stretching in quinones or carboxylic anhydrides, whereas, the band at 1247 cm^{-1} refers to C–O–C stretching in ethers or ether bridges between rings. After carbonization and activation processes, the intensity of those bands decreased, and even disappeared on ACPKS-H_3PO_4 and ACPKS-KOH respectively. All the two activated carbons show the wide band at about 3485–3475cm^{-1}, which is assigned to O–H stretching mode of hexagonal groups and adsorbed water. On the ACOPS-KOH, the band at 2626 cm^{-1} which is assigned to the asymmetric C–H band representing the alkyl groups such as methyl group is observed. The two activated carbons also show the band around 1625-1150 cm^{-1}, which is attributed to C–O stretching vibrations of carbonyl groups.



Adsorption isotherms: The Langmuir's parameters obtained from adsorption equilibrium constants are lower for 4CPh and 2NPh which seems a rapid increase on the long plateau stage, thus a good possibility of removal of 4CPh and 2NPh by adsorption [22]. The adsorption of 4CPh onto the two samples of activated carbons is well described by the Langmuir model with the values of the correlation coefficient closed to unity. This observation confirmed the presence of a long plateau indicating a weak formation of multiple layers. The conformity of experimental data with the Langmuir model also indicates homogeneous character at the surface of the two activated carbons, thus the adsorption process follows is a normal Langmuir isotherm.



The value of correlation coefficient $R^2 = 0.9755$ seems to show that the adsorption of Ph on ACPKS-H_3PO_4 can also be described by the Freundlich adsorption model, with the possibility of formation of multilayer of Ph on ACPKS-H_3PO_4. Additionally, the Freundlich model is obeyed here even with the constant 1/n less to unity. The experimental data fitted the Freundlich adsorption isotherms well, moreover chemisorptions is more predominant that physisorption.



The table 2 summarizes the parameters of adsorptions models used in this work.

Table 2: Adsorption parameters for Ph, 4CPh and 2NPh on ACPKS-H_3PO_4 and ACPKS-KOH

	Models	Langmuir			Freundlich		
Samples	Parameters	Q_m (mg/g)	K_Lx10^{-3}(L/mg)	R^2	K_F(L/g)	1/n	R^2
ACPKS-H_3PO_4	Ph	44.84	5.30	0.9751	1.40	0.54	0.9755
	4CPH	46.08	9.65	0.9936	1.60	0.42	0.8946
	2NPh	84.03	4.95	0.9842	1.30	0.46	0.9971
ACPKS-KOH	Ph	79.36	6.32	0.9629	1.39	0.69	0.9203
	4CPH	113.63	5.66	0.9835	1.63	0.14	0.9416
	2NPh	238.09	3.08	0.9080	1.42	0.54	0.8660

Mechanisms involved in removal of phenolic compounds: The highest adsorption capacity is obtained for the removal of 2NPh (238.09 mg/g). It's well known that the more soluble specie is, the less it is adsorbed, which is the case of Ph with a solubility of 95g L^{-1}. Therefore, the weak solubility of 4CPh (28 g L^{-1}) and 2NPh (2 g L^{-1}) can explain their better adsorption. Manole showed in 2007 that, the adsorption of aromatic molecules is a result of several possible mechanisms among which intervenes the π- π system of the aromatic cycle [23]. This interaction occurs between the π system of the aromatic cycle which is electrons deficient and the π system on the surface of activated carbons which is rich in electrons thus electron donor. The 2NPh and 4CPh almost have the hydroxyl functional group (–OH), they also have nitro (-NO_2) and chloro (–Cl) functional groups respectively. The nitro (-NO_2) and hydroxyl (-OH) functional groups attached to the aromatic ring of 2NPh possess inductive and attractive mesomeric effects. Consequently, these two effects participate to reduce the electronic density and favoring the interaction of donor (activated carbons) to acceptor (aromatic ring molecules). Whereas the chloro (-Cl) functional group possesses both attractive inductive and mesomeric donating effects, of course the mesomeric effect predominate on the inductive effect [24]. This trend conducts to increase and to reduce simultaneously interactions. Additionally, the ionic forms can create the interactions/repulsions of electrostatic nature depending of the type activated carbon. In this present work the pH is varied between ($4.5<pH<5.6$), thus the predominant form of Ph found is the non-ionic form. In this case, mainly the repulsive interactions are very strong, thus the weak adsorption observed. In literature, other authors obtained the Ph adsorption capacity of 166 mg/g on the activated carbon prepared with the same precursor [25]. The intramolecular and intermolecular hydrogen bonds can be formed between the oxygen atoms and the protons of water molecules in one side and also with hydroxyl functional group (-OH) of phenolic compounds with the nitro functional group (–NO_2) in other side. This observation has been demonstrated by others authors [23]. The hydrophobicity and hydrophilicity of substituents attached on the aromatic ring also influence the adsorption capacities of activated carbons in aqueous solution [23].This is because a hydrophobic substituent located near a hydroxyl (-OH) functional group of a phenolic compound like 2NPh always forms intramolecular bonds with the proton of the hydroxyl (–OH) functional group. In addition, the same oxygen atom also forms the intermolecular bonds with the proton of water molecules and with those of the nitro (-NO_2) functional group. Whereas, for 4CPh, the formation of intramolecular hydrogen bonds is not possible this consequently increases its adsorption capacity. Thus the good adsorption of 4CPh and 2NPh against Ph can be explained by the hydrophobic and hydrophilicity characters of the substituents. The presence of acidic functional groups on the activated carbons surface with their mesomeric attractive effect can weakly reduce the π- π interactions responsible for adsorption mechanism of phenolic compounds [24]. It can be concluded that, the presence of substituents seems not only to be directly implicated in the interactions with the surface of activated carbons, but it is their donor-acceptor electrons characteristics which is also involved in the π electrons delocalization on the aromatic ring. The same trend was also observed by others authors [23].

CONCLUSIONS

The adsorption capacities of activated carbons were influenced by the surface functional groups, the hydrophobicity and less by the solubility of adsorbates. Thus, the activated carbons have showed less affinity with molecule of high solubility (Ph) contrary with 4CPh and 2NPh which is less soluble in water. This explained why the phenolic compounds adsorption is governed by the interactions between the basal plan of activated carbons and the π electrons of aromatic ring. In this mechanism of donor to acceptor, the activated carbons surface plays the role of electrons donor while the aromatic ring acts as the electrons acceptor. The kinetic adsorption of phenolic compounds can also be explained by the steric clutter and the presence of substituents with different inductive and mesomeric effects.

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