

Adsorption ability of carbon black for nickel ions uptake from aqueous solution

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Abstract

Surface modification can be performed by adsorption of certain organic compounds on the surface of carbon. The main objective of this work was to compare the adsorption ability of acid-modified carbon black with the non-modified one. The modification process was performed by adsorption of acetic acid onto commercial carbon black surface. A batch adsorption system was applied to study adsorption reactions of acetic acid and Ni(II) onto carbon black. Adsorption isotherms of acetic acid and Ni(II) adsorption onto the non-modified and modified carbon black were fitted by classical adsorption models, such as Freundlich and Langmuir models. Modified carbon black surface become more active for Ni(II) ions removal from aqueous solutions. The results showed that modification by acetic acid increases the adsorption capacity of carbon black from 18.3823 to 86.9566 mg Ni(II) g⁻¹. SEM analysis enabled the observation of any surface changes in the carbon black that had occurred due to either acid modification or Ni(II) adsorption.

Keywords: carbon black, Ni(II), adsorption, surface modification.

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Carbon black (CB) is the accepted generic name for a family of small particle size carbon pigments, which are formed in the gas phase by the thermal decomposition of hydrocarbons. The usual feedstock is heavy distillates or residual petroleum products, especially those with high aromatic contents. The common processes for carbon black production are channel, thermal, acetylene and furnace processes. Of these, the oil furnace process is by far the most important today [1].

Carbon black is composed of particles that are either spherical or spheroidal with a pronounced ordering of the carbon layers. Since CB is produced from hydrocarbons, the dangling bonds at the edges of the carbon layers are saturated mostly by hydrogen. The surface properties of CB are influenced to an extent by the foreign elements fixed on the surface, in particular by oxygen [1]. Carboxylic, phenolic, carbonylic, hydroxylic and other functional groups are located at the edges of carbon layer plane and are responsible for surface reactivity of CB [2].

Approximately 80% of the worldwide market for carbon black is consumed in automotive related rubber product applications due to its reinforcing effect. Other uses are found in printing inks, paints, plastics and dry electric cells [2,3].

Industrial activities (e.g., mining, painting, car manufacturing, metal plating, tanneries, etc.) and agricultural activities are the main sources of waste con-

taining heavy metals. Heavy metals are considered to be one of the most hazardous water contaminants. According to the World Health Organization (WHO), cadmium, chromium, copper, lead, mercury and nickel are listed among the most toxic metals [4]. Nickel can cause serious health and ecological problems when released into the environment [5]. The health effects of nickel include higher chances of the development of lung, nose, larynx, respiratory failure and birth defects. Certain compounds of nickel have been recognized as carcinogenic. Environmental regulations related to the discharge of nickel and other heavy metals demand development of novel methods for their removal from wastewater.

There are several methods to treat the metal contaminated effluent such as precipitation, ion exchange, electrolysis, separation by membrane, adsorption, etc., but the selection of the wastewater treatment methods is based on the concentration of waste and the cost of treatment [6].

Adsorption is one of the most popular methods for the removal of metals ions from the aqueous solutions. It is a surface phenomenon, in which molecules of adsorbate are attracted and held on the surface of an adsorbent until an equilibrium is reached between adsorbed molecules and those still freely distributed in the bulk of the gas or liquid. The adsorption phenomenon depends on the interaction between the surface of the adsorbent and the adsorbed species.

The primary requirement for an economic adsorption process is the selectivity of adsorbent, its high adsorption capacity and low cost. Activated carbons are known as very effective adsorbents due to their highly

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developed porosity, large surface area (that can reach $3000 \text{ m}^2 \text{ g}^{-1}$), variable characteristics of surface chemistry and high degree of surface reactivity [7,8]. The aqueous-phase adsorption of both organic and inorganic compounds has been a very important application of activated carbon. The adsorption process with activated carbon is very attractive due to the effectiveness for the removal of heavy metal ions at trace quantities. However, because of its high cost, the less expensive non-conventional adsorbents are being studied including their surface modification [3,9–12], which can be performed by adsorbing specific organic compounds on the surface of carbons.

Until recently, very little research has been focused on metal ion adsorption by activated carbon in the presence of organic acids. Thus, the objective of this work was to compare the adsorption ability of acid-modified carbon black with to the non-modified one. Acetic acid was used as organic acid for the modification of commercial carbon black. It is a biodegradable and widely used low cost acid in the food and pharmaceutical industries.

Based on the literature data, it is possible to conclude that there have been no reported research studies on nickel adsorption onto carbon black modified by acetic acid. Therefore, the present study was directed toward the evaluation of the adsorption potential of modified and non-modified carbon black for the adsorption of nickel ions from aqueous solution.

EXPERIMENTAL

Preparation and characterization of samples

Commercial carbon black was produced by oil furnace process in Croatia. The oil furnace process is based on partial combustion of highly aromatic hydrocarbon fraction. The raw materials were the aromatic oil derivatives produced by secondary petroleum refining processes. Carbon black is formed in the furnace by partial oxidation and cracking of oil feedstock at $1300\text{--}1400 \text{ }^\circ\text{C}$ and is carried by gas flow to a heat exchanger and a quench cooler, where the temperature is reduced to about $200 \text{ }^\circ\text{C}$.

The basic properties of granular carbon black are presented in Table 1.

Table 1. The properties of commercial carbon black

Property	Value	Unit	Test method
Heating loss	0.57	%	ISO 1126
Iodine number	119	mg g^{-1}	ISO 1304
Ash content	0.26	%	ISO 1125
Sulfur content	1.0	%	DIN 51400
Density	340	kg m^{-3}	ISO 1306
pH value	9.2	–	ASTM D 1512
Tint strength	116	% ITRB	ISO 5435

The CB samples were ground and sieved to retain the particle size $\leq 0.1 \text{ mm}$.

Acetic acid – HAc ($\text{H}_3\text{C-COOH}$) – was used as adsorptive carbon black surface modifier.

Carbon black modification

CB modification was done in batch experiments under the following conditions: 100 mL HAc solutions of known concentration (in range: $c_i = 0.05\text{--}0.35 \text{ mol L}^{-1}$) were added to reagent bottles with 1 g of dry CB. The suspension was shaken at a rotation speed of 60 rpm for a period of 30 min. Although the experiments were performed within the time from 0 to 48 h, the equilibrium was established after 24 h.

Acid concentrations before and after equilibrium were determined by titrating with a 0.1 mol L^{-1} NaOH solution and phenolphthalein as indicator.

The acid modified carbon black was then washed with deionized water until no turbidity could be observed when a 0.1 mol L^{-1} lead nitrate solution was added to the washed liquid.

The carbon black modified with 0.05 mol L^{-1} acetic acid, was dried at $105 \text{ }^\circ\text{C}$ for 1 h and used as adsorbent (MCB) for nickel ions.

Batch mode of Ni (II) adsorption

The adsorption of nickel ions from aqueous solution onto the adsorbents (CB and MCB) was performed using a batch technique. A standard solution, containing 1000 mg L^{-1} of Ni(II) was prepared by dissolving $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ with deionized water.

Different initial concentration of Ni(II) solutions was prepared by dilution from 1000 mg L^{-1} Ni standard.

For determination of the adsorption isotherm, 50 mL of each solution containing known concentration of nickel ($50\text{--}500 \text{ mg L}^{-1}$) was added 0.25 g of each adsorbent in glass flasks and shaken (at the speed of 60 rpm) for 30 min.

The time required for reaching the equilibrium condition was estimated at regular intervals of time until equilibrium was reached (48 h).

After the reaction period, the suspension were filtered through Whatman filter paper No. 44 and the supernatant was analyzed for Ni(II) concentration following standard method for examination of water and wastewater [13]. The nickel concentration was determined by a Perkin-Elmer M 54-Coleman (Perkin-Elmer, USA) spectrophotometer by measuring the absorbance at λ_{max} of 450 nm from solutions with pH value 7.5. The pH of the solution was adjusted by adding 0.1 mol L^{-1} HCl and 0.1 mol L^{-1} NaOH solution. Experiments were performed at $20 \text{ }^\circ\text{C}$. All chemicals used in this study were analytical grade (Merck, Germany). All samples and their respective blanks were run in duplicate.

Surface properties

The surface morphology of the CB samples before and modification was visualized by scanning electron microscope (JOEL JXA 50A, Nippon). SEM enables the direct observation of any changes in the CB surface morphology that would have occurred due to the modification.

The specific surface area was determined from nitrogen adsorption data acquired on a Quantachrome-Nova 1000 apparatus (Quantachrome Instruments, USA).

Data analysis

The equilibrium quantity, Δc , of adsorbate (HAc and nickel ions, respectively) onto carbon black samples was calculated as the difference between initial concentration, c_i , and concentration at equilibrium, c_e .

The amount adsorbed at equilibrium i.e. the adsorption capacity, q_e (mg g^{-1}), was calculated according to the formula:

$$q_e = \frac{\Delta c}{m} V \quad (1)$$

where q_e is adsorption capacity (mg g^{-1}), Δc – quantity of adsorbed adsorbate (mg L^{-1}), V – volume of solution (L), m – adsorbent mass (g).

The Freundlich and Langmuir isotherms are used to interpret adsorption equilibrium data.

RESULTS AND DISCUSSION

Adsorption isotherms

Equilibrium relationships between adsorbent and adsorbate are described by adsorption isotherms, usually as the ratio between the quantity adsorbed and that remaining in the solution at a fixed temperature at equilibrium.

Linear regression is frequently used to determine the best-fitting isotherm. The linear least-squares method with linearly transformed isotherms has also been widely applied to confirm experimental data and isotherms using correlation coefficients of determination. In this study, two classical adsorption models were employed to describe the adsorption equilibrium. The adsorption data have been correlated with Langmuir and Freundlich isotherm equations.

The Freundlich equation is an empirical relationship, where it is assumed that the adsorption energy of ion binding to a site on an adsorbent depends on whether or not the adjacent sites are already occupied [14].

The Freundlich isotherm is expressed as:

$$q_e = K_F c_e^{1/n} \quad (2)$$

where q_e is adsorption capacity (mg g^{-1}), c_e – the equilibrium concentrations of adsorbate (mg L^{-1}), K_F and n – the Freundlich constants.

Constants can be determined from a linearized form in the above equation, represented by:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln c_e \quad (3)$$

The Langmuir adsorption model [15] is based on the assumption that maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface, with no lateral interaction between the adsorbed molecules.

The common form of the Langmuir isotherm was expressed as:

$$q_e = \frac{q_m K_L c_e}{1 + K_L c_e} \quad (4)$$

where q_e is adsorption capacity (mg g^{-1}), c_e – the equilibrium concentration of adsorbate (mg L^{-1}), q_m – Langmuir maximum adsorption capacity of the adsorbents (mg g^{-1}), K_L – Langmuir constant.

The values of constants q_m and K_L were determined graphically from a linearized form of Langmuir isotherm represented by the equation:

$$\frac{1}{q_e} = \frac{1}{K_L q_m c_e} + \frac{1}{q_m} \quad (5)$$

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor, R_L , that is given by the following equation [16]:

$$R_L = \frac{1}{1 + K_L c_m} \quad (6)$$

where R_L is separation factor, c_m – highest adsorbate concentration (mg L^{-1}), K_L – Langmuir's constant.

The value of R_L indicates the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$).

Adsorption isotherms of acetic acid onto carbon black

The obtained data for adsorption HAc by CB fit the linear relationship of both Freundlich (Figure 1) and Langmuir (Figure 2) isotherm well.

The values of Langmuir and Freundlich constants and correlation coefficients R^2 were calculated and shown in Table 2. The high R^2 values (0.9983) obtained for adsorption of acetic acid onto the CB indicates that equilibrium data fitted well to the Freundlich isotherm. Constants K_F and n are indicators of adsorption capacity and adsorption intensity, respectively. The value of the exponent, $1/n$, gives an indication of the favorability of adsorption. Values of $1/n$ smaller than unity represent favorable adsorption [17]. In the present case $1/n$ is equal to 1.033, which is quite close to 1.0 indicating some lower adsorption capacity of the CB for acetic

acid. The results of the Langmuir isotherm indicate the satisfactory good correlation between this model and the experimental data (Table 2, $R^2 = 0.9998$). Obviously, it can be seen that the Langmuir model yields a somewhat better fit than the Freundlich model.

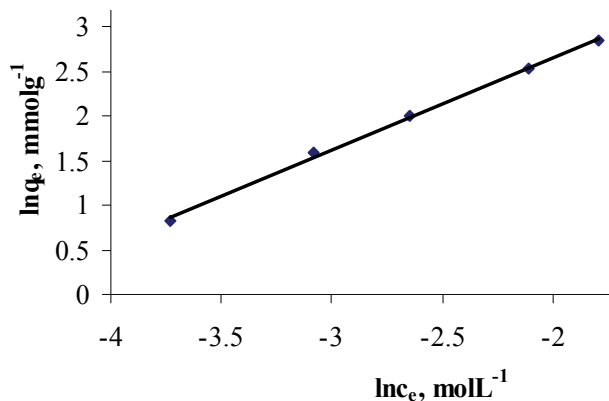


Figure 1. Freundlich isotherm obtained for acetic acid (HAc) adsorption onto carbon black (CB).

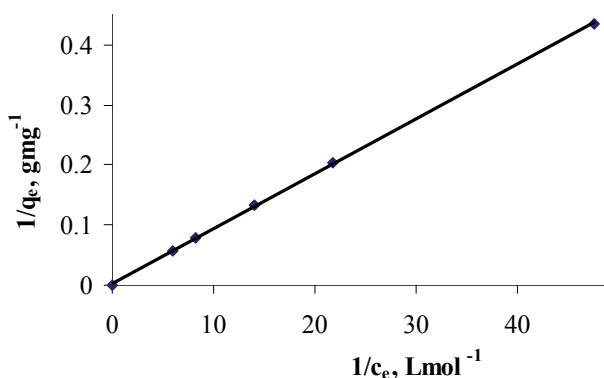


Figure 2. Langmuir isotherm obtained for acetic acid (HAc) adsorption onto carbon black (CB).

Adsorption isotherms of Ni(II) ions onto the CB and MCB

The equilibrium adsorption isotherm of Ni(II) ions on the carbon black before and after modification is shown in Figure 3. It can be seen that in both cases the adsorption of Ni(II) increases with the increase of initial concentration of Ni(II) ions in aqueous solutions. The adsorbed amount of Ni(II) on MCB was higher than those on the CB. Carbon black modification with HAC improved adsorption capacity for Ni(II).

To determine the constants K_F and n , the linear form of the Eq. (3) may be used to produce a graph of $\ln q_e$ against $\ln c_e$ (Figure 4). Values of K_F and n are calculated from the intercept and slope of the plot (Figure 4). The values of the calculated Freundlich constants K_F and n are listed in Table 3. The adsorption isotherms of Ni(II) on the CB and MCB fit the Freundlich isotherm well (R^2 is 0.9780 and 0.9582, respectively). The value of $1/n$ was < 1 , indicating that the adsorption was favorable.

The Langmuir adsorption isotherm of Ni(II) adsorption on the CB and MCB is shown in Figure 5. The values of Langmuir constant K_L , maximum adsorption capacity q_m and correlation coefficients were determined, and are shown in Table 4. The maximum adsorption quantities calculated by applying the equation (5) for Ni(II) were 18.3823 and 86.9566 mg g^{-1} , respectively. Maximum adsorption capacity values reported in literature [10] using different adsorbents are shown in Table 5. The comparison of literature data reveals that maximum adsorption capacity of CB and MCB for Ni(II) is of the same or similar order of magnitude than those obtained for other types of adsorbents.

Table 2. The values of Langmuir and Freundlich constants and correlation coefficients for adsorption of acetic acid onto carbon black

$q_{\max} / 10^{-3} \text{ mol g}^{-1}$	Langmuir		Freundlich		
	$K_L / \text{L mol}^{-1}$	R^2	$K_F / \text{mol g}^{-1} (\text{L mol}^{-1})^{1/n}$	$1/n$	R^2
0.3125	0.3521	0.9998	0.1126	1.033	0.9983

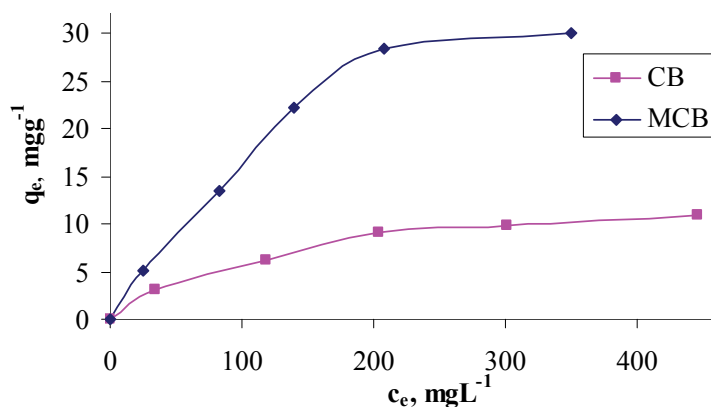


Figure 3. Adsorption isotherms of Ni(II) onto carbon black before (CB) and after modification (MCB).

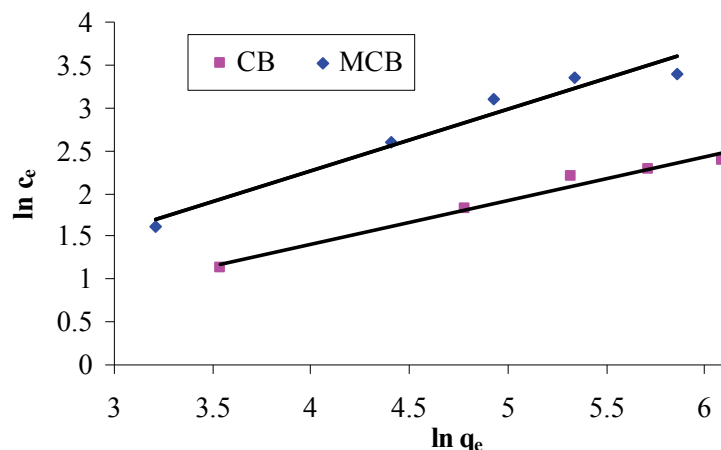


Figure 4. Freundlich isotherm of Ni(II) adsorption onto carbon black before (CB) and after modification (MCB).

The value of $R^2 = 0.9906$, suggesting the better fit of the data to the Langmuir model for Ni(II) adsorption on the MCB samples. The value of R_L was found to be 0.2632 and 0.4545, respectively indicating that the adsorption of Ni(II) on the both CB and MCB are favorable.

Table 3. Parameters of equilibrium Freundlich model of Ni(II) adsorption onto carbon black

Sample	$K_F / (\text{mg g}^{-1}) \cdot (\text{L mg}^{-1})^{1/n}$	$1/n$	R^2
CB+Ni	0.5328	0.5116	0.9780
MCB+Ni	0.5487	0.7184	0.9582

According to the obtained results, the Freundlich model better describes Ni(II) adsorption on carbon black than the Langmuir model. The Freundlich isotherm is an empirical equation employed to describe heterogeneous systems. Hence, it confirms that the surface of CB is more heterogeneous than that of the MCB. The main assumption of the Langmuir model is that adsorption occurs uniformly on the active sites of the adsorbent surface, and when a molecule is adsorbed on a site, the latter does not have any effect upon other incident molecule [17].

Table 5. Adsorption capacity values, q_m , of different adsorbents for Ni(II), reported in literature [10]

Adsorbent	Adsorption capacity, mg g^{-1}
Magnetite	18.43
Fly ash	0.99
Activated red mud	160
Calcined phosphate	15.53
Red mud	13.69
Clarified sludge	14.30
Activated slag	30
Carbon anode dust	8.64

Adsorbents, characterized by high adsorption capacity and surface reactivity, are better for uptake of metal ions from solutions. CB exhibits chemical heterogeneity due to the presence of several types of heteroatoms such as oxygen, hydrogen, nitrogen, and/or sulfur, present within the carbon matrix. Oxygen forms surface functional groups bonded on the edge of carbon (carboxylic, carbonyl, hydroxyl, phenolic, lactonic etc.), and has a strong influence on solution adsorption [18]. The adsorption of heavy metals by carbon black occurs through the electrostatic interactions between positive metal species and negative functional groups of carbon black.

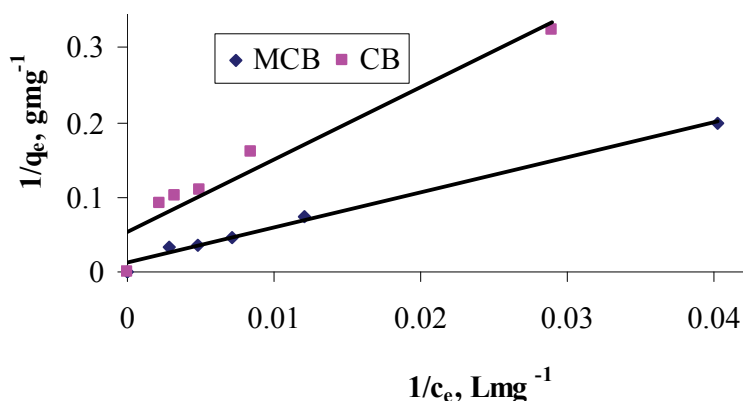
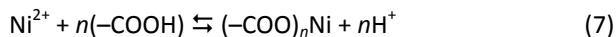


Figure 5: Langmuir isotherm of Ni(II) adsorption onto carbon black before (CB) and after modification (MCB).

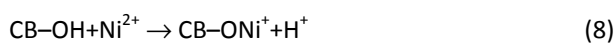
Modification by adsorption of HAc onto CB improves nickel removal from solution. Adsorption capacity of MCB for nickel ions is higher than those of CB (Figure 3). Literature data confirm that the affinity of the carboxylic functional groups for metal ions is very high [12,19].

The physicochemical interactions that might occur during nickel removal could be expressed as follows:



where $(-\text{COOH})$ represents the surface functional group of CB and MCB; n is the coefficient of the reaction component depending on the oxidation state of metal ions [20].

The interaction of nickel with carbon black can be also described as [19]:



SEM Micrographs

Results of SEM analysis of CB and MCB samples investigation are shown in Figures 6–9.

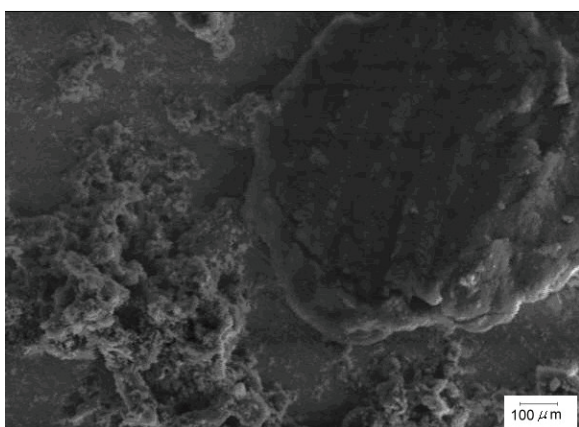


Figure 6. SEM Image of the CB sample surface before the modification.

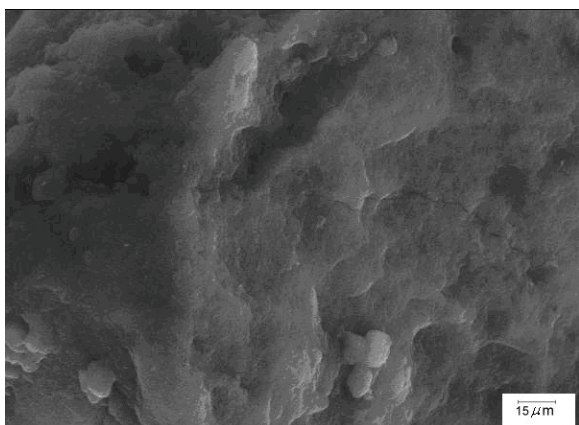


Figure 7. SEM Image of the MCB sample surface modified with 0.05 mol L^{-1} acetic acid (HAc).

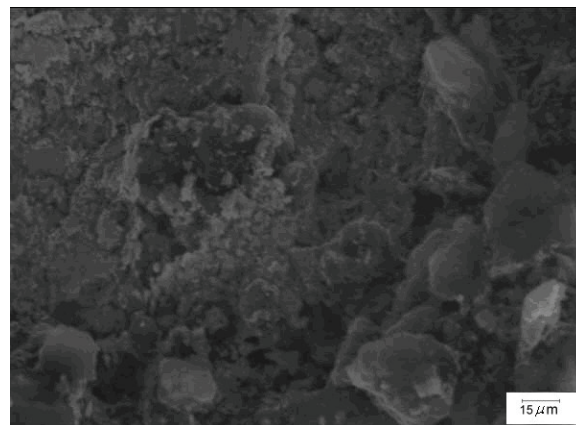
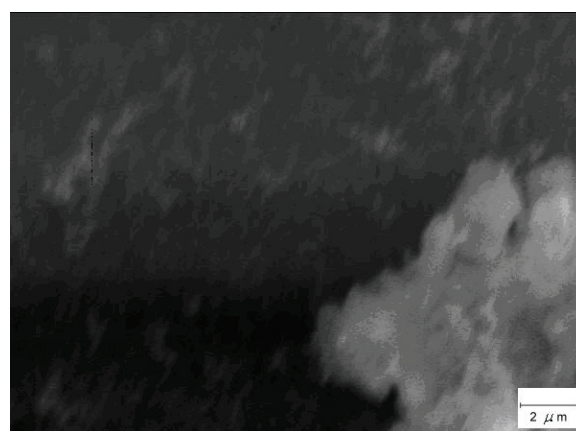
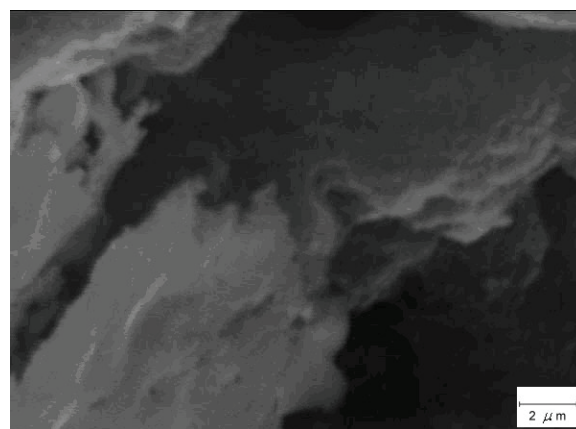


Figure 8. SEM Image of the CB surface after the contact with nickel ions solution ($c_i = 350 \text{ mg L}^{-1}$, $t = 24 \text{ h}$).



(a)



(b)

Figure 9. SEM Image of the MCB surface after contact with nickel ions solution: a) $c_i = 350 \text{ mg L}^{-1}$, $t = 24 \text{ h}$; b) $c_i = 150 \text{ mg L}^{-1}$, $t = 48 \text{ h}$.

Carbon black consists essentially of fine carbon particles, semi-graphite in structure [2]. It is usually macro- and mesoporous materials that exhibit low micropore volume [21]. The particle size of CB is partially in the nanometer range with average values between 20 and 300 nm from different sources [22].

Figure 6 shows the SEM image of the surface of non-modified CB sample with clearly visible mostly round particles, but of different sizes. Aggregates formed by coalescence of elemental particles. Fusing of aggregates by van der Waals forces resulted in the formation of new structures-agglomerates, as previously described in literature [2,23]. From Figure 6 is visible that there are cavities, cracks and some of pores on the CB surface. Treatment of carbon black by acetic acid resulted in a modified appearance of its surface (Figure 7). Because of its small molecular size, acetic acid can easily access to pore structure of carbon material [24]. The acetic acid modification reduces the number of cracks and pores on the CB surfaces, which are clogged by adsorbed acetic acid. Such explanation is supported by the data about the specific surface area determined before ($121.3 \text{ m}^2 \text{ g}^{-1}$) and after ($60.2 \text{ m}^2 \text{ g}^{-1}$) treatment of carbon black by acetic acid. Comparing to the non-modified CB adsorbent, the changes caused by Ni(II) ions adsorption are more pronounced on the surface of MCB as accumulations and deposits, not depending either on initial concentration, c_i or the contact time (Figures 8 and 9). Accordingly, the specific surface area of the carbon black is decreased, but its surface becomes more homogeneous.

CONCLUSION

Results of batch equilibrium studies provide information about the retention capacity and the simple possible mechanism by which the nickel ions are held onto carbon black. The chemical modification based on acetic acid adsorption onto carbon black increased its adsorption capacity for Ni(II) ions from aqueous solutions.

The equilibrium adsorption isotherm of acetic acid onto carbon black is well described by the Freundlich ($R^2 = 0.9983$) and Langmuir ($R^2 = 0.9998$) models. The adsorption of Ni(II) onto non-modified and modified carbon black is also described by Freundlich and Langmuir isotherm models. However, the Langmuir isotherm model showed better agreement with the equilibrium data, especially for Ni(II) uptake onto MCB ($R^2 = 0.9906$).

Results obtained by SEM analysis enabled the observation of surface changes in the carbon black that have occurred due to either acid modification or Ni(II) adsorption. The specific surface area of carbon black is decreased, but its surface becomes more homogeneous after modification.

The carbon black modified by this method has potential for non-conventional applications in areas of heavy metal removing from aqueous solutions.

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IZVOD

ADSORPCIONA SPOSOBNOST ČAĐI ZA VEZANJE JONA NIKLA IZ VODENOG RASTVORA

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(Naučni rad)

Čađ je proizvod hemijske industrije isključivo namenjen industriji gume. Svrha ovog rada je upozoriti na mogućnost drugačije upotrebe komercijalne čađi. Pokazana je adsorpciona sposobnost čađi za vezivanje Ni(II) jona iz vodenog rastvora jednostavnom „batch“ tehnikom. Upoređeni su rezultati vezivanja Ni(II) jona prije i nakon modifikacije površine čađe. Modifikacija površine čađi je izvedena adsorpcijom biorazgradive i netoksične sirćetne kiseline. Za razliku od sirćetne kiseline, nikal je toksičan i štetan čak i u malim količinama, pa je poželjno smanjiti njegovu koncentraciju u vodenim rastvorima. Štetnost nikla za zdravlje ljudi kreće se od blažih respiratornih smetnji do kancerogenih promena. Nakon modifikacije sirćetnom kiselinom postignut je veći kapacitet adsorpcije čađi za Ni(II) jone. Eksperimentalni rezultati su fitovani sa klasičnim ravnotežnim adsorpcionim modelima, Freundlich-ovim i Langmuir-ovim. Bolje slaganje eksperimentalnih podataka za vezivanje Ni(II) jona na čađi prije modifikacije površine dobijeno je korišćenjem Freundlich-ovog ravnotežnog modela. Primena Langmuir-ovog ravnotežnog modela je pokazala bolje fitovanje eksperimentalnih podataka za vezivanje Ni(II) jona na čađi modifikovanoj sirćetnom kiselinom. Te rezultate dopunjuju ispitivanja morfologije površine, koja nakon adsorpcije sirćetne kiseline postaje izmenjena, tj. homogenija. Dobijeni rezultati su doprinos proširenju korišćenja čađi u obradi vodenih rastvora zagađenih metalima.

Ključne reči: Čađ • Ni(II) • Adsorpcija • Modifikacija površine