AIP Conference Proceedings

scitation.org/journal/apc

Volume 2668

# The 3rd International Conference on Mathematics and Sciences (The 3rd ICMSc)

## A Brighter Future with Tropical Innovation in the Application of Industry 4.0

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**East Kalimantan, Indonesia** • 12–13 October 2021

**Editors** • Rudy Agung Nugroho, Veliyana Londong Allo, Meiliyani Siringoringo, Surya Prangga, Wahidah, Rahmiati Munir and Irfan Ashari Hiyahara

The 3rd ICMSc

2021

FMIPA UNMUL

International Conference on Mathematics and Sciences

AIP Publishing

October 2022

THE 3RD INTERNATIONAL CONFERENCE ON MATHEMATICS AND SCIENCES (THE 3RD ICMSc): A Brighter Future with Tropical Innovation in the Application of Industry 4.0




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Conference date: 12–13 October 2021
Location: East Kalimantan, Indonesia
ISBN: 978-0-7354-4214-6
Editors: Rudy Agung Nugroho, Veliyana Londong Allo, Meiliyani Siringoringo, Surya Prangga, Wahidah, Rahmiati Munir and Irfan Ashari Hiyahara
Volume number: 2668
Published: Oct 11, 2022

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# Synthesis and characterization of $TiO_2$ from Lampung’s iron sand using acid leaching method with variation of HCl concentration

Cite as: AIP Conference Proceedings **2668**, 060003 (2022); https://doi.org/10.1063/5.0111689
Published Online: 11 October 2022

**Roniyus Marjunus, Adi Sucipto, Diah Purwarini, et al.**

# Synthesis and Characterization of $TiO_2$ from Lampung's Iron Sand Using Acid Leaching Method with Variation of HCl Concentration

Roniyus Marjunus[a)], Adi Sucipto, Diah Purwarini, and Iqbal Firdaus

*Department of Physics, Faculty of Mathematics and Natural Sciences, University of Lampung, Jl. Sumantri Brojonegoro No. 1, Bandar Lampung, Indonesia*

[a)] Corresponding author: roniyus.1977@fmipa.unila.ac.id

**Abstract.** This research has been done to determine the content of $TiO_2$ from iron sand of Lampung by the acid leaching method. Previously, iron sand was mixed with sodium hydrogen carbonate ($NaHCO_3$) and roasted at 700ºC for an hour. This research used five samples to determine the $TiO_2$ content of Lampung's iron sand, with variations in HCl concentration of 4, 6, 8, 10, and 12 M in leaching process for 2 hours at 70ºC and 110ºC. Then the water leaching process was carried out using 50 ml distilled water. Afterward, all samples were characterized using XRD and XRF to determine Lampung's iron sand content. The highest $TiO_2$ content of Lampung's iron sand in samples with 12 M of HCl concentration at leaching temperature 110 ºC i.e., 33,737%.

## INTRODUCTION

Indonesia has many irons sand on the south of Java, Borneo, Celebes island and Papua [1]. In Sumatera island, the iron sand can be found at Lampung Province, exactly at West Lampung Regency. According to Soepriadi et al. (2013), the amount of iron sand at West Lampung Regency reachs 1.773.480 ton. This massive volume of iron sand causes the research regarding iron sand has been developing. The main composition of the iron sand i.e., Fe and Ti, but it usually bound with other elements such oxygen, then forms ilmenite ($FeTiO_3$), hematite ($Fe_2O_3$), dan magnetite ($Fe_3O_4$) [1] and titanium dioxide ($TiO_2$).

$TiO_2$ has a variety of crystalline phases i.e, anatase, rutile, and brookite. Every phase shows different physical properties such as refractive index, chemistry reactivity and photochemistry reactivity [2]. The anatase phase can change to a rutile phase if heated until 500 – 600 ºC [2,3,4,6].

$TiO_2$ has several characteristics, including light, strong, non-flammable, does not cause toxic effects, has high biocompatibility, is resistant to high temperatures, corrosion, and is an inorganic salt [7-9]. Various advantages of $TiO_2$ cause many application of $TiO_2$, including heterogeneous catalysts (photocatalysts), solar cells [10], gas sensors, sunscreens, pigments in paints and cosmetic products, protective coatings corrosion, optical coatings, and electrical equipment.

Extraction of $TiO_2$ from iron sand has been done in several methods. One of them is hydrometallurgy [11]. Hydrometallurgy normally uses chloride acid solution or sulfate acid. The purpose of the acid is to remove the unwanted stuffs.

Based on the mentioned background, research related to the extraction of $TiO_2$ from natural iron sands needs to be carried out. The study will report how the effect of HCl concentration variation to the iron sand leaching process. Variations in the concentration of HCl that would be used in this study are 4 M, 6 M, 8 M, and 10 M at a temperature of 70 ºC, and 110 ºC. In this study, leaching with HCl will also be carried out twice, according to Setiawati's research

*The 3rd International Conference on Mathematics and Sciences (The 3rd ICMSc)*
AIP Conf. Proc. 2668, 060003-1–060003-7; https://doi.org/10.1063/5.0111689
Published by AIP Publishing. 978-0-7354-4214-6/$30.00

(2016). Oil bath was also used in order to uniform the solution temperature. The advantage of this research compared to the previous researches is to find the optimum concentration of HCl in order to get the high purity of $TiO_2$.

## METHOD

Iron sand was from the coast of West Lampung Province was washed with distilled water to remove impurities contained in the iron sand. After washing, the iron sand was then dried in an oven at 100 $^oC$ to dry. After drying, the iron sand was crushed with a mortar. The iron sand that has been pulverized was sieved with a mesh of 200 (70 microns). Referring to research Setiawati et al. (2013), $NaHCO_3$ was used as a reducing agent in the pyrometallurgical method, which will damage the ilmenite crystal structure in the iron sand so that iron compounds are more soluble in the leaching process. The iron sand which has been mixed with $NaHCO_3$, was then roasted at a temperature of 700 $^oC$. After the iron sand was roasted, it was leached using a HCl solution twice. Variations of the HCl concentration were 4 M, 6 M, 8 M, and 10 M at temperature 70 $^oC$, and 110 $^oC$. Then the samples were analyzed by X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) to be compared with iron ore sand before leaching and to determining the titanium purity.

## RESULTS AND DISCUSSION

The results of XRF characterization of iron sand leached at temperature of 70 $^oC$ can be seen in Table 1. The XRF results indicate that the $TiO_2$ contained in the iron sand before leaching was 13.808%, and the hematite ($Fe_2O_3$) content in the iron sand was 65.852%. After leaching with HCl, the $TiO_2$ content tends to increase, and the hematite content decreases. The highest $TiO_2$ was obtained at 10 M HCl of 28.787%.

**TABLE 1**. The XRF results of iron sand leaching HCl at 70 $^oC$.

| Compound | Unit | Iron Sand | Concentration of HCl | | | |
|---|---|---|---|---|---|---|
| | | | 4 M | 6 M | 8 M | 10 M |
| $SiO_2$ | % | 11.876 | 9.590 | 10.370 | 16.055 | 29.068 |
| $TiO_2$ | % | 13.808 | 17.791 | 19.567 | 27.971 | 28.787 |
| $Fe_2O_3$ | % | 65.852 | 66.076 | 63.346 | 47.420 | 33.864 |
| Rest | % | 8.464 | 6.543 | 6.717 | 8.554 | 8.281 |

If the results at Table 1 is plotted in a graph of HCl concentration ($C_{HCl}$) versus percentage of $TiO_2$ (%$TiO_2$) then as presented in (Fig. 1).

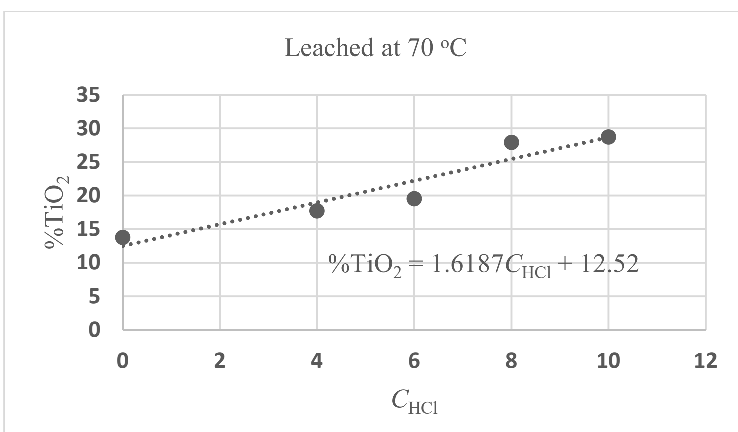


**FIGURE 1**. HCl concentration ($C_{HCl}$) versus percentage of $TiO_2$ (%$TiO_2$) at 70 $^oC$.

According to (Fig. 1), it can be seen that the linear dependency of $TiO_2$ percentage to the HCl concentration as revealed in Eq. (1),

$$\%TiO_2 = 1.6187C_{HCl} + 12.52. \quad (1)$$

Then based on XRD result of raw material and for the leached samples using 4 M, 6 M, 8 M, and 10 M of HCl at 70°C, as given in (Fig. 2), it is seen that brookite phase of $TiO_2$ is found in raw material. After leaching, rutile is produced. The existence of the rutile phase agrees with the XRF results, which shows that the purity of $TiO_2$ is increases as the increament of HCl concentration.

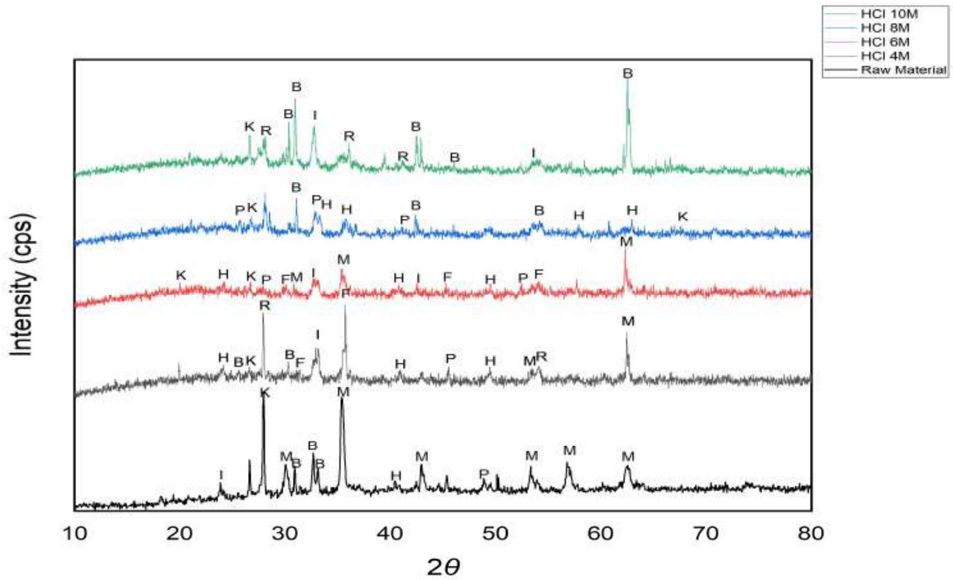


**FIGURE 2**. XRD result of iron sand before leaching (raw material) and after *leaching* for 2 hours at 70°C used 4 M, 6 M, 8 M and 10 M HCl, where K: Quartz, R: rutile, A: anatase, B: brookite, I: ilmenite, M: magnetite, m: maghemite, F: fayalite, H: hematite dan P: pseudobrookite.

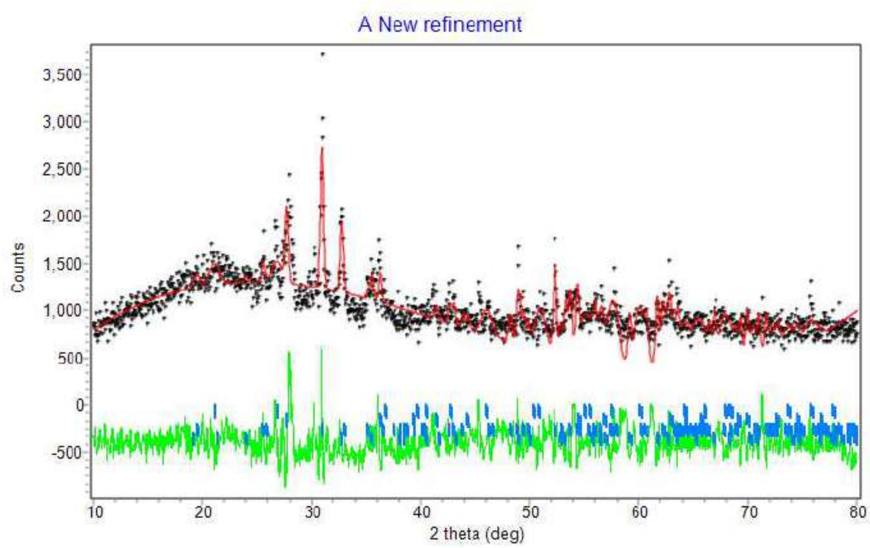


**FIGURE 3**. The refinement using Reietica of leached iron sand using 10 M HCl at 70 ºC

Based on the refinement by Reietica of leached iron sand using 10 M HCl at 70 ºC (Fig. 3), it is obtained the percentage $TiO_2$ phase (rutile and brookite) is revealed in Table 2. According to Table 2, the percentage of those $TiO_2$ phases is 42.73%.

**TABLE 2**. Phase percentage using 10 M HCl at 70 ºC.

| Phase | % |
|---|---|
| Quartz | 15.09 |
| Rutile | 8.34 |
| Brookite | 34.39 |
| Ilmenite | 42.19 |

Afterward, in order to complete this research at 70 ºC, leaching was also done at 110 ºC. The XRF result at 110 ºC can be seen in Table 3. According to Table 3, it shows that the percentage of $TiO_2$ is also increasing at 110 ºC as the raising of HCl concentration, as well as at 70 ºC (Table 1).

**TABLE 3**. The XRF results of iron sand leaching HCl at 110 ºC.

| Compound | Unit | Iron Sand | HCl Concentration | | | | |
|---|---|---|---|---|---|---|---|
| | | | 4 M | 6 M | 8 M | 10 M | 12 M |
| $SiO_2$ | % | 11.876 | 9.101 | 11.256 | 15.775 | 29.391 | 25.411 |
| $TiO_2$ | % | 13.808 | 17.833 | 19.294 | 27.999 | 28.036 | 33.737 |
| $Fe_2O_3$ | % | 65.852 | 66.967 | 62.617 | 47.798 | 34.112 | 33.429 |
| Sisa | % | 8.464 | 6.099 | 6.833 | 8.428 | 8.461 | 7.423 |

The results at Table 3 is plotted in a graph of HCl concentration ($C_{HCl}$) versus percentage of $TiO_2$ (%$TiO_2$) then as presented in (Fig. 1).

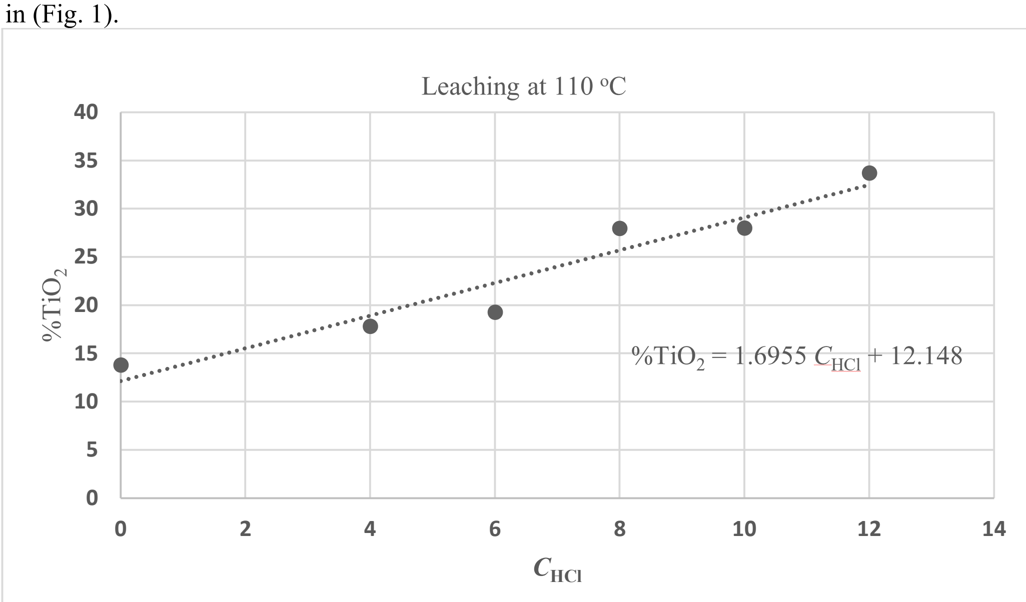


**FIGURE 4**. HCl concentration ($C_{HCl}$) versus percentage of $TiO_2$ (%$TiO_2$) at 70 ºC.

According to Fig. 4, it is presented that the linear dependency of $TiO_2$ percentage to the HCl concentration as revealed in Eq. (2),

$$\%TiO_2 = 1{,}6955\ C_{HCl} + 12{,}148. \quad (2)$$

If Eq. (2) is compared with Eq. (1), it can be concluded that the dependency of the $TiO_2$ purity to the HCl concentration is not influenced significantly because of the leaching temperature.

Then, based on XRD result of raw material and for the leached samples using 4 M, 6 M, 8 M, 10 M, and 12 M of HCl at 110°C, as given in Fig. 5, it is seen that rutile phase of $TiO_2$ is also found after leaching as at 110 C.

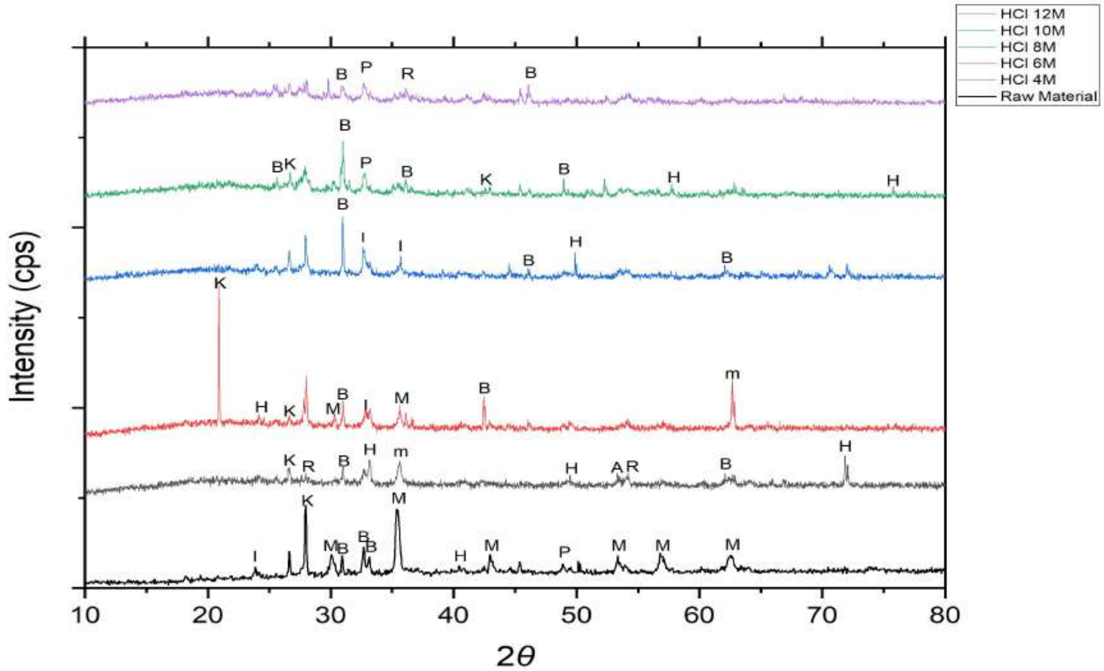


**FIGURE 5**. XRD result of iron sand before leaching (raw material) and after *leaching* for 2 hours at 110°C used 4 M, 6 M, 8 M, and 10 M HCl, where K: Quartz, R: rutile, A: anatase, B: brookite, I: ilmenite, M: magnetite, m: maghemite, F: fayalit, H: hematite dan P: pseudobrookite

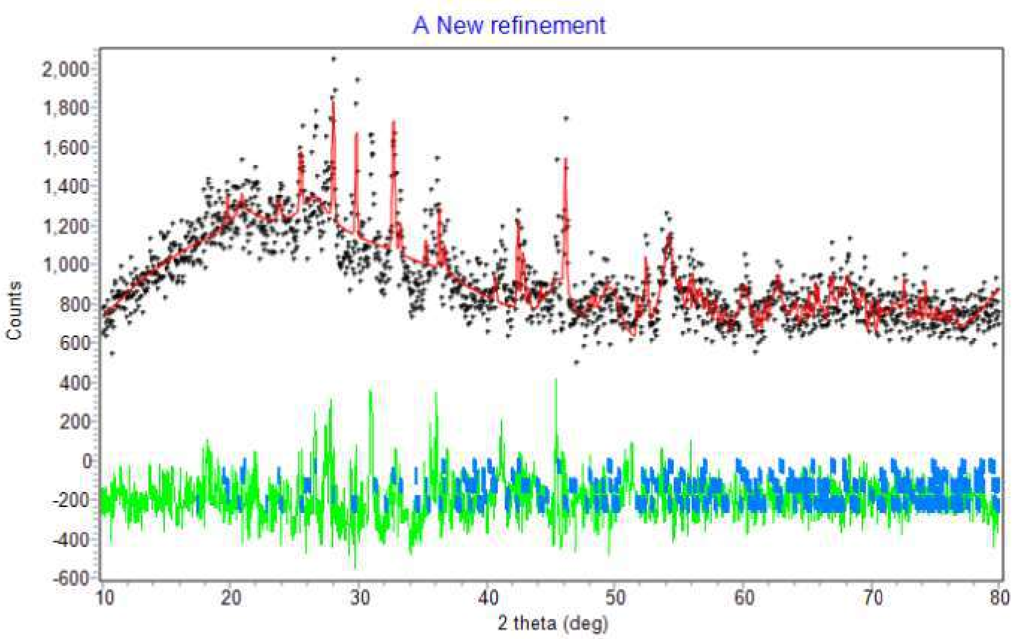


**FIGURE 6**. The refinement using Reietica of leached iron sand using 10 M HCl at 110 °C

Based on the refinement by Reietica of leached iron sand using 10 M HCl at 110 °C (Fig. 6), it is obtained the percentage $TiO_2$ phase (rutile and brookite) as revealed in Table 4.

**TABLE 4**. Phase percentage using 10 M HCl at 110 ºC.

| Phase | % |
|---|---|
| Quartz | 10.97 |
| Ilmenite | 31.03 |
| Brookite | 23.80 |
| Rutile | 5.75 |
| Fayalite | 28.44 |

The results of XRF analysis on Lampung's iron sand have the main compounds $Fe_2O_3$ and $TiO_2$ with relative percentages of 65.852% and 13.808%, respectively. The results of XRF analysis shows that the highest percentage of $TiO_2$ was found in HCl with a concentration of 12 M at 33.737%. $TiO_2$ extraction process using HCl was carried out to dissolve Fe (iron) in the $FeTiO_3$ structure and produce $TiO_2$ and was able to increase the percentage of $TiO_2$, the silicon element in $SiO_2$ was difficult to dissolve in the leaching process. HCl with concentration variations can dissolve various impurities and does not dissolve titanium so that the acid solution (HCl) can be refused.

From XRD pattern and the result of refinement, our result suggests that iron sand exhibits multiple phases, including titania. The rutile phase and brookite are formed when the iron sand is activated by thermal treatment [6]. Based on XRD analysis, the employed method, in general, has successfully improved $TiO_2$ content. However, impurity phases, such as $SiO_2$, remain in the obtained sample.

## CONCLUSIONS

In summary, variations in the concentration of HCl at temperature of 70 ºC and 110 ºC are able to increase the purity of $TiO_2$ i.e. 28.787% and 28.036% respectively, with variations of HCl 10 M. For variations of HCl 12 M at temperature 110ºC increases the amount of $TiO_2$ i.e. 33.737%. The formed phases are rutile and brookite. However, the impurity phases, such as iron oxide and silica, still presents in the sample.

## ACKNOWLEDGMENTS

The authors would like to thank the Grant of DIPA BLU University of Lampung Year 2021 that has funded this research.

## REFERENCES

1. L. D. Setiawati, T. P. Rahman, D. W. Nugroho, Nofrizal, R. Ikono , Suryandaru, Yuswono, Siswanto, N. T. Rochman. *Ekstraksi Titanium Dioksida ($TiO_2$) dari Pasir Besi dengan Metode Hidrometalurgi.* Proceeding of SEMIRATA Universitas Lampung, Lampung, 2013), 465-468.
2. D. He and F. Lin. Materials Letters **16**(16): 3385-3387 (2007).
3. M. Hirano, K. Ota and H. Iwata. Chemistry of Materials 16(19): 3725-3732 (2004).
4. J. Nair, P. Nair, F. Mizukami and Y. Oosawa. Materials Research Bulletin **34**(8): 1275-1290 (1999).
5. I. Firdaus, A. Stevani, Y.N. Handayani, N. Febriyanti, R. Marjunus, P. Manurung, *Jurnal Fisika dan Aplikasinya* **17** (2), (2021): 37 – 40.
6. A. M. Ruiz, G. Sakai, A. Kornet, K. Shimanoe, J. R. Morante and N. Yamazoe, Sensors and Actuators B: Chemical **103**(1-2): 312-317 (2004).
7. R. Ermawati, S. Naimah, and E. Ratnawati, *Jurnal Kimia dan Kemasan* **33**(2): 131-136 (2011).
8. L. Liang, K. Li, K. Lv and W. Ho. *Chinese Journal of Catalysis* **38**(12): 2085-2093 (2017).
9. X. Zou, S. Li, X. Lu, Q. Xu, C. Chen, S. Guo and Z. Zhou. Materials Transactions, **58**(3): 331–340 (2017).
10. J. H. Yum, S. S. Kim, D. Y. Kim and Y. E. Sung. *Electrophoretically deposited $TiO_2$ photo-electrodes for use in flexible dye-sensitized solar cells.* Journal of Photochemistry and Photobiology A: Chemistry **173**(1): 1-6 (2005).

11. R. Vasquez and A. Molina. *Leaching of Ilmenite and Pre-oxidized Ilmenite in Hydrochloric Acid to Obtain High Grade Titanium Dioxide.* (17th International Metallurgical & Materials Conference Proceedings-Metal. Venezuela, 2008),