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Optimization Of Titanium Recovery From Tin Tailings Using Flotation Route

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Abstract

Titanium has developed to large applications in modern industry. Due to its high corrosion resistance, titanium has been applied in dental apparatus and surgical instruments, as implant in bones, and as sea vessel component in harsh marine condition. Titanium has also been used as engine component in high thermal environment. Since its weight is lighter than that of steel, therefore, titanium has been used in many construction materials replacing the role of stainless steel. Tin tailings in Bangka Island, Indonesia, has been investigated possessing titanium, therefore, titanium analysis from Bangka tin tailings is very substantial. Since the froth flotation method is broadly used in mineral analysis on account of simple, rapid, and low cost, therefore, this study applied froth flotation method to analyze titanium from tin tailings using sodium oleate frother/collector and sodium chlorate depressant yielding concentrate and tailing. The mass ratio (g) of depressant and collector at fixed collector are varied, i.e. 1:10, 5:10, 10:10, and 15:10, and at fixed depressant, i.e. 10:3, 10:6, 10:9, and 10:12. The highest titanium concentration (2.03 %) obtained at mass ratio of 10:12, and the optimum titanium recovery (45.51%) in the collector (concentrate) was achieved at equal amount (3.75 g) of respective depressant and collector or at mass ratio 10 :10 at 15 min. flotation time and neutral pH. The XRF (X-Ray Fluorescence) and XRD (X-Ray Diffraction) examinations revealed the initial tin tailing was attributed to silicate and zircon minerals in the presence of small amount titanium addressing to rutile, ilmenite, and titanate. This study is really valuable for future titanium industry.

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1. Introduction

Titanium is a highly valuable metal that has broad applications due to its remarkable corrosion resistance, moderately good strength, and acceptable density. In medical field, titanium material has been applied as implants in dental organ, surgical devices, and bone joints. In industry, titanium material has been used for pipes, heat exchangers, and instrumentations due to its excellent corrosion resistance to high temperatures. Titanium has been utilized as engine components on account of its suitable performance and light weight. Titan has also used in marine field due to its high resistance in harsh marine environment. Titanium shows benefit properties for jewelry and sports applications on account of its aesthetic appearance, long life time, and low weight. On reason of titanium broad applications, this work attempts to focus a study on titanium recovery.

Titanium has richly found in mineral rutile (TiO_2) and ilmenite ($FeTiO_3$) in tin tailings. From geological site, rutile and ilmenite are found in metamorphic and igneous rocks, as well as in sedimentary deposits. Igneous rocks are commonly found in ultramafic rocks such as basalt. Sedimentary deposits are formed due to weathering and erosion process. Figure. 1 shows rutile and ilmenite minerals.

Dahani (2023) reported high grade ilmenite (90.46%) in Bangka tin tailing applying magnetic separator [1]. With regard to ilmenite separation, Miao [2] used Fenton oxidant in flotation method for ilmenite recovery, Xiao [3] applied combined flotation and adsorption methods to separate ilmenite and titanaugite, Tian [4] used carboxylated starch to separate ilmenite and forsterite applying flotation route, and Sitepu [5] reported effect of electrical current in magnetic separator on ilmenite recovery. On the other side, with regard to rutile recovery, Wahyuningsih (2016) reported rutile recovery from Bangka tin tailing applying leaching process with HCl [6], Ismael (2020) reported rutile recovery (79 %) applying combined alkaline roasting and acid leaching from solid waste tailing [7], and Kurniawan (2022) reported rutile recovery (92.6%) from tin ore applying chemical separation technique related to peroxidation and reductive leaching with HCl [8]

(a) (b)

Figure 1 (a) Rutile and (b) Ilmenite minerals

In the context of titanium enrichment, Zhang (2022) concentrated Nb and Ti from carbonatite ore through calcining-slaking followed by gravity separation [9]. Along with the rapid development the demand of titanium and titanium alloy is gradually increasing and the price for titanium materials is rising. Therefore, the titanium recovery from secondary resources such as lithium titanate waste and residual waste using extraction and leaching methods attracts great interest in order to reduce environmental impact, to advantage waste of secondary resources, and to sustain titanium industry [10]. The understanding of secondary resources is related to electronic waste such as obsolete or damaged smartphone, lap top, television, batteries, and so on. Recovery of desired material from electronic waste is in relation to waste recycling. Primary resources is related to mining rocks or mining tailing that undergone exploration and exploitation to separate the interested mineral applying several methods.

Although titanium recovery can be obtained by acid leaching method as reported by Dahani [11] applying sulfuric acid to recover lead from galena mineral, however, this study attempts to analyze titanium from tin tailing after crushing and screening to obtain given particle size followed by hydrometallurgy method, i.e. the froth flotation method. Sodium oleate was used as frother and collector, while sodium chlorate used as depressant based on hydrophobic and hydrophilic characters to improve titanium recovery and reduce any impurities. Previous investigations applied flotation methods as Nie (2023) reported flotation separation of cassiterite and calcite using cinnamon hydroxamic acid as collector [12], Tian (2022) reported flotation method to separate forsterite and ilmenite applying starch depressant [4], while Feng (2021) used hydroxyethyl cellulose depressant in flotation route to separate chalcopyrite and galena [13], and Feng (2019) conducted flotation technique to separate galena and pyrite applying serpentine depressant [14]. Nowadays, numerical and statistical methods have been introduced in many analytical methods to improve the accuracy of the research results. Sierra & Korotenko [15] implemented statistical analysis in shaking table method in order to improve the recovery of mineral elements based on different specific gravity of those elements. Keshun [16] integrated algorithm system of learning machine and shaking table method to increase mineral recovery in industry and mining. Since the flotation method is simple, fast, and low cost for mineral recovery, therefore, this study applied flotation route to analyze titanium from tin tailing.

2. Experimental and Procedures

2.1. Physical separation

Initially, the ore sampling taken from Bangka tin tailing was crushed using a jaw crusher to obtain particles with smaller size. Then, the smaller particles were grinded using a Hammer mill to get finer particles. Finally, the fine particles were filtered using a sieve shaker yielding a powder with given size of 150 mesh and was ready for chemical process. The particle size of 150 mesh was chosen since usually mining dressing applied varied particle size in the range of 80 mesh – 200 mesh.

2.2 Chemical separation

This study used a hydrometallurgy method for chemical separation, i.e. the froth flotation route. The flotation process was conducted applying a flotation machine (Figure. 2). The sample powder, sodium oleate frother/collector, sodium chlorate depressant, and HCl as pH regulator were put in a flotation cell of the machine. The solution pH was set at pH 7.0. The pH 7.0 was selected in this study on the reason of using aqueous media related to neutral pH.

The addition of sodium oleate and sodium chlorate was adjusted to obtain varied ratios as given. Table 1 shows the given ratio of sodium chlorate depressant and sodium oleate frother/collector. Figure. 3 reveals the flow chart of this study.

Figure 2 Denver flotation machine

Table 1. Varied ratio of sodium chlorate depressant and sodium oleate collecto. 150 mesh.pH 7.0.

Sodium chlorate depressant (g)	Sodium oleate collector / frother (g)	Ratio of sodium chlorate and sodium oleate
0.375	3.75	1 : 10
1.875	3.75	5 : 10
3.750	3.75	10 : 10
5.625	3.75	15 : 10
2.50	0.75	10 : 3
2.50	1.50	10 : 6
2.50	2.25	10 : 9
2.50	3.00	10 : 12

2.3 XRF characterization

This study applied a PANalytical, type Minipal 4 XRF (X - Ray Fluorescence) Spectrophotometer to examine qualitatively and quantitatively chemical composition of tin tailing sample of the highest recovery of titanium using flotation route at varied concentration of sodium oleate and sodium chlorate.

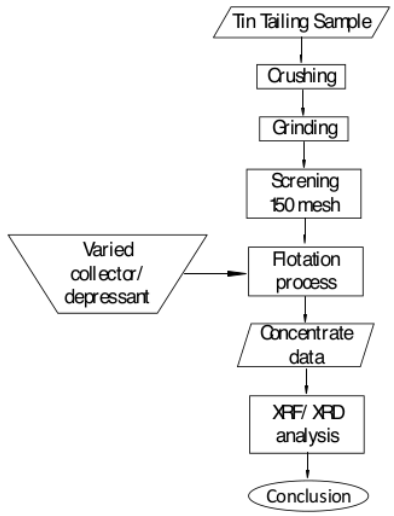

Figure 3 Flow chart of this study

2.4 XRD characterization

This study applied PANalyticalX'pert PRO XRD with CuKα incident radiation (1.54060 A), from a Cu anode operated at 40 kV and 30 mA for XRD characterization.. The XRD patterns of samples were conducted in continuous mode at a speed of 0.02°/min. in the range of 2θ 10° – 90°.

3. Results and Discussion

3.1. Froth Flotation

Table 2 shows the results of titanium concentration in the concentrate related to the varied ratio of sodium chlorate depressant and sodium oleate frother/collector at pH 7.0. The titanium concentration was determined by XRF.

Table 2. Results of titanium content (%) by XRF. pH 7.0. 150 mesh.

Varied of ratio NaClO3 and Na-oleate	Titanium concentration (%)
1 : 10	1.52
5 : 10	0.99
10 : 10	1.18
15 : 10	0.42
10 : 3	1.14
10 : 6	1.28
10 : 9	1.07
10 : 12	2.03

Data of Table 2 were used in bar chart (Figure. 4) based on constant mass of sodium oleate frother/collector and varied sodium chlorate depressant.

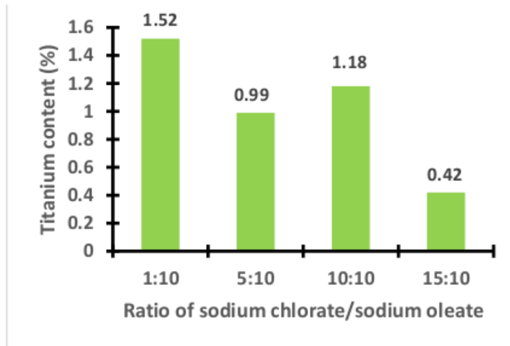

Figure 4. Titanium concentration based on ratio sodium chlorate depressant/ sodium oleate collector in constant mass collector. pH 7.0.

Figure 4 reveals that increased sodium chlorate depressant yielding decreased titanium concentration in the concentrate. This finding is due to weaken hydrophobic interaction between sodium oleate frother and the titanium resulting decreased titanium concentration in the concentrate. Flotation method involved with the concept of “like dissolve like”. Sodium oleate has dominantly non polar character or hydrophobic, on the other hand, sodium chlorate has dominantly polar character or hydrophilic. The principle method of froth flotation is to attract the desired mineral together with the frother go up to solution surface, on the other hand, the depressant polar behavior attracted impurities to go down to the flotation cell bottom, and therefore, the separation of the desired mineral and the impurities can be achieved. In relation to this phenomenon, Amalia [17] investigated hydrophobic and hydrophilic character in rare earth element separation applying active carbon/palmitic acid collector.

On the other hand, Fig. 5 reveals that increased sodium oleate frother yielding increased titanium concentration in the collector. This finding is due to stronger hydrophobic interaction between sodium oleate frother and the titanium resulting increased titanium concentration in the collector.

The separation process in flotation route is based on weaker or stronger hydrophobic or hydrophilic attraction leading to polar – polar attraction or non polar – non polar attraction. In this case, sodium oleate frother/collector is assumed to have more non polar character, on the other hand, sodium chlorate depressant is assumed to have more polar character. In this matter, the degree interaction between titanium and frother or between titanium and depressant is influenced by the mass concentration of frother and depressant. In this case, sodium oleate has two functions, i.e. as frother and collector. Many other flotation works applied separated frother and collector chemical agent [12 – 14].

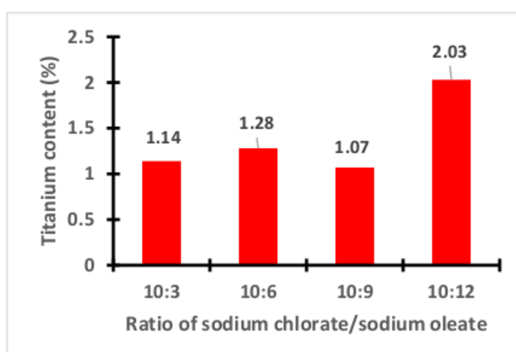

Figure 5 Titanium concentration based on ratio sodium chlorate depressant/ sodium oleate collector in constant mass depressant pH 7.0.

3.2. Titanium Recovery Equations

The general formula for mineral recovery is described as follows: 143

$$R = \frac{C\,.\,c}{F\,.\,f}\ x\ 100\% \qquad (1)$$

R = recovery
C = concentrate mass (g)
c = concentrate concentration (%)
F = feed mass (g)
f = feed concentration (%)

For titanium recovery,

C = concentrate at given ratio of depressant/collector (g)
c = concentration of titanium at given ratio of depressant/collector (%)
F = feed of initial sample (250 g)
f = concentration of titanium at initial sample (0.50 %)
Note: (i) the concentrate at given ratio of depressant/collector obtained from material balance (C) as stated in equation (2) below; (ii) data of material balance are not included here; (iii) the concentration of titanium at given ratio of depressant/collector (c) obtained from data Table 2.

Material balance:

$$F - C + D \qquad (2)$$

F = Feed; C = Concentrate; D = Depressant

Example:

1. The titanium recovery (R) of ratio depressant/ collector (10:10) from Table 2 using equations (1) and (2) is described as follows:

$$R = \frac{(48.22\text{g x } 1.18\%)}{(250g\ x\ 0.50\%)} = 45.51\% \qquad (3)$$

2. The titanium recovery (R) of ratio depressant/ collector (10:12) from Table 2 using equations (1) and (2) is described as follows:

$$R = \frac{(27.59\text{g x } 2.03\%)}{(250g\ x\ 0.50\%)} = 44.80\% \qquad (4)$$

Having used similar way as described in equations (3) and (4) for other varied ratio of sodium chlorate depressant and sodium oleate collector, it yielded data of titanium recovery (Table 3).

With regard to Table 3, data of titanium concentration (%) obtained from XRF measurements (Table 2), while data of titanium recovery (%) obtained by calculations related to equations (1) – (4). Addressing to titanium recovery (Table 3), the optimum titanium result (45.51%) related to the highest titanium recovery was obtained at mass ratio of depressant and collector, i.e. 10 : 10, on the other hand, the optimum result achieved at the same mass of depressant and collector, respectively, i.e. 3.75 g (as stated in Table 1). 144

Table 3. Results of titanium recovery (%). pH 7.0. 150 mesh. 145

Ratio of depressant /collector	Titanium concentration (%)	Titanium recovery (%)
1 : 10	1.52	27.12
5 : 10	0.99	39.49
10 : 10	1.18	45.51
15 : 10	0.42	39.88
10 : 3	1.14	43.45
10 : 6	1.28	30.68
10 : 9	1.07	32.82
10 : 12	2.03	44.80

It is not surprising, the equal quantity of depressant and collector at given mass value attracted equally to titanium in froth solution at neutral pH (pH 7.0), however, sodium oleate with long organic chain showed a little dominant to trap titanium in foam solution in order to reduce energy of surface tension.

3.3. XRF Results

Fig. 6 shows the XRF presentation of initial tin tailing sample. The presence of titanium peak indicated by the red arrow in the figure.

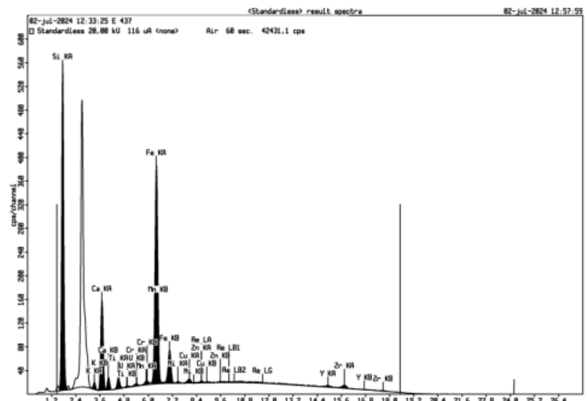

Figure 6 XRF presentation of tin tailing sample from Bangka Island

Table 4 reveals the elements found in the initial tin tailing sample with silicon (83.80 %), calcium (6.68 %) , and iron (5.28 %) as mayor elements. Addressing to high content of silicon, the sample is attributed to quartz mineral sample. Table 4 also shows little amount of titanium (0.50 %). Since titanium found in ilmenite and rutile minerals, Dahani [1] reported high grade ilmenite mineral (21.36 %) in Bangka tin tailing as located at the same land of this study taken sampling. The zircon (2.10 %) was also detected in this work attributed to zircon mineral as Dahani [1] reported 5.85 % zircon in previous study. Moreover, this study also reported the existence of little amount of transition elements, i.e. yttrium (0.48 %) and rhenium (0.10 %), however, the existence of rare earth elements was not detected.

Table 4. Elements found in initial tin tailing sample. XRF analysis.

Element	Concentr. (%)
silicon	83.80
calcium	0.67
potassium	6.68
titanium	**0.50**
vanadium	0.01
chromium	0.08
manganese	0.11
iron	5.28
nickel	0.04
copper	0.10
zinc	0.01
yttrium	0.48
zircon	2.10
rhenium	0.10

3.4. XRD Results

Fig. 7 reveals the results of XRD analysis addressing to some minerals presence in the initial tin tailing sample with respect to geological names, i.e. biotite identified as silicate mineral, nafertisite referred to iron sand, aenigmatite referred to mineral containing sodium, iron, titanium, and silicon, chondrodite containing calcium and manganese, and titanite containing titanium in calcium silicate. The presence of the characteristic minerals is supported by XRF analysis as already reported in Table 4. Some minerals were formed in igneous and metamorphic rocks in relation to sedimentary deposits caused by geochemical reactions related to migration, weathering and corrosion process occurred in very long time.

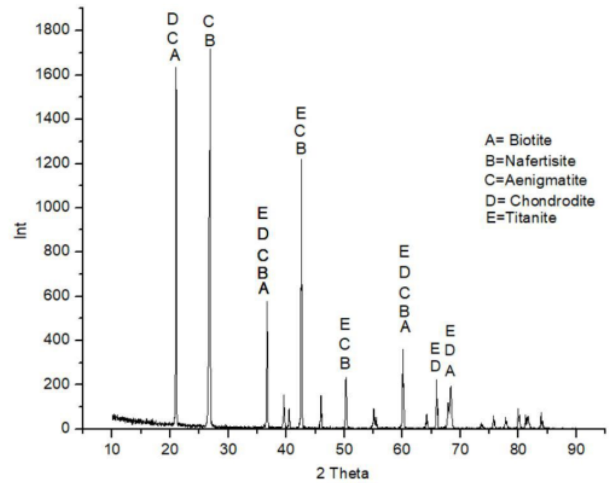

Figure 7 XRD presentation of tin tailing sample from Bangka Island**.**

4. Conclusions

The study gives new finding in relation to ratio effects of sodium chlorate depressant and sodium oleate frother/collector applying flotation route on titanium analysis from tin tailing. The optimum result of titanium recovery (45.51%) was achieved at equal mass amount of depressant and frother/collector using 3.75 g, respectively, at 15 min. flotation time and pH 7.0. The XRF characterization revealed that the interested mineral containing titanium in dominantly silicon and calcium minerals. The XRD characterization showed that the mineral mostly attributed to quartz mineral with the small amount of rutile and ilmenite. In addition, the application of low cost chemicals gives another benefit. Future insight gives valuable promising related to titanium recovery using more variables such as particle size, pH, and flotation time, as well as modified synthetic frother or depressant to improve the results. In mineral dressing, flotation method is integrated with the application of carbon adsorbent to enhance the recovery of interested mineral. Many investigations started to apply natural products such as nano carbon from bamboo shell and sodium palmitate derived from crude palm oil on account of economic reason.

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