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The effect of magnetite nanoparticles synthesis conditions on their ability to separate heavy metal ions

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Abstract: Magnetite nanoparticles have become a promising material for scientific research. Among numerous technologies of their synthesis, co-precipitation seems to be the most convenient, less time-consuming and cheap method which produces fine and pure iron oxide particles applicable to environmental issues. The aim of the work was to investigate how the co-precipitation synthesis parameters, such as temperature and base volume, influence the magnetite nanoparticles ability to separate heavy metal ions. The synthesis were conducted at nine combinations of different ammonia volumes – 8 cm^3, 10 cm^3, 15 cm^3 and temperatures – 30°C, 60°C, 90°C for each ammonia volume. Iron oxides synthesized at each combination were examined as an adsorbent of seven heavy metals: Cr(VI), Pb(II), Cr(III), Cu(II), Zn(II), Ni(II) and Cd(II). The representative sample of magnetite was characterized using XRD, SEM and BET methods. It was observed that more effective sorbent for majority of ions was produced at 30°C using 10 cm^3 of ammonia. The characterization of the sample produced at these reaction conditions indicate that pure magnetite with an average crystallite size of 23.2 nm was obtained (XRD), the nanosized crystallites in the sample were agglomerated (SEM) and the specific surface area of the aggregates was estimated to be 55.64 m$^2 \cdot$g^{-1} (BET). The general conclusion of the work is the evidence that magnetite nanoparticles have the ability to adsorb heavy metal ions from the aqueous solutions. The effectiveness of the process depends on many factors such as kind of heavy metal ion or the synthesis parameters of the sorbent.
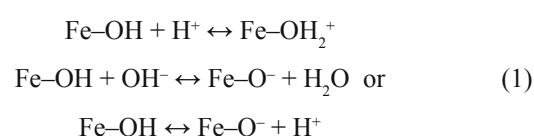
Introduction

In recent years, nanomaterials have become an interesting and promising research object for many scientists due to their unique properties and applicability in various fields.

Among extensively studied nanomaterials, iron oxide nanoparticles can be distinguished. In the natural environment, iron oxides are available in many different forms. The most common ones include hematite α-Fe$_2$O$_3$, maghemite γ-Fe$_2$O$_3$ and magnetite Fe$_3$O$_4$ (Teja et al. 2009). Synthesis of iron oxide nanoparticles are currently widely studied not only because of purely academic interest in their properties but also because of their practical application possibilities (Dave et al. 2014). Many works and articles describe various methods of the synthesis, modifications as well as physical and chemical properties characterization of magnetite and maghemite nanoparticles (Salado et al. 2008, Urquijo et al. 2011, Petcharoen et al. 2012, Mascol et al. 2013). The aim of studies is usually to obtain nanoparticles with the most suitable characteristics for the particular application field. Advances in the researches of nanomaterial usage in various fields proved among others the possibility of their application to environmental issues. A lot of attention is mainly focused on the synthesis and surface functionalization of nanoparticles with sizes below 50 nm which have a very large potential as adsorbents of contaminants such as heavy metals or organic compounds (Matei et al. 2011).

Heavy metals present in the aquatic environment pose a significant threat to human health. Many techniques are used to remove them from wastewater. Among them adsorption on iron oxide nanoparticles seems to be very attractive. Nanoparticles, as adsorbents, have many specific and useful features, such as small size, magnetic properties or high surface area (Dave et al. 2014). The adsorption mechanism between iron oxides and heavy metal ions is quite simple. In the water the iron oxides are completely hydrated. Their surface is covered by -OH functional groups which can react with H$^+$ and OH$^-$ ions. Depending on the pH, the oxide surface can undergo pronation and behave as an acid or deprotonation – obtaining the base characteristics. The protonation/deprotonation reactions are presented below:

$$Fe-OH + H^+ \leftrightarrow Fe-OH_2^+$$
$$Fe-OH + OH^- \leftrightarrow Fe-O^- + H_2O \quad or \quad (1)$$
$$Fe-OH \leftrightarrow Fe-O^- + H^+$$

As a result, the above reactions can lead to the electrostatic attraction between heavy metal ions and the iron oxide surface. The important characteristic for considered adsorbent is pH of point of zero charge (pzc). The value point of zero charge pH, depending on the literature source, can be placed somewhere between 6–6.8. When pH is above this value the surface of magnetite is negatively charged and more likely to attract cations, in turn, below this value, the surface of the adsorbent is positively charged and more likely to adsorb anions (Dąbkowska-Naskręt 2009, Horst et al. 2015, Illés et al. 2006, Liu et al. 2008)

There are various iron oxide nanoparticles synthesis technologies. They can be divided into two main streams or experimental approaches: "top-down" and "bottom-up". The "top-down" approach, currently rarely used, is based on crushing or grinding of the material in ball mills. The "bottom--up" is more prevalent than the "top-down" approach mainly due to easier, less time-consuming, cheaper and more efficient synthesis of nanoparticles which, as a result, have better quality and smaller size distribution. There are a variety of "bottom--up" methods. They are mainly based on the chemical synthesis of nanomaterials, however, some physical processes are also in use. Thus, one can point out such "bottom-up" techniques as chemical precipitation, pyrolysis, sol-gel method, thermolysis of precursors or hydrothermal reactions etc. (Khan et al. 2011, Runowski et al. 2014).

One of the most popular is the method of magnetite nanoparticles synthesis by their chemical co-precipitation from a mixture of iron(II) and (III) salts by addition of a base in the form of aqueous ammonia and sodium or potassium hydroxide (Maity et al. 2006, Mascolo et al. 2013). In the literature, one can also find precipitation methods with only Fe(II) or Fe(III) salt solutions instead of their mixtures. For example, if the precipitation of iron oxides involves the use of Fe(II) salts only, the partial oxidation of Fe(II) to Fe(III) may be conducted by H_2O_2 addition – oxidative co-precipitation (Hu et al. 2010). In the case of Fe(III) salts, the reduction of Fe(III) to Fe(II) can be initiated by Na_2SO_3 – reduction co-precipitation (Qu et al. 1999).

The popularity of chemical precipitation can be explained primarily by its simplicity, efficiency and low costs or productivity. This method enables the production of small, stoichiometric nanoparticles of high purity. Furthermore, the ability to control the synthesis conditions, such as pH of the reaction solution, the reaction temperature, speed of base addition, the concentration of solutes and the ionic strength of the reaction solution makes it possible to control the size and shape of the resulting nanoparticles (Vayssie'res et al. 1998, Khan et al. 2011, Mascolo et al. 2013). It was proved that particle sizes decrease with increasing pH and decreasing temperature of reaction (Mascolo et al. 2013, Petcharoen et al., 2012).

The objective of this work was to synthesize the magnetic iron oxide (magnetite) nanoparticles in different conditions by the co-precipitation method. After synthesis and post synthesis treatment nanoparticles were examined as heavy metal ions adsorbent. The main aim was to investigate how the co--precipitation synthesis conditions of magnetite influence its heavy metal ions separation ability. In the experiments, seven heavy metals were taken into consideration, i.e. Cr(VI), Pb(II), Cr(III), Cu(II), Zn(II), Ni(II) and Cd(II).
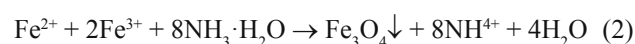
Experimental

Chemicals

The following chemicals were used for magnetite nanoparticles synthesis: $FeCl_3 \cdot 6H_2O$ (Warchem, Poland), $FeSO_4 \cdot 7H_2O$ and $NH_3 \cdot H_2O$ 25% solution (Stanlab, Poland). Heavy metal solutions were prepared using their salts, i.e. $Cr(NO_3)_3 \cdot 9H_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, $Cd(NO_3)_2 \cdot 4H_2O$, $Cu(NO_3)_2 \cdot 3H_2O$, $Pb(NO_3)_2$, $Zn(NO_3)_2 \cdot 9H_2O$ (POCH S.A., Gliwice, Poland), $K_2Cr_2O_7$ (Merck, Germany).

Nanoparticles preparation and characterization

The method used for magnetite synthesis was based on their co-precipitation from a mixture of Fe(II) and Fe(III) salt solutions with the addition of 25% ammonia solution to complete the following reaction:

$$Fe^{2+} + 2Fe^{3+} + 8NH_3 \cdot H_2O \rightarrow Fe_3O_4 \downarrow + 8NH^{4+} + 4H_2O \quad (2)$$

The synthesis reaction was conducted in different temperature/ammonia quantity conditions. The base volume was the factor which affected the pH of the reaction mixture. Moreover, the reaction was conducted under aerobic conditions so the partial oxidation of Fe(II) to Fe(III) was predicted. Because stoichiometric value of Fe(II):Fe(III) molar ratio in magnetite equals 1:2, to compensate the process of oxidation during the synthesis reaction, the initial ratio of Fe(II):Fe(III) in the reaction solution was decreased to 1:1.5.

The amounts of iron salts used during the synthesis were prepared following Liu et al. method (Liu 2008) – 6.1 g $FeCl_3 \cdot 6H_2O$ and 4.2 g $FeSO_4 \cdot 7H_2O$ were dissolved in 100 cm³ of distilled water. The mixture was placed in water bath and heated to reach a required temperature. Then, starting the mechanical stirring of the mixture, the appropriate volume of base in the form of 25% ammonia water was added to initiate magnetite precipitation. The magnetite syntheses were conducted at three temperatures – 30°C, 60°C and 90°C and for each temperature, three volumes of ammonia solution were added – 8 cm³, 10 cm³ and 15 cm³ (thus nine synthesis reactions were carried out). The quantities of the reagents in comparison to the stoichiometric reaction are presented in Table 1.

After base addition, the stirring was continued for 30 minutes whilst keeping the mixture at stable temperature conditions. Afterwards, the mixture was cooled to room temperature, its pH was measured and the black precipitate of magnetite nanoparticles was washed approximately six times with 50 cm³ of distilled water with help of a neodymium magnet, in order to remove the unreacted residues of the chemicals. The final pH of the mixtures after iron oxides precipitation as well as the temperature and ammonia volumes conditions of each synthesis are given in Table 2.

Then, following the synthesis treatment described by Schwertmann et al. (2000), the magnetite sample was dried in an oven at 40°C and crushed in an agate mortar to break up aggregates and to obtain the powder.

The sample synthesized at 30°C with 10 ml of ammonia was used for further characterization. Qualitative and quantitative analysis of the sample as well as its particle size analysis were carried out by X-ray powder diffraction (XRD) in a Bruker Advance D8 powder diffractometer (Bruker, USA). The morphology and structure of the sample were investigated

Table 1. Amounts of chemicals used in synthesis reactions

Fe^{2+}	Fe^{3+}	$NH_3 \cdot H_2O$		Fe^{2+}	Fe^{3+}	NH_3H_2O
mol	mol	cm³	mol	molar ratios of chemicals		
				1	2	8*
0.015	0.023	8.0	0.057	1.0	1.5	3.8
0.015	0.023	10.0	0.071	1.0	1.5	4.7
0.015	0.023	15.0	0.107	1.0	1.5	7.1

*stoichiometric amounts of chemicals

Table 2. Experimental data

Number of syntheses/combinations	Temperature	Ammonia water volume	Final pH after synthesis
	°C	cm³	
1	30		7.31
2	60	8	5.81
3	90		5.31
4	30		8.84
5	60	10	8.53
6	90		6.95
7	30		9.37
8	60	15	9.16
9	90		7.92

with Scanning Electron Microscope FEI Quanta 650 FEG (FEI, USA). Specific surface area and pore distribution of the sample were evaluated by Brunauer-Emmett-Teller (BET) and Barrett--Joyner-Halenda (BJH) method using TriStar II 3020 (V1.03) surface area and porosity measurement system (Micromeritics Inc., USA).

Adsorption procedure

Batch adsorption studies were conducted in order to examine the differences in heavy metal separation from the solution by magnetite nanoparticles synthesized in different temperature/ammonia quantity conditions. For each of the seven heavy metals, the adsorption process was conducted on each of the nine sorbents with three repetitions.

In every removal procedure, 50 ±1 mg of previously prepared (at one of the nine synthesis reactions) dry magnetite powder was placed in a small plastic container and poured over with 20 cm³ of one metal solution at 10 mg·dm⁻³ concentration. During the sorption process pH was not corrected and maintained as it settled itself. The pH values were measured immediately after pouring metal solution to the container and after sorption process at the adsorption equilibrium. So prepared adsorption reactor was placed on a mechanic shaker and shaken for 8 hours at room temperature. After that, the reactor was put on the neodymium magnet in order to help adsorbent sedimentation and 10 cm³ of supernatant was collected.

Atomic absorption spectrometer SepctrAA 880 (Varian) with atomization in the acetylene-air flame was used to determine the content of metal in the solution after the sorption process.

Results and discussion

Sorption results

Based on the concentration of heavy metal ion in the solution before and after sorption, the process efficiency η was evaluated according to the following equation:

$$\eta = \frac{C_i - C_f}{C_i} \cdot 100\% \qquad (3)$$

where C_i and C_f are metal ion concentrations before and after sorption (initial and final concentration) respectively. At Figures 1–7 the separation efficiencies for each metal using iron oxides synthesised at different combinations of temperatures and base volumes, as well as pH values, are presented. At every diagram, for each combination only one pH value is shown because it turned out that it has remained almost unchanged during the sorption process.

While analysing the results, pH during the adsorption process is the first issue that should be taken into consideration as it is one of the main factors which influence process efficiency. Because the aim of this study was to compare adsorbents, which were precipitated in different conditions, it was important to maintain the stable pH in the reaction containers within each metal ion, during the sorption process. As it arises from the diagrams (Fig. 1–7) the pH values showed only slight fluctuations. For most of the ions they stayed in the range of 4–5 and do not exceed the pH of point of zero charge of magnetite. Moreover, the pH values were also low enough to avoid the metal hydroxides precipitation. Taking into account

the above, it was assumed that during the sorption process the pH conditions were stable within each individual ion, so the data of sorption abilities of nine considered materials could be compared.

Comparing charts data of separation efficiencies, it is noticeable that the least differences in sorption on iron oxides synthesized under different conditions and, at the same time, the highest values of the metal ion removal were obtained for Cr(VI). Its separation using all adsorbents was above 90%, reaching almost 100%. For this metal it is hard to indicate which adsorbent and hence which synthesis conditions are more favourable.

High levels of separation were also reached for Pb(II) and Cr(III) ions. Their removal efficiencies varied from 76 to 92% and from 66 to 77% respectively. Moreover, it is visible that for Pb(II) ions material synthesised at 30°C with 10 cm^3 of ammonia is more effective than the others. The usage of this adsorbent brought slight separation increase from a few to several percent when comparing it with the adsorption on the magnetite synthesised in other conditions. In turn, the adsorbent produced also at 30°C but with 15 cm^3 of ammonia solution seems to be more favourable for Cr(III) ions removal.

The separation effectiveness for the rest of ions, i.e., Cu(II), Zn(II), Ni(II) and Cd(II), is evidently smaller. But on the other hand, for this group of metals, the adsorbent co-precipitated at 30°C and 10 cm^3 of ammonia can be marked out, as the one whose separation ability is higher than that of the others. The sorption on this material led to 26% separation of Cu(II) and

Zn(II), 21% of Cd(II) and 14% of Ni(II), while the removal of those ions using the other tested adsorbents reaches form 0 to only few percent.

Summing up, it can be said that lower temperatures are more suitable to synthesize the efficient magnetite adsorbent. The materials produced at 30°C were generally more effective than the others. It can be explained by the fact that the temperature may cause the particle growth acceleration what was proved in the literature (Petcharoen et al. 2012). Moreover, the lower the particles size, the bigger surface area of the material (Mascolo et al. 2013), what, in turn, improves its heavy metals adsorption ability.

It is suspected that the mechanism of the reactions which occurred during sorption experiments was the electrostatic attraction described in introduction. It is worth to mentioned that for most of the ions (cations) the process was led in not the most favourable pH levels – pH of the solutions was below point of zero charge (pzc) of magnetite It means that the adsorbents were more suitable to attract anions – predominant groups on their surface were Fe-OH$_2^+$. This is the reason why the highest removal efficiencies were achieved for Cr(VI) anions.

On the other hand, it can be said that pH in reaction containers for the rest of ions was not significantly far from pzc. Because of this fact the materials also demonstrated bigger or smaller affinity for cations – Fe-OH$^-$ groups were still present. It is especially noticeable for Pb(II) and Cr(III) ions whose separations are high.
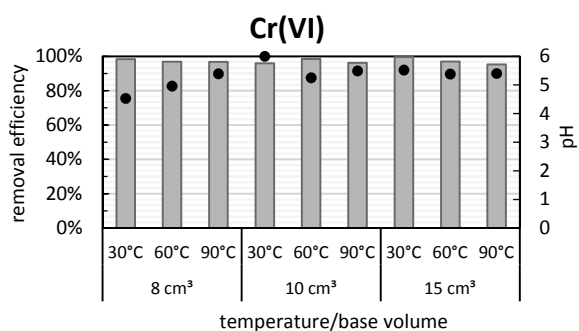

Fig. 1. Removal efficiency of Cr(VI) ions by iron oxides synthesised at different ammonia volume/temperature conditions
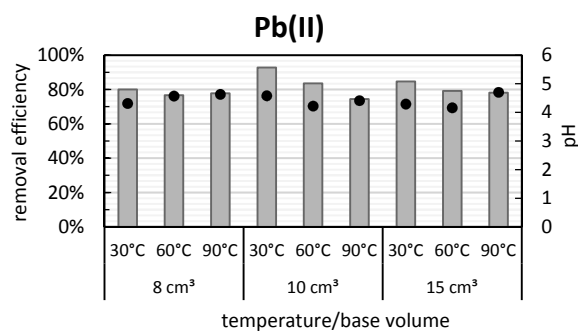

Fig. 2. Removal efficiency of Pb(II) ions by iron oxides synthesised at different ammonia volume/temperature conditions
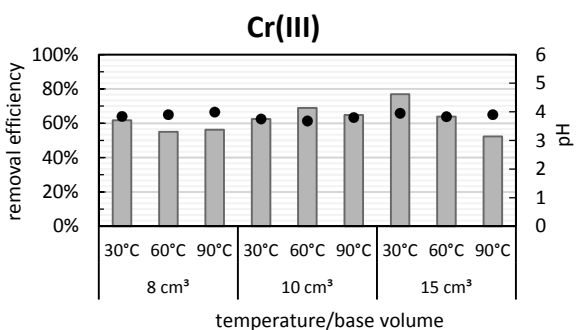

Fig. 3. Removal efficiency of Cr(III) ions by iron oxides synthesised at different ammonia volume/temperature conditions
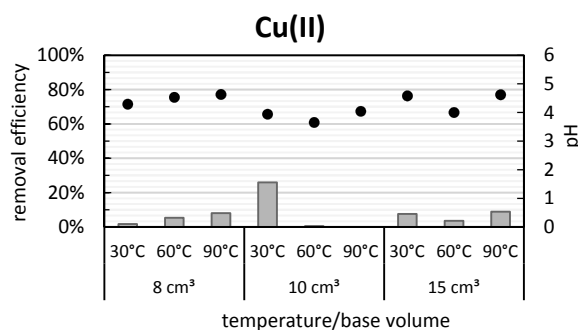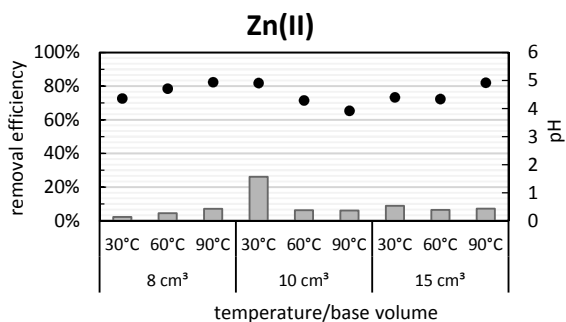

Fig. 4. Removal efficiency of Cu(II) ions by iron oxides synthesised at different ammonia volume/temperature conditions
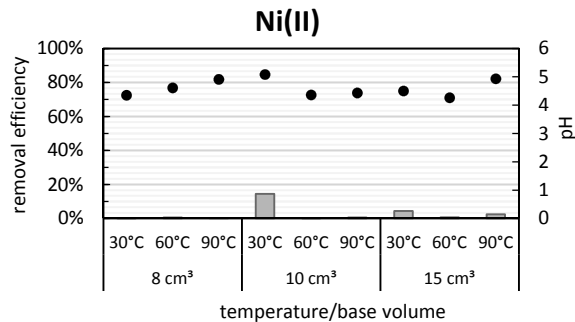
Zn(II)

Fig. 5. Removal efficiency if Zn(II) ions by iron oxides synthesised at different ammonia volume/temperature conditions

Ni(II)

Fig. 6. Removal efficiency of Ni(II) ions by iron oxides synthesised at different ammonia volume/temperature conditions
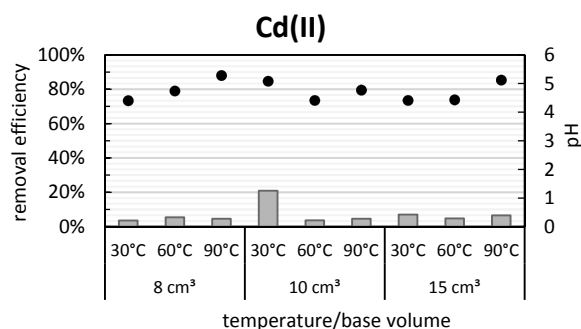
Cd(II)

Fig. 7. Removal efficiency of Cd(II) ions by iron oxides synthesised at different ammonia volume/temperature conditions

Sample characterization

Characterization was made for the iron oxide sample synthesised at 30°C with 10 cm³ of ammonia addition which, for most of the examined heavy metal ions, had the best sorption ability. In Figure 8, the XRD pattern of the sample is shown. The obtained diffraction peaks fit well the standard XRD magnetite pattern from JCPDS – ICDD data base. Quantitative Rietveld analysis indicated magnetite to be the main 100% crystalline compound in the sample. The crystallite size was estimated at 23.2 nm from the most intense reflection according to the Scherrer's formula.

Figure 9 shows SEM images. The irregular, micrometric (from a few to several hundred micrometers in size) aggregates of the magnetite sample crushed in a mortar are visible. Each of them is the agglomeration of nanosized iron oxides. This form of the sample is due to its after-synthesis treatment procedure. Drying of iron oxides in the form of wet precipitate causes their agglomeration, thus magnetite powder has a form of aggregated agglomerates in micrometric sizes.

Based on argon adsorption/desorption isotherms determined by BET method, the specific surface area of the sample was determined to be 55.64 g·m^{-2}. BJH desorption pore size and volume analysis are presented in Figure 10. Two prevailing pore sizes were distinguished – 6.81 and 2.55 nm. The pore size is affected by the size of individual particle of magnetite. In the case of research material the pore sizes indicates that the material is mesoporous (according to IUPAC classification).

The agglomeration of the iron oxides nanoparticles, even when they are left in the suspension form (without drying), is
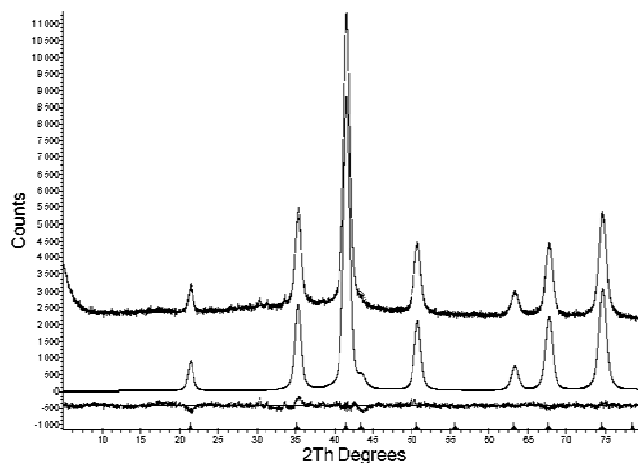

Fig. 8. X-ray diffraction pattern of magnetite synthesised at 30°C with 10 ml ammonium hydroxid
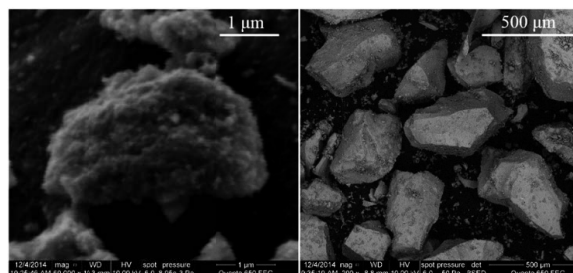

Fig. 9. SEM images of mortar crushed magnetite agglomerates used in heavy metal separation process
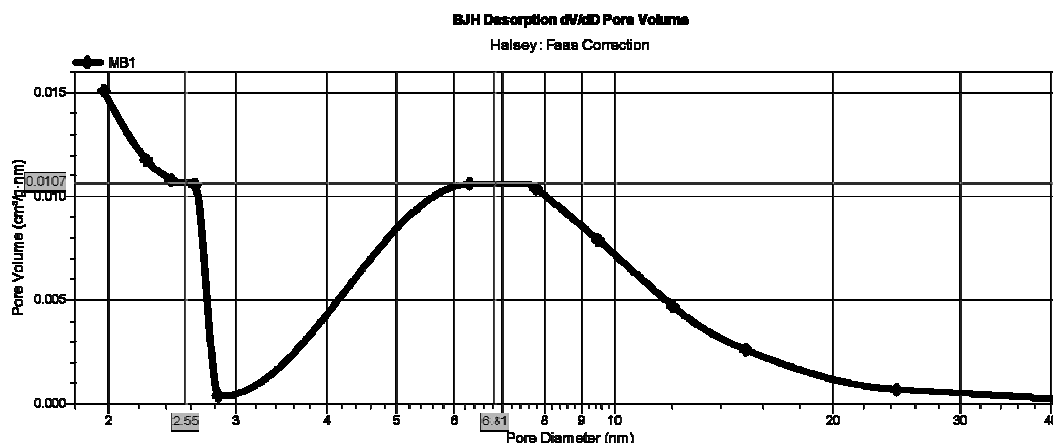
Fig. 10. BJH desorption pore size distribution curve

widely reported in the literature. This phenomenon is especially common when the nanoparticles are synthesised without surfactant addition. It is worth to mention that although the researched material aggregates were large in size, their surface area was comparable to the results obtained in other studies. In the work of Liu et. al. the aggregates of 10 nm nanoparticles in average size of 120 nm give surface area equal to 62 g·m^{-2} (Liu et al. 2008). In turn, Mascolo et al. proved that the nanoparticles in average sizes of 11.5 nm agglomerate giving the mesoporous material with the surface area of about 90 g·m^{-2} (Mascolo et al. 2013).

Conclusions

The first thing which is noticeable in the adsorption tests results is the general tendency of heavy metal ions removal. Although all tested magnetites were precipitated separately, each of them gave very good results in Cr(VI), Pb(II) separation, slightly worse in Cr (III) removal and much worse in separation of the rest metal ions considered, i.e. Cu(II), Ni(II), Zn(II) and Cd(II)

Moreover, it was proved that co-precipitation conditions of adsorbent in some cases influence its separation ability. The material synthesized in the reaction at 30°C and with 10 cm^3 of ammonia was more suitable for removal of Pb(II), Zn(II), Ni(II), Cd(II) and Cu(II) ions than other materials.

It should be also mentioned that the sorption processes were led without pH correction so it is highly probable that retention of some metal ions was not as high as it would be possible in more favourable pH conditions. The sorptions of all the ions were conducted below point of zero charge of the adsorbent, when its surface is in general positively charged and more likely attracts anions. That is why the best separation effectiveness was obtained for Cr(VI) ions. Nonetheless, due to the presence of some negatively charged sites on adsorbent at pH below pzc, bigger or smaller affinity for the rest of the ions (cations) was also noticeable. To conclude, in the work heavy metal ions were retained in the following order: Cr(VI) > Pb(II) > Cr(III) > Zn(II) > Cu(II) = Cd(II) > Ni(II)

Representative magnetite sample synthesized at 30°C and with 10 cm^3 of ammonia was subjected to a further testing. XRD pattern proved that the iron oxides produced in these conditions formed pure, crystalline magnetite with the grain size of about 23 nm. Because of the post-synthesis treatment of the sample

– drying and crushing in the mortar, nanoparticles underwent strong aggregation. It was especially noticeable in the SEM images – the magnetite powder used in the sorption process consists of micrometric agglomerates of magnetite nanoparticles, which, according to BHJ and BET testing, constitute mesoporous material with a specific surface area of 56 g·m^{-2}.

Acknowledgement

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Wpływ warunków syntezy nanocząstek magnetytu na ich zdolność do separacji jonów metali ciężkich

Streszczenie: Nanocząstki magnetytu stanowią obiecujący materiał badań ze względu na możliwość ich praktycznego zastosowania w różnorodnych dziedzinach. Wśród wielu metod ich syntezy, jako jedną z najwygodniejszych, najmniej pracochłonnych oraz ekonomicznych, wyróżnić można chemiczne współstrącanie. Efektem metody chemicznego współstrącania są drobne nanocząstki tlenku żelaza o dużej czystości, odpowiednie do zastosowań w zagadnieniach środowiskowych. Celem pracy było badanie w jaki sposób warunki syntezy chemicznego współstrącania, takie jak temperatura reakcji czy też ilość użytej zasady wpływają na zdolności separacyjne nanocząstek magnetytu względem jonów kilku metali ciężkich. Syntezy prowadzone były w dziewięciu kombinacjach przy różnej objętości dodawanego amoniaku – 8 cm³, 10 cm³, 15 cm³ oraz temperaturze – 30°C, 60°C, 90°C. Powstałe podczas każdej z syntez tlenki żelaza były badane jako adsorbenty jonów siedmiu metali ciężkich: Cr(VI), Pb(II), Cr(III), Cu(II), Zn(II), Ni(II) and Cd(II). Ponadto próbka magnetytu zsyntezowanego w wybranych warunkach była badana przy użyciu metod XRD,

SEM oraz BET. Analizując wyniki procesu sorpcji stwierdzono, iż najbardziej efektywny materiał, dla większości metali ciężkich, powstał w temperaturze 30°C przy użyciu 10 cm³ amoniaku. Dodatkowa charakterystyka powstałego w tych warunkach sorbentu wykazała, iż stanowił on czysty magnetyt o średniej wielkości ziarna 23,2 nm (XRD). Ponadto wykazano, iż pojedyncze ziarna w badanej próbce są zaglomerowane (SEM) a powierzchnia właściwa agregatów wynosi 55,64 m²/g (BET). Ogólnym wnioskiem z pracy jest dowód, iż warunki syntezy badanego adsorbentu mają wpływ na jego zdolność separacyjne względem niektórych jonów metali ciężkich.