Application of Taguchi optimization on the titanium recovery from iron sands using flotation process

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Abstract. Titanium is very important metal which commonly used in industrial process, automotive, electronic devices and other application. Titanium is not pure element in nature but mixed with other elements such as in iron sands. Hence, it is required to do research to gain high concentration titanium and also eliminate its impurities. One of method which able to overcome this problem is the flotation method. On this research, titanium recovery process is done from natural iron sands of Lampung using oleic acid as a collector in the flotation process. The iron sands which used for each variation are 10, 15 and 20 wt% with a size of 60, 100 and 150 mesh. Those iron sands were mixed into 0,10; 0,20; 0,30 lb/tons of collector by varying pH condition of 6, 8 and 10. After that, each variation product was characterized by XRF characterization and then calculated based on Taguchi method. From the research that had been done, it is known that the optimum values are at the content: oleic acid = 0,1 lbs/ton, ore 20 wt%, pH 8, the Particle size of 150 mesh.

1. Introduction
Titanium is a very important material which can be alloyed with other metal such as iron, aluminum, vanadium, and molybdenum, among other elements, to produce strong and lightweight alloys. The titanium alloys commonly used in aerospace, military, industrial processes, automotive, agrifood, medical prostheses, orthopedic implants, dental and endodontic instruments and files, dental implants, sporting goods, jewellery, mobile phones, and other applications [1,6].
Table 1. The analysis result of XRF from Lampung Iron Sand

| Number | Compositions of Iron Sand | Value +/- |
|--------|---------------------------|-----------|
| 1      | Fe                        | 53,883    | 0,306     |
| 2      | Le                        | 26,185    | 0,370     |
| 3      | Ti                        | 3,828     | 0,040     |
| 4      | Cl                        | 2,028     | 0,071     |
| 5      | Si                        | 4,772     | 0,042     |
| 6      | Al                        | 2,004     | 0,132     |
| 7      | K                         | 0,636     | 0,088     |
| 8      | Ca                        | 1,344     | 0,011     |

Titanium can be produced from a natural material which available on earth such as iron sands. Titanium which produces from iron sand such as in Uni Soviet, Australia and South Africa[2]. Lampung province (Indonesia) has a lot of iron sand which contains of 3.83% titanium as listed in Table 1. One of the methods which able to increase the titanium contained in the sand is froth floatation. Froth flotation is a mineral separation process into ore from its impurities by floating ore to the surface through its bonds with froth. This process is used for some ores such as Cu, Pb, Zn, Ag, Au and Ni. The procedures are done by blowing air into mineral grains which mixed with water and frothing substances. The hydrophobic mineral grains then will be flown by air to the surface. The flotation process will be more effective if it is added to a collector substance. One of economical and effective collector for titanium floatation is oleic acid [3]. The parameters which have significant effect towards titanium floatation were studied and analyzed using Taguchi methods such as oleic acid concentration, ore concentration, pH in floatation cell and particle size. In this work, Taguchi method for four parameters and three levels only need 9 experiments [4,5]. This method needs less experiments than the full factorial methods which need 81 experiments.

2. Methodology

The sand used in this work was from iron sand from West Lampung beach which contains titanium as shown in Table 1. An oleic acid from Brataco Chemika Co. Ltd. (Jakarta, Indonesia) was used as a collector. Pine oil or surfactant Frother chemical which used in this work was purchased from Brataco Chemika Co. Ltd. (Jakarta, Indonesia). Lime (CaO) (Mutiara co. Ltd., Lampung, Indonesia) was used to adjust pH in floatation cell. In this work, iron sands were Iron sand which will be used is gained from Lampung that is separated by magnetseparator then grilled by a ball mill. Then, sieved by sieve in size of 60, 100 and 150 mesh. Flotation process was conducted in 6 Liters capacity of froth floatation cell (Quinn process equipment co., Denver, United States). The pH inside froth floatation cell was measured using Universal indicator from Merck (German). the composition of tailing and product of froth Flotation were analyzed using handheld X-ray fluorescent (XRF) analyzers from Olympus, (Massachusetts, US).

All experiments were conducted by following design experiment of the Taguchi orthogonal arrays (OA) method for four parameters and three levels as shown in Table 2. This method was used to determine the optimum operating condition and to study the effect of parameters towards a recovery of titanium [6,7]. The orthogonal arrays of the L9 type which has 9 experiments were chosen to study four parameters and three levels as shown in Table 2. The benefit using Taguchi orthogonal arrays (OA) this project only has 9 experiment, meanwhile when if this project using full factorial, its need 81 experiment. The optimum condition obtained was obtained using Signal to Noise (S/N) ratio of “larger the better” which was calculated using equation as follows:

\[ S/N = -10\log(\Sigma(1/Y^2)/n) \]

where Y = responses for the given factor level combination and n = number of responses in the factor level combination [4]. All experiments were conducted for 10 minutes operations. During the experiment, the opening of the gas valve was kept constant to ensure all experiments were conducted in the same condition of the gas inlet. The froth from flotation cell was collected with an automatic scraper, which is installed in the flotation cell. At the end of an experiment, the gas inlet valve was
closed and the compressor was turned off. Then concentrate sample or product of flotation and the
tailing of floatation was sun-dried. Then, the drying products were analyzed by XRF and weighed
using the analytical balance. The schematic diagram of froth flotation cell was used in this research
was shown in Fig. 1. Percent of titanium recovery was calculated using formula as follows:

\[
\text{Recovery} = \frac{\text{Ti product} \times \text{product weight}}{\text{tailing} \times \text{tailing weight}} \times 100\% 
\]

![Diagram of Froth Flotation](image)

**Figure 1. Diagram of Froth Flotation**

| Run | Oleic acid (lb/ton) | Ore (wt %) | pH | Particle Size (mesh) | Ti product (%) | Ti tailing (%) | Product Weight (gram) | Tailing Weight (gram) | Recovery (%) |
|-----|---------------------|------------|----|----------------------|----------------|---------------|-----------------------|-----------------------|--------------|
| 1   | 0.1                 | 10         | 6  | 60                   | 3.8            | 3.0           | 360                   | 390                   | 18,1631      |
| 2   | 0.1                 | 15         | 8  | 100                  | 5.1            | 4.4           | 700                   | 460                   | 36,8741      |
| 3   | 0.1                 | 20         | 10 | 150                  | 4.3            | 4.3           | 1240                  | 340                   | 41,1661      |
| 4   | 0.2                 | 10         | 8  | 150                  | 2.9            | 4.7           | 570                   | 180                   | 41,1661      |
| 5   | 0.2                 | 15         | 10 | 60                   | 3.0            | 3.5           | 980                   | 130                   | 10,8112      |
| 6   | 0.2                 | 20         | 6  | 100                  | 4.9            | 4.8           | 1270                  | 280                   | 35,9132      |
| 7   | 0.3                 | 10         | 10 | 100                  | 4.8            | 4.3           | 700                   | 80                    | 6,2504       |
| 8   | 0.3                 | 15         | 8  | 150                  | 4.1            | 4.0           | 700                   | 389                   | 27,1697      |
| 9   | 0.3                 | 20         | 6  | 60                   | 4.3            | 3.7           | 1280                  | 270                   | 37,1039      |

3. Result and Discussion

3.1. The effect of oleic acid concentrations towards titanium recovery
The effect oleic acid concentration towards titanium recovery is shown in Fig. 2. The figure shows the larger means of S/N ratio of titanium recovery for oleic acid concentration is at level 1. This result indicates that the best flotation recovery at 0.1 lb/ton of oleic acid. The means of SN ratios decreased from level 1 to level 3 for oleic acid concentration. This caused that the flotation recovery oleic acid act as a surfactant that increases the hydrophobic characteristic of the mineral. In nature, titanium commonly binds to sulfide material which has a hydrophobic characteristic. Oleic acid cause these sulfide materials become more hydrophobic hence, these hydrophobic materials easily float together with gas bubbles. However, the means of SN ratios decreases at levels 2 and 3. This decrease of percentage recovery is due to the excess of oleic acid as a surfactant will cause a lot of non-sulfide minerals which do not contain titanium will turn into hydrophobic, hence these float with gas bubbles. This phenomenon led to the decrease in the recovery percentage.

3.2. The effect of ore concentration towards titanium recovery
The effect of ore concentration towards titanium recovery is shown in Figure 3. The figure shows that the increment of ore concentrations will enhance the titanium recovery. The figure also shows that the optimum ore concentration (wt %) is at level 20 wt % of ore. This caused by more titanium particle in the mixture of sand and water, hence more titanium in the sand which able to separate using flotation process.
3.3. The effect of pH towards titanium recovery

The correlation of pH towards titanium recovery in flotation process of iron sand is shown in Fig. 4. The figure shows that the increase in pH will increase the titanium recovery. However, at the pH of 10, the titanium recovery was decreased. This caused by the increased pH as the increase of CaO concentration which leads to the increase of impurities on the product floatation. Hence, the titanium recovery was decreased as the increase in pH. The surface of titanium ore in froth floatation process is affected by pH, at the pH of 8 the attraction of collectors to the titanium ore surface become more effective as shown in Fig. 4 [8].

3.4. The effect of particle size towards titanium recovery

The titanium recovery on flotation process was affected by particle size as shown in Fig. 5. The figure shows that the increase of mesh or the decreased of the particle will enhance the titanium recovery on froth flotation process. This decrease of individual particle size caused the decrease of mass gravitation than these particles were lifted up by the gas bubbles easily. The smaller particle size has a good the degree of liberation which the most of valuable mineral particles has been separated with other mineral impurities. Hence, collector reagent interacts with the surface of the precious mineral particles and resulting the recovery will increase. However, if the particle size is too small it will cause high consumption of reagent or cause the poor selectivity due to mechanical entrainment as well as entrapment of ore with gangue or vice versa [9]. From the figure, it is shown that the optimum condition of flotation process for titanium is at 150 of mesh.
3.5. **Optimum Condition of Flotation**

The largest titanium recovery percentage indicates the best flotation process. The titanium recovery percentage has been analyzed using Taguchi analysis "Larger the better ". The S/N ratio and mean analysis result was listed in Table 2 and 3, respectively. The data of the responses for the means and the values of the delta are shown in Table 2 and 3. Delta is the difference in values between the biggest to the smallest means or SN ratio of percentage titanium recovery. It represents the most parameter, which affects percentage gold recovery. From Table 3 and 4, pH has the biggest delta value, which means that the pH is the most significant parameter to the percentage of titanium recovery. This is followed by the ore concentration, particle size and oleic acid concentration. Based on Taguchi method which is shown in table 4.2, the optimum condition is the highest value of SN ratio of "larger the better". The optimum values are at the content: oleic acid = 0.1 lbs/ton, 20 wt% of ore, pH 8, the Particle size of 150 mesh.

![Figure 5. The effect of particle size on the means of SN ratios](image)

**Table 3.** Graph of SN ratio analysis of Taguchi method by using Minitab

| Level | Oleic acid | Ore | pH | Particle Size |
|-------|------------|-----|----|---------------|
| 1     | 29.60      | 24.46| 28.32 | 25.75         |
| 2     | 28.02      | 26.90| 31.67 | 26.12         |
| 3     | 25.33      | 31.59| 22.96 | 31.09         |
| Delta | 4.27       | 7.13 | 8.71  | 5.34          |
| Ranking | 4         | 2    | 1    | 3             |

**Table 4.** Graph of analysis of mean recovery result from Taguchi Method by Minitab

| Level | Oleic acid | Ore | pH | Particle Size |
|-------|------------|-----|----|---------------|
| 1     | 32.07      | 21.86| 27.08 | 22.03         |
| 2     | 29.30      | 24.95| 38.38 | 26.35         |
| 3     | 23.51      | 38.06| 19.41 | 36.50         |
| Delta | 8.56       | 16.20| 18.97 | 14.47         |
| Ranking | 4        | 2    | 1    | 3             |
4. Conclusion
The flotation process of titanium from iron sand has been conducted using Taguchi design of the experiment. Based on Taguchi analysis it is found that the pH is the most significant parameter to the percentage of titanium recovery than followed by the ore concentration, particle size and oleic acid concentration. The optimum condition also enable to determine using SN ratio of “larger the better”. The optimum values are at the content: oleic acid = 0.1 lbs/ton, ore 20 wt%, pH 8, the Particle size of 150 mesh.

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