

**KINETIC AND ISOTHERM ADSORPTION STUDIES OF COBALT (II) ON
MESOPOROUS ALUMINOSILICATE MATERIALS AND MESOPOROUS CARBON
OF MCM-41 TYPE**

S. Boumessaidia^{1,2,3}, O. Mohammedi², B. Cheknane², N. Bouchenafa-saib², K. Bachari³

¹Faculty of Exact Science, Hamma Lakhdhar University El Oued, Algeria

²Laboratory Physical Chemistry of Interfaces of Materials Applied to the Environment, Saad Dahleb University Blida, Algeria

³Center for Scientific and Technical Research in Physico-Chemical Analysis, Bousmail Tipaza, Algeria

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ABSTRACT

The main objective of this work is to study the use of silica and alumina materials and coals mesoporous for the adsorption of cobalt ions from a synthetic industrial effluent. Three types of mesoporous materials MCM-41, Al-MCM-41 (10), Al-MCM-41 (20) and one carbon CMK-3M were synthesized. The characterization is carried out by X-ray diffraction and nitrogen adsorption-desorption (BET). Efficiency of prepared adsorbents was tested toward the adsorption of cobalt (II) ions. Modeling of the adsorption kinetics of cobalt by the four materials in an aqueous medium under optimal operating conditions is pseudo first order. The adsorption isotherm on the four materials shows that the Langmuir model is favorable only with CMK-3M coal. On the other hand, the model of Freundlich is favorable with the four matrices studied with values of adjustment coefficient close to unity.

Key words: mesoporous materials, alumino-silicates, nanocasting, carbons, isotherm, adsorption.

Author Correspondence, e-mail: boumessaidiaselman@mail.com

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1. INTRODUCTION

The elimination of heavy metals released by industrial water in environments is very interesting because of their toxicity and industrial activities in large quantities of non-degradable heavy metals [1]. Cobalt is one these pollutants which can be found in water, soil and air as a result of industrial releases and because of its use in the manufacture of alloys and other fields. It has been found that exposure to a very high concentration of cobalt can nevertheless be detrimental to health. Pulmonary conditions (asthma, pneumonia, wheezing) have been observed in workers who had been breathing heavily cobalt-polluted air.

Adsorption [2] has the advantage that it can be applied to the treatment of various effluent composed of organic and / or inorganic pollutants. In this context, several adsorbents are used for the purification of these effluents.

Organized mesoporous silicas are one of the effective ways to remove a wide range of contaminants because of their texture characteristics. Since their discovery in the early 1990s, mesoporous silica [3-7] has attracted a lot of interest in many fields of research from around the world, the physicochemical characteristics of these materials then allow their use in various fields such as catalysis [8], adsorption [9], separation [10], chromatography [11,12] and biochemistry as a support for enzymatic immobilization [13] or the release of drugs [14].



Fig.1. The MCM-41 formation mechanism proposed by Beck et al. [15]

Mesoporous coals synthesized from mesoporous materials by the mechanism of nanocasting; nanocasting has recently been developed as an alternative way to obtain a negative replica from a mesoporous materials used as a rigid mold [16].

The principle of nanocasting can be summarized by three synthesis steps schematized in Figure 2.

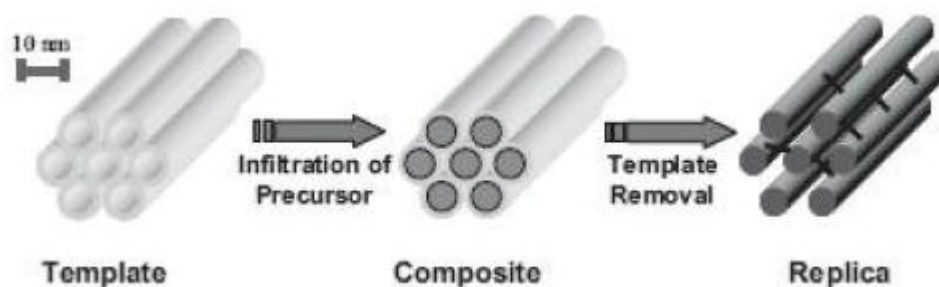


Fig.2. Illustrative diagram of the nanocasting method [17]

The present study focus on the comparative study of the adsorption capacity of the Co^{2+} metal ion on the mesostructured materials of the MCM-41, Al-MCM-41 (10) and Al-MCM-41 (20) type and the mesoporous coal CMK-3M synthesized from MCM-41.

2. EXPERIMENTAL

2.1. Synthesis of MCM-41 Material

The synthesis of the MCM-41 phase is carried out in a basic medium from an aqueous solution containing the cationic surfactant (hexadecyl-trimethyl ammonium bromide (CTAB)) and the source of tetraethylorthosilicate silica (TEOS) [18,19]. It therefore involves mechanisms involving S+/I- type interactions. In order to prepare these adsorbents we followed the protocol given by 'Bagshaw and Brucea'.

A quantity of 0.8 g of CTAB in distilled water is dissolved in a beaker until a translucent colorless solution is obtained. After that we added a volume of 2.66 mm of ammonia in a room temperature. The addition of 3.33 ml of TEOS dropwise gives a whitish cloudiness and thickening; this phenomenon followed by a sudden precipitation of silicic species. Just before the end of the addition, the opaque white mixture is homogenized (stirring for two hours) before hydrothermal treatment. The gel is placed in a Teflon autoclave at 100 °C under static conditions for 3 days. The suspension containing the mesophase is cooled, the white solid filtered on Buchner and washed extensively with distilled water until a filtrate pH around a value of 7. The solid washed and dried in the oven (40 °C, 24h), ground and then calcined in a muffle furnace under a stream of dry air at 550 °C for 13 h (ramp 1 °C / min).

2.2. Synthesis of Al-MCM-41 Material

All the mesoporous materials Al-MCM-41 with Si / Al ratios (20 and 10), are obtained directly, using an ionic surfactant, CTAB (commonly used for the preparation of MCM-41 material).

The synthesis is carried out from a micellar solution of CTAB surfactants ($C_{16}H_{33}N + (CH_3)_3Br$ - Aldrich) in water. The solution is left under stirring with enough time for the dissolution of the surfactant. After that we added gradually the ammonium hydroxide, to adjust the pH (with a value of 11). Then, the aluminum tetraethoxysilane $[(CH_3)_2CHO]_3Al$ is added, in order to obtain respectively the Si / Al ratios of 10 and 20. Obtained mixture is stirred for two hours at room temperature of (25°C). Then, the gel is placed in the Teflon sheath of an autoclave to perform a hydrothermal treatment, at a fixed temperature and for a given time (usually 48 hours at 100 °C). After this treatment, the gel is transferred to a Buchner for filtration and washing. The recovered powder is dried at a temperature of about 40 °C. The resulting solid is then calcined at 550 °C (1 °C / min) in air to remove the surfactant [20].

2.3. Synthesis of Mesostructured Carbon

CMK-3M is obtained following the replication of MCM-41 pure mesoporous silica. A quantity of 1.25 g of sucrose is dissolved in 5 ml of H_2SO_4 (0.28 M). Obtained solution is introduced into 1 g of pure silica and the whole is heated in air at 100 °C for 6 h and then at 160 °C for 6 h. A second impregnation is then transported by introducing 5 ml of the sucrose solution in H_2SO_4 (0.18 M containing 0.8 g of sucrose). In the same way, the mixture obtained is heated at 100 °C. for 6 hours and then at 160 °C. for 6 hours. The recovered black solid is pyrolyzed at 900 °C. for 8 hours. The composite (SiO_2/C) is immersed for 24 hours in 80 ml of a 5% solution of HF to remove the silica. The resulting carbon is then filtered, washed with water and air is discharged at room temperature for 24 hours [21].

2. CHARACTERIZATION

2.1. X-ray diffraction

The small angle diffractogram (Fig.3) from 1° to 5° of the MCM-41 silica exhibits a first intense diffraction peak is observed at 2.30 and two other peaks are less intense at 3.90 and 4.46, and for the CMK-3M coal the presence of a main peak at 2.44 ° and two smaller ones placed around 4.05 ° and 4.52 °. The first peak represents the reflection of the plane (100) and the other two of the planes (110) and (200). This demonstrates the formation of organized porous networks of the hexagonal symmetry $P6mm$ [22].

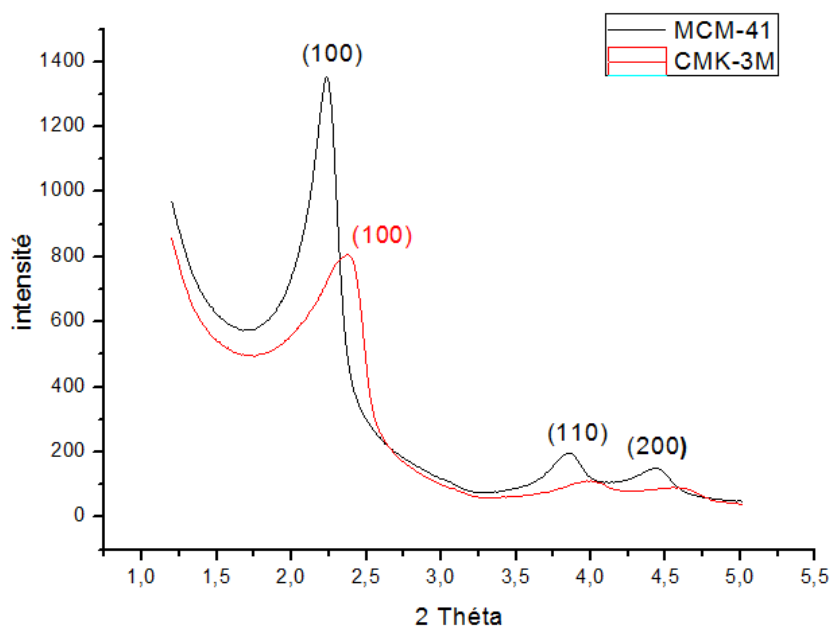


Fig.3. X-ray Diffractograms of MCM-41 and CMK-3M at low angles

From this analysis, we have been able to determine the interreticular distances d_{hkl} and the mesh parameters a_0 for the two silicas ($a_0 = 2 / \sqrt{3} \cdot d_{100}$). We used the interplanar distance between the planes (100) to calculate the corresponding mesh parameters. Table 1 summarizes the results obtained.

Table 1. Structural Properties of MCM-41 and CMK-3M:

	d (nm)	a_0 (nm)
MCM-41	3,96	4,57
CMK-3M	3,73	4,31

The values of the mailer parameter a_0 of carbon are smaller than those obtained on their mold. This decrease with an order of (0.2 nm), is due to the contraction of the structure under the effect of the heat treatment applied at high temperature (900 °C) to develop the carbon.

According to the DRX diffractograms (Figure 4), the introduction of aluminum into the MCM-41 framework does not affect the organization of the material. Indeed, the intensity of the characteristic peaks of the MCM-41 is reduced in the presence of aluminum. In addition, it is observed that these characteristic peaks are even lower as the amount of aluminum introduced increases.

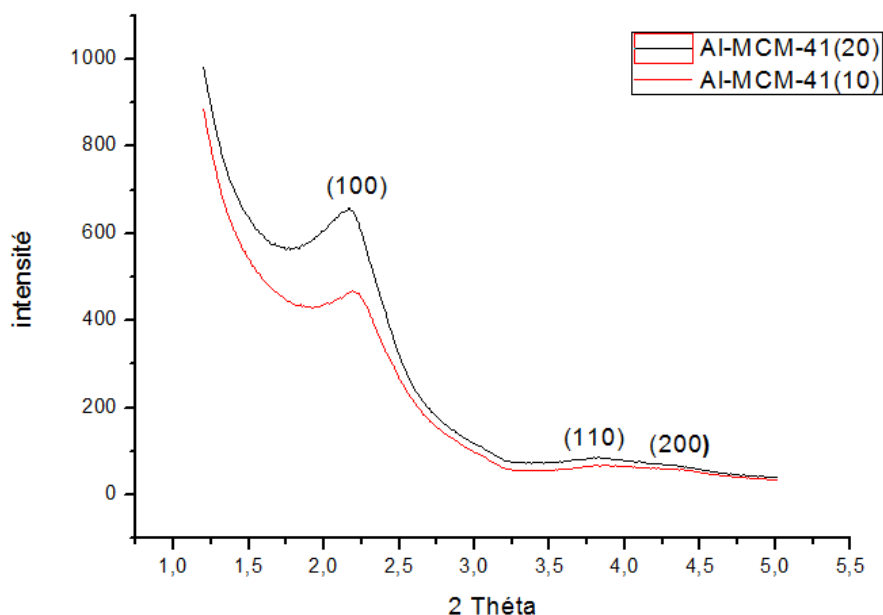


Fig.4. X-ray diffractogram of Al-MCM-41 (20/10) at low angles

Table 2 collates all the X-ray powder diffraction data of the synthesized mesoporous aluminosilicate samples.

Table 2. List of parameters d (100) and a_0 in nm determined by DRX for each of the compounds

	d (nm)	a_0 (nm)
Al-MCM-41 (20)	4,03	4,65
Al-MCM-41 (10)	4,10	4,73

For Al-MCM-41 materials, a displacement of the main peak d_{100} towards the small angles thus towards the higher inter-reticular distance giving a higher parameter of mesh. This is explained by the fact that the Al ions have been incorporated in the tetrahedral positions of Si. Because the Al-O bond is longer than the Si-O bond.

It is also observed that the increase in the value of the α -mesh parameter is proportional to the increase in aluminum contents of the Al-MCM-41 materials.

2.2. Nitrogen Adsorption-Desorption Porosimetry at 77 K (BET Analysis)

The textural properties of prepared materials were determined at low temperature nitrogen adsorption / desorption isotherm (77K). Figure 5 shows the characteristic isotherms of the matrices studied. We can see clearly all isotherms are type (IV) according to the classification (IUPAC), which confirms the mesoporosity of our materials [23,24]. The hysteresis loops,

which appear more clearly in the isotherms of MCM-41, Al-MCM-41 (20), Al-MCM-41 (10) and CMK-3M, correspond to an H1 type hysteresis. The mesopores of our silicic materials are therefore uniform in size.

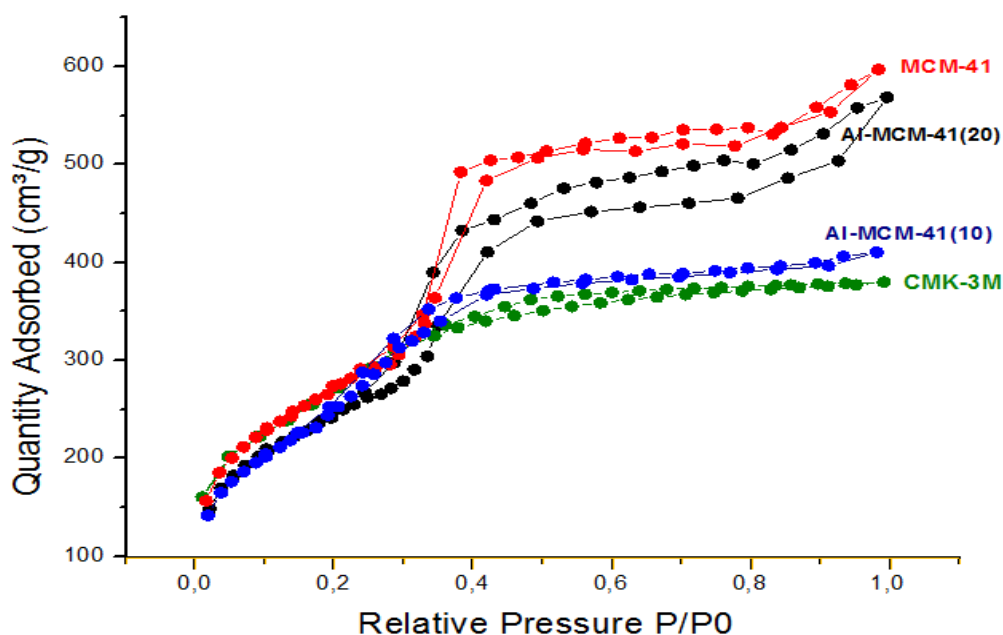


Fig.5. Nitrogen adsorption / desorption isotherms curves of the solids studied

On the other hand, our silicas have a high specific surface (877-1108 m² / g) (Table 3) which explains their ability to remove heavy metals from water. From the results given by the Table 3, we can see clearly CMK-3M has the largest surface area with a value of 1108.01 m²/g.

Table 3. The different values of specific surface area and pore size obtained

<i>Enchantions</i>	<i>Specific surface</i> (m ² /g)	<i>Pore diameter</i> (nm)	<i>Pore volume</i> (cm ³ /g)
MCM-41	968,82	1,91	0,925
Al-MCM-41 (10)	887,19	1,44	0,641
Al-MCM-41 (20)	877.49	2,00	0.879
CMK-3M	1108.01	2,42	0.705

2.3. Thickness of the Walls

Combining the results of the diffraction and nitrogen adsorption / desorption techniques, we can estimate the wall thickness of amorphous material, denoted by "e". In the case of hexagonal symmetry (P 6mm), this is the difference between the mesh parameter and the pore diameter (e = a₀ - dp). Table 4 shows the wall thickness values for the four materials

considered. The CMK-3M coal was obtained by mesoporous silica replication, the thickness of the walls of the starting silica must correspond to the pore size of the coal and this hypothesis is perfectly verified. Indeed, they have a pore diameter of value ($d_p = 2.42$ nm) are comparable to that of the thickness of the wall of their silicic mold ($e = 2.66$ nm for MCM-41) with a difference that does not exceed 0.3 nm.

Table 4. Structural parameters of mesoporous materials obtained by association DRX-Adsorption / desorption of nitrogen

<i>Enchantions</i>	<i>a₀ - DRX (nm)</i>	<i>Pore diameter (nm)</i>	<i>e (nm)</i>
MCM-41	4,57	1,91	<u>2,66</u>
Al-MCM-41 (10)	4,65	1,44	3,21
Al-MCM-41 (20)	4,73	2,00	2.73
CMK-3M	4,31	<u>2,42</u>	1,89

3. APPLICATION TO ADSORPTION

In order to evaluate the adsorption efficiency of prepared adsorbents we used cobalt as pollutant. For that, we studied the kinetics as well as the adsorption equilibrium.

3.1. Adsorption Kinetics

For the kinetic study, a series of dark flasks is placed in a linear stirrer, where 10 mg of adsorbent, 5 ml of the aqueous solution of Co^{2+} [11.4 mg/l] are introduced at pH 6.4. The temperature is kept constant at 22 °C and the mixture is stirred at 250 rpm.

The adsorption kinetics for each adsorbent is illustrated in FIG.6. Obtained by plotting the amount adsorbed Q_e as a function of time (b) $Q_e = f(t)$.

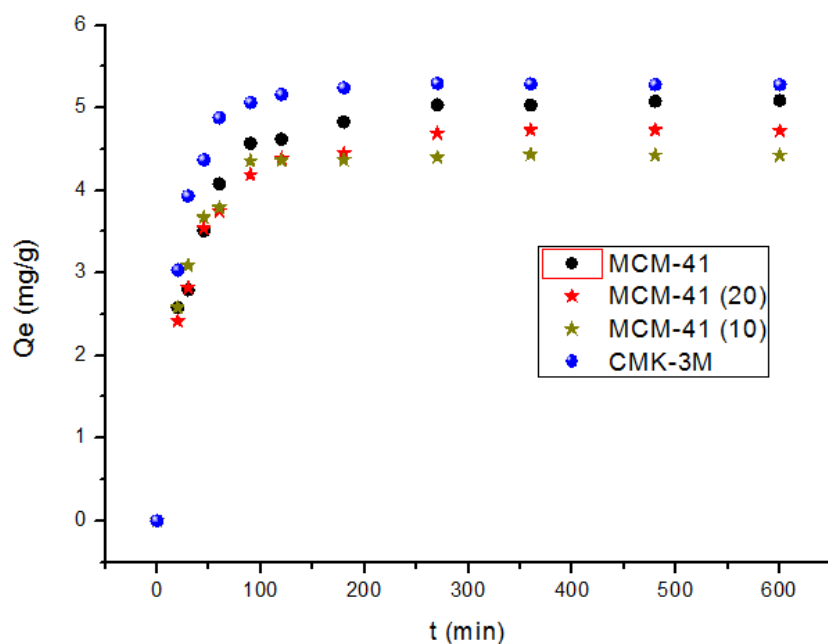


Fig.6. Cobalt adsorption kinetics

The adsorption of cobalt for all our solids has shown that the insertion of aluminum into the silicic framework has decreased the adsorption rate of cobalt. On the other hand, the increase in adsorption rate is observed in the case of CMK-3M mesoporous coals.

The maximum adsorption rates of cobalt (II) are of the order of 75.09% (4.6 mg/g) for both Al-MCM-41, 80.41% (5.03 mg/g) for MCM-41 and 91.54% (5.29 mg/g) for CMK-3M.

The binding rate of cobalt on the CMK-3M coal is the highest of the three mesoporous materials because of its high specific surface area, and the equilibrium was reached around 120 min for all the samples.

3.2. Modeling of Cobalt Adsorption Kinetics

Monitoring of kinetics and measurement of equilibrium are two important steps in the adsorption phenomenon. The adsorption of heavy metals from the liquid phase to the solid phase can be considered as a reversible equilibrium reaction between the two phases [25]. Lagergren's pseudo-first-order model, Equation (1), and Ho's pseudo-second-order model, Equation (2), were applied to the data.

$$q = q_e (1 - e^{-k_1 t}) \quad (1)$$

$$q = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \quad (2)$$

Where q_e and q (mg g⁻¹) are the amount of dye adsorbed at equilibrium and time t (min), k_1 (min⁻¹) is the pseudo-first rate constant and k_2 (g mg⁻¹min⁻¹) is the pseudo-second-order model.

Modeling results show clearly (see table 5) that the pseudo-first-order model can represent very well experimental results with an adjustment coefficient ($R^2 > 0.97$). We can see also that, all our prepared adsorbents present a constant ($k_1 > 0.04$) compared to that obtained for the adsorption of basic dyes onto activated carbon [26], this can be explained by the structural properties of prepared adsorbents.

Table 5. Kinetic parameters of both models

Adsorbants	First-order pseudo model				Second order pseudo model		
	Qe (exp)	Qe	K ₁	R ²	Qe	K ₂	R ²
MCM-41	5,03	4,83	0,029	0,983	5,29	0,008	0,988
Al-MCM-41(10)	4,69	4,52	0,059	0,975	4,67	0,052	0,952
Al-MCM-41(20)	4,35	4,39	0,040	0,997	4,67	0,014	0,973
CMK-3M	5,06	5,21	0,040	0,992	5,56	0,012	0,976

Based on the regression values and calculated maximum theoretical amounts of these two models, we conclude that the adsorption kinetics of cobalt by the four materials in an aqueous medium prefer to follow the pseudo first order model.

3.3. Determination of adsorption isotherms

The adsorption isotherms are obtained by the graphical representation of $Q_e = f(C_e)$ (Q_e and C_e are respectively the amount of cobalt adsorbed and the equilibrium concentration of this metal). The results are shown in the figures 7.

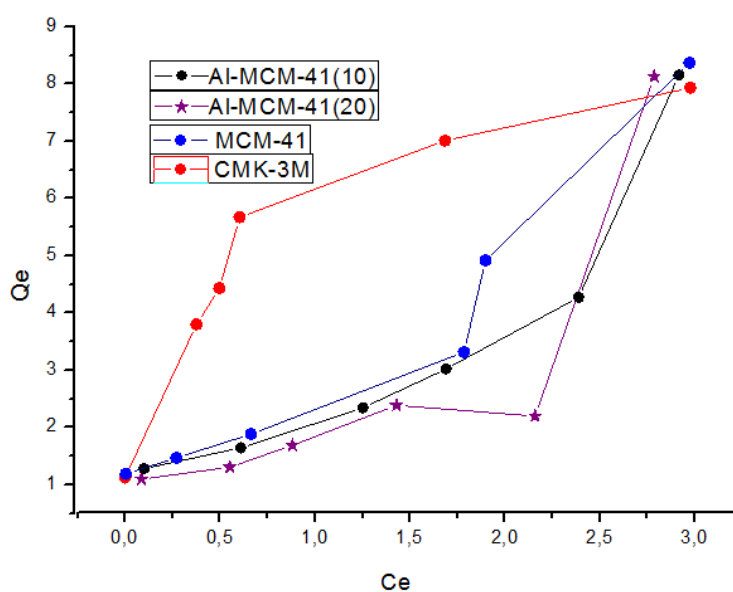


Fig.7. Cobalt adsorption isotherms

According to the classification Giles *et al.* [27] the curves obtained show that the CMK-3M coal isotherm is of type L. This curve can be mathematically described by the Langmuir or Freundlich equation. The more sites occupied by solute molecules, the more adsorption of new molecules is difficult.

The isotherm of materials MCM-41, Al-MCM-41 (20) and Al-MCM-41 (10) are of type S. The amount of cobalt Co^{2+} adsorbed is practically zero at a low concentration of solution because the concentration of the solution increases the amount adsorbed increases rapidly. Thus, the adsorbed molecules promote the subsequent adsorption of other molecules (cooperative adsorption due to the attraction between the molecules of the solute by Van der Waals forces).

3.4. Modeling of adsorption isotherms

Adsorption isotherms in our case were analysed using the Langmuir, Equation (3), and Freundlich, Equation (4), models:

$$q_e = q_m \frac{k_L C_e}{1 + k_L C_e} \quad (3)$$

$$q_e = k_F C_e^n \quad (4)$$

where q_e (mg g^{-1}) and C_e (mg L^{-1}) are, respectively, the cobalt concentration adsorbed and in solution, q_m (mg g^{-1}), k_L (mg^{-1}) are the Langmuir constants relating to the sorption capacity and energy, respectively, K_F is the Freundlich constant and n relates to the sorption intensity or surface heterogeneity. Non-linear regression analyses were performed using Origin 8.1.

Modeling results show clearly that the Langmuir model fits very well at the experimental points with the CMK-3M carbon, and that the value of the correlation parameter is close to 1 ($R^2 = 0.97$). The calculated value of RL is less than 1, to conclude that the adsorption of Co^{2+} is favorably carried out on the carbon to be studied.

On the other hand, we can see also that, for the matrices MCM-41, Al-MCM-41 (20) and Al-MCM-41 (10), the value of $(1/n)$ obtained by applying the Freundlich model to our experimental results is greater than 1 ($1/n = 1.48, 2.55$ and 2.71 successively). This shows that the isotherms are of type S.

These experimental results show that the Freundlich model is reliable. At low solute concentrations, the adsorption remains lower and becomes larger as the concentration of cobalt (II) increases, thus confirming that solute-solute interactions are stronger than solute-adsorbent interactions.

The value of the parameter $1/n$ obtained by the Freundlich model is less than 1 in the case of coal, which shows that the isotherms are of type L. The results obtained experimentally in the Freundlich model perfectly describe the adsorption process cobalt on CMK-3M.

The results of the various adsorption models are summarized in Table 6.

Table 6. isotherms parameters of both models

<i>Parameters of models</i>		MCM-41	Al-MCM-41(10)	Al-MCM-41(20)	CMK-3M
<i>Langmuir</i>	K_L (L/mg)	$6,01 \cdot 10^{-5}$	$4,97 \cdot 10^{-5}$	$5,29 \cdot 10^{-5}$	1,1
	Q_m (mg/g)	22123	33202	32856	4,04
	R^2	0,94	0,90	0,97	0,97
	R_L	0,99	0,99	0,99	0,07
<i>Freundlich</i>	K_f (mg/g)	0,72	0,29	0,35	1,94
	$1/n$	1,48	2,71	2,55	0,45
	R^2	0,936	0,96	0,87	0,92

4. CONCLUSION

The application of MCM-41, Al-MCM-41 (20), Al-MCM-41 (10), and CMK-3M to the adsorption of cobalt (II) ions has been studied to test their retention capacities vis-a-vis this metal. The maximum adsorption rates of cobalt (II) are of the order of 75.09% (4.6 mg/g) for both Al-MCM-41, 80.41% (5.03 mg/g) for MCM-41 and 91.54% (5.29 mg/g) for CMK-3M. The curves of the isotherms obtained show that the CMK-3M charcoal isotherm is of the L type, and that the isotherm of the materials MCM-41, Al-MCM-41 (20) and Al-MCM-41 (10) is type S. Adsorption isotherms of Co^{2+} on the four solids are modeled using the isotherms of Langmuir and Freundlich. Results of monetization using the non-linear regression method shows, that these models can describe well the adsorption phenomenon onto the four matrices (values of the correlation parameters very close to 1); except in the case of the Langmuir model which is applicable only with CMK-3M coal. All results shows the efficiency of CMK-3M toward cobalt (II) compare to the other prepared mesoporous materials.

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