Journal of Metallurgical Engineering and Processing Technology, Vol. 5, No. 2
February, 2025
P-ISSN: 2723-6854, E-ISSN: 2798-1037, page 121-134
DOI: https://doi.org/10.31315/jmept

Analysis of the Effect of Increasing Sn^{2+} Content in the Electrolyte Solution from Leaching on the Recovery of the Electrorefining Process at PT. Timah Tbk

Andeliani Saputri[1], Desi Cahyani[2], Muhammad Rifqi Ihsanuddin[3]
[1]Universitas Pembangunan Nasional "Veteran" Yogyakarta, Indonesia, [2]PT Timah Tbk, Indonesia
[3]Bachelor's Program in Metallurgical Engineering, Faculty of Mineral Technology, Yogyakarta
116220003@student.upnyk.ac.id[1], 116220007@student.upnyk.ac.id[2], 116220071@student.upnyk.ac.id[3]

Received 16/06/2025; Revised 00/00/0000; Published; 00/00/0000

Abstract

Iron tin in this study is one of the by-products from the flame oven. This iron tin undergoes testing, yielding Sn content of 40.85% and Fe content of 31.32%. Tin Crystal is a product of pyrorefining, a process where tin is separated from various impurities such as As, Ni, Pb, Fe, among others. The study involves leaching process using 37% HCl solvent with varied volumes. From the leaching process, $SnCl_2$ is obtained containing Sn^{2+}. This solution undergoes titration to determine the Sn content. Subsequently, $SnCl_2$ solution is added to an initial electrolyte solution containing 22 g/l to increase Sn^{2+} concentration. Electrorefining experiments were conducted using electrolyte solutions: PT Timah Tbk electrolyte, electrolyte with leaching solution 1, electrolyte with leaching solutions 1 and 2, and electrolyte with Tin Crystal leaching solution. Conditions included fixed parameters: voltage at 0.24V, experiment duration of 8 hours with hourly checks. Electrorefining process mirrored PT Timah Tbk's refining process, optimizing with 0.24V voltage for best results. Research findings showed leaching process produces $SnCl_2$ solutions containing 80 to 130 g/l for iron tin (FeSn) samples and 150 g/l for Tin Crystal samples. Electrorefining yielded products of varying weights depending on Sn^{2+} concentration in the electrolyte. Quality differences were significant, suggesting the need for optimization towards superior products.

Keywords: Iron tin, Tin Cristal, Electrorifining, Leaching

Introduction

The mining industry is one of the sectors that contributes significantly to Indonesia's market capitalization growth. The total contribution of the mining industry to

P-ISSN: 2723-6854, E-ISSN: 2798-1037, page 121-134
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the Gross Domestic Product (GDP) reaches 7.2%. The GDP generated from the mining industry in Indonesia reaches $13.8 million and is the highest in Southeast Asia (Irzon, 2021). Indonesia has a long history of tin mining and is currently the second-largest tin producer in the world after China. The presence of tin also has an impact on the economic and social conditions of the community. PT. Timah Tbk is currently the largest company engaged in exploration, exploitation, and processing in the Indonesian Tin Islands (Estefania et al., 2021).

95% of tin production in Indonesia is exported as pure tin in the form of ingots. Derivative tin products with higher economic value are rarely produced domestically. Therefore, the government imposed a ban on the export of pure tin in June 2023 in accordance with Law No. 3 of 2020 to encourage tin down streaming. The policy of banning the export of raw tin needs to be accompanied by mastery of the technology for down streaming tin products. One of the largest tin producers in Indonesia is PT. Timah Tbk(Alia Badra Pitaloka et al., 2023), a state-owned enterprise (BUMN) engaged in tin mining and listed on the the Indonesia Stock Exchange since 1995. The company's headquarters are located in Pangkal pinang, Bangka Belitung Province, and it operates in the provinces of Bangka Belitung Islands, Riau, South Kalimantan, Southeast Sulawesi, and Cilegon, Banten (Bayu Muhammad Ilham & Fadhilah, 2021).

Tin is a chemical element with the compound name Sn with element number 50. Tin is a metal with low hardness, easy to form, and quite resistant to corrosion. Tin is usually obtained through the mineral cassiterite which is in the form of tin oxide (SNO2). Tin is usually found in primary deposits in granite rocks in metamorphic deposit areas, alluvial and elluvial deposits (Muh Anhar & Betti Ses Eka Polonia, 2020)

Iron-tin (FeSn) compounds produced through the flame oven process are currently not further processed to reach optimal quality. This process results in products with suboptimal tin content, and there remains untapped potential for further purification. One important step in tin purification is the electrorefining process, which aims to improve the purity of tin metal. However, in the current electrorefining process, the electrolyte solution used still contains Sn^{2+} ions at a concentration of around 22 g/l, which is significantly lower than the target standard of 40 g/l. Lower Sn^{2+} concentrations can affect the efficiency of the purification process, resulting in suboptimal final tin product quality.

Therefore, in this study, experiments were conducted to improve the efficiency of the leaching process on FeSn samples using HCl. The leaching process is expected to increase the concentration of Sn^{2+} ions in the electrolyte solution, helping it meet the required standard. In addition, electrorefining tests using electrolyte solutions with higher Sn^{2+} concentrations were also carried out to evaluate their effect on the quality of the tin product. Through this approach, the study aims to find solutions to improve the efficiency and quality of the tin electrorefining process.

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Research Methods

In this study, two important experimental processes were carried out: the Leaching process and the Electrorefining process. The Leaching process involves the extraction of metals from impurities through dissolution using an appropriate solvent. In this context, 37% HCl was used as the solvent reagent to dissolve tin minerals from cassiterite ore (SnO), resulting in Sn^{2+} ions in the form of $SnCl_2$ solution. The Electrorefining process is a metal purification stage using electric current to obtain pure tin. At PT Timah Tbk, the electrorefining process is conducted in an electrolytic cell using crude tin as the anode and tin crystal as the cathode.

For this research, various tools and materials were used to support the experimental process. The equipment used included a laboratory-scale electrorefining cell, power supply, digital scale, disc mill backlog, muffle furnace, beakers, measuring cylinders, and titration apparatus. The materials used in this study included iron-tin (FeSn), distilled water, tin crystal, 34% HCl solution, H_2O_2 reagent, NaOH reagent, cathode, anode, and emulsion.

This research process was divided into two stages: the first stage was the leaching process, and the second stage was the electrorefining process. The leaching process began with the preparation of the equipment and materials to be used. This preparation included sampling FeSn and tin crystal samples, preparing a 34% HCl solution, weighing the FeSn and tin crystal samples, and grinding some of the FeSn samples.Next, twelve leaching experiments were conducted six for the FeSn samples and six for the tin crystal samples. The experiments were carried out by varying the molarity, temperature, and duration of the process. After the leaching process, a concentration test was performed on each result using iodometric titration with KIO_3 reagent (0.08N). The results of the concentration tests were then analyzed and used as a basis for consideration in the next stage of the process.

After the leaching process, the electrorefining process was carried out, beginning with the preparation of the experimental tools and materials. This preparation included the fabrication of the anode and cathode, preparation and measurement of the electrorefining cell tank, and homogenization of the electrolyte solution.Subsequently, four electrorefining experiments were conducted: one experiment using a sample from PT Timah, two experiments using FeSn samples, and one experiment using a tin crystal sample. The experiments were performed using constant variables, namely a current of 0.24V and a duration of 8 hours, while the independent variable was the variation in Sn concentration in each electrolyte solution.
After obtaining the cathode from the experiment, melting and casting were performed, followed by a content analysis of the electrorefining product. The content analysis of the electrorefining product was carried out using the Spark ARL Spectroscopy instrument.

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After obtaining the composition data of the electrorefining products, the process producing the best product was then determined.

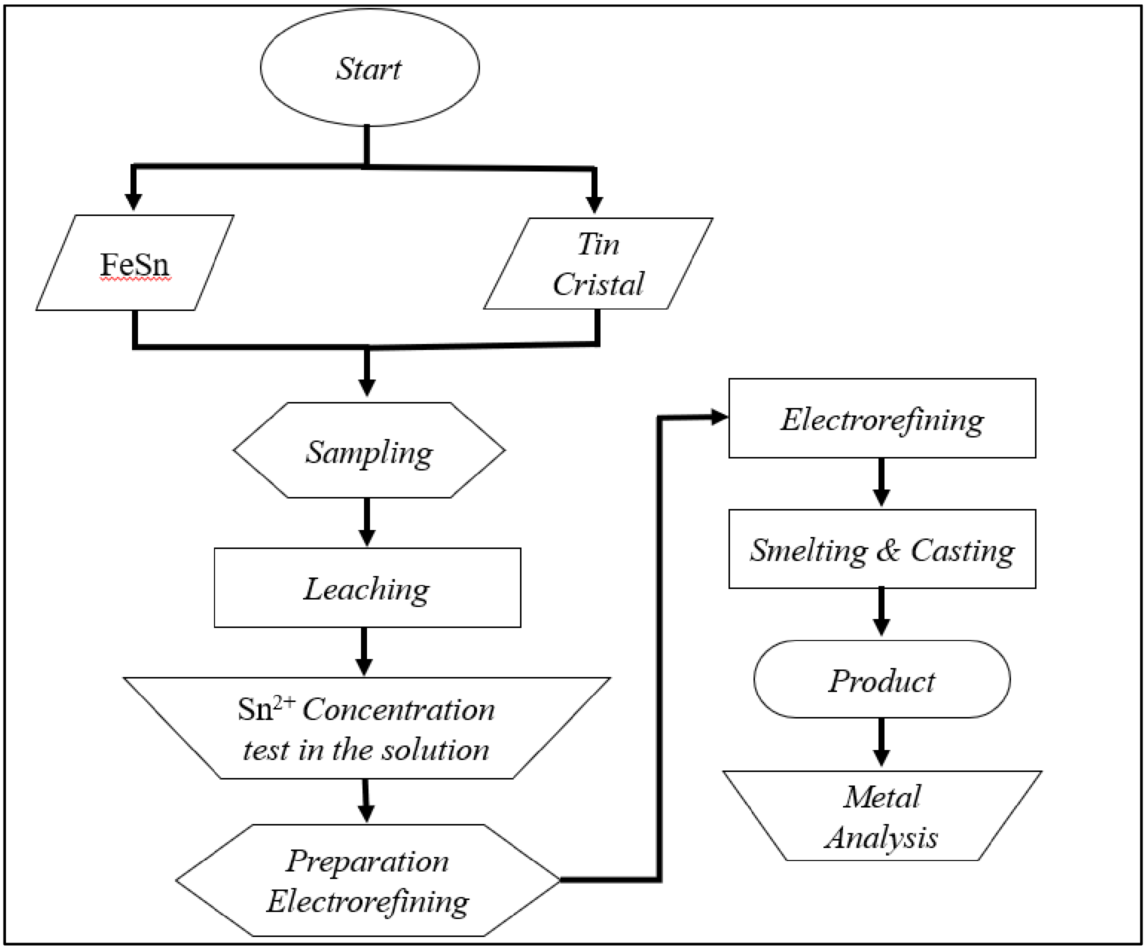

Figure 1 Research flow diagram

Result and Discussion

Results of Research

Based on the experiments in this study, the following are experimental data for the leaching and electrorefining processes. The leaching process itself was carried out 12 times and the electrorefining process was carried out 4 times.

Table 1. Leaching Process Data

Testing	Sample	HCl (ml)	Aquadest (ml)	H_2O_2 (ml)	Information	Time
1	FeSn 1kg	1,200	300	-	Not Heated	1 H

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2	FeSn 1kg	800	400	-	Not Heated	4 H
3	FeSn 1kg	600	200	-	Not Heated	2.5 D
4	FeSn 1kg	800	50	30	Not Heated	12 H
5	FeSn 1kg	500	50	25	Heated	1 H
6	FeSn 1kg	300	150	30	Heated	1 H
7	*Tin crystal* 0,025 kg	125	20	5	Not Heated	5 H
8	*Tin crystal* 0,023 kg	75	100	30	Not Heated	5 H
9	*Tin crystal* 0,5 kg	1000	50	5	Heated	5 H
10	*Tin crystal* 0,5 kg	500	50	5	Not Heated	5 H
11	*Tin crystal* 0,5 kg	200	50	5	Not Heated	5 H
12	*Tin crystal* 0,5 kg	200	50	5	Not Heated	12 H

After the leaching process, the electrorefining process is carried out, where the electrorefining process data carried out is as follows.

Table 2. Electrorefining Process Data

No	Volt	Ampere	Mass Teoritis
1	0.24	4.65	10.322
2	0,24	5.32	11.809
3	0,24	6.87	15.249
4	0,24	6.45	14.317
5	0,24	6.88	15.271
6	0,24	6.88	15.271
7	0,24	6.89	15.294
8	0,24	6.84	15.183
			112.716

For the levels of Sn^{2+} contained in the electrorefining experiment solution

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itself, including:

Table 3.

No	Testing	Levels	Information
1	Electrolyte solution 1	22,06	Before Experiment Electrorefining
	Electrolyte solution 1	21,83	After Experiment Electrorefining
2	Electrolyte solution 2 + Sample 3	26,7	Before Experiment Electrorefining
	Electrolyte solution 2 + Sample 3	27,07	After Experiment Electrorefining
3	Electrolyte solution 3 + sample 1	30,87	Before Experiment Electrorefining
	Electrolyte solution 3 + sample 1	30,90	After Experiment Electrorefining
4	Electrolyte solution 4 + Sample Tin crystal	37,12	Before Experiment Electrorefining
	Electrolyte solution 4 + Sample Tin crystal	34,46	After Experiment Electrorefining

Based on the experiments that have been carried out, then a content test was carried out for the results of the leaching process for FeSn and tin crystal samples. The test was carried out using iodometric titration using 0.08% KIO3 reagent. For the results of the electrorefining process itself, it was tested using a spectroscopy tool in the form of SPARK ARL 3460. Then from the test results, a re-examination was carried out to obtain a discussion of this study. In the leaching process, there are 4 key factors that affect the efficiency of the process:

1. Particle Size, Optimal particle size enlarges the contact surface area and accelerates mass transfer. However, too fine particles can cause problems in circulation and dregs removal.
2. Solvent, Choose a solvent that is effective, does not damage the solute or

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residue, and has a low viscosity to facilitate circulation.
3. Operating Temperature, Higher temperatures can increase solubility and diffusion, thereby accelerating extraction. However, it is necessary to avoid excessively high temperatures that may damage the material.
4. Stirring, Sufficient stirring can increase diffusivity and mass transfer speed, and prevent precipitation.

Based on the research of which aims to increase the recovery of tin (Sn) ore from mining waste with a SnO2 content of 3.6%. Based on the research of (Elbert et al., 1964) which aims to increase the recovery of tin (Sn) ore from mining waste with a SnO2 content of 3.6%. The extraction method used a 5-15%wt HCl solution with a liquid-solids ratio of 5/1-7/1. The results showed 80-90% of tin ore was extracted. Operating conditions included solution temperature of 60-105°C and leaching time of 15-180 minutes. Analysis showed that HCl concentration and leaching time affected the extraction percentage, but not significantly.

1. **Analysis of leaching process**

The experiment was conducted by leaching using HCL reagent with a concentration of 34%. The use of HCl as a solvent reagent is because the chloride compounds in HCl can bind with sn to form Sn^{2+} ions in $SnCL_2$ solution. the overall reaction that occurs as follows :

$$SnO_2 + 2HCl \rightarrow SnCl + H_2O_2$$

However, the presence of Fe can also bind to chloride compounds that form FeCl, therefore this must be considered in the next process. In this experiment, the leaching process was carried out with HCl variations of 1 liter, 800 ml, 600 ml, and 400 ml for iron tin (FeSn) samples. Then experiments were carried out with HCl variations of 1 liter, 500 ml, and 200 ml. To optimize this leaching process, some HCl solutions were heated at 80oC and H2O2 was added. for data from the leaching process can be seen in table 1.

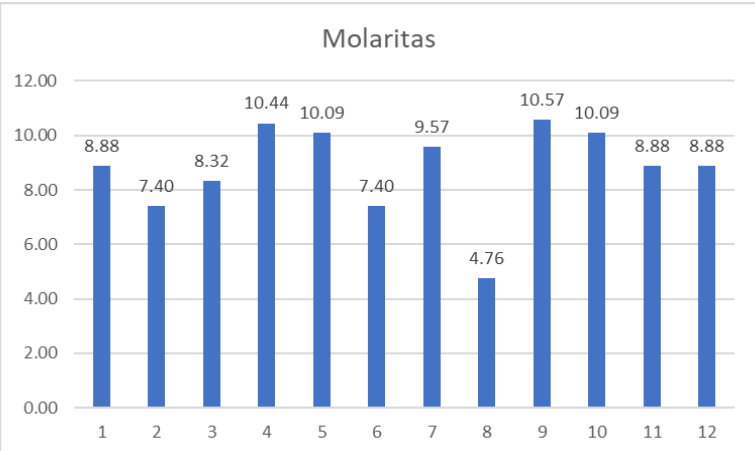

Figure 2. Graph Mole Solution

Figure 2 shows the molarity graph of 12 leaching solution samples where for experiments 1-6 using FeSn samples and experiments 7-12 using Tin Crystal samples. The

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leaching results showed that the highest molarity range was reached in sample 9 at 10.57 mol/L, the lowest molarity in sample 8 at 4.76 mol/L and the optimal molarity ranged from 8-9 mol/L in samples 1, 3, 11 and 12. ini. Process time and temperature are not a reference in this leaching process. Instead, these two specs will affect the saturation speed of the HCl solution used. Higher temperatures will accelerate the reaction rate so that the HCl solution will also saturate faster.

2. Analysis of Electrorefining Process

The experiment was conducted with an electrorefining process for 8 hours with sampling at intervals of every 1 hour. After sampling, iodometric titration was carried out to determine the levels of Sn2+ still present in the solution. After the process is complete, the cathode of the electrorefining product will then be melted and then the spectroscopy testing process will be carried out using SPARK ARL 3460.

From the tests that have been carried out in this experiment, it is then used to determine the increase in current efficiency based on the increase in product weight. Current efficiency itself is assessed based on the comparison between actual weight and theoretical weight. The following is data related to actual weight, theoretical weight, and a graph of the increase in current efficiency.

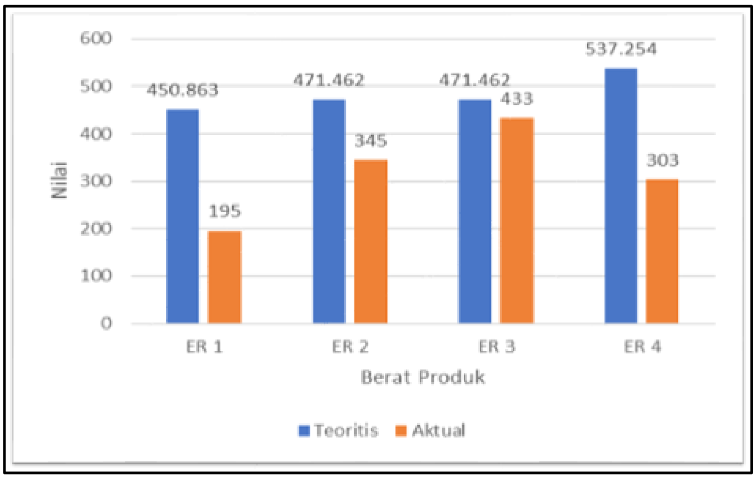

Figure 4. Comparison of Theoritical Weight and Actual Weight

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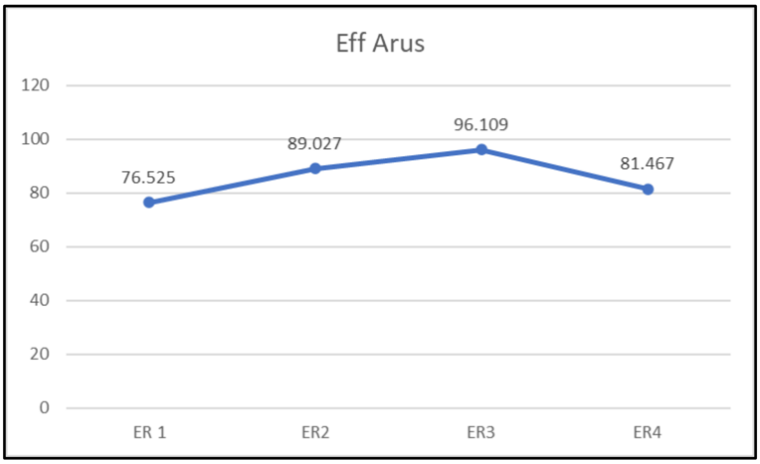

Figure 5. Flow Efficiency Data

Based on figure 5 shows an increase in actual weight in each electrorefining process. For the experiment with the highest actual weight, namely in the third process with the highest increase in Sn2+ levels. while for the comparison related to electrorefining experiments with the addition of FeSn samples and tin crystal samples can be seen in experiments 2 and 4. Where the increase in product weight with the addition of FeSn samples is higher than the experiment with the addition of tin crystals.

Next, from Figure 6, the current efficiency graph shows an increase up to experiment three and decreases in experiment four. This is because from experiments 1 to 3, the actual weight is much closer to the theoretical weight. While in experiment 4, the actual weight decreases, causing the comparison with the theoretical weight to be further away. From the graph, it can be seen that experiment 3 is the most efficient experiment.

3. Analysis Product

Based on the research results, after the electrorefining process is carried out, the next stage is tested using SPARK ARL 3460 to identify the content of impurities that are still present in the Product. SPARK ARL 3460 results can be seen in table 4.

Table 4. Impurity data from the electrorefining process

No	Elements	Sample I (ppm)	Sample II (ppm)	Sample III (ppm)	Sample IV (ppm)
1	Fe	1	45	34	3
2	As	1	34	15	5
3	Pb	16	18	16	19
4	Cu	14	20	18	14
5	Sb	10	10	11	10

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6	Ni	2	5	4	4
7	Bi	1	1	1	1
8	Co	1	1	1	1
9	Cd	1	1	1	1
10	Zn	1	1	1	3
11	Al	1	1	1	0
12	Ag	1	1	1	1
13	In	2	16	11	9

Analysis of impurities in sample I products which are electrorefining processes using pt tin electrolyte solutions which are then compared to the addition of leaching solutions that have been carried out in samples II, III, and IV. These results show that the highest increase in impurities is in the elements Fe, As, Pb, Cu, Sb, and In. The increase in Fe in the impurity content is due to the sample used FeSn which still contains a lot of Fe, then the increase in As, Pb, Cu and Sb is due to the sample used in the leaching process still contains these elements. So it is necessary to re-separate the results of the leaching process for optimization. While the increase in indium elements occurs due to the leaching process using HCL which results in the indium element being dissolved so that the indium element in the solution increases. The increase in indium in the solution causes indium impurities in the electrorefining product to increase (Fan et al., 2024).

Conclusion

Based on the results of the experiments that have been carried out, the following conclusions are obtained:

1. Based on the results of the research, the leaching process applied to FeSn samples produced electrolyte solutions with the highest levels of 4 g/l and 112.14 g/l. Meanwhile, the tin crystal sample produced an electrolyte solution with a level of 132.20 g/l.
2. To maximize the leaching process, optimization of several important factors is required. An increase in HCl molarity proved effective in accelerating the reaction rate and increasing efficiency. The use of H_2O_2 as a reducing agent also plays a role in accelerating the reaction rate by creating stronger oxidative conditions.
3. Based on the experimental results, it was found that there was an increase in the weight of the sample with the addition of leaching solution. This increase in weight indicates that the compound successfully dissolved and moved into the solution, thus increasing the concentration of Sn^{2+} in the electrolyte solution.
4. The electrorefning product using PT Timah electrolyte solution shows the best quality compared to other variations. This indicates that the composition of the electrolyte from PT Timah is optimal in supporting the metal refining process.

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However, in the experiment with the addition of leaching solution, there was a significant increase in Fe and As content in the final product.

Acknowledgements

The authors would like to thank the metallurgists at PT Timah tbk muntok unit for their guidance and direction during this research. Thanks also goes to the head of the processing and refinery division of PT tin tbk who has provided guidance so that this research can be carried out properly.

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