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Adsorption of cyanide ions in aqueous solution using raw and oxidized coke

Z'hour Hattab[a], Naima Filali[a], Radia Mazouz[a], Kamel Guerfi[a], Nacer Rebbani[a,*], Atika Nafa[b], Salah Kheriaf[b]

[a]*Laboratory of Water Treatment and Valorization of the Industrial Waste, Faculty of Sciences, Department of Chemistry, Badji-Mokhtar University, B.P.12, Annaba 23000, Algeria, Tel. +213 797598522; email: zoumourouda20012000@yahoo.fr (Z'. Hattab), Tel. +213 792764612; email: filali_naima@yahoo.com (N. Filali), Tel. +213 698675131; email: jihen2002@hotmail.com (R. Mazouz), Tel. +213 666132069; email: k_guerfi@yahoo.fr (K. Guerfi), Tel. +213 771250811; email: nacer.rebbani@univ-annaba.dz (N. Rebbani)*

[b]*Laboratory of Water and Raw Material Quality, Environment Department, Central Laboratory, Direction Metallurgy–Quality, Complex Arcelor-Mittal, Annaba, Algeria, Tel. +213 770956685; email: atika.nafa@arcelormittal.com (A. Nafa), Tel. +213 770997617; email: kheraif@arcelormetal.com (S. Kheriaf)*

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ABSTRACT

The objective of this study was the use of raw and modified coke material for the adsorption of cyanide ions (CN^-) in water. The surface functions of this material (carboxyl, lactones, phenol, and carbonyl) were modified by oxidation with sulfuric acid, nitric acid, and $KMnO_4$ solution at varying concentrations. This chemical surface treatment improved the adsorption capacity. The experimental results of adsorbent/adsorbate equilibrium show that the adsorption isotherms follow the Freundlich model and the kinetic adsorption follows the pseudo-second-order model. Overall, coke material oxidized by $KMnO_4$ has shown the best adsorption of CN^- ions with an adsorption capacity $q_e = 1.28$ mg g^{-1} at $T = 20$°C and pH 11.

Keywords: Adsorption; Cyanide ions; Coke modified; Surface functions; Water treatment

1. Introduction

Cyanide ions, due to its huge uses, can exist in wastewater which is discharged from different factories [1,2]. However, many cyanide compounds including cyanide anion are highly toxic. Several technologies are available for the removal of cyanide ions from water such as precipitation, complexation, separation by foam, and ion exchange. Furthermore, the destruction of cyanide compounds in industrial effluents can also be carried out by oxidation using different reagents like hypochlorite of sodium [3,4], chlorine gas [5], peroxide [6,7], ozone [8,9], and Caro's Acid [10] leading to the formation of cyanate which is not toxic [11]. Of these mentioned techniques, the adsorption can be used for fixing of cyanide ions in wastewater [12,13]. Usually, the commercial granular-activated carbon is used as an adsorbent for water treatment thanks to its effectiveness. However, the high production cost and the difficulty of its regeneration are still the major drawbacks of this material [14–18]. Recently, several researchers have been interested in using cheaper natural materials as an alternative adsorbent for water treatment [19–22]. The coke is an abundantly available material as a discharged

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*Corresponding author.

waste from the cast iron manufactures. This material which has a large specific surface could be used successfully as an adsorbent. However, few studies have reported its adsorption capacity in liquid medium for the purification of aqueous solutions.

In this work, we have improved the sportive properties of the coke material by the chemical modification of its superficial functional group (carboxyl, lactones, phenol, and carbonyl). For this reason, three chemical oxidizing agents were used: sulfuric and nitric acids, and potassium permanganate. The adsorption of cyanide ions on raw and oxidized/modified coke has been tested. Some process parameters (contact time, pH, temperature, and initial substrate concentration) have been studied.

2. Materials and methods

The raw coke material used in this study as an adsorbent was provided by the metallurgical complex Arcelor-Mittal manufactory (located in Annaba, Algeria). Its chemical composition was determined by wet process. The distribution of the sizes of particles was measured by a laser granulometer (MASTER SIZER MALVERN). Only particles of size less than 110 µm were used. The tests of adsorption were carried out in static mode. The determination of the concentration of cyanides was realized by direct ionometry, using a potentiometer (PHILIPS PW 9420) equipped with a cyanide specific electrode, whose membrane consisted of the mixture $AgI + Ag_2S$, marketed by Tacussel (Ref. PCN 2 M) in association with a reference electrode of saturated calomel $Hg/Hg_2Cl_2\ Cl_2/KCl$ (Ref. Tacussel C8). The lower detection limit of this electrode is 10^{-7} M.

2.1. Method of modification of the raw coke

Several authors [23–25] have studied both the modification of texture and character and/or content of functional groups on the surface of different adsorbents. Therefore, several chemical oxidizing agents with different actions and particular effects on the surface have been tested: $HNO_3, H_2O_2, KMnO_4$, air, ozone, etc.

In this work, the content modification of various functional surface groups was carried out by oxidation using H_2SO_4 at 40%, HNO_3 at 40%, and finally $KMnO_4$ at 1 N.

2.1.1. Oxidation by H_2SO_4 *and* HNO_3

The same procedure was used for the oxidation with H_2SO_4 and HNO_3. Firstly, the raw coke was dried at 110˚C. Then, 20 g of raw coke and 250 mL of oxidation solution of sulfuric or nitric acid solution at a concentration of 40% were mixed in a balloon of 1 L equipped with a cooling system. The mixture was stirred under reflux system for 4 h. After separation, the wet solid obtained was washed by double-distilled water until neutrality and filtered. Then, it dried at 110˚C for 24 h.

2.1.2. Oxidation with the permanganate of potassium

In a 1 L Pyrex reactor, 20 g of raw coke was mixed with 250 mL of a normal solution of $KMnO_4$ in a 4 N sulfuric medium under agitation until complete discoloration of the solution. Then, a purification of the sample was carried out using the same procedure described by Donnet [26]. The obtained material was filtered and dried at a temperature ranging between 105 and 110˚C.

2.2. Kinetics of elimination of cyanide

The elimination of the cyanide was carried out by contacting a coke mass (1 g) with the cyanide solution (50 mL) in a polyethylene reactor with double envelope while maintaining constant temperature using a thermostatic bath (Julabo A6—Julabo 3U). The cyanide solutions (Merck) with a concentration of 26 mg L^{-1} were prepared at different pH values: 10, 11, and 12. Furthermore, the adsorption process was tested under three various temperatures: 20, 30, and 40˚C under mechanical agitation for 24 h. The residual concentration of cyanide is given in the supernatant which is separated by centrifugation.

The adsorbed quantities of cyanide q_e were calculated using Eq. (1).

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

where q_e (mg g^{-1}) is the capacity of adsorption of equilibrium; C_0 and C_e are, respectively, the initial and equilibrium concentrations (mg L^{-1}); V (L) is the volume of the solution; m (g) the mass of the adsorbent.

2.3. Adsorption isotherm of cyanide

The adsorption isotherms were investigated by mixing of 1 g of raw or oxidized coke with 50 mL of cyanide solution at different concentrations, under the optimal conditions obtained in the kinetic study (pH 11, $T = 20°C$, stirring speed constant = 200 rpm/min, and contact time = 4 h). The models of Langmuir and Freundlich were exploited in the study of the adsorbent capacity of the coke.

3. Results and discussion

3.1. Characterization of material

Elemental analysis of raw and oxidized coke was carried out by wet process. The obtained results are reported in Table 1.

The chemical surface functions of the different coke samples were determined by the method of Boehm [27–29]. The obtained results of the consumed quantity of base and the distribution of the functional groups of our samples are presented, respectively, in Table 2 and Fig. 1.

The results show that the stronger the base is, the larger is the consumed quantity of base and that it is larger for the coke oxidized with potassium permanganate than for the coke oxidized with nitric and sulfuric acid.

Fig. 1 demonstrates that the content of surface functional group (carboxyl, lactones, phenol, and carbonyl) increases after oxidation by different agents where the phenolic and carbonyl groups are more pronounced. Furthermore, the oxidation by potassium permanganate showed the highest improvement of the functional groups of coke material.

The results presented in Table 3 show that the predominant functions on the surface of raw and oxidized coke are acid type.

By considering the coke of the form $R - C_xH_yO_z$, where $C_xH_yO_z$ represent a functional group such as COOH, CHO, or CH_2OH and R is the coke surface, we could explain variations in pH by the two following acido-basic equilibriums:

$$R - C_xH_yO_z + H_2O = R - C_xH_{y-1}O_z^- + H_3O^+ \tag{2}$$

$$R - C_xH_yO_z + H_2O = R - C_xH_{y+1}O_z^+ + OH^- \tag{3}$$

These equilibriums are accompanied by a variation of the number of charge carriers on surface of the solid and in the liquid. The electro neutrality of these two phases is respected (equal number of charges on the surface and in the solution).

In the case of a mineralized aqueous solution, electric neutrality of each phase is respected by exchanging, either a proton or a hydroxide ion with an ion of the solution such as Na^+ or CN^- for example. Thus in the case of a cation, equilibrium (Eq. 2) will be modified as follow:

$$Na^+ + R - C_xH_yO_z + H_2O \rightarrow R - C_xH_{y-1}O_z^-Na^+ + H_3O^+ \tag{4}$$

For anions, exchange sites of the solid are probably carboxylates or phenolates, such as the case of activated carbon [30]. The exchange of anions such as CN^- is done with OH^- according to reaction 5:

$$CN^- + R - C_xH_yO_z + H_2O \rightarrow R - C_xH_{y+1}O_z^+CN^- + OH^- \tag{5}$$

3.2. Influence of temperature and pH

The results of the influence of pH and temperature on the adsorption of cyanide on the raw and oxidized coke samples are shown in Figs. 2–5. From these results, we can conclude that, the optimum temperature and pH are, respectively, 20°C and pH 11.

Table 1
Chemical composition of raw and oxidized coke

	Chemical composition										
	Coke							Ashes			
Coke samples	C	Ashes	Ashes/dry	VM	VM/dry	S	H_2O	SiO_2	Al_2O_3	Fe_2O_3	CaO
Raw	85.63	11.19	11.15	0.94	0.57	0.56	0.37	40.50	33.58	14.58	5.64
Oxidized by H_2SO_4	88.15	10.15	10.12	0.81	0.42	0.53	0.39	38.00	27.10	6.70	2.82
Oxidized by HNO_3	87.37	8.98	8.90	2.35	1.47	0.50	0.88	58.10	22.94	9.79	6.97
Oxidized by $KMnO_4$	69.79	23.18	22.99	5.99	5.15	0.39	0.84	55.03	20.39	11.41	11.62

Notes: VM: volatile matter. The chemical composition of raw and oxidized coke is obtained by summing C + Ashes + MV + S. SiO_2, Al_2O_3, Fe_2O_3 and CaO represent the chemical composition of the ashes.

Table 2
Quantities of base consumed by the raw and oxidized coke (expressed in μeq g^{-1}) in order to determine the functional groups of surface

Coke samples	$NaHCO_3$	Na_2CO_3	NaOH	$NaOC_2H_5$
Raw	39	48	72	155
Oxidized by H_2SO_4	79	49	138	279
Oxidized by HNO_3	99	86	114	328
Oxidized by $KMnO_4$	156	130	221	415

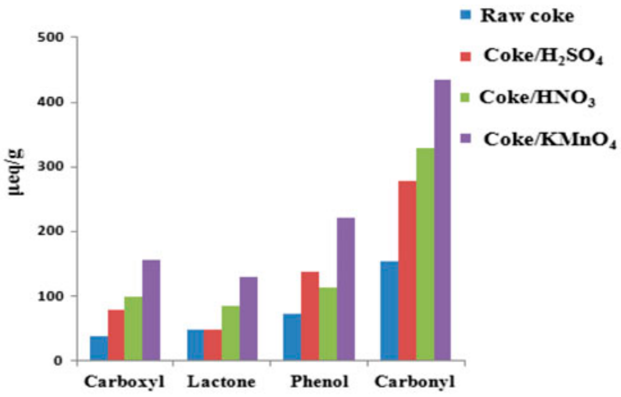

Fig. 1. Amounts of surface functions of the raw and oxidized coke.

3.3. *Kinetic study of the adsorption of cyanide on the raw and oxidized coke*

The results of kinetic adsorption of cyanide on different samples (raw and oxidized coke) are shown in Fig. 6. From this figure, the adsorption equilibrium was practically reached at about four hours of contact, which is reduced to more than half for the coke oxidized by $KMnO_4$. After that, the adsorption was not changed after 24 h of contact and this quantity may be slightly higher for the coke oxidized by $KMnO_4$.

Furthermore, we can deduce that the coke oxidized by $KMnO_4$ has an adsorption speed faster compared to the other samples. This is due to fact that $KMnO_4$ is a stronger oxidant than HNO_3 and H_2SO_4, which creates more number of surface functions.

3.4. *Kinetic modeling*

Several kinetic models are developed in order to highlight the essential parameters of the kinetics of adsorption. The adsorption of cyanides on the raw coke and oxidized samples was modeled by using the equations of the pseudo-first-order [31] (Eq. 6) and of the pseudo-second-order [32] (Eq. 7).

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{6}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{7}$$

where t: contact time; k_1: constant of adsorption speed of the kinetics of pseudo-first-order; k_2: constant of adsorption speed of the kinetics of pseudo-second-order; q_t and q_e: adsorption capacities, respectively, at the moment t and at equilibrium.

The study of the linear regressions presented in Fig. 7 and represented in Table 4 shows that the elimination of CN^- by the raw and oxidized coke follows the two model kinetics but with better adequacy for the model of the pseudo-second-order (R varying from 0.9937 to 1).

We notice that the coke oxidized by $KMnO_4$ has an adsorption speed faster compared to the other samples. The CN^- ions are better adsorbed at a temperature of 20°C and an initial pH around 11, pH for which all the cyanide is in the form of free CN^- [3].

The results of Table 4 show that the theoretical values of q_e are better estimated by the theoretical model of second-order than by that of first-order which indicates that the adsorption of the CN^- ions by the raw or oxidized coke is dominated by a chemical process.

Table 3
Amounts of surface functional groups of the raw and oxidized coke

Coke samples	Raw	Oxidized by H_2SO_4	Oxidized by HNO_3	Oxidized by $KMnO_4$
Total of acid functions (μeq g^{-1})	314	545	627	922
Basic groups (μeq g^{-1})	131	70	87	175

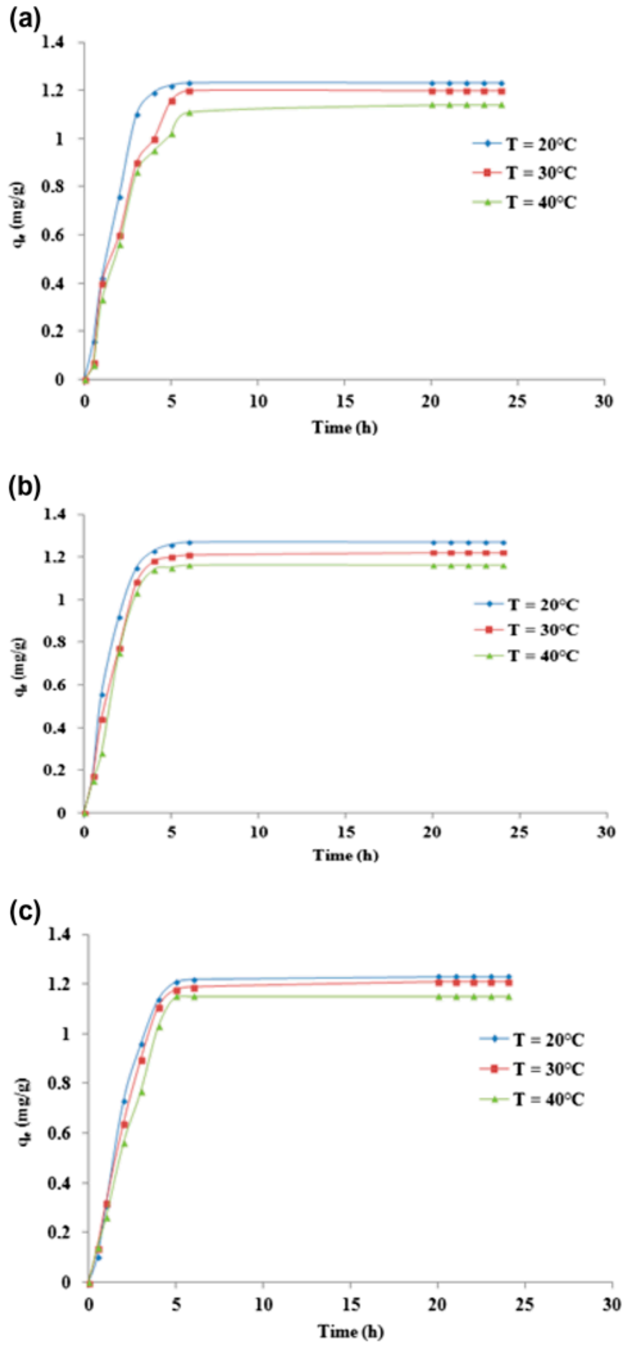

Fig. 2. Effect of temperature and pH on the adsorption of cyanide on raw coke (a) pH 10, (b) pH 11, and (c) pH 12 ([CN$^-$] = 25.85 mg L^{-1}, V = 50 mL, m = 1 g).

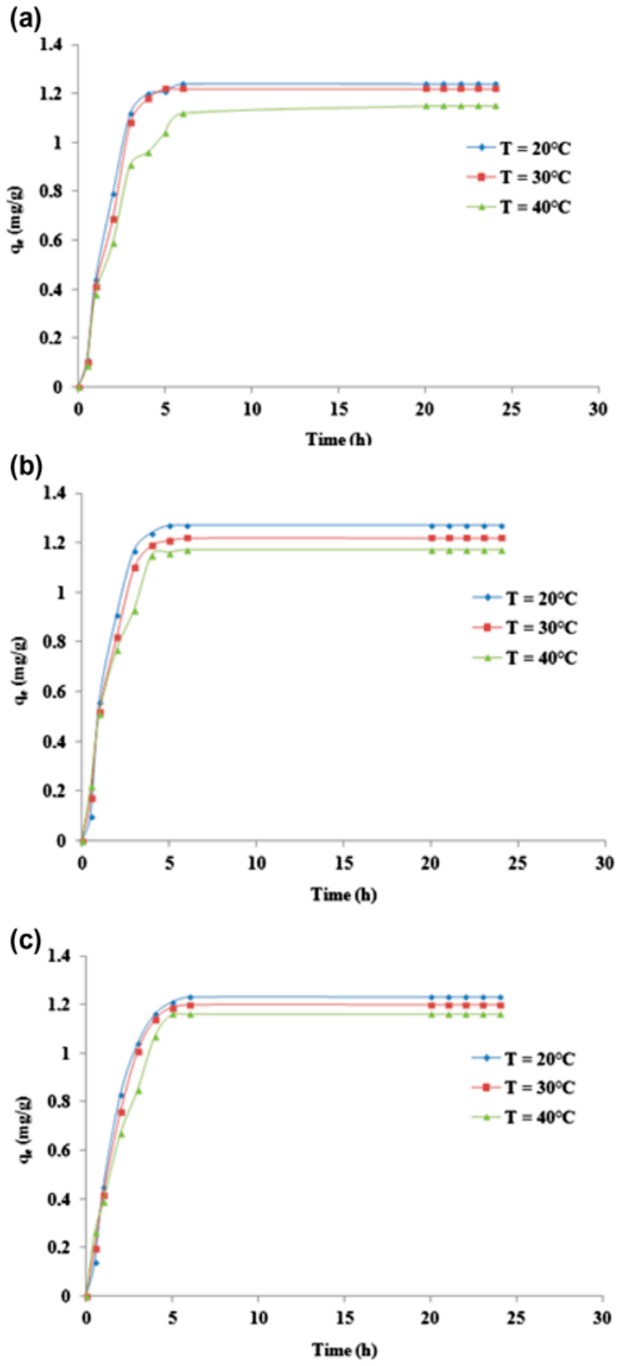

Fig. 3. Effect of temperature and pH on the adsorption of cyanide on coke oxidized by H_2SO_4 (a) pH 10, (b) pH 11, and (c) pH 12 ([CN$^-$] = 25.85 mg L^{-1}, V = 50 mL, m = 1 g).

3.5. Adsorption isotherm

The establishment of adsorption isotherms enables to calculate the maximum quantity adsorbed by the solid and also the identification of the type of adsorption. The experimental results obtained show that the isotherm is of type S, which corresponds to the classification of Gilles et al. [33]. This last, indicates a growth of adsorption when the concentration of the adsorbate increases (Fig. 8).

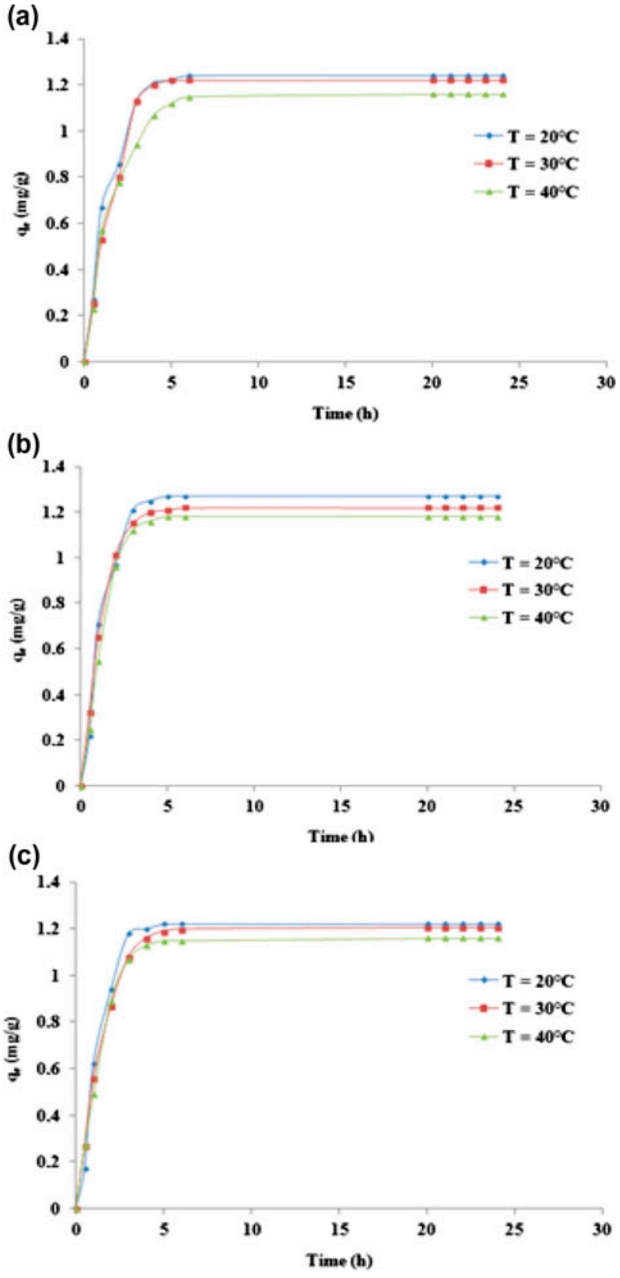

Fig. 4. Effect of temperature and pH on the adsorption of cyanide on coke oxidized by HNO_3 (a) pH 10, (b) pH 11, and (c) pH 12 ($[CN^-] = 25.85$ mg L^{-1}, $V = 50$ mL, $m = 1$ g).

The examination of the isotherm of Langmuir [34] and that of Freundlich [35] allows us to calculate the maximum quantity adsorbed by the solid. The modeling equations of the adsorption of Langmuir (Eq. 8) and Freundlich (Eq. 9), in their linearized forms, are as follows:

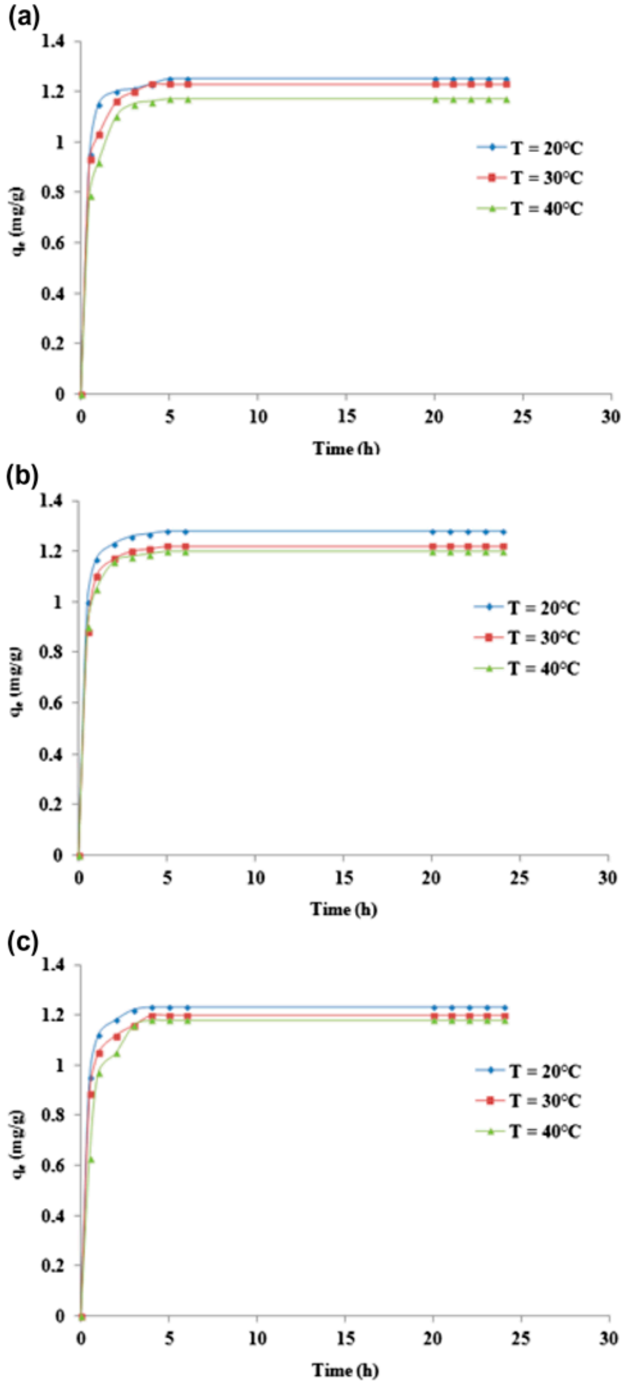

Fig. 5. Effect of temperature and pH on the adsorption of cyanide on coke oxidized by $KMnO_4$ (a) pH 10, (b) pH 11, and (c) pH 12 ($[CN^-] = 25.85$ mg L^{-1}, $V = 50$ mL, $m = 1$ g).

$$\frac{C_e}{q_e} = \frac{1}{bq_m} + \frac{C_e}{q_m} \tag{8}$$

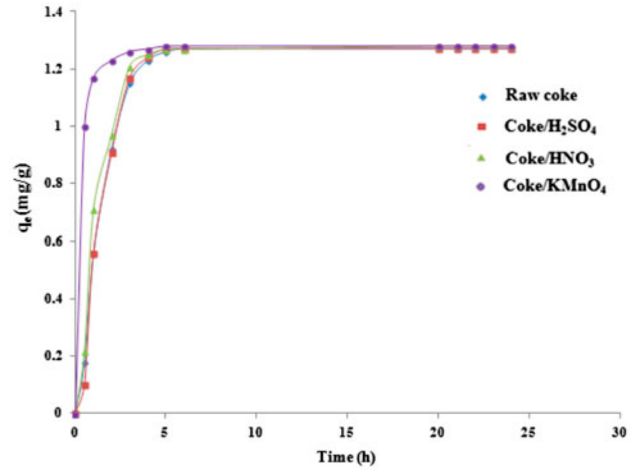

Fig. 6. Adsorption kinetics of CN^- on the raw and oxidized coke ($[CN^-]$ = 25.85 mg L^{-1}, V = 50 mL, m = 1 g, T = 20˚C, pH 11).

$$\ln q_e = \frac{1}{n} \ln C_e + \ln k_f \tag{9}$$

where q_e: adsorption capacity of the solute per gram of adsorbent at equilibrium (mg g^{-1}); q_m: Langmuir maximum adsorption capacity (mg g^{-1}); C_e: concentration of the solute at equilibrium (mg L^{-1}); b: constant of Langmuir connected to the energy of adsorption (L mg^{-1}); k_f and n: empirical constants of Freundlich representing, respectively, the capacity and the intensity of adsorption.

The study of Langmuir and Freundlich adsorption isotherms are represented in Fig. 9 and Table 5. From these results, according to the values of R^2 (Table 5), it is clear that the Freundlich model is the better. The values of the constant of Freundlich k_f indicate that there is a strong adsorption of CN^- and it increases with the oxidation of the coke (H_2SO_4, HNO_3, and $KMnO_4$).

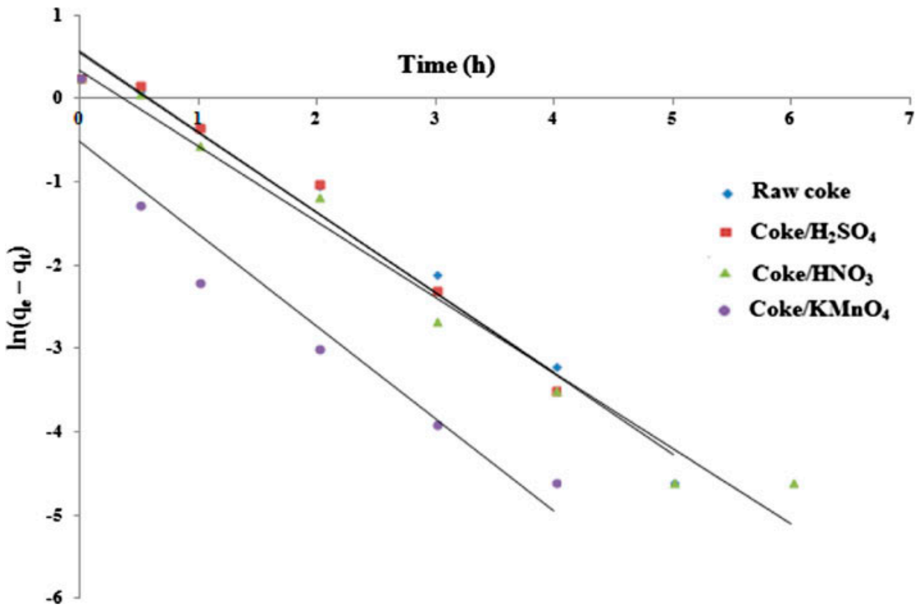

Fig. 7. Representation of the kinetic model of pseudo-second-order for adsorption of CN^- on raw and oxidized coke (pH 11, V = 50 mL, T = 20˚C, $[CN^-]$ = 25.85 mg L^{-1}, m = 1 g).

Table 4
Adsorption kinetic parameters of CN^- on raw and oxidized coke

	Model						
		PFO			PSO		
Coke samples	$q_{e,exp}$	k_1 (min^{-1})	$q_{e,cal}$ (mg g^{-1})	R^2	k_2 (g mg^{-1} min^{-1})	$q_{e,cal}$ (mg g^{-1})	R^2
Raw	1.27	0.9690	1.7726	0.9811	0.6979	1.3377	0.9937
Coke/H_2SO_4	1.27	0.9584	1.7292	0.9752	0.4362	1.3764	0.9742
Coke/HNO_3	1.28	0.9076	1.4028	0.9763	0.9371	1.3310	0.9962
Coke/$KMnO_4$	1.28	1.1104	0.5977	0.9269	10.9831	1.2843	1

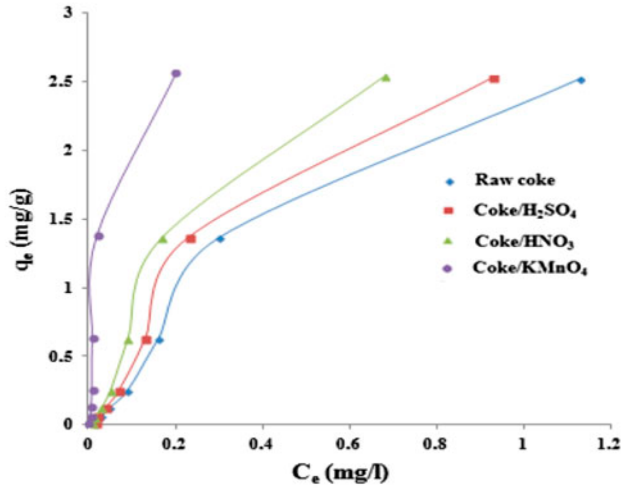

Fig. 8. Adsorption isotherms of CN^- ions on raw and oxidized coke (V = 50 mL, m = 1 g, T = 20˚C, pH 11).

For the Freundlich model, the constant $1/n$ is an indicator of the intensity of adsorption. The values of n ranging between 1 and 10 represent a favorable adsorption [36]. In this study, the coke oxidized by $KMnO_4$ is the only one to provide a value of n greater than 1 (1.2889) demonstrating a favorable condition for elimination of CN^- by this modified coke.

From an experimental point of view, the achieved results show that the coke oxidized by $KMnO_4$ is more effective for the treatment of cyanided water. We confirmed these results while carrying out an experimental checking, on the scale of the laboratory, of water containing 13 mg L^{-1} of cyanide.

The method consists of putting in contact, in a reactor, 1 g of raw or oxidized coke, with 50 mL of the cyanide solution. Every hour, the solution is filtered and the CN^- concentration measured, then 1 g of coke is added to this solution and after an hour it is again filtered and the CN^- concentration measured. These operations are repeated, for each coke sample, until reaching the limit of detection of the method of the assay.

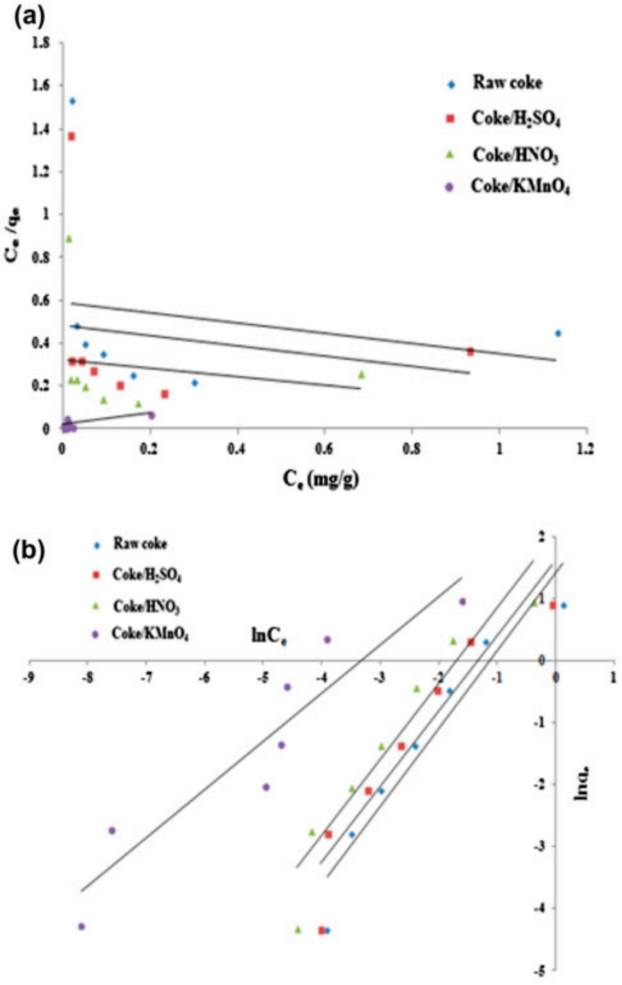

Fig. 9. Adsorption isotherm of cyanide on raw and oxidized coke according to the model (a) Langmuir and (b) Freundlich (V = 50 ml, m = 1 g, T = 20˚C, pH 11).

The standards of $[CN^-]$ recommended by WHO for a drinking water are 0.07 mg L^{-1} and in accordance with the recommendation of the USEPA, concentration limit of free cyanide in the rejection of effluents is of 0.2 mg L^{-1} [37].

Table 5
Values of adsorption parameters of CN^- on the raw and oxidized coke

	Langmuir			Freundlich		
Coke samples	R^2	q_e (mg g^{-1})	b (L g^{-1})	R^2	$1/n$	k_f (L g^{-1})
Raw	0.0444	−4.1476	−0.4087	0.9186	1.2535	4.1263
Coke/H_2SO_4	0.0334	−4.2698	−0.4870	0.8940	1.2258	5.2033
Coke/HNO_3	0.0327	−4.9285	−0.6241	0.9040	1.2285	8.1181
Coke/$KMnO_4$	0.6016	3.8343	11.6430	0.8786	0.7758	13.2951

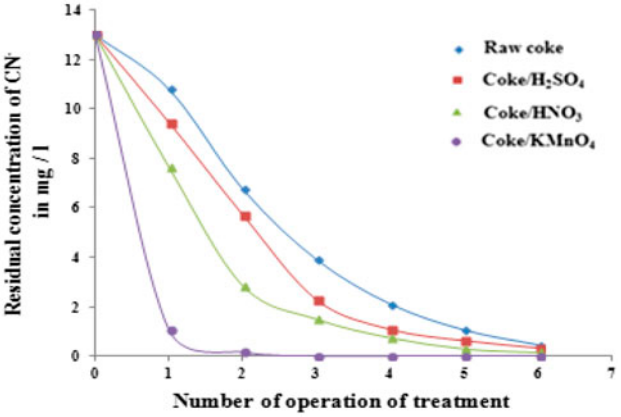

Fig. 10. Evolution of the residual concentration of CN^- with the number of operations realized.

Table 6
Comparison of the adsorption of the coke for CN^- with other adsorbants

Materials	Quantity of adsorbent ($g\ L^{-1}$)	pH	$[CN^-]_0$ ($mg\ L^{-1}$)	% of cyanide elimination	Reference
Residual coal	40	9	20	82	[37]
Fly ash	40	9	20	78.89	[38]
Pistachio-green hull wastes α-	20	7.34	10	89	[39]
Alumina (E. MERCK)™	1.5	10	100	99	[40]
Coke oxidized by $KMnO_4$	10	11	25.85	99	/

We note, according to Fig. 10, that for the case of the coke oxidized by $KMnO_4$ only two operations are necessary to reduce, below the recommended standards, the initial concentration of CN^-. On the other hand, to reach the same result, five operations are necessary for the coke oxidized by the nitric acid and six for the raw coke or the coke oxidized by the sulfuric acid.

Since each operation lasts for 1 h, coke oxidized by $KMnO_4$ enables the treatment of cyanided water three times faster than a coke oxidized by H_2SO_4 or HNO_3.

Moreover, as shown in Table 6, we compared our results obtained with the coke oxidized by $KMnO_4$ with other materials reported in the literature for CN^- adsorption. From this table, the coke oxidized by $KMnO_4$ shows a high potential for CN^- removal from water with a rate in the same order as the reported materials.

4. Conclusion

In this work, we showed the adsorption capacity of raw and oxidized coke by different agents (H_2SO_4, HNO_3, and $KMnO_4$) for the removal of cyanide ions from water. The results demonstrate that the oxidation improved the sorption properties of the raw coke by developing its surface functional group (carboxyl, lactones, phenol, and carbonyl). The application of the Boehm's method permitted the determination of the surface functional group of the different samples and showed that the oxidation creates especially phenolic and carbonyl functions. The adsorption kinetics is better described by the pseudo-second-order model. According to the high values of correlation coefficients (R^2), the adsorption isotherm of CN^- on different samples of coke follows the model of Freundlich. The coke oxidized by $KMnO_4$ has a high effectiveness for the elimination of CN^- with a maximum adsorption capacity of $q = 1.28$ mg g^{-1}.

Nomenclature

b	— empirical equilibrium constant of Langmuir isotherm
C_0	— initial concentrations of CN^-
C_e	— equilibrium concentration of CN^-
t	— time
q_e	— the amount of CN^- adsorbed at saturation
q_t	— the amount of CN^- adsorbed at time t
q_m	— maximum adsorption capacity
k_f	— the Freundlich constant
k_1	— rate constant of the first-order reaction kinetics
k_2	— pseudo-second-order rate constant
$1/n$	— adsorption intensity
m	— amount of the coke
V	— volume of the solution
R^2	— correlation coefficients
VM	— volatile matter
PFO	— pseudo-first-order
PSO	— pseudo-second-order
WHO	— World Health Organization
USEPA	— United States Environmental Protection Agency

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