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## RECOVERY OF RARE EARTH ELEMENTS AND YTRIUM FROM NON-MAGNETIC COAL FLY ASH USING ACETIC ACID SOLUTION

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### *Abstract*

The increasing need of REY has been observed in industries such as catalyst industry, metallurgy, military, health and renewable energy resources. REY such as Ce, Nd, Y, and Eu has a prominent evidence to improve material properties such as high electropositive, good conductivity and renewable energy. Due to this increasing need of REY, exploration of alternative source has been conducted. One of the promising alternative sources is coal fly ash. The conventional method to recover REY from coal fly ash using inorganic acids is harmful to the environment. Thus, the exploration of REY extraction from coal fly ash using organic acid has been proposed in this study. The leaching agent was acetic acid applied for all operating condition. Regarding to the mineralization of REY in the form of siliceous minerals (non-magnetic coal fly ash). Silicate digestion was conducted using sodium hydroxide 8 M with solid-to-liquid of 25% to decompose the siliceous mineral. Further, the REY bound siliceous mineral will change to REY(OH)<sub>3</sub>. Temperature as one of the operating conditions was varied. The maximum recovery of leaching 20.58%, 43.53%, 17.38%, 40.96%, 18.45% and 32.74% were achieved for Ce, Dy, La, Nd, Y and Yb respectively at temperature of 90°C pH 1.74 and 120 minutes. For some metals, increasing the temperature higher than 70°C does not provide any significant effect for REY recovery such as La and Ce.

Keywords: REY, non-magnetic CFA, leaching, recovery

### **INTRODUCTION**

Rare Earth Elements such as Ce, Dy, La, Nd, Yb and Yttrium (Y) becomes important issue in the recent year. The increasing need of REY has been observed in industries such as catalyst industry, metallurgy, military, health and

renewable energy resources (Zhou et al. 2017; Harjanto et al. 2013). Alonso et al (Alonso et al. 2012) reported that REY demand increase 5.3% every years from 2010 until 2020. REY has a prominent evidence to improve material properties such as a high electropositive, good

conductivity and renewable energy. Due to this increasing need of REY, exploration of alternative source has been conducted. There are some several alternative sources of REY such as red mud, spent catalyst, electronic waste, tin residue coal, coal fly ash and zircon sand (Abhilash et al., 2014; Aung and Ting, 2005; Borra et al., 2015; Sinha et al., 2014; Virdhian and Afrilindia Eva, 2014, Prameswara et al., 2020, Manurung et al., 2020). One of the promising alternative sources is coal fly ash and coal (Anggara et al., 2018; Anggara and Petrus, 2016; Peramaki, 2014). Anggara et al. (Ferian et al. 2018) recently reported that fly ash and bottom ash from Coal Power Plant in Java are promising raw material for REY extraction.

The existence of REY on coal fly ash were influenced by the coal rank and mineral matter or detrital mineral such as quartz, clay mineral, sulfide and phosphate (Finkelman et al 2018; Wang et al. 2008). Due to high temperature in combustion process, REY were melting and distributed into the fly ash component such ash quartz ( $\text{SiO}_2$ ), mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) and amorphous phase (Kashiwakura et al. 2013).

The mathematical formulation to estimate the prospect of REY from coal fly ash has a several parameters such as resources of metal, possibility of beneficiation, simplicity and hydrometallurgical recovery (Vladimir V Seredin and Dai 2012). The cut-off-grade for REY extraction should be minimum or  $>1000$  ppm. However, due to the increasing need of REY, the recent prices much higher than in the past. Moreover, at previous decade, this cut-off grade could be lowered to 800-900 ppm. The present criterion to evaluate the REY in the coal fly ash based on the individual composition of the metal (elements). It was divided in three classification such as critical (Nd, Eu, Tb, Dy, Y and Er), uncritical (La, Pr, Sm and Gd) and excessive (Ce, Ho, Tm, Yb and Lu) (V V Seredin 2010). Critical is an element with very low amount and high demand, uncritical is a medium and excessive is the most abundant element occurrences. For preliminary estimation of raw material quality, there was a ratio of relative amount of critical REY in total REY to the relative amount of excessive REY. The index called outlook coefficient of REY. The equations used to calculate the outlook is as follow (equation 1):

$$C_{\text{out}} = \frac{(\text{Nd} + \text{Eu} + \text{Tb} + \text{Dy} + \text{Er} + \text{Y})/(\sum \text{REY})}{(\text{Ce} + \text{Hp} + \text{Tm} + \text{Yb} + \text{Lu})/(\sum \text{REY})} \dots\dots (1)$$

The minimum  $C_{\text{out}}$  index is 0.7. The higher  $C_{\text{out}}$  the more promising REY raw material to extract (V V Seredin 2010).

Hydrometallurgy is the most efficient method for extract some metal for low concentration, because it consumes little energy, emits small amounts of gases and high recovery. Leaching is one of hydrometallurgy process, where acid used as the leaching agent to extract element from the coal fly ash. Conventionally, inorganic acids used as the leaching agents such as  $\text{H}_2\text{SO}_4$ , HCl, and  $\text{HNO}_3$  (Kashiwakura et al. 2013; Peramaki 2014). Anyway, inorganic acid in the leaching process produces harmful byproduct. In the recent years, researchers have investigated the use of organic acid to substitute the inorganic acid as a leaching agent such as citric acid, acetic acid, oxalic acid and nitric acid (Wanta et al. 2016; Wanta et al. 2018; Lazo et al. 2017; Setiawan et al. 2019; Sekar et al. 2018; Sari et al. 2018). The use of organic acid often called as an environmentally friendly leaching agent.

This study aims to investigate the use of acetic acid as an environmentally friendly leaching agent. Acetic acid has been demonstrated to leach neodymium from the scrub magnetic leaching (Behera and Parhi 2016). Moreover acetic acid also has been demonstrated to leach Li and Co from spent lithium-ion batteries (Setiawan et al. 2019). Regarding to the mineralization of REY in the form of siliceous minerals (non-magnetic coal fly ash). Silicate digestion was conducted using sodium hydroxide to decompose the siliceous mineral and enable the leaching of REY. Further the REY bound siliceous mineral will change to  $\text{REY}(\text{OH})_3$ .

## RESEARCH METODOLOGY

### Material

Coal fly ash (CFA) were collected from Coal Fired Power Plan (PLTU) Tuban East Java. Particle size less than  $38 \mu\text{m}$  (- 400 mesh) were used as a raw material. Non-magnetic component and magnetic component were separate using wet magnetic separator with 2 A current at Mineral and Coal Technology Reaseach and Development (PUSLITBANG TekMIRA) Bandung west Java.

XRD analysis were applied to identify the material characterization using XRD X'Pert 3 Powder instrument. The Non-magnetic component analysis using ICP-MS both major elements and REY in ALS Global - Geochemistry Analytical Lab in North Vancouver, BC, Canada.

### Silicate Digestion

The digestion was done using sodium hydroxide NaOH pro-analysis produced by Merck Inc. The digestion process was conducted in a three-necked flask by mixing 62,5 g non-magnetic component of CFA with 250 mL (solid-to-liquid 25% w/v) of sodium hydroxide 8 M (pH 13,9). The reagent was heated using waterbath equipped with reflux condenser and stirrer operated at a speed 500 rpm than heated to 90°C at atmospheric condition. They were digested for 120 minute to breakage the silicate content. The digested residue were washed using destilated water (60x) using dry sample wight based and dried at temperature 100°C during 5 hours using oven. This solid were used as a material for acid leaching.

### Metal Leaching

Acetic acid (100vol%) pro-analysis was used as a leaching reagent, produced by Merck Inc. The leaching process was conducted in a three-necked flask by mixing 25 g sample with 250 mL (solid to liquid 10% w/v) of acetic acid 50% (pH 1.72). The reagent was heated using a mantle heater equipped with reflux condenser and with a stirred operated at speed 500 rpm than heated at 30°C (room temperature), 70°C and 90°C. Temperature as one of the operating condition was varied. They were leached for 120 minutes to dilute the REY from non-magnetic CFA. The peocess flow-sheet was presented in Figure 1 from raw material until leaching step.

### ICP-EAS Analysis and XRD Analysis

Sample preparation was carried out by diluting 2 mL of leached to 10 mL (5 x dilution) using aqua for injection. The samples was filtered using a 0.22 µm micro filter to separate any suspended solid in the filtrate. The filtrate was analyzed using ICP-AES (Simadzu Europe) in LPPT UGM to find the amount REY recovered by leaching. Sample preparation for XRD analysis was carried out by weighing dry samples 10 gr for raw material (non-magnetic coal fly ash), after

silicate digestion and after acid leaching using XRD X'Pert 3 Powder instrument.

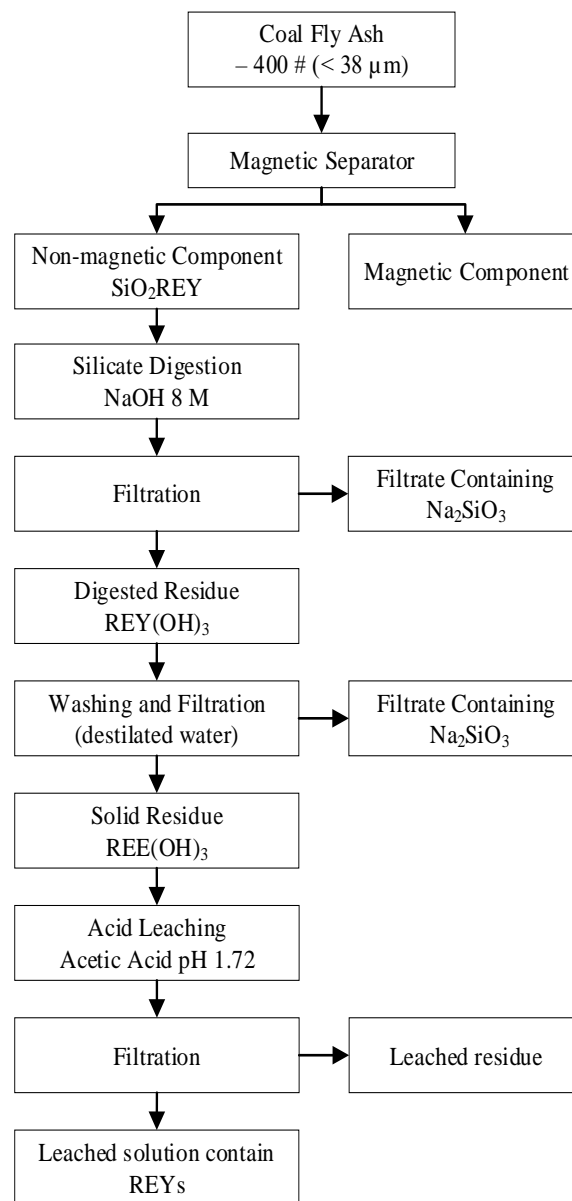


Figure 1. Process flow-sheet for extracting REE from coal fly ash

## RESULT AND DISCUSSION

### Raw Material Characterization

Raw material characterization was conducted using XRD analysis and ICP-MS instrument for major composition and rare earth concentration. Based on the major element analysis shows that non-magnetic coal fly ash contain SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, CaO and MgO as a

major mineral and K<sub>2</sub>O, Cr<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, MnO, P<sub>2</sub>O<sub>5</sub>, SrO and BaO as a minor mineral (Table 1). SiO<sub>2</sub> is the most abundance major element for non-magnetic coal fly ash. The rare earth elements concentration were presented in Table 2. Cerium is the higher concentration for excessive and Yttrium is critical. Based on the XRD analysis shows that quartz, mulite, hematite and khamosite (Figure 3 sample A). Rare earth elements were not appear in this analysis due to very small amount (ppm) and rare earth element were not mineralized but only distributed and sticky on the silicate and glass phase after combustion process (Kashiwakura et al. 2013; Peramaki 2014).

Table 1. Major element in nonmagnetic component of CFA

Major Element	Wt (%)	Major Element	Wt (%)
SiO <sub>2</sub>	53.20	Cr <sub>2</sub> O <sub>3</sub>	0.01
Al <sub>2</sub> O <sub>3</sub>	31.90	TiO <sub>2</sub>	1.15
Fe <sub>2</sub> O <sub>3</sub>	5.58	MnO	0.06
CaO	3.08	P <sub>2</sub> O <sub>5</sub>	0.24
MgO	2.11	SrO	0.08
Na <sub>2</sub> O	0.90	BaO	0.07
K <sub>2</sub> O	0.99	LOI	1.09

Table 2. REY concentration in non-magnetic coal fly ash

REY Elements	Unit (ppm)
Ce	98.3
Dy	9.32
La	49
Nd	45.2
Y	53.2
Yb	6.08

Based on the Table 2 (another elements concentration not shown), the Cout can be calculated using equation (1):

$$C_{out} = \frac{(Nd + Eu + Tb + Dy + Er + Y) / (\sum REY)}{(Ce + Hp + Tm + Yb + Lu) / (\sum REY)}$$

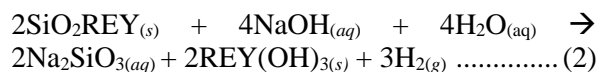
$$C_{out} = 1.068.$$

Cout = 1.068 (Cout > 0.7) indicate that REY in non magnetic coal fly ash are promising to extract.

### Studies for Silicate Digestion and recovery

The presence of silicate component in non-magnetic coal fly ash decrease the REY

recovery. Dissolving silicate component become an essential point to remove. Silicate digestion can be achieved by alkaline fusion or alkaline leaching. NaOH solvent was used to as a leachant to breakage and solubilize the silicious component (Manurung et al., 2020). During the silicate digestion of coal fly ash, its constituent's at elevated temperatures takes place according to the reaction (Ian McGill 2012).



Where SiO<sub>2</sub>REY was transformed to natriumsilicate salt and REY change the bound as a REY(OH)<sub>3</sub>. The filtrate were analyzed to identify the amount of silicate and REY in the liquor. Based on the analysis data shows that SiO<sub>2</sub> were dilute 20% until 40% during silicate digestion.

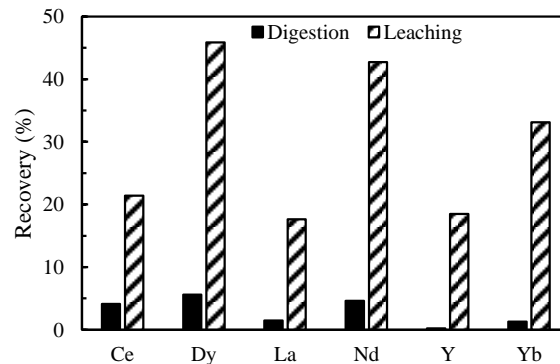


Figure 2. Recovery of REY after digestion process and leaching at temperature 90°C

The recovery of REY during silicate digestion were presented in Figure 2. The REY recovery during silicate digestion 5.5%, 4.6% and 4.1% were achieved for Dy, Nd and Ce respectively. It shows that only small amount of REY were diluted during silicate digestion compare to acid leaching at the same temperature (90°C). Furthermore, when silicate component were decomposed, rare earth elements were transformed to rare earth oxide (REY(OH)<sub>3</sub>) (Ian McGill 2012). Gupta et al. (Nagaiyar dan Gupta 2016) also reported that alkaline fusion using NaOH solid was applied to recovery rare earth element from monazite (CePO<sub>4</sub>) to removed phosphate as a pretreatment to form cerium-oxide before acid leaching. Beside that, alkaline fusion

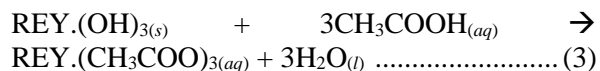
or roasting using NaOH were applied to recovery rare earth element from xenotime and bastnasite to convert rare earth elements to rare earth oxide (Rekha et al. 2014; Huang et al. 2017).

The XRD analysis shows that there were no significant peak differences for raw material and after silicate digestion for quartz and mullite (Figure 3; sample A and sample B). It shows that silicate digestion will dissolve the amorphous phase and glass mineral such as quartz and mullite (Kashiwakura et al. 2013).

### Acid Leaching of REY from Non-magnetic Coal Fly Ash

The non-magnetic coal fly ash was leached using acetic acid. Acid leaching was conducted at pH 1.72 at various temperatures (30°C, 70°C and 90°C) in 120 minutes. The acid

leaching using acetic acid were used to leach the REY from REY(OH)<sub>3</sub>. The general reaction for acid leaching using acetic acid can be described as equation (3) (Krishnamurthy dan Gupta 2016).



Where REY(OH)<sub>3</sub> is transformed to REY(CH<sub>3</sub>COO)<sub>3</sub> in a liquid phase. Ion H<sup>+</sup> from acetic acid was successfully to replace the REY bound and dissolve the REY. In order to assure the chemical reaction, XRD analysis was conducted (Figure 3; sample C, D and E). It shows that there were no significant peak differences for each mineral and REY not visible due to very low concentration.

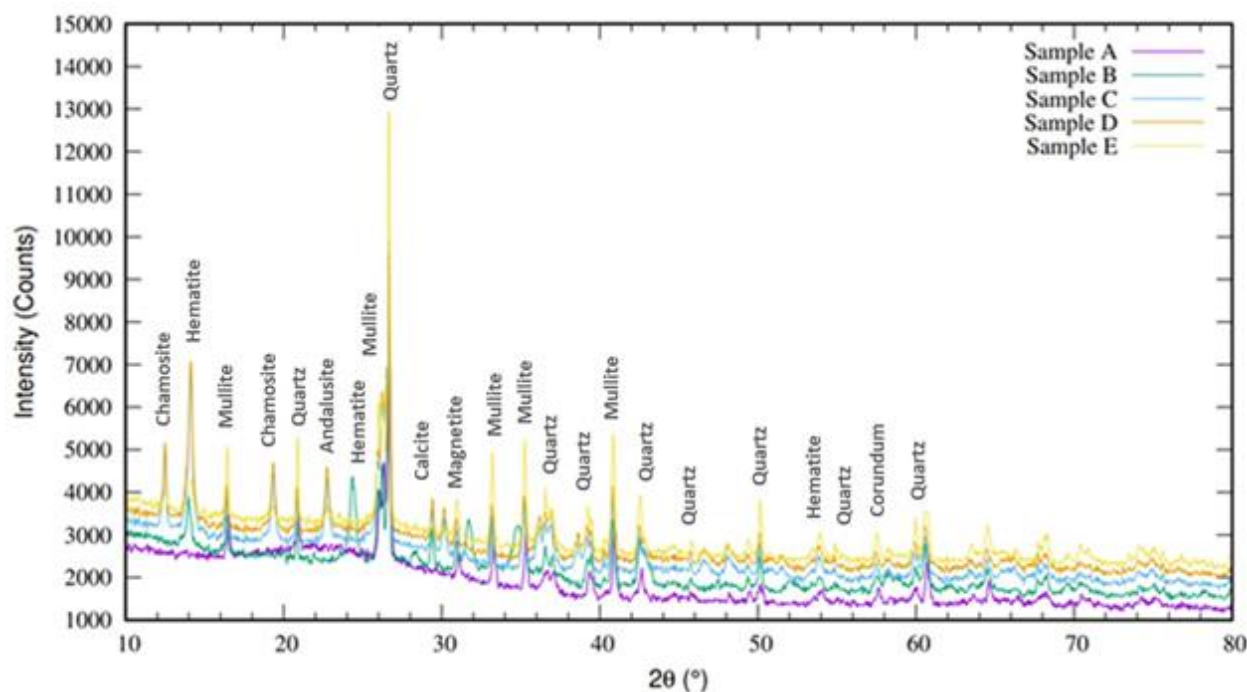


Figure 3. XRD analysis Pattern for coal fly ash (Sample A: raw material, Sample B: after silicate digestion, Sample C: acid leaching at temperature 90°C, Sample D: acid leaching at temperature 60°C and Sample E: acid leaching at temperature 30°C)

The recovery of REY metal such as Ce, Dy, La, Nd, Y and Yb were calculated in certain temperature. Based on the Arrhenius rule shows

that, temperature is one of the main parameters on chemical process due to process rate. The effect of temperature on REY recovery is shown in Figure.

4. It shows that temperature influence the REY recovery. Increasing temperature, the rate of REY recovery also increased (Manurung et al., 2020). This phenomenon is consistent with the Arrhenius rate law, which states that an increase the temperature will increase the rate of reaction, increase in temperature of 10 degrees can increase the reaction rate twice (Fogler 2005).

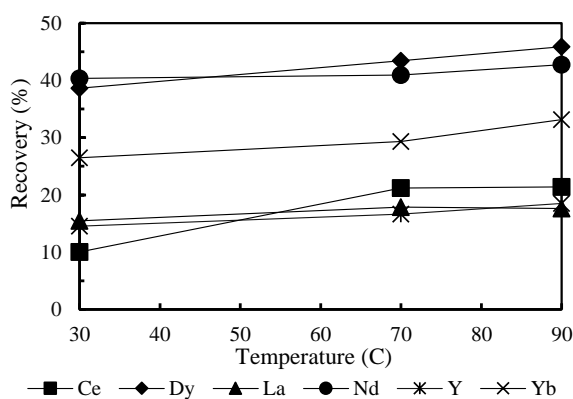


Figure 4. Recovery of REY at various temperature (pH 1.72, 500 rpm, S/L 10%, 120 min)

The temperature need to achieve the maximum recovery of each REY element were different. For Cerium (Ce), the recovery were excessively increase at temperature 30°C to 70°C, the resulting recovery increase from 9.68% to 20.41% (increase 10.72%). Increasing temperature until 90°C was slightly increase the Ce recovery, it was from 20.41% to 20.58%. Lyberopulu and Ochsenkiihn (Lyberopulu dan Ochsenkiihn 1996) reported that leaching cerium from red mud achieved the optimum conditions at 80°C using HNO<sub>3</sub>, HCL and H<sub>2</sub>SO<sub>4</sub>, the recovery were 29%, 32% and 24% respectively. There were 10% recovery different between organic and inorganic acid

Dysprosium (Dy) element shows that, increasing the temperature from 30°C to 70°C and 90°C were increase the Dy recovery, it was 36.69%, 41.24% and 43.53%. The Dy recovery was increase step by step due to increase the temperature. For Lanthanum (La) element shows that increasing temperature from 30°C to 70°C were increase the recovery from 15.29% to 17.62%. However at temperature 90°C the La recovery decrease from 17.62% to 17.38%. For

Neodymium (Nd) element shows that, increasing temperature from 30°C to 70°C were increase the recovery from 38.67% to 39.22%. Increasing temperature until 90°C was increase the recovery from 39.22% to 40.96%. There were no significant effect of temperature to Nd recovery. Migdisov and William (Migdisov dan Williams-Jones 2007) reported that neodymium solubility increase above 150°C in F-bearing aqueous solutions, due to high stability of neodymium.

Yttrium (Y) element shows that, increasing temperature from 30°C to 70°C and 90°C were increase the recovery of yttrium from 14.51%, 16.63%, and 18.45% respectively. For Yb element shows that temperature influence the Yb recovery from temperature 30°C, 70°C and 90°C were increase from 26.18%, 28.98% and 32.74%.

## CONCLUSION

Based on the experiments and discussion conclude that temperature (30°C, 70°C and 90°C) influence the REY recovery. Overall the higher temperature the higher REY recovery. The maximum recovery 20.58%, 43.53%, 17.38%, 40.96%, 18.45% and 32.74% were achieved for Ce, Dy, La, Nd, Y and Yb respectively at temperature 90°C and 120 minutes. Otherwise for some metal, the increasing temperature more than 70°C does not have a significant effect for REY metal recovery such as La and Ce.

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