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Special: Edelstahl, Nickel, Chrom

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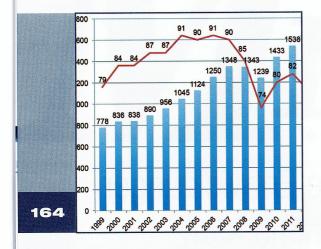
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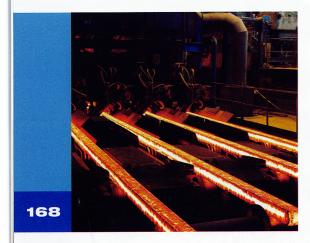
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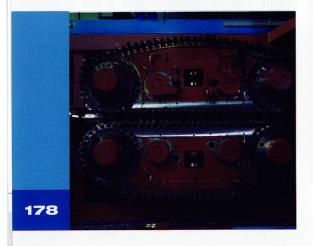


70. Jahrgang · Mai 2016 GDMB Verlag GmbH ISSN 0026-0746







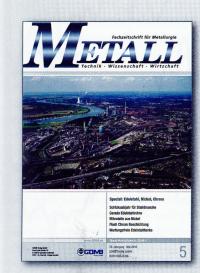


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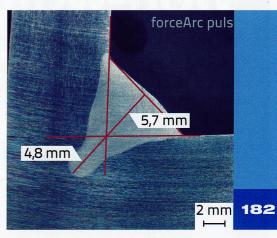
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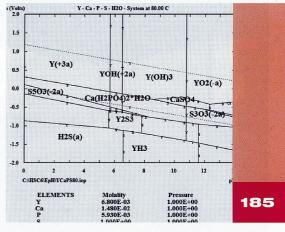


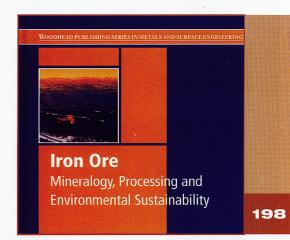
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Leaching of Spent Nd-Fe-B Magnetas in Acidic Media

Palencar, M.; Miskufova, A.; Kochmanova, A.; Horvathova, H. (1)

Neodymium was identified by the European commission as one of the deficient Rare earth elements (REE). This element is an irreplaceable element for permanent sintered Nd-Fe-B production. This work deals with kinetic and thermodynamic study of leaching the spent sintered Nd-Fe-B magnets obtained from hard disc drives of personal computers. The main aim is to find suitable conditions for transfer of neodymium into the mineral acid solutions and at the same time to verify behavjour of iron and nickel. Before leaching spent Nd-Fe-B magnets were subjected to thermal demagnetizing (heating up to Curie temperature of 340 °C) and after that to crushing and milling. The results showed that neodymium was leached at room temperature (20 °C) in the 0.5 M of sulfuric acid with the maximum extraction of 95 wt.% into the solution after 15 minutes of leaching. In the 0.1 M of hydrochloric acid the highest extraction of neodymium (31.7 wt. %) after 30 minutes of leaching at room temperature was achieved. In the 0.5 M H,SO, extraction 100 wt. % of iron into the solution was achieved. In the 0.1 M HCl the highest iron extraction of 32 wt. % was achieved. In the case of nickel the extraction around 1.2 wt. % in 0.5 M H_2 SO₄ and 0.2 % (0.1 M HCI) of leaching after 120 minutes was achieved.

ecause of the constant development and modernization of information technologies there is a trend to substitute older equipment with new and more powerful equipment. The consequence of this is a continuous increase of waste production from electrical and electronic equipment. This

Fig. 1: Schematic view of the leaching apparatus [15] 1- Stirrer engine; 2- propeller; 3-pulp; 4-sampler; 5- thermometer; 6- feeder; 7- water seal; 8-water thermostat; 9- Nd-Fe-B sample

type of waste represents a valuable secondary raw material especially for non-ferrous metals. Among these metals rare earth element (REE) are of great importance mainly in terms of critical raw materials. REE have been identified by European Commission as critical raw materials for the European Union [1,2]. Neodymium is also one of these deficient REE. At present this element can not be replaced by other elements and its resources in open deposits are limited. It's global reserves are estimated to be 16.7 million tons. The yearly production of neodymium amounts to approximately 7300 tons. For this reason it is necessary to focus on recycling of this element from secondary raw materials, which are the used Nd-Fe-B magnets [3,4]. Nd-Fe-B magnets were placed on the market after the year 1980. In comparison with other magnets (Alnico, SmCo) neodymium magnets have a higher value of maximal energy product. This lead to the highest performance per volume unit, which is a characteristic difference to others types of magnets available on the market [5]. The greatest usage (31%) and also the greatest secondary raw material source of these magnets are discarded hard disc drives (HDD) from personal computers [6]. The lifetime of one HDD is on average about 5 years. Statistics indicated (in 2009), that the yearly production of HDD is at the level of 600 million

pieces. One HDD contains from 10 to 20 g of Nd-Fe-B alloy. That means, that every year 6,000 to 12,000 tons of this alloy are produced. The current level of neodymium recycling varies from 1 to 10 % [7]. For this reason it is necessary to search new effective methods for the recycling of spent Nd-Fe-B magnets. These methods would save primary raw materials resources.

At the stage of laboratory research have been realized different pyrometallurgical, hydrometallurgical or combined methods for spent Nd-Fe-B magnets processing. Hydrometallurgical methods of treatment seem to be the most promising regarding energy and environmental impact. These methods are similar to the process of obtaining REE from primary raw materials and do not require high operating costs.

In the research sphere several results of leaching in the acidic medium have been published [8].

Lyman et. al. leached crushed Nd-Fe-B scrap, slag, swarf and waste from primary production of these magnets. Leaching was realized in 2M solution of $\rm H_2SO_4$ without stirring at ambient temperature (25 °C) for 24 hours. Under these conditions the 98 % yield of neodymium was achieved [9].

According to Lee et. al. after mechanical pretreatment (crushing and milling) Nd-Fe-B magnets were leached in the solutions of $\rm H_2SO_4$ and HCl. In both media at concentration of acids solution 3M ($\rm H_2SO_4$ a HCl), leaching temperature 27 °C, ratio L:S = 0.02 and leaching time 120 minutes, 99.5 % yields of neodymium were achieved in both of media [10].

Itakura et. al. treated Nd-Fe-B scrap in the mixed solutions of acids 3M HCl and 0.2 M (COOH)₂ at temperature of 110 °C. After leaching and simultaneous precipitation during 6 hours neodymium in the form of precipitate $Nd_2(C_2O_4)_3 \cdot xH_2O$ with purity of 99.8 % was obtained [11].

Vossenkaul et. al., used in leaching experiments sulfuric acid and hydrochloric acid solutions with concentrations 1, 2 and 3 M during 3 hours. The L:S ratio was 10:1. The leaching results showed, that the highest recovery of REE (Dy, Nd, Pr) in the 2M sulfuric and in the 3 M hydrochloric acid were achieved [12].

Hoogerstraete et. al. leached pre-treated sample of Nd-Fe-B magnets by roasting (at the temperature of 950 °C during 6 hours). Leaching was realized in a mixture of 12 M HCl and 3.5 M NH₄Cl. This mixture was intensively mixed for 3 days at temperature

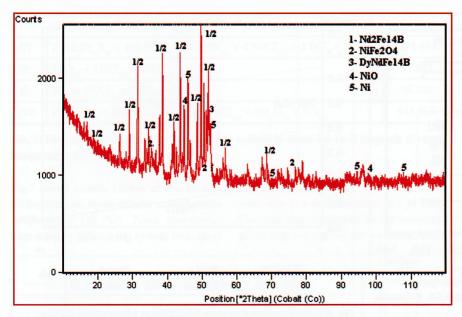


Fig. 2: XRD pattern of representative sample of Nd-Fe-B magnets

100 °C in a closed vessel. By this process 90 % yield of neodymium and 10 % yield of dysprosium and iron were achieved [13]. For the recovering of REE from Nd-Fe-B magnets Önal et. al. used a procedure, which included selective roasting and leaching in demineralized water. Based

acidic solutions (H_2SO_4 and HCl) high yields of neodymium into the solution at low temperatures were achieved. However, in these works kinetics and thermodynamic aspects of leaching neodymium and others elements (Fe and Ni) into the solution were not studied.

dr all	Reaction	ΔG° [kJ]
(1)	$2Ni(s) + O_{2(g) \text{ from air}} = 2NiO(s)$	-365.364
(2)	$Ni(s) + 2Fe_{\text{(from Nd2Fe14B)}} + 2O2_{\text{(g) from air}} = NiFe_2O_4(s)$	-840.520

Tab. 2: Thermodynamic data for reactions of NiO and NiFe $_2{\rm O}_4$ formation at temperature of demagnetization (340 °C)

No. 21 E	Reaction	ΔG° [kJ]
(3)	$Nd_{2}Fe_{14}B_{(s)} + 17H_{2}SO_{4(1)} = Nd_{2}(SO_{4})_{3(s)} + 14FeSO_{4(s)} + 17H_{2(g)} + B_{(s)}$	-16 984. 256
(4)	$NiO_{(s)} + H_2SO_{4(l)} = NiSO_{4(l)} + H_2O_{(l)}$	-88.936
(5)	$Ni_{(s)} + H_2SO_{4(l)} = NiSO_{4(l)} + H_{2(g)}$	-72.189
(6)	$NiFe_2O_{4(s)} + H_2SO_{4(l)} = NiSO4_{(s)} + Fe_2O_{3(s)} + H_2O_{(l)}$	-77.843

Tab. 3: Thermodynamic study for leaching system of spent Nd-Fe-B magnets at room temperature 20 $^{\circ}\text{C}$ in sulfuric acid

on achieved results the optimum conditions and parameters were selected: concentration of $\rm H_2SO_4$ 14.5 M (25°C), drying (110 °C/ 24 h), selective roasting (1h, 750°C), leaching in the demineralized water (ratio magnet to demineralized water 0.02 g·ml⁻¹, 1h, 25°C). Under these conditions, the extraction efficiencies for Nd, Dy, Pr, Gd, Tb and Eu 95-100 % were achieved [14].

From experimental studies it is clear, that mechanical pretreatment of the spent Nd-Fe-B magnets followed by leaching in the

The aim of this work is a thermodynamic and kinetic study of leaching demagnetized samples of spent Nd-Fe-B magnets in selected acidic media.

Material and methods

For hydrometallurgical treatment sintered Nd-Fe-B magnets from spent hard disc drives were used. After their thermal demagnetization in the chamber furnace (Curie temperature 340 °C for 30 minutes) they were crushed and milled in a vibra-

Element	Nd	Fe	Ni
[%]	38.23	56.39	2.99

Tab.1: The results of chemical analysis of demagnetized and mechanically treatment sample of spent Nd-Fe-B alloy

tion mill (for 2 minutes). A representative sample was obtained by quartering. This sample was then analysed by AAS (atomic absorption spectrometry Varian AA240+) for concentration estimation of Nd, Fe and Ni. Phase analysis of sample was realized by XRD diffraction phase analysis with powder diffractometer Panalytical X-Pert Pro. The results of phase analysis were evaluated by HighScore plus software.

Leaching experiment were carried out by use of 4g of sample Nd-Fe-B magnets and 400 ml of leaching medium (L:S= 100). For experimental leaching study solutions of $\rm H_2SO_4$ with concentration of 0.5 M and HCl with concentration 0.1M were selected.

Experiments were realized in a standard leaching apparature (Fig.1) at conditions: room temperature ($20\,^{\circ}\text{C}$), $250\,\text{rpm}$, leaching time 120 min. The aim of the experimental study was the leaching behaviour of Nd, Fe and Ni after thermal demagnetization and mechanical treatment of Nd-Fe-B magnets in selected acid media.

Thermodynamic study was realized by use of software HSC Chemistry 6.1 The thermodynamic data for leaching reactions and E-pH diagrams for the systems Nd-S-H₂O, Fe-S-H₂O, Ni-S-H₂O, Nd-Cl-H₂O, Fe-Cl-H₂O, Ni-Cl-H₂O have been calculated and studied. For E-pH diagrams construction the concentration values given by content of elements in the experimental samples were chosen.

Result and discussion

Sample characterization

In Tab. 1 the results of chemical analysis of representative demagnetized sample of sintered Nd-Fe-B magnet are shown. From this analysis is clear, that the sample of Nd-Fe-B magnets contain 38.23 % Fe, 56.39 % Fe and 2.99 % Ni. Nd-Fe-B magnets except of given elements can contain also other elements depending on their application, for example Dy, Pr, Tb, B, Cu, Co, Si and Al. Fig.2 shows the diffraction pattern of representative sample of Nd-Fe-B magnets. In terms of phase composition the main compound Nd₂Fe₁₄B was identified. The

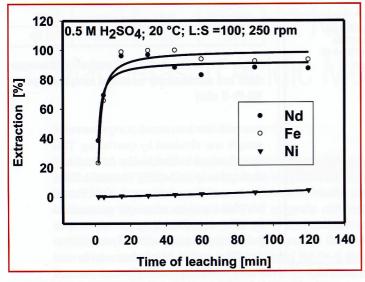


Fig. 3: Kinetic curves of leaching Nd, Fe and Ni in 0.5 M $\rm H_2SO_4$ at temperature 20 °C

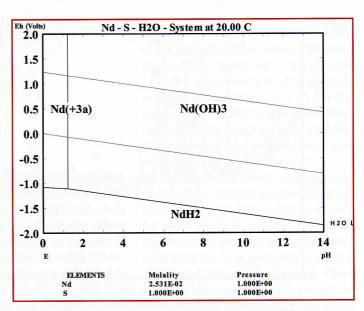


Fig. 4: E-pH diagram of system Nd-S-H2O at temperature 20 °C

other phase present was elemental nickel, because all sintered Nd-Fe-B magnets contain a protective layer of nickel which acts against oxidation. NiO and NiFe₂O₄ were created probably due to the oxidation and reactions in the solid state at the process of thermal demagnetization (reaction 1, 2 in

ture 20 °C are shown. From thermodynamic study of leaching reactions (2,3,4 and 5 in Tab. 3), it is clear that all these reactions are probable and spontaneously running in the direction of products formation. From results it is clear, that neodymium is leached in the $0.5~\mathrm{M}~\mathrm{H}_2\mathrm{SO}_4$ at room tem-

Time [min]	2	5	15	30	45	60	90	120
рН	0.17	0.21	0.23	0.33	0.36	0.40	0.45	0.46

Tab.4 pH values depending on time during leaching in 0.5 M H,SO, at temperature 20 °C

Tab. 2). In addition, the DyNdFe₁₄B phase was identified too.

Leaching in sulfuric acid

In Fig. 3 kinetic curves of leaching Nd, Fe and Ni in 0.5 M sulfuric acid at tempera-

perature (20°C). The process is fast and the highest extraction of neodymium (95 %) after 15 minutes of leaching was achieved. It can be seen from E-pH diagram of the system Nd-S-H₂O at temperature 20 °C (Fig. 4), neodymium in the region of water stability is in the form of Nd³⁺

to pH around 1.3. It corresponds to pH at leaching in 0.5 M H₂SO₄ which was in the range of values from 0.17 to 0.46. In the case of iron (Fig.3) the extraction reached 100 % after 15 minutes of leaching. It can be seen from the E-pH diagram of the system Fe-S-H2O at temperature 20 °C (Fig. 5), iron was in the form of Fe³⁺ as well as Fe2+ within the region of water stability at selected conditions (in the pH range from 0.17 to 0.46). The presence of individual iron species depends also on the electrochemical potential. In the case of nickel (Fig. 3) efficiency only reached only 1.2 % after 120 minutes of leaching. In the E-pH diagram of the Ni-S-H₂O system at the temperature 20°C nickel is in the range of pH values at leaching (from 0.17 to 0.46) in the form of Ni2+.

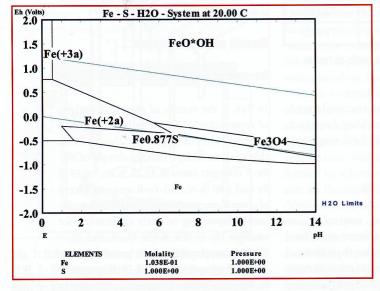


Fig. 5: E-pH diagram of system Fe-S-H_sO at temperature 20 °C

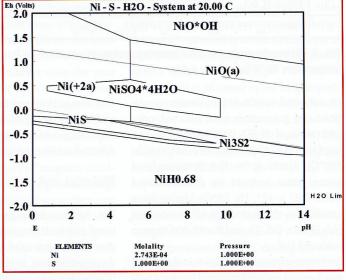


Fig. 6: E-pH diagram of system Ni-S-H₂O at temperature 20 °C

	Reaction	ΔG° [kJ]
(7)	$2 \text{ Nd}_2 \text{Fe}_{_{14}} \text{B}_{_{(8)}} + 102 \text{ HCl}_{_{(I)}} = 28 \text{ FeCl}_{_{3(I)}} + 4 \text{ NdCl}_{_{3(I)}} + 2 \text{ BCl}_{_3} + 51 \text{ H}_{_{2(g)}}$	-873.454
(8)	$NiO_{(s)} + 2HCl_{(1)} = NiCl_{2(l)} + H_2O_{(l)}$	-79.632
(9)	$Ni_{(s)} + 2HCl_{(l)} = NiCl_{2(l)} + H_{2(g)}$	-53.730
(10)	$NiFe_2O_{4(s)} + 8HCl_{(l)} = NiCl_{2(s)} + 2FeCl_{2(s)} + 4H_2O_{(l)} + 3Cl_{(g)}$	343.465

Tab. 5: Thermodynamic study for the leaching system of spent Nd-Fe-B magnets at room temperature 20 °C in hydrochloric acid

Fig.7 shows kinetic curves of Nd, Fe and Ni leaching in hydrochloric acid at room temperature (20 °C). From thermodynamic study of leaching system (reactions The reason of this is the weak acid solution with very low concentration (0.1 M HCl). This was also confirmed by the measured pH of solution during the leaching, which

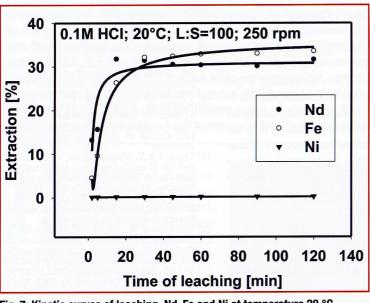
2.0

Nd(+3a)

range of pH values at leaching (from 1.08 to 6.75) in the form of Ni⁽²⁺⁾. The extraction of nickel was very low and achieved only 0.2 % after 120 minutes.

From the results of experiments is clear, that neodymium is leached relatively fast in the acidic media (H_2SO_4 and HCl) at high liquid to solid ratio (L:S = 100) with the extraction of 95 % in the solution of 0.5 H₂SO₄ and with extraction about 30 % in 0.1M HCl. In the case of iron leaching, extraction of 100 % in the 0.5 M H₂SO₄ was achieved. In the case of 0.1 M HCl the extraction of iron was quite low (only 32 %).

Nd - Cl - H2O - System at 20.00 C



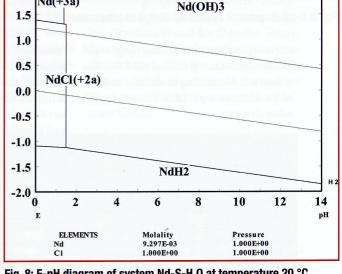


Fig. 7: Kinetic curves of leaching Nd, Fe and Ni at temperature 20 °C

Fig. 8: E-pH diagram of system Nd-S-H₂O at temperature 20 °C

7,8,9; Tab. 5), it is clear that given reactions (except the reaction 10), are probably and spontaneously running in the direction of products formation.

From results is clear, that neodymium is only partially leached in the 0.1 M HCl at room temperature (20°C). The highest extraction of neodymium (31.70 %) after 30 minutes of leaching was achieved. It can be seen from the E-pH diagram of Nd-Cl-H₂O at temperature 20 °C (Fig. 8) that neodymium in the region of water stability is in the form of specie NdCl(2+) to pH around 1.9. It corresponds to pH at leaching in 0.1M HCl which was in the range of pH values from 1.08 to 1.59 after 15 minutes of leaching. After 30 minutes of leaching, the pH increased to values higher than 5. That means that hydrochloric acid in the solution was slowly consumed.

had values from 5.32 (after 30 minutes) to 6.75 after 120 minutes of leaching (Tab. 6). It can be seen from the E-pH diagram of Nd-Cl-H₂O at temperature 20 °C (Fig. 8), neodymium in the region of water stability at selected conditions and pH higher than 1.9 is in the form of solid compound Nd(OH),.

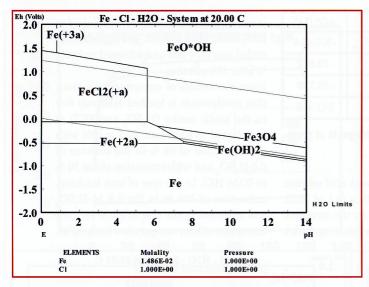
In the case of iron (Fig. 7) at selected conditions the extraction was approximately the same after 30 minutes of leaching (32 %). It can be seen from the E-pH diagram of Fe-Cl-H₂O at temperature 20 °C (Fig. 9), iron in the region of water stability at selected conditions and pH in the range of pH values from 0 to 5.9 is in the form of species FeCl(2+). It is obvious that only bivalent iron species are present in the solution. In the E-pH diagram of the Ni-Cl-H₂O system at temperature 20 °C is nickel in the

Time [min]	2	5	15	30	45	60	90	120
pН	1.08	1.07	1.59	5.32	6.42	6.47	6.52	6.75

Tab.6: pH values depending on time during leaching in 0.1M HCl at temperature 20 °C

The extraction of nickel in both of media was very low (up to around 0.2 %).

For the next experimental study is necessary to study metals extraction kinetics, especially neodymium at various leaching conditions (different concentration of leaching medium, leaching temperature, L:S ratio, leaching time, stirring speed). After finding the optimal leaching conditions it is necessary to search the suitable way for neodymium recovery from achieved leachate (precipitation, solvent extraction etc.). From this point of view, the main problem is presence of iron in the leaching solution. Iron coincidentally precipitates together with neodymium. This fact causes problems in recovery of neodymium from solution by decreasing the quality of final product. From thermodynamic study followed that in chloride media neodymium could be removed by precipitation more selectively towards iron. At pH around 2 iron should stay in solution in the form of FeCl⁽²⁺⁾. In the sulphate media seems to be more complicated due to the fact that iron





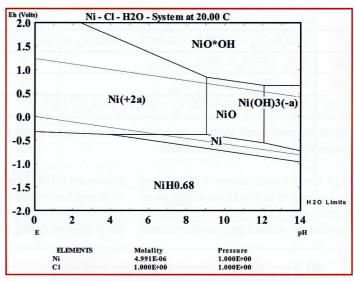


Fig. 10: E-pH diagram of system Ni-Cl-H₂O at temperature 20 °C

starts to precipitate in the same range of pH like neodymium, e.g. around pH 1. For this reason it is demanding to search a method of its selective separation from iron and other elements.

Conclusions

Nd-Fe-B magnets are in some types of equipment an irreplaceable commodity. Neodymium is a very important component of it and its primary resources are limited. For this reason the question of recycling is very important. An effective recycling of Nd-Fe-B magnets in a practical scale is still missing and recycling processes are studied only on a laboratory scale. The most viable way of recycling of spent Nd-Fe-B magnets is a hydrometallurgical treatment, which serves from an environmental and economical point of view as the best option, including the waste quantity aspect. The thermodynamic study showed some interesting aspects and differences concerning to leachability and precipitation of Nd, Fe and Ni species in sulphate and chloride media. In the experimental part of this work the kinetic study of the leaching process of used Nd-Fe-B magnets obtained from HDD of personal computers in the acidic solutions (H2SO4 and HCl) was realized. The results indicated high extractions of neodymium and iron in diluted sulphuric acid (0.5 M) and a fast leaching process. In opposite, a low concentration of HCl (0.1 M) is not suitable for a total extraction of neodymium and iron. As the iron is leached and precipitates along with neodymium in acid solutions, the pH is an important factor influencing iron precipitation during leaching and

finally the quality of a Nd product. E-pH diagrams confirm this assumption. It is recommended to verify in more details the influence of individual leaching parameters on neodymium and iron extraction as well as possibilities for selective leaching or their selective recovery from the solution in a following next study.

Acknowledgements

This work was supported by Ministry of Education of the Slovak Republic under grant MŠ SR 1/0293/14. Article is the result of the Project implementation: University Science Park TECHNICOM for Innovation Applications Supported by Knowledge Technology, ITMS: 26220220182, supported by the Research & Development Operational Programme funded by the ERDF. This work was supported by the Slovak Research and Development Agency under the contract No. APVV-14-0591. This contribution is the result of the project implementation Research excellence centre on earth sources, extraction and treatment, which was supported by the Research & Development Operational Programme funded by the ERDF".(ITMS: 26220120017)

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