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## CONTROLLED SYNTHESIS OF IRON OXIDE NANOPARTICLES USED AS AN EFFICIENT HEAVY METAL IONS ADSORBENT

### KONTROLOWANA SYNTEZA NANOCZĄSTEK TLENKÓW ŻELAZA STOSOWANYCH JAKO EFEKTYWNY ADSORBENT JONÓW METALI CIĘŻKICH

**Abstract:** In this paper a few attempts of iron oxide nanoparticles synthesis via chemical co-precipitation are presented. Iron(II) and (III) salts were used as a precursors and aqueous ammonia as a precipitation medium. In the literature it was proved that there are some factors, like reaction temperature, base amount (pH of the reaction mixture) or the concentration of iron salts, which affects characteristics of formed nanoparticles such as their size distribution. The aim of presented research was to select such reaction parameters which would lead to the production of the most effective sorbent for few heavy metal ions, such as: Cr(VI), Pb(II), Cr(III), Cu(II), Zn(II), Ni(II) and Cd(II). The synthesis of nanoparticles were carried out for three temperatures (30, 60, 90°C) and three ammonia volumes (8, 10, 15 cm<sup>3</sup>) for each temperature. Furthermore the influence of iron salts molar ratio Fe(II) : Fe(III) in the reaction mixture on resulting sorbent was examined. Because the syntheses were lead in the oxidizing environment, in order to compensate partial oxidation of Fe(II) to Fe(III), a few molar ratios Fe(II) : Fe(III) beyond the stoichiometric value (which is 1 : 2 for magnetite Fe<sub>3</sub>O<sub>4</sub>) were investigated. Additionally for the magnetite synthesized in the selected conditions the effect of pH on the sorption of heavy metal ions were examined. The pH conditions were estimated to not exceed 7 in order to avoid metal precipitation.

**Keywords:** adsorption, heavy metals, magnetite, nanoparticles

#### Introduction

Heavy metals are used by human beings from thousands of years. Despite the awareness about their toxicity and negative impact on the living organisms the exposure to heavy metals is still significant, especially in the developing countries.

Most of heavy metals in the environment have industrial origin. The serious environmental problems constitute the industrial plants which discharge effluents containing such heavy metals as Cd, Cr, Cu, Ni, Pb or Zn. These heavy metals tend to accumulate in the living organisms, causing many health disorders like organs and nervous system damages, tumours, reduced growth and development of children and in some cases, even death [1, 2].

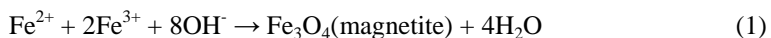
In the last years intensification of research on the economical and efficient technologies in order to reduce quantities and improve qualities of wastewater discharged into environment is noticeable. There are many conventional technologies for heavy metal removal such as ion exchange, chemical precipitation, flotation, chemical decomposition or adsorption [1]. Considerable attention is paid on the development of applicable and cost effective adsorbents. Among various researches on adsorbents, many works confirm that iron oxides are those which can be used for efficient heavy metal removal [3-6]. Iron oxides

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\* Contribution was presented during ECOpole'15 Conference, Jarnoltówek, 14-16.10.2015

in form of magnetite, maghemite or goethite are the natural minerals which occur in earth crust. They are characterized by very reactive surface. Active sites and large surface area made them able to bound contaminants in the form of cations and anions as well [7]. Iron oxides application in wastewater treatment area is especially attractive when their size is limited to nanoscale. As nanoparticles they provide high efficiency, fast reaction kinetics and high reactivity with respect to the separated ions. These properties are caused by the extremely small particles size and a large specific surface area. Besides, those nanoparticles have also another important feature - magnetism. This feature is especially important in water and wastewater treatment systems as it provides a great convenience in the process of separation of the nanoparticles from the water after adsorption process. Separation can be carried out using a small magnetic field or even handheld magnet [8, 9].

One of the simplest and the most economical methods of the magnetite nanoparticles production is chemical co-precipitation. The method is founded on co-precipitation from Fe(II) and Fe(III) salts by addition of a base [10]. The stoichiometric reaction can be written as follows:



The properties of nanoparticles produced during chemical co-precipitation, such as their size, chemical composition or morphology, strongly depend on the synthesis reaction parameters. Among them one can point out such parameters as reaction temperature, pH and type of base, mixing rate, ionic strength of medium, presence of the coating agent (*eg* surfactant), reaction mixture deoxidization by for example nitrogen gas bubbling or Fe(II) : Fe(III) molar ratio value [11, 12]. For instance, it was proved that the size of the nanoparticles increase with the temperature of the synthesis reaction and decrease with the increasing amount of base [12, 13].

The aim of this study was to investigate how the synthesis conditions influence the resulting magnetite efficiency as a heavy metal sorbent. Thus syntheses reactions were led at different combinations of temperatures and base amounts. The properties of the iron oxides were examined on the basis of following ions separation efficiencies - Cu(II), Ni(II), Zn(II), Cd(II), Pb(II), Cr(III) and Cr(VI). After choosing the reaction conditions which seemed to be most reasonable for the efficient sorbent production, the pH effect of the sorption process on its efficiency regarding to four ions, *ie* Zn(II), Cu(II), Cd(II) and Pb(II), was investigated.

## Experimental

### *Chemicals and methods*

The syntheses of magnetite nanoparticles were conducted using iron salts -  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  (Warchem),  $\text{Fe}_2\text{SO}_4 \cdot 7\text{H}_2\text{O}$  (Stanlab) and base solution -  $\text{NH}_3 \cdot \text{H}_2\text{O}$  25% (Stanlab).

Heavy metal solutions at a concentration of 20 and 50 mg/dm<sup>3</sup> were prepared based on the following, mainly nitrate, salts -  $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ,  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ ,  $\text{Pb}(\text{NO}_3)_2$ ,  $\text{Zn}(\text{NO}_3)_2 \cdot 9\text{H}_2\text{O}$  (POCH S.A.),  $\text{K}_2\text{Cr}_2\text{O}_7$  (Merck). For pH correction base - NaOH (0.1M) and acid -  $\text{HNO}_3$  (0.1M) solutions were used (POCH S.A.).

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

### *Magnetite nanoparticles preparation*

Nanoparticles syntheses were conducted following modified Liu et al method [5]. General procedure of all syntheses includes succeeding steps:

- dissolution of appropriate  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  salts amounts in  $100 \text{ cm}^3$  of distilled water,
- placing the mixture in the water bath followed by heating in order to reach required temperature,
- starting the mechanical stirring of the mixture,
- rapid addition of  $50 \text{ cm}^3$  distilled water and appropriate volume of the base to initiate magnetite precipitation,
- vigorous stirring for 30 minutes whilst keeping the mixture in the same temperature conditions,
- cooling the mixture to the room temperature (optional pH measurements of mixtures after synthesis),
- washing of black precipitate of magnetite by distilled water with the help of a neodymium magnet in order to separate sorbent from unreacted residues of chemicals,
- preparation of approximately  $5 \text{ g/dm}^3$  suspension of magnetite nanoparticles.

### *The synthesis at different temperature and pH conditions*

In first part of the research the influence of temperature and volume of precipitating medium, which affected pH of reaction mixture, on resulting adsorbent efficiency were examined.

Magnetite synthesis conditions at different temperature/base volumes

Table 1

Number of syntheses/combinations	Temperature	Ammonia water volume	Molar ratios of chemicals $\text{Fe}^{2+} : \text{Fe}^{3+} : \text{NH}_3 \cdot \text{H}_2\text{O}$	Final pH after synthesis
	[°C]	[ $\text{cm}^3$ ]		[-]
1	30	8	1 : 1.5 : 3.8	7.31
2	60			5.81
3	90			5.31
4	30	10	1 : 1.5 : 4.7	8.84
5	60			8.53
6	90			6.95
7	30	15	1 : 1.5 : 7.1	9.37
8	60			9.16
9	90			7.92

The molar ratio of iron salts was according to Liu et al [1] maintained stable, equal  $\text{Fe(II)} : \text{Fe(III)} = 1:1.5$  (4.2 g  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and 6.1 g  $\text{Fe}_2\text{SO}_4 \cdot 7\text{H}_2\text{O}$ ) whereas temperature of syntheses was settled at 30, 60 or 90°C and base was added in volume of 8, 10 or 15  $\text{cm}^3$ .

Summarizing, nine syntheses combinations at different temperature/base volumes conditions were carried out. The amounts of chemicals used in each combination in comparison to the stoichiometric synthesis reaction, which for pure magnetite is  $\text{Fe}^{2+} : \text{Fe}^{3+} : \text{NH}_3 \cdot \text{H}_2\text{O} = 1 : 2 : 8$ , and final pH of a mixtures after each synthesis were collected in Table 1.

#### *The synthesis at different Fe(II)/Fe(III) molar ratio conditions*

The aim of second part of the research was to investigate how the molar ratio of iron salts effects the resulting adsorbent efficiency. Because of the fact that syntheses were conducted with an oxygen access, the partial oxidation of Fe(II) to Fe(III) were predicted. Therefore a few syntheses with different Fe(II) and Fe(III) salts amounts were conducted. Four Fe(II) : Fe(III) ratios below stoichiometric one were chosen, that is 1 : 1, 1 : 1.25, 1 : 1.5, 1 : 1.75, one equal to the pure magnetite stoichiometry - 1 : 2 and one ratio above - 1 : 2.25. The molar ratios and corresponding iron salts amounts were collected in Table 2.

Table 2

Magnetite synthesis conditions at different Fe(II)/Fe(III) molar ratios

Number of syntheses	Iron salts molar ratio		Iron salts amount	
	Fe(II)	Fe(III)	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$
			[g]	[g]
1	1	1	4.20	4.08
2	1	1.25	4.20	5.10
3	1	1.5	4.20	6.13
4	1	1.75	4.20	7.15
5	1	2	4.20	8.17
6	1	2.25	4.20	9.19

All syntheses with different iron salts amounts were conducted under the most appropriate temperature/base volume conditions obtained from the first part of the research - that is at 30°C and with 15 cm<sup>3</sup> of ammonia solution.

#### *Batch sorption tests*

In order to examine how the synthesis conditions of magnetite, *ie* the temperature, base volume and molar ratio of iron salts added to the reaction mixture, effect resulting adsorbent the batch adsorption tests were conducted. In every heavy metal removal procedure 5 cm<sup>3</sup> of magnetite particles suspension (5 g/dm<sup>3</sup>) was placed in plastic container and poured over with 20 cm<sup>3</sup> of metal solution (Cu(II), Zn(II), Ni(II), Cd(II), Cr(VI), Cr(III) or Pb(II)) at concentration of 20 mg/dm<sup>3</sup> (for temperature/base volume testing) and 50 mg/dm<sup>3</sup> (for molar ratio testing). Every sorption experiment was performed in triplicate without pH correction. Then the reactors were placed on mechanical shaker and shaken for 8 hours at room temperature. In the next day the adsorbents were separated via external magnetic field and supernatants were collected for metal concentration measurement.

Moreover, following the procedure of sorption described above, the influence of pH during the separation process on its efficiency was examined for four metal ions, *ie* Cu(II), Zn(II), Cd(II) and Pb(II). The sorbent was prepared at 30°C, 15 cm<sup>3</sup> of base and

Fe(II) : Fe(III) molar ratio equal to 1 : 1.5, heavy metal solutions were used in concentration of 50 mg/dm<sup>3</sup>. The pH conditions during sorption process was established at different values for each test, namely 3, 4, 5, 6, 7.

## Results and discussion

The separation process efficiency  $\eta$  was evaluated according to the following equation:

$$\eta = [(C_i - C_f)/C_i] \cdot 100\% \quad (2)$$

where  $C_i$  and  $C_f$  are metal ion concentrations before and after sorption (initial and final concentration), respectively.

### *The results for synthesis at different temperature and base volume conditions*

Results of each ion separation effectiveness are shown at Figures 1-7. Analysed ions can be divided into two groups. The first group - Cu(II), Zn(II), Cd(II) and Ni(II) ions, which removal is substantially smaller than separation of the second group - Pb(II), Cr(III) and Cr(VI) ions.

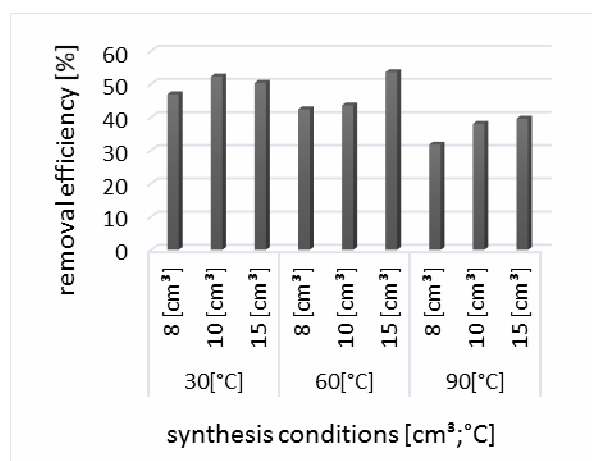


Fig. 1. Removal efficiency of Cu(II) ions by iron oxides synthesised at different ammonia volume/temperature conditions

While analysing the first group of ions it can be observed that the maximum separation efficiencies amounted to more than 50% for Cu(II) and Zn(II), over 30% for Ni(II) and about 20% for Cd(II). Concerning the effect of the base volume added during syntheses, it can be said that the smallest removal of these ions for the whole range of temperatures, were achieved using 8 cm<sup>3</sup> of ammonia solution. It is related to the too low pH of the reactions environments and thus to insufficient amounts of -OH groups required for proper synthesis of iron oxides. The pH values measured immediately after syntheses was in the range from 5 to over 7 (Table 1). Especially large diversity in separation results was obtained for Zn(II) ions. Their removal using magnetite synthesised at 30 and 60°C

increased from 20% while using 8 cm<sup>3</sup> of base to over 50% while using 15 cm<sup>3</sup> of ammonia solution. Similar trend can be observed for Ni(II) ions.

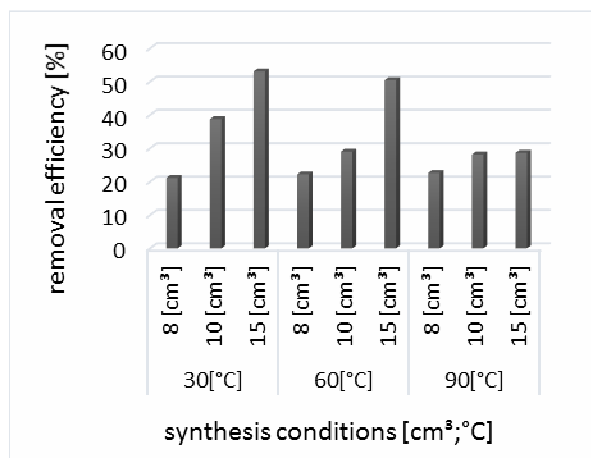


Fig. 2. Removal efficiency of Zn(II) ions by iron oxides synthesised at different ammonia volume/temperature conditions

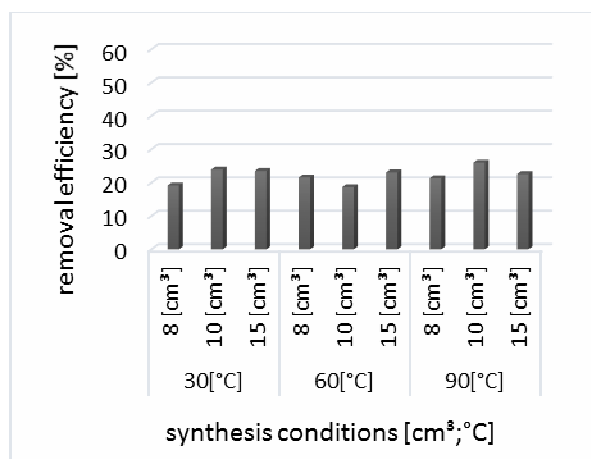


Fig. 3. Removal efficiency of Cd(II) ions by iron oxides synthesised at different ammonia volume/temperature conditions

The effect of temperature conditions on resulting sorbent separation efficiency was also most noticeable for Zn(II) ions sorption - the highest removal was obtained while using magnetite produced at 30°C. Similar tendency was observed for Ni(II) ions with less noticeable removal efficiency decrease while using sorbent synthesised at 60°C and more noticeable for iron oxides synthesised at 90°C. In case of Cu(II) ions, although the biggest removal was obtained during sorption on the magnetite produced at 60°C, the results was

not so differ from those observed for material synthesised at 30°C. In turn, the noticeable decrease of Cu(II) ions removal was observed while using sorbent made at 90°C. The smallest differences at sorption by using sorbents obtained at different temperatures was noticed for Cd(II) ions.

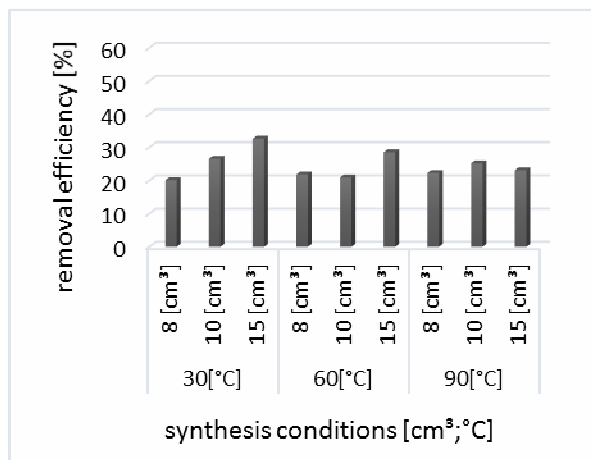


Fig. 4. Removal efficiency of Ni(II) ions by iron oxides synthesised at different ammonia volume/temperature conditions

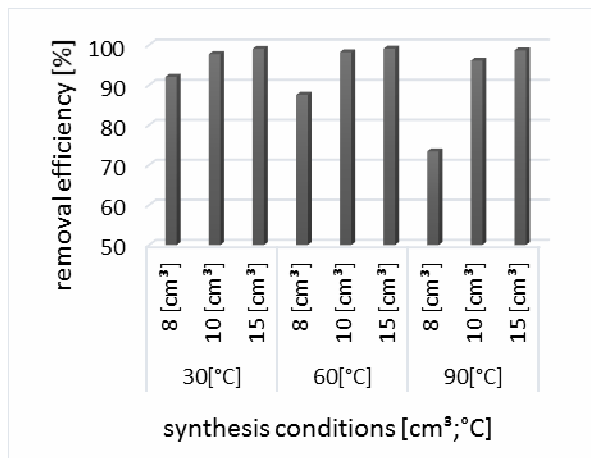


Fig. 5. Removal efficiency of Pb(II) ions by iron oxides synthesised at different ammonia volume/temperature conditions

Analysing the results for second group of ions - Cr(VI), Pb(II) and Cr(III), their high, up to 100%, removal is noticeable. Despite small differences in removal efficiencies while applying sorbents synthesised in different temperature/base amounts conditions some tendencies also can be observed. Especially with regard to the amount of base addition.

Similar as for ions from the first group, the 8 cm<sup>3</sup> of ammonia was insufficient to produce efficient sorbent. While using this amount of base at temperature increasing up to 90°C, a clear decrease in the sorption properties of material was noticeable (due to increased evaporation, pH of the reaction mixtures during the synthesis decreased). In turn, for the majority of these metals there was no significant difference between sorbent synthesised with 10 and 15 cm<sup>3</sup> of base. The quite observable difference can be seen only for Cr(III), wherein the removal properties of the sorbent synthesized at 90°C with 10 cm<sup>3</sup> of ammonia usage was less than with 15 cm<sup>3</sup> of base applied. Slight differences can also be noticed while analysing the effectiveness of the Pb(II) removal.

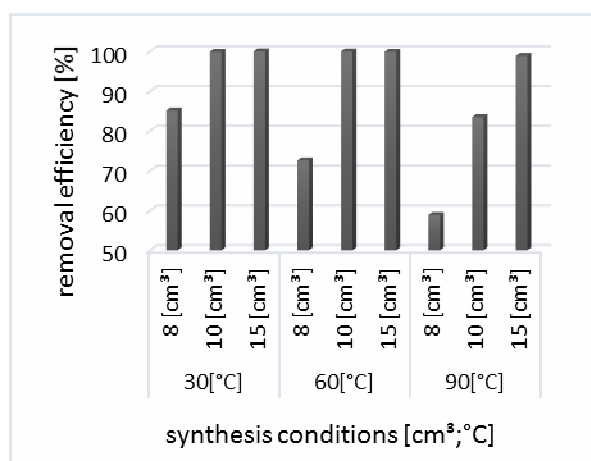


Fig. 6. Removal efficiency of Cr(III) ions by iron oxides synthesised at different ammonia volume/temperature conditions

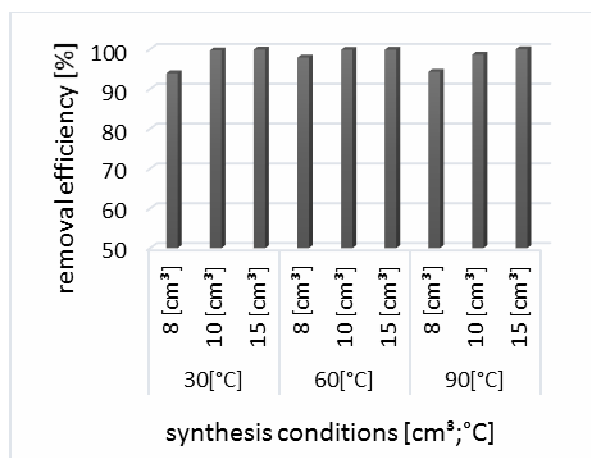


Fig. 7. Removal efficiency of Cr(VI) ions by iron oxides synthesised at different ammonia volume/temperature conditions



Given the above analyses, for further research the magnetite syntheses was carried out at 30°C with the 15 cm<sup>3</sup> of a base addition.

*The results for synthesis with different amount of Fe(II) and Fe(III) salts*

The results of ions separation using magnetite synthesized under optimal temperature and base volume conditions (selected in the previous research) but with different molar ratios of iron salts added to the reaction mixture are shown at Figure 8.

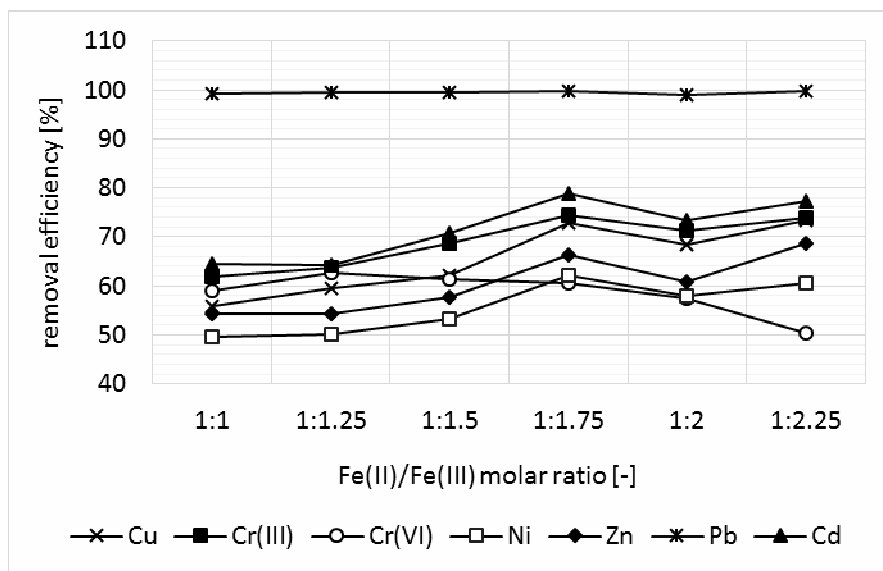


Fig. 8. Removal efficiency of heavy metal ions by iron oxides synthesised at different Fe(II) : Fe(III) molar ratios

Except Pb(II) ions, which removal regardless of the molar ratio Fe(II) : Fe(III) used during sorbent production was maintained in the range of 100%, the differences in separation efficiencies for other ions can be observed.

The least effective sorbent for most of heavy metal ions (Cu(II), Zn(II), Ni(II), Cd(II), Cr(III)) was obtained with a molar ratio Fe(II) : Fe(III) equal to 1 : 1 and 1 : 1.5. Over this ratio, while trivalent iron salt amount increased up to proportion 1 : 1.75, the sorption capacity of magnetite also increased. At Fe(II) : Fe(III) ratio equal to 1 : 2, which is stoichiometric one for pure, solid magnetite, the separation efficiencies decrease and then increase again at ratio 1 : 2.25. Considering mentioned results, Fe(II) : Fe(III) molar ratios between 1 : 1.5-1 : 1.75 seem to be the most optimal.

Although good separation effectiveness was also obtained for the ratio of 1 : 2.25, this proportion of iron salts is not recommended because of the product purity uncertainty. The aim of the study was the usage of iron oxides as the sorbent meanwhile the product obtained at 1 : 2.25 was characterized by slightly brownish colour and slower or incomplete settling on a neodymium magnet that can indicate worse magnetic properties. It can be the evidence of other iron forms presence such as iron hydroxides.

*The pH of sorption process effect on Cu(II), Zn(II), Cd(II) and Pb(II) ions separation efficiency*

The results of Cu(II) Zn(II), Cd(II) and Pb(II) ions separation at different pH conditions are shown in Figure 9.

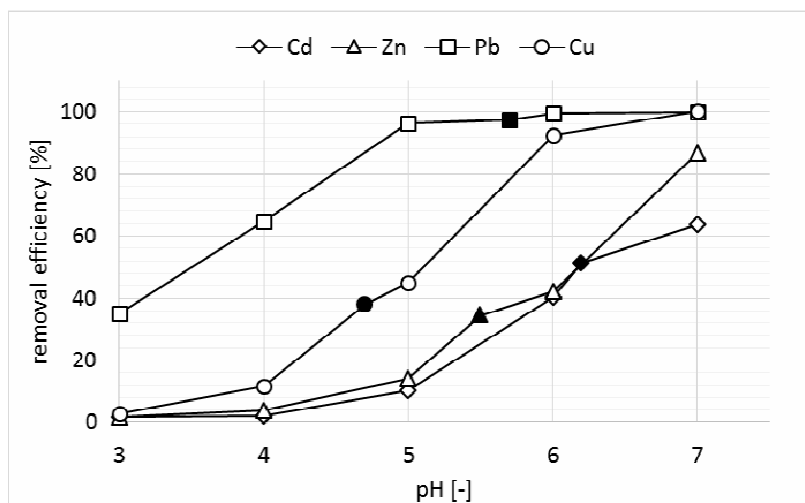


Fig. 9. The effect of pH on heavy metal ions separation effectiveness

The sorption was carried out at the following pH values: 3, 4, 5, 6, 7 and, for comparison, without pH adjustment after pouring the suspension of nanoparticles with a solution of heavy metal (marks filled in black at Fig. 9).

Analysing series of data at the graph, the close relationship between pH of the sorption process and separation efficiencies of each metal ion can be noticed.

The low pH values result in the small removal of Cd(II), Zn(II) and Cu(II) in range of 2-3% and about 35% for Pb(II). The gradual separation increase with the rise of the alkalinity was noticed for all ions. In case of Cd(II) from 3% at pH 3 to 60% at pH 7, for Zn(II) ions from 2 up to 87%, for Cu(II) ions from 3 to 92% and finally for Pb(II) ions from 35 to 100%.

Moreover, for Pb(II) ions the pH of 5 was enough to achieve almost 100% separation and more than 90% of Cu(II) ions were removed at pH 6. It is an important information, because especially in case of Pb(II) ions, the increasing pH values of its solutions above 6, cause their visible precipitation. Thus the high removal efficiency at pH 7 could be not the result of sorption by itself but also chemical precipitation process.

## Conclusions

It can be concluded that the sorbents synthesized under different temperature/amount of base conditions are characterized by relative different separation properties with respect to various ions. It turns out that too small amount of ammonia seems to be generally insufficient to produce the effective sorbent. It was proved that usage of 10 or 15 cm<sup>3</sup> of

base with an indication of the latter volume is most justified. It was confirmed mainly during the Zn(II), Ni(II) and also Cr(III) ions separation analysis. Moreover, considering the effect of synthesis temperature on the magnetite efficiency, it appears to be reasonable to carry out the syntheses at the temperature of 30°C. Increasing the temperature to 60°C and 90°C does not appreciably change the sorption properties of magnetite (ions Cd(II), Cr(VI), Cd(III)) or worsen it more or less noticeably (Cu(II), Zn(II), Ni(II)).

Also the Fe(II) : Fe(III) ratio in the reaction mixture affects separation properties of the sorbent. It comes out that the most effective sorbent was synthesised while keeping Fe(II) and Fe(III) in the proportion between 1 : 1.5 and 1 : 1.75.

During the sorption process, the important element is to maintain pH at proper level. For all ions considered in this part of research, *ie* Cu(II), Zn(II), Cd(II) and Pb(II) the gradual increasing tendency in their separations was visible while increasing pH from 3 up to 7. In summary, it can be said that during the sorption the pH values should be maintained at high, efficient for the process level but also at level low enough to prevent heavy metal precipitation.

### Acknowledgements

This work was supported by the Silesian University of Technology project No. BKM-547/RIE4/2014 and BKM-539/RIE4/2015.

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## KONTROLOWANA SYNTEZA NANOCZĄSTEK TLENKÓW ŻELAZA STOSOWANYCH JAKO EFEKTYWNY ADSORBENT JONÓW METALI CIĘŻKICH

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**Abstrakt:** W pracy przedstawiono próby syntezy nanocząstek tlenków żelaza poprzez ich chemiczne współstrącanie. Jako medium strącające zastosowano wodę amoniakalną. W literaturze udowodniono wpływ takich czynników, jak temperatura reakcji, ilość zasady czy też stężenie soli żelaza Fe(II) i Fe(III) w mieszaninie reakcyjnej na charakterystykę, między innymi wielkość, powstających nanocząstek. Celem niniejszej pracy było dobranie takich parametrów reakcji, aby w efekcie uzyskać najbardziej efektywny sorbent względem jonów kilku metali ciężkich: Cr(VI), Pb(II), Cr(III), Cu(II), Zn(II), Ni(II) i Cd(II). Przeprowadzono syntezę nanocząstek w kombinacji trzech temperatur: (30, 60, 90°C) oraz trzech objętości amoniaku: (8, 10, 15 cm<sup>3</sup>). Ponadto sprawdzono, jaki wpływ na powstający sorbent ma stosunek molowy jonów żelaza Fe(II) : Fe(III) w mieszaninie reakcyjnej. Biorąc pod uwagę fakt, iż synteza przeprowadzana była przy dostępie tlenu, w celu kompensacji częściowego utlenienia jonów żelaza Fe(II) do Fe(III), wyjściowy stechiometryczny stosunek tych jonów, który dla magnetytu (Fe<sub>3</sub>O<sub>4</sub>) wynosi 1:2, obniżono do kilku wartości poniżej 1 : 2. Dodatkowo dla magnetytu zsyntezowanego w wybranych, optymalnych, warunkach przeprowadzono próbę wpływu pH na sorpcję jonów metali. W celu uniknięcia możliwości wytrącania się osadów maksymalne pH ustalono na nieprzekraczające wartości 7.

**Słowa kluczowe:** adsorpcja, metale ciężkie, magnetyt, nanocząstki