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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

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

AIP Conference Proceedings



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2021

International Conference on Mathematics and Sciences



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



Table of Contents

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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

< PREV NEXT >



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Regularized quasi semigroups

Risky Yoga Suratman and Christiana Rini Indrati

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SNI standard freight software design with social platform media for cost efficiency and effectiveness for product advantages for cooperative found MSME products

Diena Noviarini and Eko Arif Syaefudin

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Effect of width, thickness, and length on static bending of red meranti (*Shorea sp.*)

Irvin Dayadi

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Synthesis and characterization of TiO₂ from Lampung's iron sand using acid leaching method with variation of HCl concentration

Roniyus Marjunus, Adi Sucipto, Diah Purwarini and Iqbal Firdaus

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Rani Chahyani and Abdul Manan

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

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Synthesis and Characterization of TiO₂ from Lampung's Iron Sand Using Acid Leaching Method with Variation of HCl Concentration

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Abstract. This research has been done to determine the content of TiO₂ from iron sand of Lampung by the acid leaching method. Previously, iron sand was mixed with sodium hydrogen carbonate (NaHCO₃) and roasted at 700°C for an hour. This research used five samples to determine the TiO₂ 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 70°C and 110°C. 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 TiO₂ content of Lampung's iron sand in samples with 12 M of HCl concentration at leaching temperature 110 °C 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 reaches 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 (FeTiO₃), hematite (Fe₂O₃), dan magnetite (Fe₃O₄) [1] and titanium dioxide (TiO₂).

TiO₂ 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 °C [2,3,4,6].

TiO₂ 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 TiO₂ cause many application of TiO₂, 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 TiO₂ 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 TiO₂ 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 °C, and 110 °C. In this study, leaching with HCl will also be carried out twice, according to Setiawati's research

(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 TiO₂.

METHOD

Iron sand 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 °C 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), NaHCO₃ 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 NaHCO₃, was then roasted at a temperature of 700 °C. 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 °C, and 110 °C. 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 °C can be seen in Table 1. The XRF results indicate that the TiO₂ contained in the iron sand before leaching was 13.808%, and the hematite (Fe₂O₃) content in the iron sand was 65.852%. After leaching with HCl, the TiO₂ content tends to increase, and the hematite content decreases. The highest TiO₂ was obtained at 10 M HCl of 28.787%.

TABLE 1. The XRF results of iron sand leaching HCl at 70 °C.

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 ₂ O 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 TiO₂ (%TiO₂) then as presented in (Fig. 1).

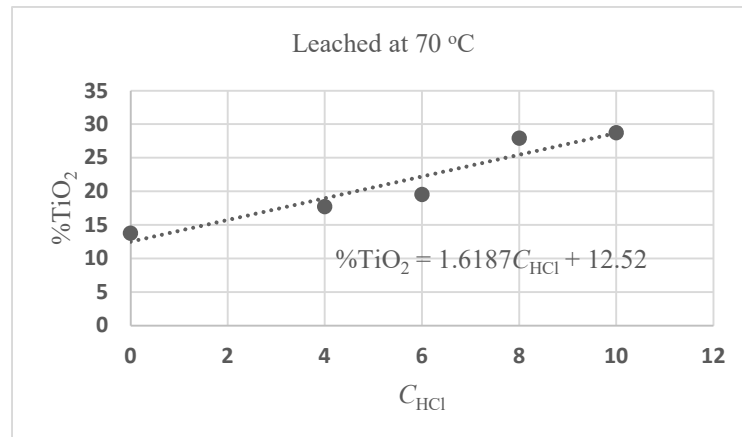


FIGURE 1. HCl concentration (C_{HCl}) versus percentage of TiO₂ (%TiO₂) at 70 °C.

According to (Fig. 1), it can be seen that the linear dependency of TiO₂ 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 TiO₂ 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 TiO₂ is increases as the increment of HCl concentration.

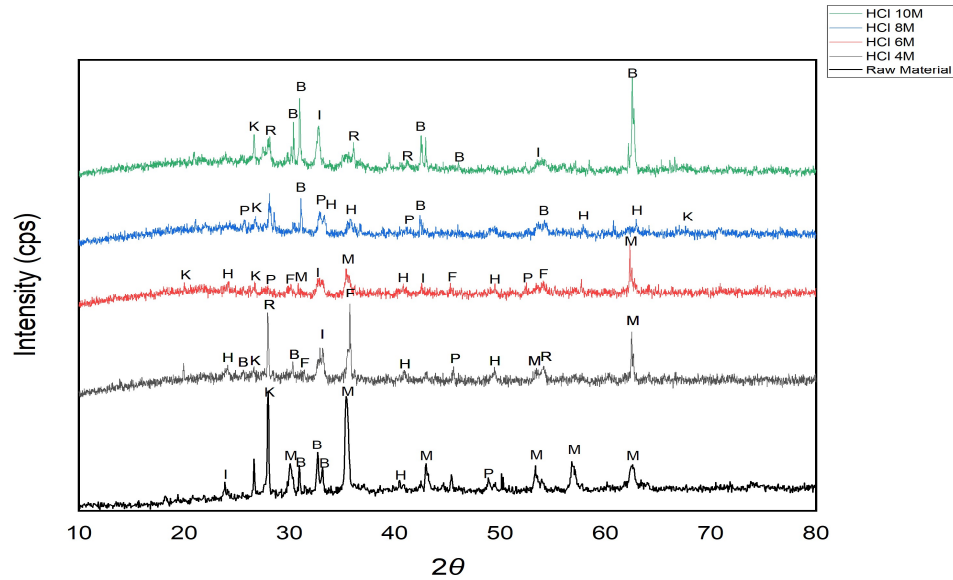


FIGURE 2. XRD result of iron sand before leaching (raw material) and after *leaching* for 2 hours at 70°C used 4 M, 6 M, 8 M and 10 M HCl, where K: Quartz, R: rutile, A: anatase, B: brookite, I: ilmenite, M: magnetite, m: maghemite, F: fayalite, H: hematite dan P: pseudobrookite.

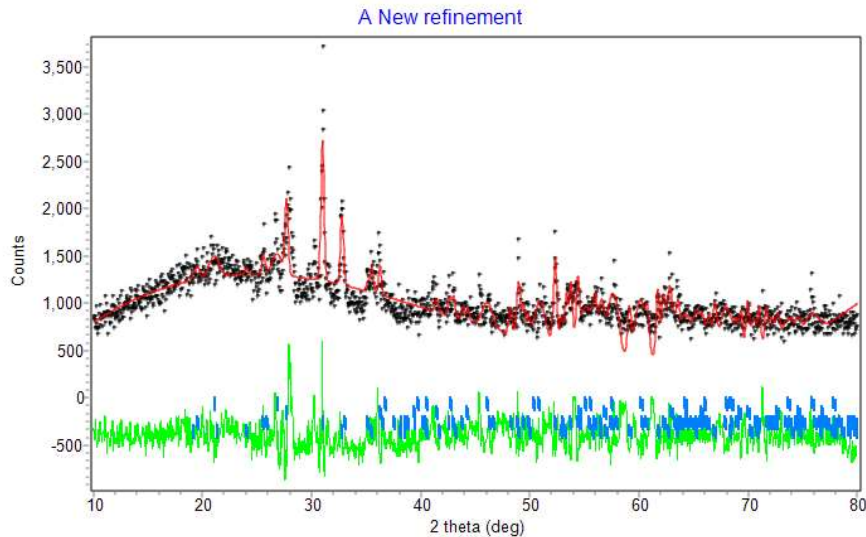


FIGURE 3. The refinement using Rietica of leached iron sand using 10 M HCl at 70 °C

Based on the refinement by Rietica of leached iron sand using 10 M HCl at 70 °C (Fig. 3), it is obtained the percentage TiO₂ phase (rutile and brookite) is revealed in Table 2. According to Table 2, the percentage of those TiO₂ phases is 42.73%.

TABLE 2. Phase percentage using 10 M HCl at 70 °C.

Phase	%
Quartz	15.09
Rutile	8.34
Brookite	34.39
Ilmenite	42.19

Afterward, in order to complete this research at 70 °C, leaching was also done at 110 °C. The XRF result at 110 °C can be seen in Table 3. According to Table 3, it shows that the percentage of TiO₂ is also increasing at 110 °C as the raising of HCl concentration, as well as at 70 °C (Table 1).

TABLE 3. The XRF results of iron sand leaching HCl at 110 °C.

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 ₂ O 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 TiO₂ (%TiO₂) then as presented in (Fig. 1).

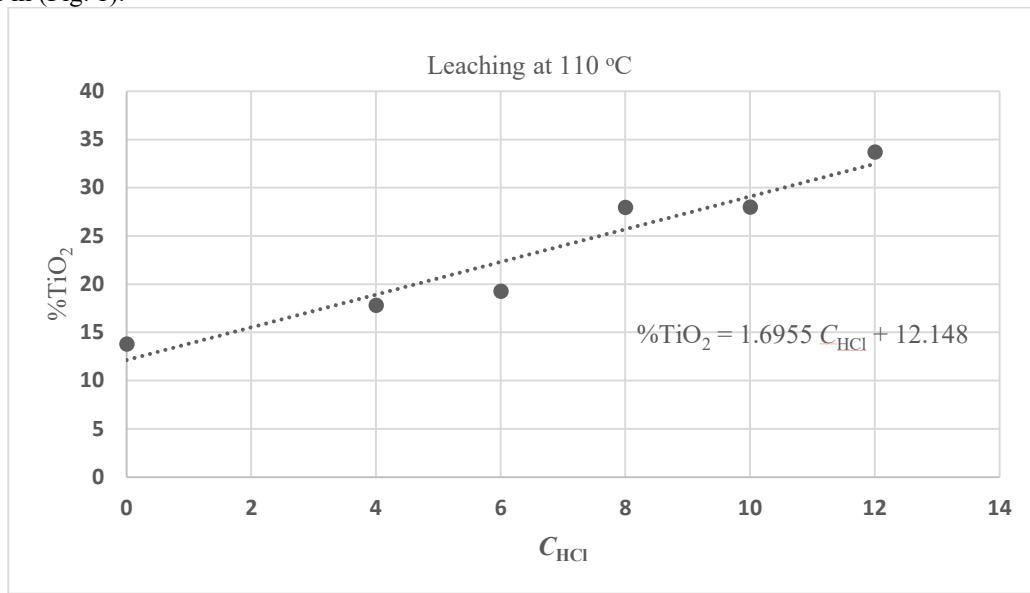


FIGURE 4. HCl concentration (C_{HCl}) versus percentage of TiO₂ (%TiO₂) at 70 °C.

According to Fig. 4, it is presented that the linear dependency of TiO₂ 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 TiO₂ 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 TiO₂ is also found after leaching as at 110 C.

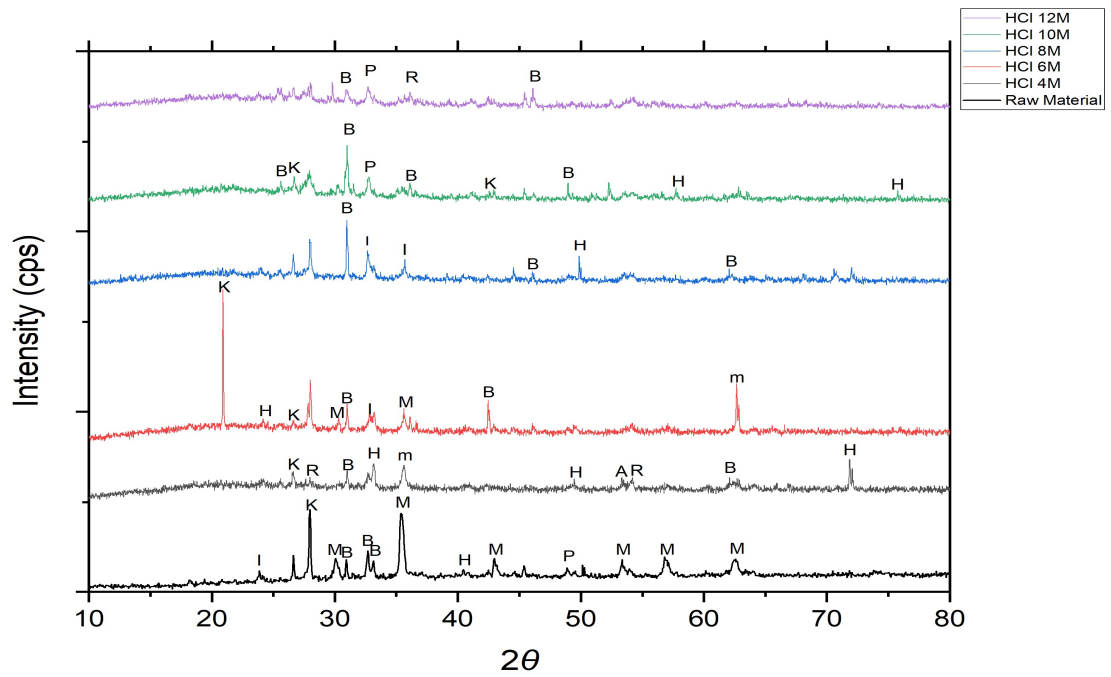


FIGURE 5. XRD result of iron sand before leaching (raw material) and after *leaching* for 2 hours at 110°C used 4 M, 6 M, 8 M, and 10 M HCl, where K: Quartz, R: rutile, A: anatase, B: brookite, I: ilmenite, M: magnetite, m: maghemite, F: fayalit, H: hematite dan P: pseudobrookite

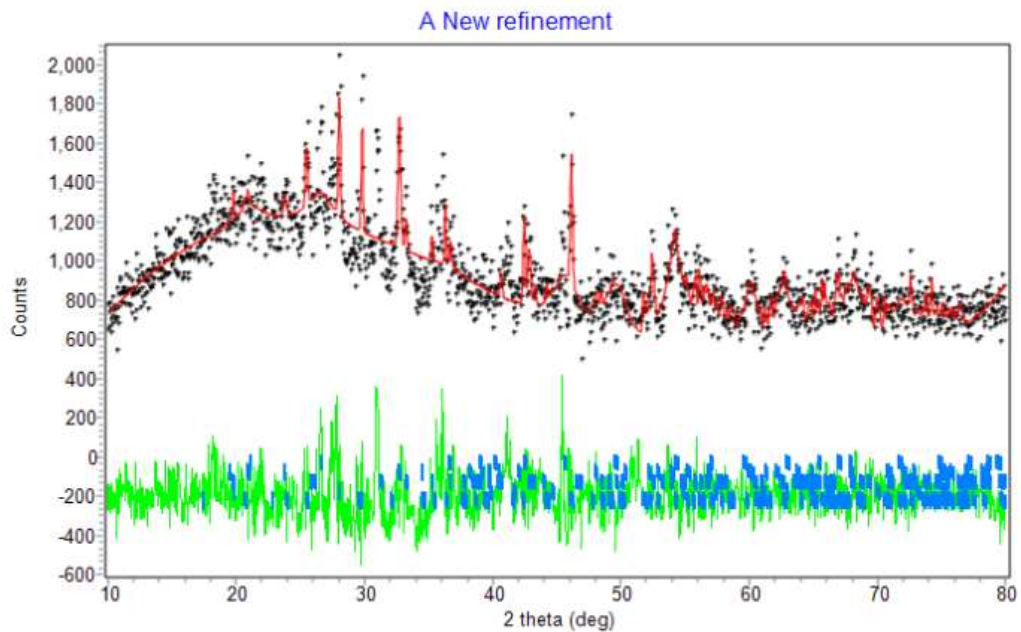


FIGURE 6. The refinement using Rietica of leached iron sand using 10 M HCl at 110 °C

Based on the refinement by Rietica of leached iron sand using 10 M HCl at 110 °C (Fig. 6), it is obtained the percentage TiO₂ phase (rutile and brookite) as revealed in Table 4.

TABLE 4. Phase percentage using 10 M HCl at 110 °C.

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 Fe₂O₃ and TiO₂ with relative percentages of 65.852% and 13.808%, respectively. The results of XRF analysis shows that the highest percentage of TiO₂ was found in HCl with a concentration of 12 M at 33.737%. TiO₂ extraction process using HCl was carried out to dissolve Fe (iron) in the FeTiO₃ structure and produce TiO₂ and was able to increase the percentage of TiO₂, the silicon element in SiO₂ 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 TiO₂ content. However, impurity phases, such as SiO₂, remain in the obtained sample.

CONCLUSIONS

In summary, variations in the concentration of HCl at temperature of 70 °C and 110 °C are able to increase the purity of TiO₂ i.e. 28.787% and 28.036% respectively, with variations of HCl 10 M. For variations of HCl 12 M at temperature 110°C increases the amount of TiO₂ 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.

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