

# Selective formation of neodymium phosphate from iron-neodymium magnet waste

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## Abstract

Neodymium-iron-boron alloy, Nd<sub>2</sub>Fe<sub>14</sub>B, is used as a material for magnets. Neodymium is one of the valuable and expensive rare earth elements and is often recovered to reduce costs and environmental impact. Recently, a method for recovering neodymium as neodymium phosphate from mixed solutions was reported. This method was very useful because the main component of rare earth ores is rare earth phosphates, and there are well-established methods for refining rare earths from rare earth phosphates. This study attempts to obtain neodymium phosphate using acid to dissolve waste magnets, reducing agent, phosphoric acid, and pH adjuster. The diluted acid (hydrochloric acid and nitric acid), amount of ascorbic acid and phosphoric acid, and the pH values were studied to clear the suitable conditions. The color of the heat-treated product, the precipitate yield, and the Fe/Nd molar ratio indicated that a low concentration of acid is suitable and ascorbic acid is required. Excessive use of phosphoric acid improved the precipitate composition. The high pH value decreased the selectivity of neodymium phosphate due to the easy formation of iron phosphate and hydroxide. The process addressed in this study is very promising because it does not require heating at high temperatures or special reagents.

**Keywords:** rare earth recovery, phosphoric acid, precipitation.

## INTRODUCTION

Neodymium-iron-boron alloys (typical formula: Nd<sub>2</sub>Fe<sub>14</sub>B) are used as permanent magnet materials in hard disk drives, hybrid car motors, magnetic resonance imaging devices (MRI), and so on [1, 2]. In the production of these rare earth transition metal magnets, a significant amount of scrap is yielded through the cutting and milling processes. On the other hand, rare earth elements are known to co-occur with some other rare earth elements in the natural ores [3]. Due to the energy-consuming procedures required to separate the desired rare earth elements from other elements and to avoid resource depletion, there is a demand from industry to recycle certain rare earth elements from scrap, which saves time and energy [4].

Rare earth elements are being utilized in a variety of functional materials, but there are some problems with their stable supply to industry [5, 6]. Rare earth ores are found only in certain parts of the world and generally contain radioactive elements [7]. Recycling rare earth elements from scrap is very important to reduce the environmental impact since mining rare earth ores causes environmental problems such as destroying land and producing radioactive elements [8]. Several processes have been reported for the recovery of rare earth elements. For instance, transition metals were removed from the mixtures with rare earth elements by solvent extraction with undiluted ionic liquids [9, 10]. However, this process demands high concentrations of acid and special reagents. As an alternative, samarium

was recovered from wastes associated with samarium-cobalt magnets by a chemical vapor transport method [11, 12]. However, this method also has the disadvantage of requiring high temperatures and special equipment. Because of these disadvantages, it is hoped that new technologies will be developed to improve current recycling methods. The precipitation method is one of the most useful techniques to obtain the target metal separated from the matrix of other metals [13, 14]. Among them, precipitation separation using inorganic reagents is particularly convenient because it does not use harmful organic solvents and is relatively easy to treat wastewater. Furthermore, the precipitation process can be easily approximated by using laboratory-simulated solutions. In general, metal hydroxides are often used in precipitation separations with aqueous solutions because they can efficiently recover large quantities of metal cations [15, 16]. However, metal hydroxides are less selective in forming precipitates in the presence of precipitating agents.

Rare earth phosphates are the main component of rare earth ores and are known to be chemically stable compounds that can occur naturally [17]. Thus, while processes for supplying rare earth elements from rare earth phosphates to industry are well established, there are few reports on processes for recycling rare earth elements as phosphates. In our previous study, a new recycling procedure was reported to recover neodymium as phosphate from iron-neodymium aqueous solutions [18]. The proposed process is relatively simple and does not need the use of hazardous organic solvents. Therefore, in this study, we attempted to dissolve waste magnets in acid and recover neodymium from the solution as phosphate. The influence of concentrations of hydrochloric acid and nitric acid, the volumes of ascorbic acid and phosphoric acid, and pH adjustment were examined

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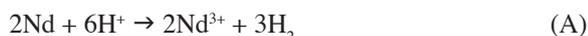
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to identify the appropriate condition. The composition of waste magnets varies depending on the manufacturer, however, in this work, we investigated the possibility of recovering neodymium phosphate using a reducing agent regardless of the composition of neodymium waste magnets.

## MATERIALS AND METHODS

**Materials:** the disposal magnets were provided by KRI Inc., Japan. All chemicals were commercially pure (Fujifilm Wako Pure Chem., Japan) and used without further purification. Specifically, the hydrochloric acid had a weight concentration of 36% and the nitric acid had a weight concentration of 60%. The weight concentration of phosphoric acid was 85%. Sodium hydroxide and ascorbic acid were over 97% and 99.6% pure, respectively.

**Procedures:** the magnetism was removed from the waste magnet by heating it at 400 °C for 1 h. The 2 g of the demagnetized alloy was solved in 40 mL of various acid solutions to achieve the following reactions:



And the approximate concentrations of each acid were 6.0, 2.4, and 1.2 mol/L hydrochloric acid and 7.0, 2.8, and 1.4 mol/L nitric acid, respectively. Some divalent iron ions were oxidized to trivalent in an aqueous solution. Then 2.28 g of ascorbic acid was added to these solutions to reduce iron cation from +III to +II condition in the following reaction [18]:



This weight was determined from the ratio of Asc/Fe=1/2 because ascorbic acid is a divalent reducing reagent. The 0.2 mol/L of phosphoric acid (20 mL) was mixed with these solutions. This was set at P/Nd=1 from the consideration that the waste magnet was Nd<sub>2</sub>Fe<sub>14</sub>B. The formations of neodymium phosphate and iron phosphate are represented by the following chemical reactions:



The pH values of the mixed solutions were adjusted to 2, 3, 4, and 5 using an 8 mol/L sodium hydroxide solution. The formed precipitates were then collected by filtration and dried at room temperature for over 3 days. A portion of samples was subjected to heating at 700 °C for 1 h for evaluation. To identify the most appropriate conditions for neodymium recovery, the volume of ascorbic acid was varied: 0, 1.14, and 4.56 g. Further, the volume of phosphoric acid was varied (10, 40, and 60 mL) to clarify the ratios for which this separation process is effective.

**Analytical methods:** the crystallographic compositions of the materials were analyzed by X-ray diffraction (XRD; MiniFlex, Rigaku, Japan) using monochromatic CuK $\alpha$  radiation (30 kV, 15 mA, 3 °/min). Infrared (IR) spectra were measured on a spectrometer (FT-IR 720, Horiba, Japan) using the KBr disk method (resolution: 4 cm<sup>-1</sup>, scan: 16 times). For the estimation of the molar ratios of neodymium and iron in the precipitates, a small portion of the sample was dissolved in a nitric acid solution. These ratios were calculated from the results by microwave plasma-atomic emission spectroscopy (4200 MP-AES, Agilent). The colors of the precipitates were estimated from the ultraviolet-visible (UV-vis) reflectance spectra (UV2100, Shimadzu, Japan; reference compound: BaSO<sub>4</sub>). The colors of the materials were also estimated with a color analyzer (TES-135 plus, TES Electrical Electr., Taiwan). The L\* value indicates the whiteness of the material, where the value of 100 corresponds to white and the value of 0 corresponds to black. The a\* value indicates the redness of the material, with positive (maximum: +128) and negative (-128) values corresponding to red and green, respectively [19]. The b\* value indicates the intensity of yellow, with positive (maximum: +128) and negative (-128) values corresponding to yellow and blue, respectively.

## RESULTS AND DISCUSSION

**Dilution ratio of HCl and HNO<sub>3</sub>:** first, the acid to be dissolved was examined. Commercial purity hydrochloric acid and nitric acid were used for convenience in the future because they are common acids. These are volatile and difficult to accurately determine, however, were considered to be approximately 12 and 14 mol/L, respectively. In this study, the lowest concentration of 10 times diluted hydrochloric acid (1.2 mol/L) corresponded to the theoretical amount for dissolving waste magnets as NdCl<sub>3</sub> and FeCl<sub>3</sub>, therefore they were diluted 2, 5, and 10 times. Table I shows the weight of the precipitate and the yield of neodymium phosphate when the composition of the waste magnet is Nd<sub>2</sub>Fe<sub>14</sub>B. Samples with low dilutions, i.e. high concentrations of acid, showed much higher yields than 100%. It was considered to be due to the coprecipitation of

Table I - Yield of NdPO<sub>4</sub> prepared with various concentrations of nitric acid and hydrochloric acid (ascorbic acid: 2.28 g, H<sub>3</sub>PO<sub>4</sub>: 20 mL, pH 3).

Acid	Dilution ratio/times	Concentration (mol.L <sup>-1</sup> )	Weight (g)	Yield (%)
HNO <sub>3</sub>	2	7.0	6.867	775.94
	5	2.8	1.253	141.56
	10	1.4	0.833	94.12
HCl	2	6.0	1.171	132.28
	5	2.4	1.208	136.54
	10	1.2	0.759	85.72

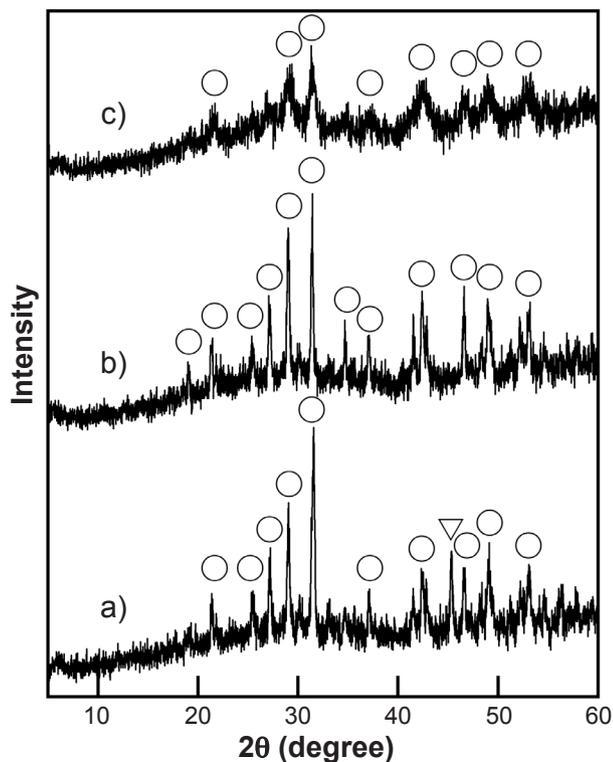


Figure 1: XRD patterns of samples prepared under various concentrations of HCl and then heated at 700 °C for 1 h: a) 6.0 mol/L; b) 2.4 mol/L; and c) 1.2 mol/L (ascorbic acid: 2.28 g, pH 3,  $\text{H}_3\text{PO}_4$ : 20 mL). ○:  $\text{NdPO}_4$ , ▽:  $\text{Fe}_2\text{O}_3$ .

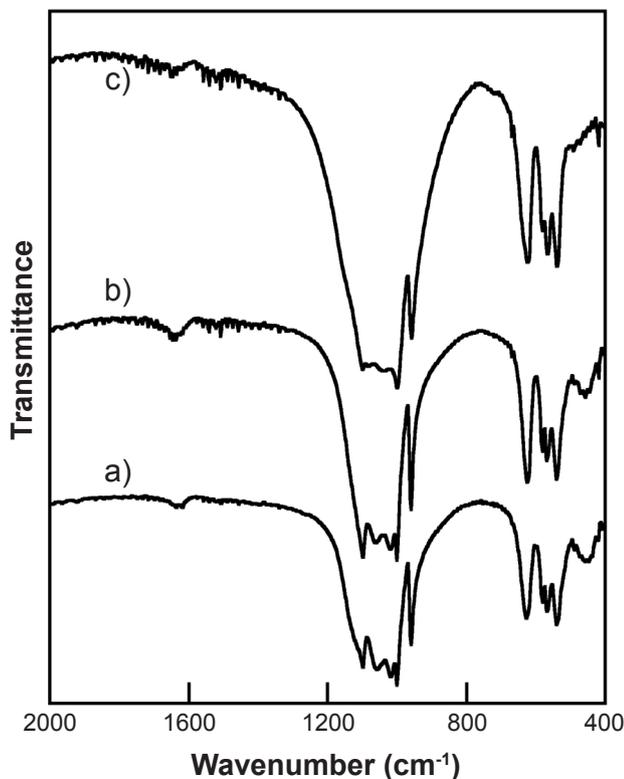


Figure 2: IR spectra of samples prepared under various concentrations of HCl and then heated at 700 °C for 1 h: a) 6.0 mol/L; b) 2.4 mol/L; and c) 1.2 mol/L (ascorbic acid: 2.28 g, pH 3,  $\text{H}_3\text{PO}_4$ : 20 mL).

iron compounds at high concentrations of acids. Therefore, it was suitable to recover neodymium phosphate by using the acids diluted to a low concentration.

The unheated sample did not show a clear peak in the XRD pattern. This was considered to be due to the fact that the material was a precipitate from the aqueous solution and thus contained water easily. The formation of amorphous precipitates was also considered. Therefore, the results of the heated sample were mainly discussed in order to clarify the properties of the sample. Fig. 1 shows XRD patterns of samples prepared with various concentrations of hydrochloric acid and then heated at 700 °C for 1 h. Samples prepared with 6.0 and 2.4 mol/L of hydrochloric acid indicated the peaks of neodymium phosphate,  $\text{NdPO}_4$ . On the other hand, the weak peaks were only observed in XRD patterns of the sample prepared with 1.2 mol/L of hydrochloric acid. Fig. 2 shows IR spectra of samples prepared with various concentrations of hydrochloric acid and then heated at 700 °C for 1 h. All samples had a similar spectrum with  $\text{NdPO}_4$  [20, 21]. The peaks due to the bending of O-P-O and the P-O stretching were observed in the region of 630-520 and 1100-960  $\text{cm}^{-1}$ , respectively [22]. From these results, the main composition of the obtained thermal products was neodymium phosphate.

The divalent iron compound had a pale color when precipitated, however by heating, the iron was oxidized to trivalent and showed a distinct color. This change facilitated the determination of the presence of iron from the color of the sample powder. Fig. 3 shows a photograph of samples prepared with various concentrations of hydrochloric acid and then heated at 700 °C for 1 h. The colors of neodymium phosphate, iron oxide ( $\text{Fe}_2\text{O}_3$ ), and iron phosphate ( $\text{FePO}_4$ )

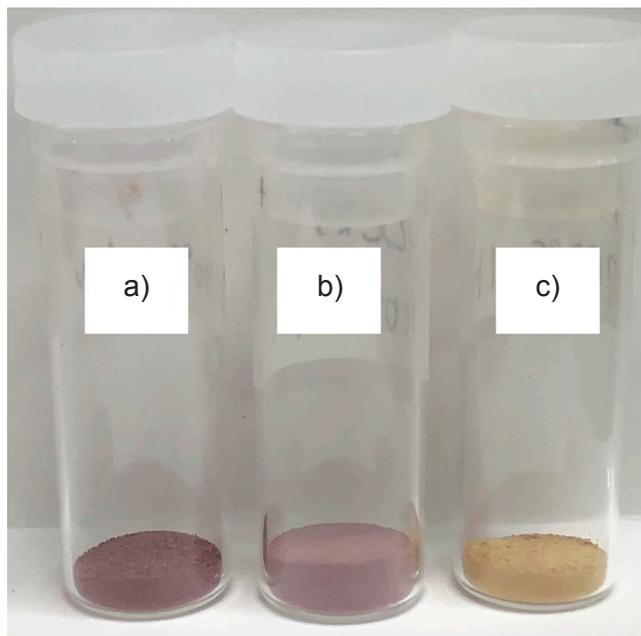


Figure 3: Photograph of samples prepared under various concentrations of HCl and then heated at 700 °C for 1 h: a) 6.0 mol/L; b) 2.4 mol/L; and c) 1.2 mol/L (ascorbic acid: 2.28 g, pH 3,  $\text{H}_3\text{PO}_4$ : 20 mL).

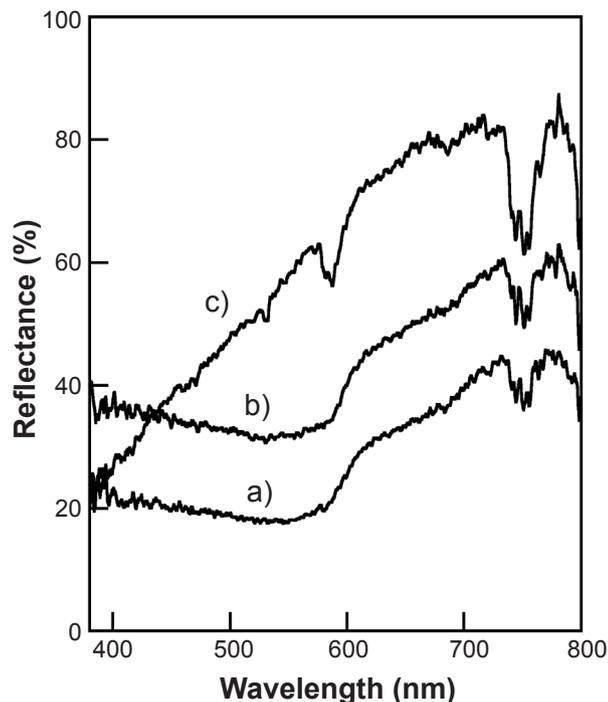


Figure 4: UV-vis reflectance spectra of samples prepared under various concentrations of HCl and then heated at 700 °C for 1 h: a) 6.0 mol/L; b) 2.4 mol/L; and c) 1.2 mol/L (ascorbic acid: 2.28 g, pH 3, H<sub>3</sub>PO<sub>4</sub>: 20 mL).

were light purple, red, and brown, respectively. From these powder colors, samples prepared with 6.0 and 2.4 mol/L of hydrochloric acid included a certain degree of iron oxide. On the other hand, iron phosphate was formed in the sample prepared with 1.2 mol/L of hydrochloric acid. Fig. 4 shows UV-vis reflectance spectra of samples prepared under various conditions. Samples prepared with 1.2 mol/L of hydrochloric acid had peaks at 535, 580, and 750 nm. These peaks were due to the  $^4I_{9/2} \rightarrow ^4G_{7/2}$  transition,  $^4I_{9/2} \rightarrow ^4G_{5/2}$ ,  $^2G_{7/2}$  transitions, and  $^4I_{9/2} \rightarrow ^4S_{3/2}$ ,  $^4F_{7/2}$  transitions of Nd<sup>3+</sup> ions, respectively [23]. This result indicated that the sample included a certain degree of neodymium compounds. Samples prepared with 6.0 and 2.4 mol/L of hydrochloric acid had lower reflectance than that prepared with 1.2 mol/L acid because these materials included a high ratio of iron compounds.

Table II shows L\*a\*b\* values of thermal products and

Fe/Nd molar ratio of precipitates prepared with various concentrations of nitric acid and hydrochloric acid. For reference, h\* (hue angle) values were also shown as calculated from a\* and b\* values [24]. Samples prepared with 1.4 mol/L of nitric acid and 1.2 mol/L of hydrochloric acid indicated higher L\* and b\* values than others. This indicated that the color of the sample was bright and yellowish. When the sample contained a large amount of iron oxide or iron phosphate, the color became dark, therefore the condition that this sample became bright was suitable for the purpose of this study. The a\* values had different changes by the concentration between nitric acid and hydrochloric acid. Because the sample prepared with 7.0 mol/L of nitric acid had a high Fe/Nd molar ratio, this condition was unsuitable to obtain neodymium phosphate selectively. The h\* values of samples prepared with hydrochloric acid varied significantly with concentration, while those with nitric acid showed relatively small changes in h\* values. The sample prepared with 1.2 mol/L hydrochloric acid was approximately 70, indicating that the color was approaching yellow. Neodymium phosphate was obtained from the waste magnet in this process. The low concentrations of acid produced Fe/Nd molar ratio lower than 0.5, which is enough low than the original neodymium magnet. The formation of iron phosphate or iron oxide produced a dark powder in color.

*Volume of ascorbic acid:* in the present study, trivalent iron ions were reduced to divalent iron ions with ascorbic acid to suppress the precipitation of iron compounds. Therefore, the amount of ascorbic acid is an important factor in this method. Table III shows L\*a\*b\* values of thermal products and Fe/Nd molar ratio of precipitates prepared with various amounts of ascorbic acid. In this process, 2.28 g of ascorbic acid was corresponding with the reduction of Fe(III) in the Nd<sub>2</sub>Fe<sub>14</sub>B magnet. With the increase of ascorbic acid, the L\* value became smaller in both acid systems. If the reduction of iron was appropriate, it was considered that the precipitation of iron compounds was reduced and the color became brighter, that is, the L\* value increased. Since the L\* value decreased when excess ascorbic acid was added, it is possible that there was some factor that darkened the color, but this was not clear. Samples prepared with nitric acid and ascorbic acid (1.14, 2.28, and 4.56 g) produced high a\* values. The changes in b\* values were small by the amount of

Table II - L\*a\*b\* and h\* (hue) values of thermal products and Fe/Nd molar ratio of precipitates prepared with various concentrations of nitric acid and hydrochloric acid (ascorbic acid: 2.28 g, H<sub>3</sub>PO<sub>4</sub>: 20 mL, pH 3).

Acid	Concentration (mol.L <sup>-1</sup> )	L*	a*	b*	h* (degree)	Fe/Nd
HNO <sub>3</sub>	7.0	38.83	6.98	11.64	59.05	4.505
	2.8	43.05	16.27	14.71	42.12	0.251
	1.4	62.59	25.73	23.11	41.93	0.405
HCl	6.0	53.75	17.23	4.01	13.10	0.471
	2.4	66.92	14.33	0.38	1.52	0.278
	1.2	73.89	8.41	23.79	70.53	0.123

Table III -  $L^*a^*b^*$  and  $h^*$  values of thermal products and Fe/Nd molar ratio of precipitates prepared with various amounts of ascorbic acid ( $\text{HNO}_3$ : 1.4 mol/L,  $\text{HCl}$ : 1.2 mol/L,  $\text{H}_3\text{PO}_4$ : 20 mL, pH 3).

Acid	Asc. ac. (g)	$L^*$	$a^*$	$b^*$	$h^*$ (degree)	Fe/Nd
$\text{HNO}_3$	0	69.00	7.49	21.11	70.46	30.83
	1.14	62.22	19.47	25.10	52.20	0.319
	2.28	62.59	25.73	23.11	41.93	0.405
	4.56	43.17	21.21	18.03	40.37	0.185
$\text{HCl}$	0	78.37	7.57	22.36	71.39	0.185
	1.14	75.09	13.31	23.21	60.27	0.743
	2.28	73.89	8.41	23.79	70.53	0.123
	4.56	67.45	18.53	22.33	50.31	0.100

ascorbic acid. As the amount of ascorbic acid increased, the  $h^*$  value decreased, which indicated a change in color from yellow to red. Sample prepared with nitric acid and without ascorbic acid indicated a high Fe/Nd molar ratio. This iron ratio was higher than that of the original neodymium magnet, indicating that selective neodymium recovery with nitric acid and without ascorbic acid was not possible. The large difference between the samples with nitric acid and hydrochloric acid was thought to be influenced by the oxidative property of nitric acid. In other words, iron was oxidized to trivalent, which made it easier to precipitate and more difficult to separate. The precipitates prepared using nitric acid had higher  $a^*$  values than the samples prepared using hydrochloric acid, suggesting that they were affected by oxidation. The large volume of ascorbic acid supported the preparation of neodymium phosphate selectively.

*Volume of phosphoric acid:* in this work, phosphoric acid was absolutely necessary because it was precipitated as neodymium phosphate. The optimum amount of phosphoric acid was investigated in order to recover neodymium more selectively. Table IV shows  $L^*a^*b^*$  values of thermal products and Fe/Nd molar ratio of precipitates prepared with various volumes of phosphoric acid. Because a too small amount of powders was obtained with phosphoric acid in  $\text{P/Nd}=0.5$  ( $\text{H}_3\text{PO}_4$ : 10 mL), it was impossible to measure  $L^*a^*b^*$  values. A large amount of phosphoric acid produced higher  $L^*$  values,

smaller  $a^*$  and  $b^*$  values, and a smaller Fe/Nd ratio than standard condition ( $\text{P/Nd}=1$ , 20 mL of  $\text{H}_3\text{PO}_4$ ). Iron oxide and iron phosphate have strong red and brown in color, respectively, on the other hand, neodymium phosphate is light purple in color [25, 26]. With the decrease of iron oxide and iron phosphate, the color of powders closed to white. No regularity to  $h^*$  values was observed with increasing amounts of phosphoric acid. This was thought to be due to the influence of the small amount of iron compounds present. The excess of phosphoric acid was advantageous to selectively recovering neodymium phosphate.

*Influence of pH:* the pH value is an important factor in the formation of precipitates in an aqueous solution. Table V shows  $L^*a^*b^*$  values of thermal products and Fe/Nd molar ratio of precipitates prepared under various pH values. As the pH value increased, the  $L^*$  value of the powder decreased, and the  $a^*$  value increased. It is considered that this was because iron compounds were precipitated as the pH was increased and iron oxide and iron phosphate were generated by heating. The  $h^*$  values also decreased with increasing pH, indicating an increase in redness. Samples prepared at pH 4 and 5 had a higher Fe/Nd molar ratio than those at pH 3. Therefore, lower pH than 3 was suitable to obtain neodymium phosphate with a lesser amount of iron compounds. Since a method to obtain rare earth metal from rare earth phosphate which is the main component of rare

Table IV -  $L^*a^*b^*$  and  $h^*$  values of thermal products and Fe/Nd molar ratio of precipitates prepared with various volumes of phosphoric acid ( $\text{HNO}_3$ : 1.4 mol/L,  $\text{HCl}$ : 1.2 mol/L, ascorbic acid: 2.28 g, pH 3).

Acid	$\text{H}_3\text{PO}_4$ (mL)	$L^*$	$a^*$	$b^*$	$h^*$ (degree)	Fe/Nd
$\text{HNO}_3$	10	-	-	-	-	0.482
	20	62.59	25.73	23.11	41.93	0.405
	40	77.61	3.40	4.98	55.68	0.192
	60	78.92	1.64	2.47	56.42	0.195
$\text{HCl}$	10	69.50	12.20	2.91	13.42	0.058
	20	73.89	8.41	23.79	70.53	0.123
	40	88.56	0.93	0.97	46.21	0.171
	60	85.13	2.55	-0.15	-3.37	0.035

Table V -  $L^*a^*b^*$  and  $h^*$  values of thermal products and Fe/Nd molar ratio of precipitates prepared under various pH values (HNO<sub>3</sub>: 1.4 mol/L, HCl: 1.2 mol/L, ascorbic acid: 2.28 g, H<sub>3</sub>PO<sub>4</sub>: 20 mL).

Acid	pH	L*	a*	b*	h* (degree)	Fe/Nd
HNO <sub>3</sub>	2	76.64	3.44	5.70	58.89	0.128
	3	62.59	25.73	23.11	41.93	0.405
	4	55.25	28.80	27.44	43.61	3.726
	5	50.94	24.60	18.34	36.71	1.207
HCl	2	84.67	3.38	7.16	64.73	0.039
	3	73.89	8.41	23.79	70.53	0.123
	4	65.33	18.08	18.45	45.58	0.185
	5	65.85	17.50	6.74	21.06	0.307

earth ore has been established, this method for recovering rare earth as phosphate is very useful for building a sustainable society.

## CONCLUSIONS

Waste magnets were demagnetized by heating, dissolved in hydrochloric or nitric acid, and ascorbic acid and phosphoric acid were added to the solution. The pH value was further adjusted with sodium hydroxide solution to selectively obtain neodymium phosphate. From the color of thermal products and the Fe/Nd molar ratio in the precipitates, low concentrations of acids were suitable, ascorbic acid was necessary, and adding the excess phosphoric acid improved the composition of the precipitate. Because iron phosphate and iron hydroxide were easily formed at higher pH values, the selectivity of neodymium phosphate decreased. This process is much promising because no heating to high temperature and no special reagents are required.

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