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Adsorption behavior of phenol on activated carbons prepared from Algerian coals

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ABSTRACT

Activated carbons were prepared by chemical activation of two Algerian coals (Menouna and Didi), using different chemical agents: Cs_2CO_3, Rb_2CO_3, and H_3PO_4. The mass losses during pyrolysis of the two coals are perfectly correlated to the heat of immersion of the obtained materials in the benzene; indeed, effective activation leads to important weight loss, developed porosity and then easy wetting by benzene. Besides, the obtained results show that the cesium hydroxide is the most effective activating agent. The efficiency of this last one is confirmed by the phenol adsorption isotherms which show a bigger capacity of adsorption. Finally, the values of adsorption capacities of the obtained activated charcoals, toward the phenol, are higher than those of the coals obtained without activating treatment.

Keywords: Activation; Porosity; Adsorption; Heat of immersion; Phenols

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1. Introduction

As is widely known, activated carbons are used for the removal of pollutants from drinking water or wastewater. The use of these materials as adsorbents of inorganic and organic pollutants is due to their unique versatility resulting from their high surface area, porous structure, high adsorption capacity, and surface chemical nature, which can be appropriately modified by physical and chemical treatments to improve adsorption process [1]. It is known that phenol is the most frequently occurring contaminants in industrial wastewaters. Phenol is a white hydro-soluble solid, used as an important intermediate agent in synthesis of pesticides, pharmaceutical, and dyes industries. It is not only carcinogenic but gives bad taste and odor even at low concentrations. The presence of phenol and phenolic compounds in water is of great public concern. The adsorption of these compounds, essentially phenol and p-nitrophenol, from aqueous solution by activated carbons has been studied [2–5].

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Preparative methods for porous carbons are conventionally classified into physical and chemical activation. The latter is carried out by heating in the presence of an added chemical agent, and the physical activation is carried out by carbonization in an inert gas up to HTT followed by activation in an oxidant gas. Among the different chemical reagents, phosphoric acid is one of the commonly used agents for the activation of various precursors, such as coals of different ranks [6,7], viscous rayon fibers [8], and lignocellulosic materials [9,10]. The formation of highly microporous carbons by pyrolysis of various types of precursors (lignites, green cokes, coals) in the presence of potassium and sodium hydroxides and carbonates is now well documented [11–14]. Alkaline and carbonate hydroxides and carbonate potassium were also used in the chemical activation of coals [13,15]. However, studies have not been devoted to chemical activation with carbonates of cesium and rubidium agents.

So, the aim of this work is to study the chemical activation with cesium and rubidium carbonates of two coals Menouna (M) and Didi (D) from the Algerian Southwest. Additionally, the adsorption properties of phenol on the prepared activated carbons using chemical agents: Cs_2CO_3, Rb_2CO_3, and H_3PO_4 were investigated.

2. Experimental

The two coals M and D are characterized by closed elemental analysis, the carbon content is high for the both coals; also, Table 1 shows that their ashes and volatile matters percentages are very different. Elemental and proximate analyses were carried out by an external laboratory (Laboratory of Microanalysis, CNRS-France).

2.1. Preparation of activated carbons

The first series of activated carbons was obtained by pyrolysis of coals M and D, at various temperatures (500, 650, and 750°C) under a stream of nitrogen, at a heating rate of 5°C/min. They are noted MT and DT, respectively, T being the temperature of pyrolysis (for example, M500 is the coal M pyrolyzed at 500°C).

The second series relates to coal impregnated by the phosphoric acid (15.10^{-3} mol/g) and pyrolyzed at various temperatures under a stream of nitrogen, at a heating rate of 5°C/min, the time impregnation being fixed at one hour. The resulting materials are then washed in the distilled water by refluxing for two hours and, finally, washed up to neutral pH. Coals are noted MHT and DHT (e.g. MH500 is the coal M impregnated by H_3PO_4 and pyrolyzed at 500°C). The third series is obtained by carbonizing the intimate mixture coal and carbonate ($8.66\ 10^{-3}$ mol/g) until 800°C, under a stream of nitrogen. The resulting materials were treated by a solution of hydrochloric acid (0.1 N) during two hours then washed in the distilled water until the negative test in the silver nitrate. Coal samples are noted MCs800, MRb800, DCs800, and DRb800 (e.g. MCs800 is the coal M impregnated by Cs_2CO_3 and pyrolyzed at 800°C).

2.2. Characterization of activated carbons

Surface areas of activated carbons were determined by applying the BET equation to the corresponding adsorption isotherms of N_2 at −196°C (S_{BET}), obtained in a volumetric apparatus of Micromeritics, ASAP 2010. The cross-sectional areas for the N_2 molecule were taken as $0.162\,nm^2$.

The heat of immersion in the benzene, at 25 ± 1°C, was measured in a typical calorimeter Calvet, MS 70, by using the method of the fragile point bulb [16]. A complete description of the experimental setup can be found elsewhere [16,17].

The weight loss was determined by weighing the coal before and after carbonization, the coal having beforehand been washed after carbonization; the weight loss (%) was obtained by dividing the absolute weight loss by the initial weight of the coal.

TPD was used to determine the amounts of CO and CO_2 evolved upon heating under a flow of nitrogen up to 800°C; a quadrupolar mass spectrometer was used to analyze the gases evolved during TPD.

Table 1
Elemental analysis of coals M and D

	Elemental analysis (%)					Ashes (%)	Volatile matters (%)	Moisture (%)
	C	H	N	S	O*			
M	81.4	5.0	1.0	1.5	11.1	3.4	32.0	2.0
D	78.1	4.0	0.4	5.5	12.0	13.0	17.0	2.0

2.3. Adsorption experiments in aqueous solution

The adsorption process from aqueous solutions of phenol on activated carbons was carried out at 25˚C. The equilibrium concentration of phenol, before and after contact with the activated carbons, was determined spectrophotometrically using a Jasco, model V-630. All adsorption isotherms were determined without adding any buffer to control the pH to prevent introduction of any new electrolyte into the system. The maximal absorption wavelength was determined as equal 269 nm in neutral solution. The amount of adsorption at equilibrium was calculated from the following equation:

$$q_e = \frac{(C_0 - C_e)V}{m} \tag{1}$$

where C_0 and C_e are the initial and equilibrium concentrations of phenol, respectively, m is the amount of adsorbent and V is the volume of solution.

3. Results and discussion

3.1. Weight loss

Tables 2 and 3 show the weight losses of the coals M and D untreated and treated by H_3 PO_4, Rb_2CO_3, and Cs_2CO_3 successively.

The weight losses of treated coals are greater than those of untreated corresponding coals. Furthermore, the coal M characterized by a low rate of ashes has a higher weight loss. Finally, the cesium carbonate leads to the most important weight loss consecutive to a more effective activation as it will be shown in section 3.4.

3.2. Heat of immersion in benzene

The values of the heat of immersion of the various carbonized and parent coals in the benzene are given in Tables 4 and 5. The heat of immersion, in benzene, gives an important indication of the porous texture of activated charcoals, particularly as regards the diameter of pores [17].

Table 2
Weight losses (%) of raw and impregnated coals at various temperatures

T(˚C)	M	MH	D	DH
500	16.3	17.1	11.8	13.2
650	27.0	29.4	19.0	22.3
750	28.7	31.0	22.0	23.9

Table 3
Weight losses (%) of coals M and D untreated (n.t.) and treated (T) with Rb_2CO_3 and Cs_2CO_3 and carbonized at 800˚C

	M		D	
	n.t.	T	n.t.	T
Rb_2CO_3	28.7	30.4	22.0	28.1
Cs_2CO_3	28.7	33.1	22.0	30.7

Indeed, the larger value of the heat of immersion, the greater accessibility of benzene molecule to the porous texture of the coals samples. Moreover, there is a good correlation between the weight loss of a given coal and its heat of immersion in the benzene.

3.3. Kinetics of phenol adsorption

Figs. 1–6 show the kinetics of phenol adsorption on the coals M and D untreated and treated by H_3PO_4, Cs_2CO_3, and Rb_2CO_3. It can be observed that the equilibrium state is reached at about 1 h.

Pseudo-second-order kinetics model given by the following equation [18].

$$q_t = \frac{k_2 \times q_e^2 \times t}{1 + k_2 \times q_e \times t} \tag{2}$$

which may be written in linear form

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{3}$$

where q_e and q_t are the amounts of adsorption (mg/g) at equilibrium and at time t, respectively, and k_2 (g. $mg^{-1}.min^{-1}$) is the rate constant of second-order adsorption model. The kinetics of phenol adsorption on activated carbons DH750, MH750, DCs, DRb, MCs, and MRb are perfectly fitted by the pseudo-second-order kinetic model. Table 6 summarizes the kinetics constants of phenol adsorption and correlation coefficients R^2 which indicated that the model is more suitable for describing phenol adsorption on these activated carbons. Similar kinetic results have been reported for the adsorption of phenol on commercial and date pits activated carbons [19,20].

The treatment of the coal D by the phosphoric acid does not entail important differences between the kinetics of the phenol adsorption. On the other hand, the differences are significant as regards the coal M. It is known that the chemical activation with phosphoric acid catalyzes dehydration reactions and promotes depolymerization reactions. Otherwise, carbonates

Table 4
Heat of immersion in the benzene (J/g) of raw and treated coals M and D with H_3PO_4 and carbonized at various temperatures

T (℃)	M		D	
	Untreated samples	Treated samples	Untreated samples	Treated samples
No pyrolyzed	0	3.54	0	2.34
500	0.27	17.45	0.19	11.42
650	0.34	23.93	0.30	14.57
750	0.50	31.67	0.35	19.22

Table 5
Heat of immersion in the benzene (J/g) of coals M and D untreated and treated with Rb_2CO_3 and Cs_2CO_3 and carbonized at 800℃

	M		D	
	Untreated samples	Treated samples	Untreated samples	Treated samples
Rb_2CO_3	0.50	132.43	0.35	76.76
Cs_2CO_3	0.50	147.72	0.35	89.13

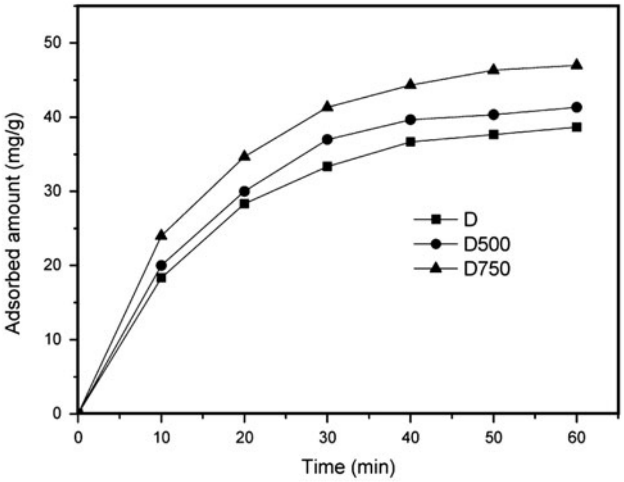

Fig. 1. Kinetics of phenol adsorption on raw coal D.

sharply increase the quantities adsorbed at the equilibrium state. Finally, the observed differences between both coals M and D are probably due to the presence of more important mineral impurities in the coal D and which may deactivate an appreciable amount of the activating agent.

3.4. Isotherms of phenol adsorption

The phenol adsorption isotherms on the two coals M and D, untreated and treated by H_3PO_4, Rb_2CO_3, and Cs_2CO_3, are presented in Figs. 7–10.

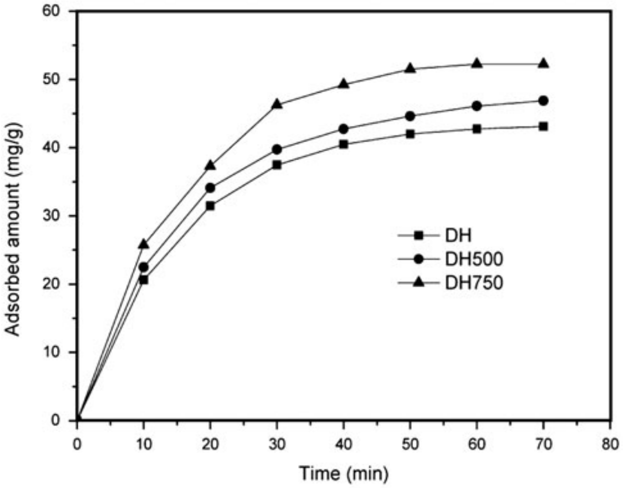

Fig. 2. Kinetics of phenol adsorption on coal D treated with H_3PO_4.

The adsorption isotherms are perfectly described by the Langmuir equation which can be written as follows:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} k_a} + \frac{C_e}{q_{\max}} \tag{4}$$

where C_e is the concentration of adsorbate in the solution at equilibrium (mg/l), q_e is the adsorbed amount at equilibrium (mg/g), $q_{\max}$ is the maximum

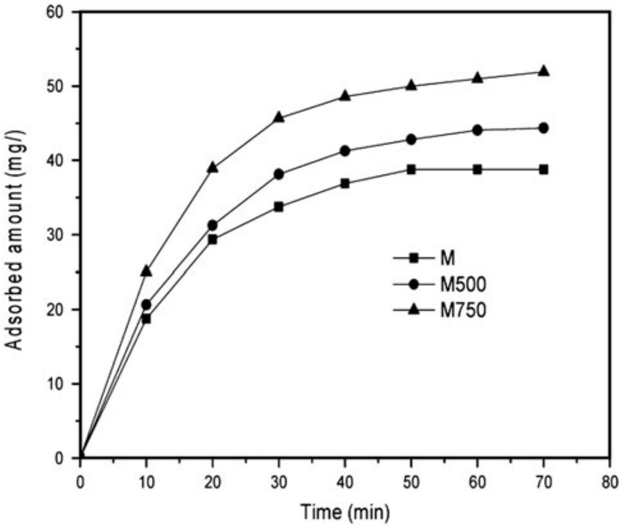

Fig. 3. Kinetics of phenol adsorption on raw coal M.

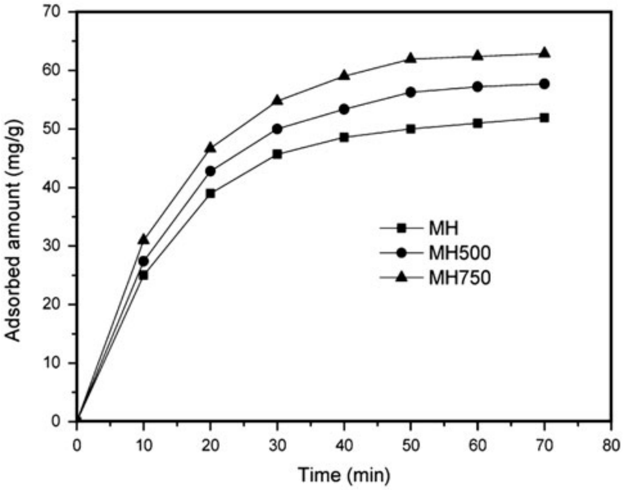

Fig. 4. Kinetics of phenol adsorption on coal M treated with H_3PO_4.

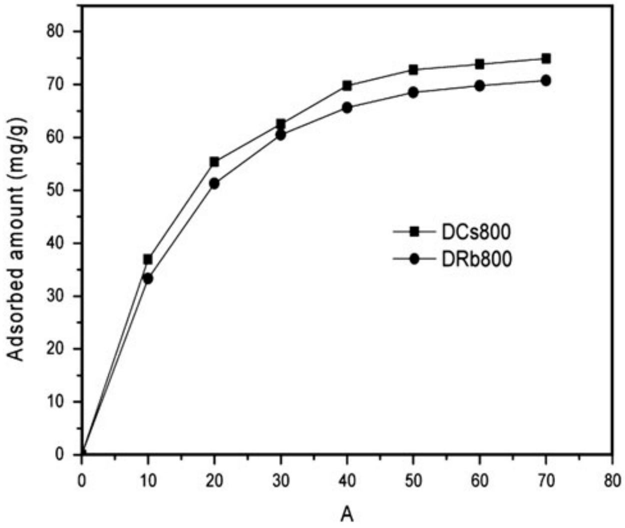

Fig. 5. Kinetics of phenol adsorption on DCs800 and DRb800.

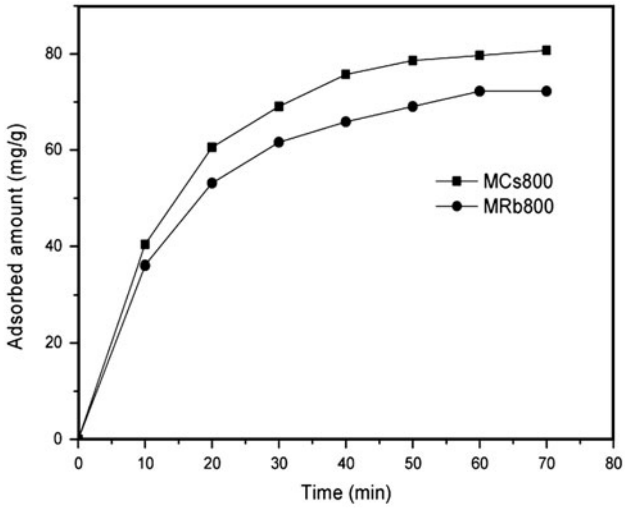

Fig. 6. Kinetics of phenol adsorption on MCs800 and MRb800.

adsorption capacity (mg/g), and k_a is the adsorption affinity constant (l/g).

Langmuir adsorption isotherms of phenol are shown in Figs. 11 and 12. The values of the adsorption capacity, q_{max}, the adsorption constant, k_a, and the correlation coefficient, R^2, are reported in Table 7.

The adsorption capacities of the treated coals M and D (DH750, MH750, DCs800, DRb800, MCs800, and MRb800) are clearly greater than those of the untreated coals D750 and M750. Different authors have reported that H_3PO_4 catalyzes dehydration reactions and promotes depolymerization reactions. However, it favors cross-link reactions and aromatization at low temperatures. It also inhibits tar formation which can obstruct some pores. Besides these effects on precursor pyrolysis, it has also been shown that H_3PO_4 produces activation through formation of phosphate and polyphosphate bridges which connect cross-link biopolymer fragments, avoiding the contraction of the material by temperature effect. Removal of the activating agent during the washing step will lead to a matrix in an expanded state with an accessible porous texture [10,21].

Regarding the activating agents Cs_2CO_3 and Rb_2CO_3, it was shown previously [15] that the quantity of CO_2, evolved during the carbonization of the

Table 6
Kinetics constants of the pseudo-second-order model for phenol adsorption

	R^2	q_e (mg/g)	$k_2 \times 10^4$
D750	0.9990	58.50	12.63
M750	0.9991	63.30	10.20
DH750	0.9980	66.42	10.13
MH750	0.9990	75.40	11.40
DCs800	0.9990	87.91	10.00
DRb800	0.9991	83.15	10.49
MCs800	0.9990	96.94	8.23
MRb800	0.9996	89.04	8.02

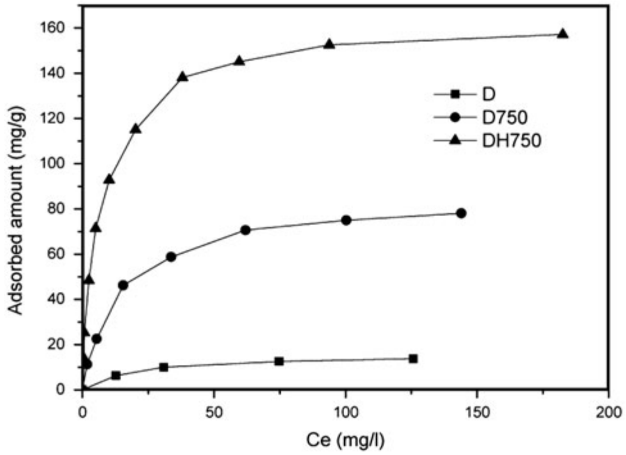

Fig. 7. Isotherms of phenol adsorption on D, D750 and DH750.

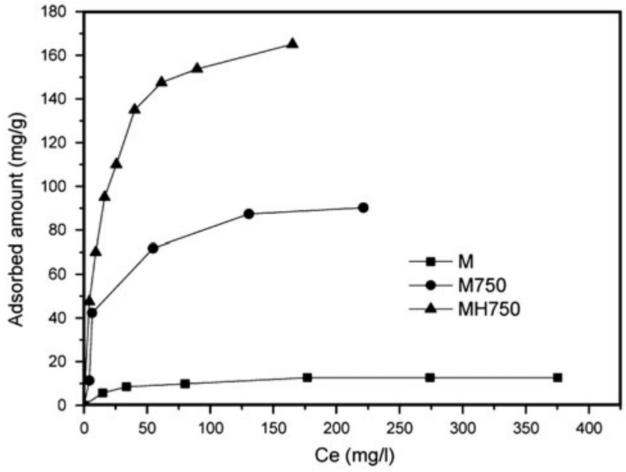

Fig. 8. Isotherms of phenol adsorption on M, M750, and MH750.

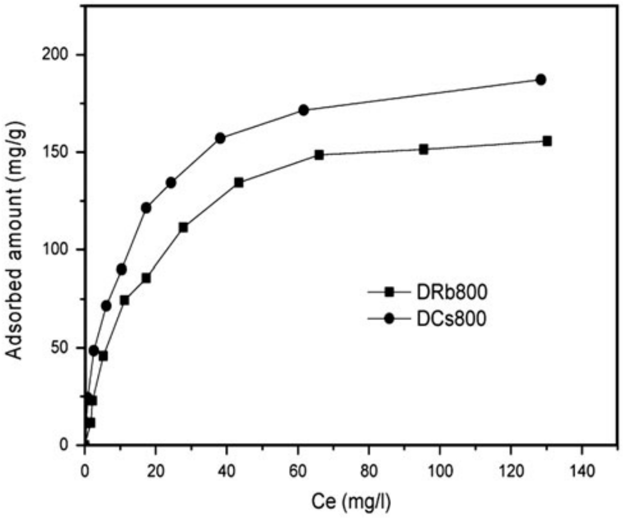

Fig. 9. Isotherms of phenol adsorption on DRb800 and DCs800.

mixture coal-carbonate, indicates the dispersion of activating agent throughout the coal and the quantity of CO evolved is dependent on the degree of activation. The total amounts of evolved CO_2 and CO during the pyrolysis up to 800°C are shown in Table 8.

Therefore, the important amounts of carbon dioxide and carbon monoxide evolved reveal that activation effect is very pronounced.

Mattson and Mark [22] assigned the mechanism of phenol adsorption as being determined by so-called π–π interactions and "donor-acceptor complex" formation. The first factor assumes that oxygen atoms bounded to the carbon reduce the π electron density and weaken the dispersion forces between phenol π

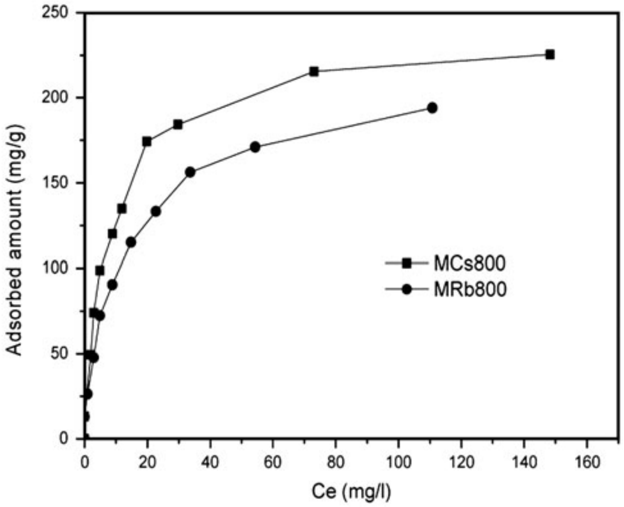

Fig. 10. Isotherms of phenol adsorption on MRb800 et MCs800.

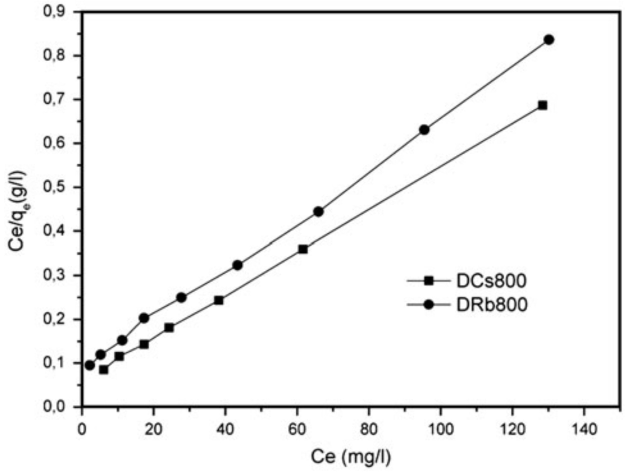

Fig. 11. Langmuir isotherm of phenol adsorption on DCs800 and DRb800.

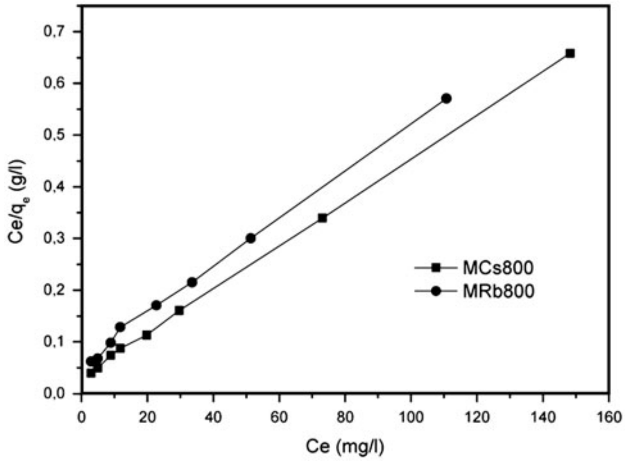

Fig. 12. Langmuir isotherm of phenol adsorption on MCs800 and MRb800.

Table 7
Parameters of the Langmuir model for phenol adsorption isotherms

	R^2	$q_{\max}$ (mg/g)	$k_a \times 10^2$
D750	0.9997	85.50	7.24
M750	0.9990	94.64	8.46
DH750	0.9999	163.30	14.06
MH750	0.9998	179.20	7.03
DCs800	0.9999	205.10	8.18
DRb800	0.9992	175.60	6.62
MCs800	0.9999	238.60	11.90
MRb800	0.9999	216.80	7.54

Table 8
Cumulative CO_2 and CO evolution during coals MCs and MRb pyrolysis up to 800°C

	CO_2 (mmol/g)	CO (mmol/g)
MCs800	5.00	19.20
MRb800	3.60	18.50

electrons ring and the π electrons of carbons. The second one postulates that the adsorption mechanism is based on the formation of donor–acceptor complexes between the surface carbonyl groups (electron donors) and the aromatic rings of phenol acting as the acceptor, and the decrease in phenol adsorption after carbon oxidation can be due to the oxidation of surface carbonyls. On the other hand, the results obtained by Terzyk [23] show that the third important factor, namely the so-called solvent effect, should be taken into account, as it strongly modifies the adsorption properties of carbons toward organics. This usually neglected effect, together with the two above mentioned ones, occurs simultaneously during adsorption on the carbons. Taking into account that the cross-sectional area of the phenol molecule is equal to 0.45 nm^2 [24] and the maximum adsorbed uptakes of phenol by MCs800 (238 mg/g) and MRb800 (216 mg/g), one deduces the following occupied areas by the phenol molecule, 685 and 622 m^2/g, respectively; otherwise, the BET surface areas of these activated carbons (MCs800 and MRb800) are 975 and 887 m^2/g, respectively. This means that a part of the porous texture of MCs800 and MRb800 is not accessible to the phenol molecule. But another explication can be done: the width of the phenol molecule is close to the one of the benzene molecule, that is 0.4 nm [17], and therefore, the phenol molecule get into the almost porous texture, the width of the pores of MCs800 and MRb800 being about 0.8 nm [15]; in this case, phenol adsorption concomitantly occurs. To confirm this last effect, experiments relating to the isotherm of phenol adsorption on the carbonaceous material characterized by a porous texture accessible to both nitrogen and phenol molecules are now in progress.

The phenol adsorption capacities of various potential adsorbents are shown in Table 9. The adsorption capacities of MCs800 and DCs800 are higher than the other adsorbents; however, they are comparable to the results obtained by Utrilla et al. [27] for a demineralized bituminous coal pyrolyzed at 1,123 K and activated in steam at 1,113 K which its weight loss (>55%) is widely larger than the one of MCs800 (33%). The same remark concerns the results of Laszlo et al. [24]. It can be concluded that Algerian coals activated with

Table 9
Comparison of adsorption capacities of phenol between activated Algerian coals and various activated carbons

Adsorbent	Adsorption capacity (mg/g)	Refs.
Commercial activated carbon	137	[19]
Date pits activated carbon (CO_2 activation, burn-off: 37%)	161	[20]
CCM 200 (carbon cryogel)	140	[25]
Activated carbon ($ZnCl_2$ activation)	55	[26]
Activated bituminous coal (steam): -AP-48 (burn-off: 48%)	152	[27]
-CP-55 (demineralized, burn-off: 55%)	218	
Grain husk activated carbon (H_2O activation, burn-off: 39%)	209	[24]
Activated carbon (MCs800)	238	This study
Activated carbon (MRb800)	216	

Cs_2CO_3 and Rb_2CO_3 could be employed as effective adsorbents for removal of phenol from aqueous solution.

4. Conclusions

The two Algerian coals investigated in this work can be used as precursors to produce activated carbons characterized by a developed porous texture. Indeed, the phenol adsorption capacity on these activated carbons is significant, especially in the case of the cesium carbonate activating agent. The kinetics of phenol adsorption on the coals M and D untreated and treated by H_3PO_4, Cs_2CO_3, and Rb_2CO_3 are well described by the pseudo-second-order model. Otherwise, the isotherms of phenol adsorption on the same activated carbons perfectly fit Langmuir equation. The mechanism of phenol adsorption can be described by so-called "π-π" interactions, "donor-acceptor complex" formation, and "solvent effect." According to the occupied surface area by the adsorbed phenol molecules, the solvent effect should play an important role in adsorption mechanism of phenol in this study.

Abbreviations

HTH	—	heat treatment temperature
M, D	—	coals Menouna and Didi, respectively
M500, M650, M750	—	coal Menouna pyrolized at 500, 650, and 750°C successively
D500, D650, D750	—	coal Didi pyrolized at 500, 650, and 750°C successively
MH500, MH650, MH750	—	coal Menouna treated with H_3PO_4 and pyrolized at 500, 650, and 750 successively
DH500, DH650, DH750	—	coal Didi treated with H_3PO_4 and pyrolized at 500, 650, and 750 successively
MCs800, MRb800	—	coal Menouna treated with Cs_2CO_3 and Rb_2CO_3 successively and pyrolized at 800°C
DCs800, DRb800	—	coal Didi treated with Cs_2CO_3 and Rb_2CO_3 successively and pyrolized at 800°C
TPD	—	temperature-programmed desorption

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