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INNOVATIONS IN WEEE RECYCLING

Evaluation of Neodymium and Praseodymium Leaching Efficiency from Post-consumer NdFeB Magnets

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Abstract

Rare earth elements (REE) are among the Critical Raw Materials classified by the European Commission due to unbalanced supply and demand. Neodymium and praseodymium belong to the REE group that plays a significant role in NdFeB magnets, which are applied in green technology devices such as wind turbines and hybrid cars, besides hard disk drives. Recent data estimate that demand for these magnets will increase in the next years, causing insufficient metals availability. Recycling is an alternative to solve the problem, however there are many difficulties, mainly economics, since there is no profitable process developed yet. This study was mainly focused on the evaluation of neodymium and praseodymium leaching efficiency from post-consumer NdFeB magnets. Three factors (solid/liquid ratio, temperature, and time) were studied to evaluate the process. Statistical design of experiments and analysis of variance were performed in order to determine the main effects and interactions among the investigated factors for the leaching efficiency of neodymium and praseodymium in sulfuric acid 2 mol/L. The results showed that the highest leaching efficiencies for neodymium and praseodymium were 90.3 and 89.6%, respectively, and they were obtained at 1/20, 70 °C and 15 min. The ANOVA showed no significant factors for praseodymium. On the contrary, temperature and time were the significant main factors with positive effect, and the interaction between the three factors had a significant negative effect for neodymium.

Keywords Recycling · Leaching · Hydrometallurgy · Rare earth metals · Magnets

Introduction

It is estimated that between 2010 and 2015 the highest growth rate of rare earth elements (REE) application was in neodymium–iron–boron (NdFeB) magnets, reaching 10–15% per year [1]. In general, about 76% of the world's neodymium production in 2010 was for magnets, of which 31% had hard disk drives (HDDs) as a destination target [2]. In 2007, the amounts of NdFeB magnets in-stock used in computers, wind turbines, hybrid cars, MRI machines, and household appliances were estimated to be 97,000 tons, representing four times the amount of REE extracted in the same year [3].

Besides neodymium (Nd), NdFeB magnets of HDDs contain other REE, such as praseodymium (Pr), dysprosium (Dy), and terbium (Tb). What make these magnets so special is their magnetic energy density of over 450 kJ/m^3, the highest ever achieved. Therefore, there is an increasing demand in specific applications like motors, generators, and others related to clean technologies, such as wind turbines and hybrid cars [4]. In the HDDs, they act as the driving force on data reading/writing.

The magnets of HDDs have on average 25.3 wt% of neodymium, 3.83 wt% of praseodymium, and 2.66 wt% of dysprosium [2]. The worldwide production of NdFeB magnets in 2010 was 45,000 tons [5] and is expected to increase to 120,000 tons by 2020 [6], largely due to green technologies.

Consequently, the European Commission (2008) lists neodymium, praseodymium, dysprosium, and terbium as Critical Raw Materials (CRMs) due to China's near-monopoly (86% market share globally in 2014) over rare

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earth metals' production and difficulties in scaling-up production. The exploration of rare earths metals has often been issued for environmental impacts related to the mining and purification processes, since these processes generate huge amounts of wastewater and involve the handling of radioactive elements [7]. At present, no mature and economically feasible technologies have been identified for recycling NdFeB permanent magnets and other REE. Most of the processing methods are still at the research and development stage [8].

Meanwhile, the lifetime of a HDD is around 5 years [9], and after this period, alongside with most household electrical and electronic devices, they are shredded. Their magnetic content, mainly containing iron and neodymium, tends to end up in the ferrous metal waste stream, which is too diluted for economically feasible REE recovery [8]. Even before shredding, the difficult collection and the lack of governmental incentives are important factors that hold back the recycling process.

There are two methods for recovering rare earth metals from magnets: hydrometallurgical and pyrometallurgical. The objective of the pyrometallurgical process is the extraction of neodymium from the magnet to the slag, since rare earth metals have a greater affinity to oxygen [10]. In hydrometallurgy, the alloy is dissolved in acidic solutions and then separated by extractions or precipitations [5]. Although it generates effluents, the hydrometallurgical process is the most used, because it is operationally easier than the pyrometallurgical process and avoids atmospheric emissions [6].

Leaching is the first step to dissolve REE from magnets. The majority of publications have used mineral strong acids, such as hydrochloric, nitric or sulfuric acid [6, 10–13]. The reactions between sulfuric acid and metals result in metal sulfates. Some metals react immediately with the acid by replacing hydronium ion. However, metals such as copper do not interact with the sulfuric acid due to its standard redox (oxidation–reduction) potential, which is nobler than hydrogen, meaning that it has low tendency of donating electrons [14]. The opposite happens with neodymium and praseodymium, since their standard redox potential is much more active than hydrogen. The hypothesized leaching reaction with sulfuric acid is shown in Eq. (1)

$$2\mathrm{REE}_{(s)} + 3\mathrm{H}_2\mathrm{SO}_4 \rightarrow \mathrm{REE}_2(\mathrm{SO}_4)_3 + 3\mathrm{H}_2, \qquad (1)$$

where REE are Dy, Nd, Pr, and Tb.

A Hydra–Medusa diagram, according to Fig. 1, shows the species that may occur and be generated when sulfuric acid and both REE interact. Hydra and Medusa are free and complementary software products. *Hydra* allows choosing the relevant chemical elements and their compounds for the study and *Medusa* allows developing chemical equilibrium diagrams from the Hydra information [15]. Between pH zero and four, the diagram shows the existence of cations and anions in solution, which is the purpose of the leaching step. The subsequent steps on recovery of REE can be performed in different ways: separation by selective precipitation with oxalic acid or sodium and ammonium salts, solvent extraction and ionic liquids.

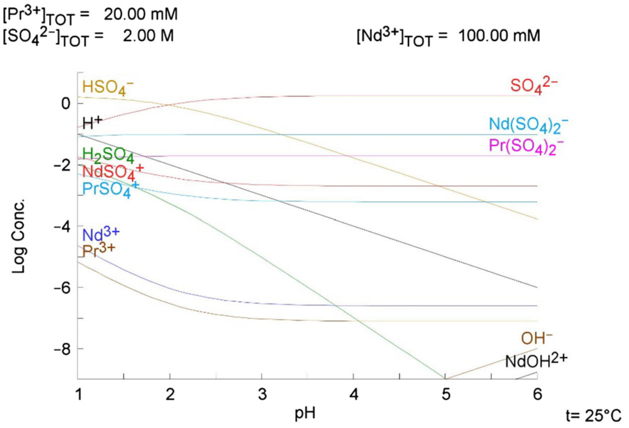

Fig. 1 Hydra–Medusa diagram of sulfuric acid and REE. (Color figure online)

Lee et al. [6], Abrahami et al. [10], and Voßenkaul et al. [16] have already developed methods and processes with sulfuric acid leaching of post-consumer magnets. According to conclusions from these publications, sulfuric acid (H_2SO_4) is among the best leaching agents for rare earth extraction from electronic scraps in general. Lee et al. [6] found good results (100%) for neodymium leaching efficiency with short times (15 min), low temperature (27 °C), low solid/liquid ratio (1/50) and acid concentration of 1.5 and 3 mol/L. Meanwhile Abrahami et al. [10] also found good results (> 95%) for neodymium leaching efficiency with acid concentration of 2 mol/L, high solid/liquid ratio (1/20), low temperature (room temperature) but long periods of time (8 h). Lyman and Palmer [13] consolidated their work regarding NdFeB magnet recycling in 1993, but the content of sulfuric acid varied between 1 and 3 mol/L, and solid/liquid ratio used was 1/10. On top of that, their effort focused on manufacturing swarf, not end-of-life magnets from different computer sources, which may be a heterogeneous sample. More than a decade later, this process is not yet diffused worldwide in terms of scaling up probably due to elevated costs. Some magnets leftovers of manufacturing are being recycled by the magnet manufacturing companies, but few details about the actual recycling processes have been disclosed [9]. Since acid leaching was established as an easy way of dissolving NdFeB magnets, many efforts have been made to reduce costs and establish an environmental friendly process to recovery rare earth metals from these end-of-life goods.

Therefore, based on the results previously reported and the fact that there is not a consolidated method regarding optimal leaching factors yet, this study focused on evaluating the main factors and possible interactions that affect the neodymium and praseodymium leaching efficiency for a scaling up unit operation from post-consumer NdFeB magnets. The best conditions would be short leaching time, low acid consumption, high solid feed rate, and room temperature, demanding less energy as well. For this purpose, in this work a leaching process was tested in accordance to a full factorial plan in which the investigated factors were solid/liquid ratio, temperature, and time.

Experimental Procedure

Materials

A sample set containing 36 post-consumer HDDs, 26 from desktops and 10 from notebooks, was manually dismantled. Total HDDs weight summed up to 13,700 g and total magnet weight summed up to 254.87 g, but all experiments demanded only 48.21 g. The magnets were separated from the HDDs and then were demagnetized according to the methodology adopted in previous work [17]. Subsequently, NdFeB magnets of desktops and notebooks were blended and ground in cutting mill (Retsch SM 300) in order to achieve a granulometry below 0.25 mm. A random sample of the ground magnet powders was digested by a microwave Multiwave (Anton Paar) according to protocol 3051A from the US Environmental Protection Agency (EPA). The content of the leachates were obtained by Inductively Coupled Plasma Optical Emission Spectrometry – ICP OES (Agilent Technologies 5110).

Leaching Tests

An experimental 2^k factorial design [18–20] of first order was used to assess the leaching efficiency of neodymium and praseodymium. Three factors ($k = 3$) each one with two levels, low and high, according to Table 1 were used to obtain the leaching efficiency as response. The significance of the effects of the factors was assessed by analysis of variance (ANOVA) using the Fisher's F-test.

Table 1 Factors and respective levels in the leaching factorial design experiments

Factors	Levels	
	–	+
A: Solid/liquid ratio (g/mL)	1/40	1/20
B: Temperature (°C)	25	70
C: Time (min)	15	30

This design allows the determination of the factor effects on the response and the effects of interactions between different factors. It takes into account all the combinations between the levels of factors during the experiment. Because there are only two levels of each factor, it is assumed that the response is approximately linear over the range of the factor levels chosen [18]. The experiment was conducted with two repetitions per set and the execution was randomized. The leaching tests were performed in a 250 mL glass beaker reactor with mechanical stirring at 300 rpm. The sulfuric acid concentration was 2 mol/L due to the high leaching efficiencies obtained by the authors previously referenced here. In specific tests, a heating plate was also employed to elevate and maintain the temperature. The amount of magnet sample employed changed according to the solid/liquid ratio: 2 and 4 g, whereas the amount of acid was constant at 80 mL.

The efficiency was calculated according to Eq. (2).

$$E(\%) = \frac{w_{l,i}}{w_{i,i}} \times 100, \tag{2}$$

where E is the efficiency (%), $w_{l,i}$ is the amount (mg/kg) after the leaching process for rare earth i, and $w_{i,i}$ is the digested in microwave sample amount (mg/kg) for rare earth i.

Results and Discussion

NdFeB Ground Sample

An original NdFeB magnet and the heterogeneous ground sample are shown in Fig. 2. The particle size was inferior to 0.25 mm (60 mesh). According to particle size theories, the smaller the size, the greater is the interfacial area between the solid and the liquid, and therefore the higher is the rate of transfer of material [21], which is directly proportional to the hydrometallurgical kinetics of the process. Although the particle size is not relevant in most studies, Lee et al. [6] used the same particle size and obtained high leaching efficiencies. Likewise Lyman and Palmer [13] established their invention considering particles below 20 mesh. Therefore, it was adopted on this study as well.

The chemical composition of this NdFeB ground sample is showed in Table 2. The anticorrosion coating layer composed of nickel, aluminum and/or copper was not removed previously, hence these metals are present in the result, though in small amounts. REE sum up to 29 wt% on average, closely to the values presented by Stuhlpfarrer

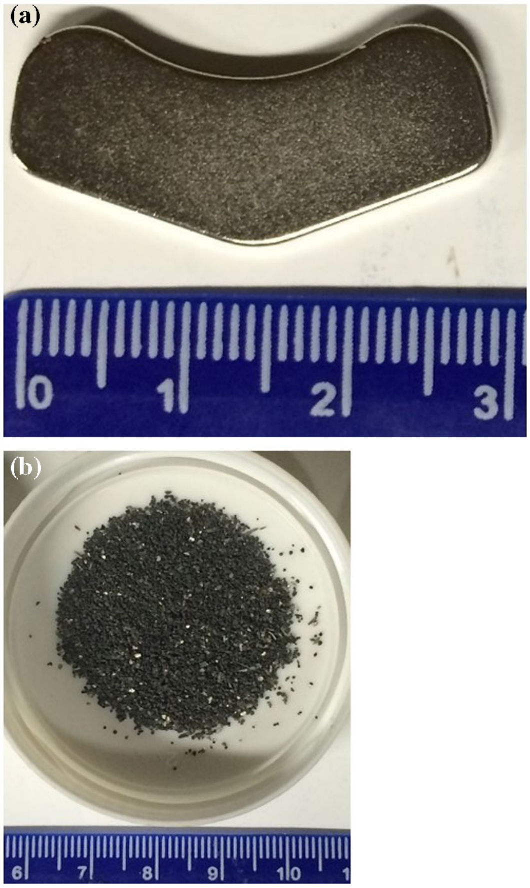

Fig. 2 NdFeB magnet (**a**) and ground sample (**b**). (Color figure online)

Table 2 NdFeB chemical composition

	Average (wt%)
Al	0.47 ± 0.17
B	0.90 ± 0.04
Co	0.87 ± 0.03
Cu	0.26 ± 0.04
Dy	0.98 ± 0.07
Fe	65.31 ± 0.17
Nd	23.95 ± 0.71
Ni	2.86 ± 1.03
Pr	4.20 ± 0.19
Tb	0.19 ± 0.05

et al. [2]. Neodymium and praseodymium are the most abundant REE in magnets, thus are the main focus on this study.

Leaching Efficiency

The results of the experimental factorial design for eight different leaching conditions are shown in Fig. 3. The response of the process was the neodymium and praseodymium leaching efficiency.

The maximum leaching efficiencies for neodymium and for praseodymium were 90.3% and 89.6%, respectively, and it happened on test number 3: solid/liquid ratio of 1/20, temperature of 70 °C and 15 min.

Good conditions for the leaching efficiency were also those of tests number 5, 7, and 8 for both neodymium and praseodymium. These tests have in common 30 min of reaction. Within this time, neodymium leaching efficiency was higher for solid/liquid ratio of 1/20 (tests 5 and 7), but as temperature increases from 25 °C (test 5) to 70 °C (test 7), the leaching efficiency seemed to decrease slightly. For praseodymium, within the 30 min of reaction, the best leaching efficiency was achieved in solid/liquid ratio of 1/40 and 70 °C (test 8), although solid/liquid ratio of 1/20 also showed high leaching efficiencies (tests 5 and 7). Tests 3 and 5 showed the highest standard deviations, around 10%, which might be related to minimum causes, e.g., dilution, samples handling, or bad balance analytic calibration.

Meanwhile, the minimum leaching efficiencies for both neodymium and praseodymium happened on tests 1 and 4, which have in common only the time of 15 min. Test 1 suggests that the arrangement solid/liquid ratio 1/20, 25 °C and 15 min is not ideal. Moreover, room temperature seems not adequate for leaching both REE. In contrast, when temperature is 70 °C, leaching efficiency is better (tests 4). So in order to maintain high solid/liquid ratio and low time, energy must be provided to elevate the temperature of the system, which ensures better leaching efficiencies.

When evaluating temperature, the results show slightly higher average leaching efficiencies on the four tests at 70 °C, around 8% for neodymium and 6% for praseodymium, according to Fig. 3. However, tests at 25 °C also showed high leaching efficiencies, and at this temperature, there is energy savings, which is important to scale up the projects. However, the temperature dependence of the sulfate solubility in the system $Ln_2(SO_4)_3 \cdot 8H_2O - H_2SO_4 - H_2O$ (Ln = Nd or Pr) shows that the acidity and the temperature have opposite effects. Accordingly, in solutions containing over 2 mol/L, the solubilities of $Pr_2(SO_4)_3 \cdot 8H_2O$ (the most soluble) at 25 and 64 °C are very close. The solubility of the less-soluble $Nd_2(SO_4)_3 \cdot 8H_2O$ at the same H_2SO_4-concentration at 64 °C becomes even higher than at 25 °C. In the same way, at 64 °C, both the solubilities of Pr and Nd are close and at 25 °C the solubility of Pr is higher than Nd [22]. This behavior is observed in experiments 1 (1/20, 25 °C, 15 min), 2 (1/40, 25 °C, 15 min), 3 (1/20, 70 °C, 15 min), 5 (1/20, 25 °C, 30 min), 6 (1/40, 25 °C, 30 min), and 7 (1/

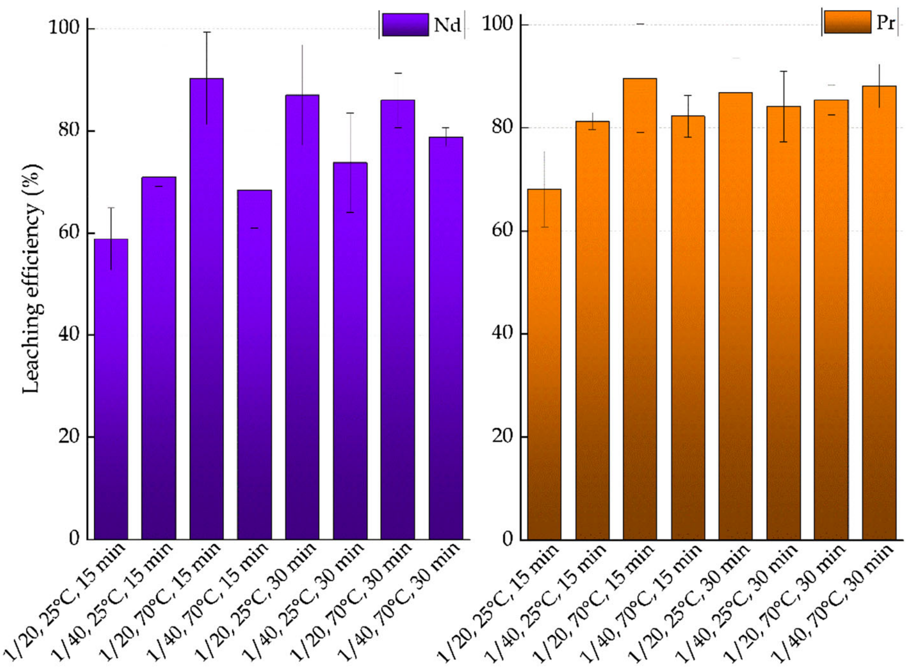

Fig. 3 Effect of solid/liquid ratio, temperature, and time on leaching efficiency of Nd and Pr. (Color figure online)

20, 70 °C, 30 min). The exceptions are experiments 4 and 8, both 1/40 and 70 °C, but 15 and 30 min respectively, which may reveal a not so good combination between these levels of factors. Room temperature is only adequate when time is longer and solid/liquid ratio is higher (1/20) for both neodymium and praseodymium. These are good conditions for scaling up, since they use less acid consumption and more solid feed rate, besides reaction time of 30 min, which is also reasonable.

In addition, Fig. 3 shows that higher time is better for leaching these REE, since three out of the four highest leaching efficiencies were achieved on 30 min for both neodymium and praseodymium, respectively. However, leaching efficiencies of 15 min were only 6 and 9% lower than leaching efficiency of 30 min for neodymium and praseodymium, respectively. Short time is important when considering the scaling up recycling, hence short reaction times allow more cycles of leaching in a batch process.

Lee et al. [6] found good results for neodymium in a much lower solid/liquid ratio (1/50) and lower time (15 min) at room temperature, which may be related to the concentration of the sulfuric acid (1.5 mol/L), in contrast to the 2 mol/L used in this work.

Analysis of Variance (ANOVA)

The leaching efficiency was used as response for the analysis of the experimental factorial design data with the assistance of the statistical software *Minitab®* [23]. The significance of factors and interactions between these factors were determined by Fisher *F*-Test method at 95% confidence level, which establishes as the most relevant factor the one with highest *F* value, and at the same time with *p* value inferior to 0.05 to represent a significant response. ANOVA data with the factors and interactions that include both significant and not significant results, based on the *p* value, are reported in Table 3 for neodymium and praseodymium. In both REE ANOVA, the residues represents more than 20% of the total sum of squares, which is not ideal.

Neodymium

The linear behavior of the neodymium leaching efficiency against the factors was not satisfactory, reaching correlation coefficient $R^2 = 80.15\%$. From the analysis of experimental results, it is possible to observe that

- Time is the most significant main factor since F value is the highest. The effect is positive, which means that when this factor increased from 15 to 30 min, the leaching efficiency also increased.
- There was positive effect of the main factor temperature. It also means that insofar as this factor increased from 25 to 70 °C, the leaching efficiency increased as well.
- The interaction of third order (solid/liquid ratio–temperature–time) had a negative effect and the highest *F* value.
- The effect of solid/liquid ratio did not show significance since its *p* value was higher than 0.05, as well as the interactions of second order, hence they had not

Table 3 Results of the effects of the factors and analysis of variance (ANOVA)

Source	DF	Effect	Sum of squares	Mean squares	F statistics	p value
	Neodymium					
A: Solid/liquid ratio	1	7.53	227.02	227.02	4.53	0.066
B : Temperature	1	8.23	271.09	271.09	5.41	0.049
C: Time	1	9.26	343.11	343.11	6.84	0.031
AB	1	6.95	193.23	193.23	3.85	0.085
AC	1	2.68	28.64	28.64	0.57	0.471
BC	1	− 6.25	156.02	156.02	3.11	0.116
ABC	1	− 10.00	400.23	400.23	7.98	0.022
Residue	8		401.10	50.14		
Total	15		2020.43			
	Praseodymium					
A: Solid/liquid ratio	1	− 1.48	8.75	8.74	0.23	0.643
B : Temperature	1	6.23	155.35	155.35	4.12	0.077
C: Time	1	5.78	133.84	133.84	3.55	0.096
AB	1	3.79	57.36	57.36	1.52	0.252
AC	1	1.46	8.51	8.51	0.23	0.648
BC	1	− 4.97	98.68	98.68	2.62	0.144
ABC	1	− 6.50	168.80	168.80	4.48	0.067
Residue	8		301.59	37.70		
Total	15		932.87			

prevail in this analysis, meaning that these specific interactions do not respond linearly with the leaching efficiency.

Praseodymium

The linear behavior of the praseodymium leaching efficiency against the factors was even poorer than neodymium, reaching correlation coefficient $R^2 = 67.67\%$. From the analysis of experimental results, it is possible to observe that all the p values are higher than 0.05, which represents poor interaction among the factors, meaning that the confidence level of 95% was not achieved. Thus these factors and levels chosen are not significant in these arrangements. In addition, sulfuric acid 2 mol/L is suitable for praseodymium leaching in any conditions aside the one from test number 1: solid/liquid ratio of 1/20, 25 °C and 15 min, which had the lower leaching efficiency (68.1%), as Fig. 3 shows. The leaching efficiency for the other seven tests are quite similar, with an average of 85.4 ± 5.2%.

Conclusions

The research described in this study was focused on evaluating the leaching efficiency of neodymium and praseodymium from post-consumer NdFeB magnets with sulfuric acid. The 2 mol/L concentration of sulfuric acid was satisfying for most leaching tests since high leaching efficiencies were achieved for both REE in specific situations. In addition, the REE showed close leaching efficiencies when considering the same arrangement of factors due to the physical–chemical properties similarity that both elements share; hence similar leaching efficiencies are expected. However, some small differences rely on the solubility, which is higher for praseodymium. According to an experimental factorial design the effect of solid/liquid ratio, temperature, and time were studied. By evaluation of p-value significant effects were determined. Both temperature and time showed slightly positive significance, whereas only one factor interaction (solid/liquid ratio–temperature–time) showed a negative significance for neodymium. For praseodymium, no factors and interactions had any significance, probably because leaching efficiencies showed values very close to each other in the eight situations performed, showing lack of variance, and hence proving that the levels of factors (temperature, time, and solid/liquid ratio) chosen were in fact not relevant in this study. Given that, the solubility of praseodymium is not influenced by these levels of factors. There was only one situation (test number 1) where the leaching efficiency was poor. Multiple best conditions were identified whereas highest leaching efficiencies for neodymium and praseodymium were observed in different arrangements. Only one level of solid/liquid ratio (1/20) appeared in all highest leaching efficiencies, meanwhile for time and temperature both levels showed high leaching efficiency: 70 °C with 15 and 30 min, and 25 °C with 30 min. At 25 °C, leaching efficiency is promising, since there are minimum energy usage.

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