RASĀYAN *J. Chem.*
Vol. 17 | No. 2 |621-630| April - June | 2024
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
http://www.rasayanjournal.co.in

SIMPLE SEPARATION OF $Ti(SO4)_2$ AND $FeSO_4$ FROM H_2SO_4-$FeSO_4$-$Ti(SO_4)_2$-H_2O SYSTEM BASED ON SOLUBILITY PROPERTIES IN METHYL ALCOHOL

Muzakky[1], Suyanti[1,✉], Kharistya Rozana[2] and M. A. Charisma Putri[2]
[1]Research Center of Mining Technology, Research Organization of Nanotechnology and Materials, National Research and Innovation Agency, Indonesia
[2]Directorate of Laboratory Management, Research Facilities, and Science and Technology Park, National Research and Innovation Agency, Indonesia
✉Corresponding Author : Suya003@Brin.Go.Id

ABSTRACT
The development of new tanning agents in the leather sector is urgently needed to cope with the increasing environmental pressure on the use of current tanning materials and processes. The best alternative tanning agent to replace chromium which is not environmentally friendly is titanium-based sulfate salts (IV). These compounds are most recommended to be developed and used in new, cleaner tanning processes for the leather industry. The main source of $Ti(SO_4)_2$ comes from the mineral ilmenite ($FeTiO_3$) extracted using H_2SO_4. The extraction process will produce a mixture of $Ti(SO_4)_2$ and $Fe(SO_4)$ in the H_2SO_4-$FeSO_4$-$Ti(SO_4)_2$-H_2O system at high acidity of H_2SO_4. The purpose of this study is to separate each of these compounds based on their solubility in methyl alcohol. This study discusses the comparison of feed and residue with FTIR, residual characteristics with XRD and XRF. While the results of quantitative analysis of each final product of $Ti(SO_4)_2$ and $Fe(SO_4)$ compounds using XRF. Qualitative results using FTIR show that $Ti(SO_4)_2$ images are different from $FeSO_4$. The %recovery result in the optimal state of H_2SO_4 extraction obtained $Ti(SO_4)_2$ was 20.6%, while $FeSO_4$ was only 9.2% from the feed of 30 g ilmenite.
Keywords: Titanium-Based Sulfate, Ilmenite, H_2SO_4-$Feso_4$-$Ti(SO_4)_2$-H_2O System, Solubility, Methyl Alcohol, FTIR, XRD, and XRF.

RASĀYAN *J. Chem.*, Vol. 17, No.2, 2024

INTRODUCTION

The process of tanning leather using chrome sulfate in the conventional way will be constantly under pressure from international regulations with the consequence that it is difficult to obtain environmentally friendly labeling.[1,2] Thus, reducing dependence on chromium sulfate is a key target for this research. Alternative compounds to replace chromium sulfate agents must be naturally abundant, easy to obtain, low price, and environmentally friendly. One of the salt compounds as a substitute for chrome sulfate is titanium sulfate ($Ti(SO_4)_2$) which is easily available and non-toxic.[3,4] The advantages of $Ti(SO_4)_2$ have long been known by Yadav that this compound has superacid (-Ho) strength with a value of 14.5. [5] While $FeSO_4$ itself by some researchers has been used as a standard for the synthesis of several compounds such as α-Fe_2O_3 nanoparticles, α-FeOOH and α-Fe_2O_3 nanorods, Fe_2O_3/CNTs supercapacitor electrodes, even as a material for shielding radioactive beta and gamma rays.[5,6,7,8,9] The main sources of $Ti(SO_4)_2$ and $FeSO_4$ are ilmenite minerals ($FeTiO_3$) spread in several regions in Indonesia, including West Kalimantan, South Kalimantan, and Bangka Belitung Islands.[10] Many researchers have done to obtain $Ti(SO_4)_2$ and $FeSO_4$ from $FeTiO_3$ through a solid-liquid extraction process using sulfuric acid. However, the procedure is too complicated and uses a lot of unnecessary chemicals.[11-16,20] Based on Liang[17] the reaction of ilmenite with sulfuric acid under sulfuric acid conditions at a temperature of 298 K.

$$FeTiO_{3(s)} + 2H_2SO_4 \rightarrow TiOSO4 + FeSO_4 + 2H_2O \qquad (1)$$

This reaction (1) will tend to shift to the right or product when the concentration of sulfuric acid is above 16 M or more, but when the concentration of sulfuric acid is less than 14 M or lower and the temperature is below 100 °C then the reaction becomes[18]

$$FeTiO_{3(s)} + 4\ H^+_{(aq)} \rightarrow TiO^{2+}_{(aq)} + Fe^{2+}_{(aq)} + 2\ H_2O \qquad (2)$$

http://doi.org/10.31788/RJC.2024.1728741

Based on Han's research,[18] there are distinctive differences in reactions (1) and (2), which is indicated by the presence of a typical 3rd phase formed in reaction (1) while not for reaction (2). The reaction product (3) is a typical layer with a colloidal form which is a solid-liquid equilibrium located in the middle layer.[18,19] Based on research by Zhang and Konigsberger, the colloid is a compound with the H_2SO_4–$FeSO_4$–$Ti(SO_4)_2$–H_2O system.[15,19,20] The characteristics of the middle layer or reaction product (1) are insoluble in sulfuric acid and ethyl alcohol but highly soluble in water.[14,15,16,21] Furthermore, the compound $Ti(SO_4)_2$ is insoluble in methyl alcohol, but $FeSO_4$ is soluble, thus the compound H_2SO_4–$FeSO_4$–$Ti(SO_4)_2$–H_2O can be separated into $Ti(SO_4)_2$ and $FeSO_4$ products, respectively.[15,18,19,22] Thus, the aim of this research is to provide information that the separation of FeSO4 and TiSO4 was successful in terms of the results of qualitative FTIR analysis and the results of semi-quantitative XRD analysis of ilmenite residue minerals extracted using methyl alcohol.

EXPERIMENTAL

Material and Methods

Ilmenite minerals from PT Timah Tbk - Bangka, have gone through the zircon sand beneficiation process using a shaker table, wet magnetic separator, and flotation machine and sifted through 200 mesh. Other materials are sulfuric acid p.a 96%, ethyl alcohol 99.9%, and methyl alcohol 99.9% which are all made by Merck. Separation method of $Ti(SO_4)_2$ and $FeSO_4$ using liquid-phase extraction.

General Procedure

Weighed as much as 30 g of ilmenite with a pass size of 200 mesh and put into a 500 mL three-neck flask reactor equipped with a condenser. Then put H_2SO_4 p.a (96% or 17.6 M) as much as 250 mL, then heated to 120 °C and stirred at a speed of 250 rpm and with 4 hours contact time. Then, into the three-neck flask added aquadest as much as 100 mL, cooled to 70 °C, and stirring continued for 60 minutes with heating at 100 °C. Furthermore, the final product in the form of 3 typical phases, is taken in the middle phase (second) with a pipette and inserted into a separate funnel with 250 mL volume. The residual phase part or ilmenite that does not react is also taken and dried for known characteristics with FTIR and XRD. Then, into the separate funnel that has been in phase 3, washed with ethyl alcohol as much as 50 mL to reduce the remaining H_2SO_4 2-3 times. Finally, washed with methyl alcohol as much as 100 mL to separate between $Ti(SO_4)_2$ and $FeSO_4$, where $Ti(SO_4)_2$ is in the water phase and $FeSO_4$ in the organic phase. Then, the two products are dried, each weighed, and % recovery is calculated with these equations.

$$\%\ \text{Recovery} = \frac{\text{Weight of } FeSO_4 \textit{ or } Ti(SO_4)}{\text{Weight of feed monazite}} \text{X } 100\%$$

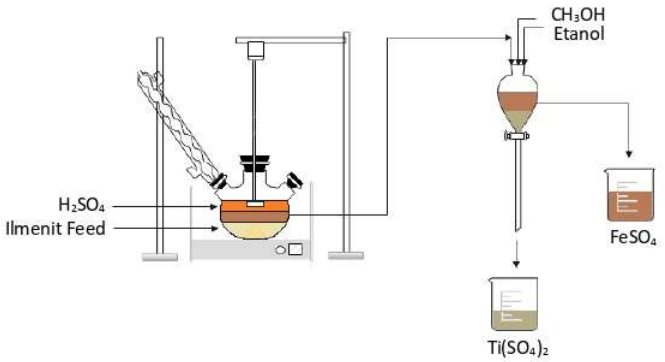

Fig.-1: Illustration of $Ti(SO4)_2$ and $FeSO_4$ Separation

Detection Method

Fourier Transform Infrared spectroscometer (FTIR, ALPHA II MODULE ATR ZnSe), X-ray Fluorescence Spectrometer (XRF Malvern PANanalytical type High-Performance ED-XRF Epsilon 4), and X-ray Diffractometer (XRD PANanalytical Aeris type DY844 equipped with 161039 databases) and Scanning Electron Microscope (SEM) Hitachi.

RESULTS AND DISCUSSION

Based on Tang, the molecular structure of ilmenite as $FeTiO_3$ is a derivative of α-Fe_2O_3 which consists of several layers that are regular and alternating with each other.[19] This regularity is characterized by the presence in each layer that is filled with Fe atoms in the plane (001) will be replaced by Ti atoms. This regularity is such that it results in densely arranged O layers, with alternating hexagonal shapes and schematic representations of $FeTiO_3$ structures shown in Fig.-2.

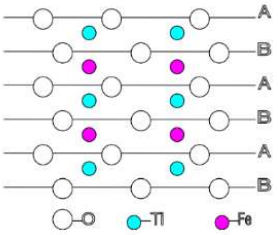

Fig.-2: The Simple Model of Ilmenite Structure as $FeTiO_3$[25]

In Fig.-3, it can be seen that the XRD analysis sample results contained ilmenite minerals 46.6%, as well as other forms of titanium minerals in the form of anastase 1.9%, rutile 6.5%, tistarite 4.8%, ilmenite-hematite mixture 29.8% and hematite 4.3%. While associated minerals such as xenotime 3.5%, monazite 0.5%, zircon 0.8% and quartz 1.2%.

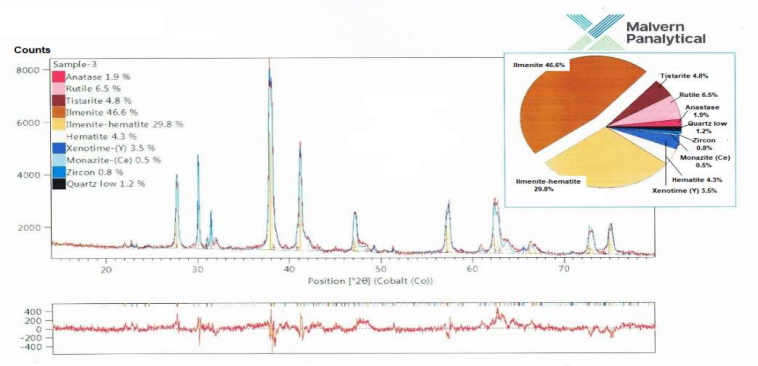

Fig.-3: XRD Image of Sample Ilmenite Feed Before Reaction (1)

Table-1: Results of XRF Analysis Before the Reaction (1)

Compound	Al_2O_3	SiO_2	P_2O_5	CaO	TiO_2	V_2O_5	Cr_2O_3	MnO	Fe_2O_3
Conc.	0.628	3.952	1.276	0.156	38.477	0.129	1.091	1.2	30.964
Unit	%	%	%	%	%	%	%	%	%
Compound	La_2O_3	CeO_2	Nd_2O_3	HfO_2	Eu_2O_3	Y_2O_3	Er_2O_3	SnO_2	As_2O_3
Conc.	0.252	0.929	0.163	0.262	0.103	0.436	0.118	4.485	0.122
Unit	%	%	%	%	%	%	%	%	%
Compound	ZrO_2	Nb_2O_5	Gd_2O_3	Ho_2O_3	PbO	Yb_2O_3	TeO_2	ThO_2	U_3O_8
Conc.	4.626	0.158	310	244.7	750.7	400	142.4	0.259	226.4
Unit	%	%	ppm	ppm	ppm	ppm	ppm	%	ppm

Shown that Table-1 is the result of the characteristics of ilmenite feed content before the reaction process is carried out (1) using an X-ray Fluorescence Spectrometer. Table-1 shows that different instruments turned out to be in accordance with what is stated in the mineral content analysis using XRD (Fig.-3) namely anatase, rutile, tistarite, ilmenite, ilmenite-hematite, xenotime, monazite, zircon, and quartz.

Comparison of Qualitative and Semiqualitative Reaction (1) Residues

Based on reaction product (1), ilmenite residues after being reacted with 16 M H_2SO_4 or more, apparently undergo many changes in several functional groups. This can be seen in Fig.-4, and it can be shown that the ilmenite feed has an O-H stretching band group at 3401.4 cm^{-1}, which is reinforced with a band at 1625.9 cm^{-1}.[25,26,27] As a result of the appearance of these two bands, at 1112.9 cm^{-1} there is a deformed Fe(or Ti)-

OH.[27] The last appearance of the band at 612.2 cm^{-1} is the tretching of Fe(or Ti)–O and Fe(or Ti)–OH of lattice water libration.[23,26] While ilmenite has been reacted with 16 M H_2SO_4 or more (reaction 1), there is a band shift at 3413.9 cm^{-1} which is still included as O-H stretching[25] which is also strengthened by the band at 1625.9 cm^{-1}.[26] While the bands at 1205.3 cm^{-1} and 1112.9 cm^{-1} constitute $V_3(SO_4)$ antisymmetric stretching and/or Fe–OH or Ti-OH deformation.[21,28] Furthermore, the band at 1014.1 cm^{-1} is a symmetric of $V_1(SO4)$ group, and the band at 612.2 cm^{-1} is a Fe(or Ti)–O and Fe(or Ti)–OH stretching of lattice water libration.[21,23,26] The appearance of the latter bands at 550.8 cm^{-1} and 476.9 cm^{-1} is symmetric of $V_2(SO_4)$.[28] With the appearance of some bands 1205.3 cm^{-1}, 1112.9 cm^{-1}, 1014.1 cm^{-1}, 612.2 cm^{-1}, 550.8 cm^{-1}, and 476.9 cm^{-1} are predicted that the surface of ilmenite has changed by being reacted with H_2SO_4 16 M or more. Furthermore, to find out the changes in content in detail, the results of reaction residue products (2) compared to feed ilmenite will be discussed in the analysis with XRD (Figure-3 and Figure-5).

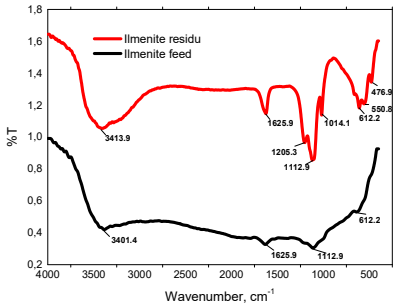

Fig.-4: FTIR Image Difference between Residual Ilmenite and Feed Ilmenite at 120℃

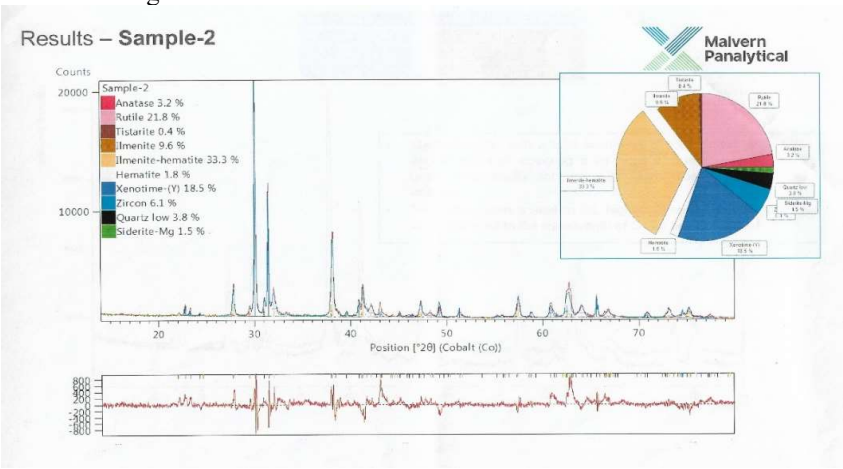

Fig.-5: XRD image of ilmenite residue after reaction (1) at 140℃

From Fig.-3 and Fig.-5 it turns out that before and after reaction (1) it can be compared that the mineral content of anatase (as a TiO_2 mineral) in the residue rises by 3.2%. While rutile in the residue is also a derivative species of the mineral TiO_2, its concentration also rises to 21.8%, where both minerals accumulate. It is based on Gao, that anatase is insoluble in H_2SO_4, so it will tend to accumulate in reaction residues (1). [29] Khan, in his research, that the resolution of anatase in sulfuric acid due to the occurrence of reverse reactions as follows[30]

$$TiO_2 + SO_3 \rightarrow TiOSO4 \quad (3)$$

$$TiOSO4 \rightarrow TiO_2 + SO_2 \uparrow + \tfrac{1}{2}\, O_2 \uparrow \quad (4)$$

While the tendency of rutile minerals to be insoluble in sulfuric acid 85% or more, has been explained in detail by Dubenko by making plots in the temperature range of 300 K-500 K versus Gibbs energy in the reaction below, [31]

$$TiO_2 + 6(H_2SO_4.H_2O) \rightarrow Ti(SO_4)_2 + 4(H_2SO_4.2H_2O) \quad (5)$$

In reaction (5) above, the change in $Ti(SO_4)_2 \rightarrow TiOSO_4$ in low acidity will not occur with a positive increase in Gibbs energy, so it is understandable that rutile is insoluble.[27] Furthermore, the mineral tistarite with the formula Ti_2O_3 (Fig.-3) which is analogous to titanium (III) oxide, has undergone dissolution in sulfuric acid quite a lot and remains 0.4% in the residue (Fig.-5), while in the feed (Fig.-3) there is 4.8% and the reaction can be written, (https://www.chemicalaid.com/tools/equationbalancer.php, 25 Sep 2022)

$$Ti_2O_3 + 3H_2SO_4 \rightarrow Ti_2(SO_4)_3 + 3\ H_2O \quad (6)$$

Furthermore, when Fig.-3 and Fig.-5 are compared, it turns out that the matrix of ilmenite-hematite is insoluble and accumulates to 33.3%. While the mineral hematite itself is well dissolved in H_2SO_4 (Fig.-5) and only 1.8% of its content. Based on Liu and Vehmaanperä, the solubility of the mineral hematite as Fe_2O_3 in H_2SO_4 reactions[32,33]

$$0.5\ Fe_2O_3 + 2H_2SO_4 \rightarrow Fe(SO4)_2^- + H^+ + 1.5\ H_2O \quad (7)$$

Then, in Fig.-5, it turns out that in the reaction residue (1), associated minerals such as xenotime and monazite as YPO_4 and (Ce, La, Nd, Th)PO_4 have undergone dissolution for monazite, while insoluble xenotime is even calculated in the residue to 18.5%.[34] According to Xi.D, the dissolution of xenotime in H_2SO_4 becomes more difficult than monazite and is carried out at a temperature of 200°C-250°C at a maximum temperature in reaction (1) of 140°C.[34] Based on Berry L, the dissolution reaction of monazite in H_2SO_4 is as follows[35]

$$2REPO_4 + 3H_2SO_4 \rightarrow RE_2(SO_4)_3 + 2H_3PO_4 \quad (8)$$
$$ThO_2 + 2H_2SO_4 \rightarrow Th(SO_4)_2 + 2H_2O \quad (9)$$
$$Th_3(PO_4)_4 + 6\ H_2SO_4 \rightarrow 3Th(SO_4)_2 + 4H_3PO_4 \quad (10)$$
$$2U_3O_8 + O_2 + 6H_2SO_4 \rightarrow 6UO_2SO_4 + 6H_2O \quad (11)$$

The minerals of zircon and quartz in the reaction residue (1) increased by 6.1% and 3.8%, respectively. This is estimated to be calculated during dissolution with H_2SO_4 and its insolubility. The solution turned out to be by El Fatah's research, that zircon and silica minerals would as insoluble residues in the purification of thorium in monazite using H_2SO_4. [36] While in the residue after the reaction (1) (Fig.-5), it turned out that the appearance of the mineral siderite-Mg was 1.5%. The formation of this mineral based on Romanek C can occur due to the gradual cooling of temperature after reaction (1) is complete, with an atmosphere of < 100°C.[37] It turns out that the changes before and after the reaction (1) in Fig.-3 and Fig.-5 have been by research by Jia L.[38] By evaluating the data above, the $TiOSO_4$ source of reaction (1) comes not only from the mineral ilmenite but also from its associated minerals such as tistarite. As for the source of origin of $FeSO_4$ in the reaction result (1), only from ilmenite and hematite.

Weight Loss and Surface Change of Reaction (1) Residue

Based on Jia L, the residual weight of the reaction (1) will be affected by several variables such as temperature and contact time.[38] While Purwani MV, in simple terms, the weight change can be observed weighing the result of the residue from the reaction (1).[39]

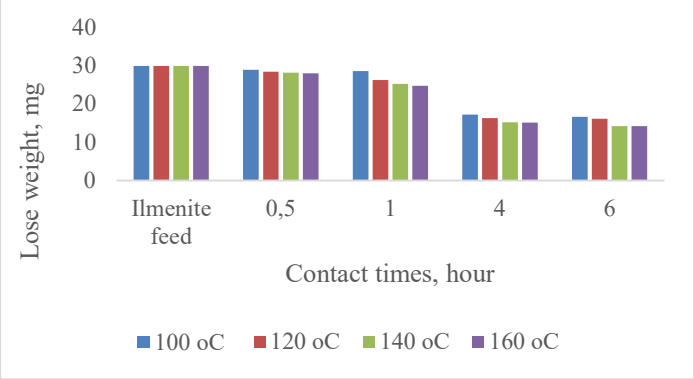

Fig-6: Product Lose Weight of Reaction (2) Residues

In Fig.-6, it can be shown that ilmenite feed taken as much as 30 g will continue to lose weight during reaction (1). As a function of increased contact time and temperature, this has been in accordance with Jia L's research.[38] In Fig.-6, it can be seen that after contact times of 240 minutes and 360 minutes and carried out at temperatures of 240℃ and 160℃, respectively, it is seen that the change in weight loss will tend to be constant.

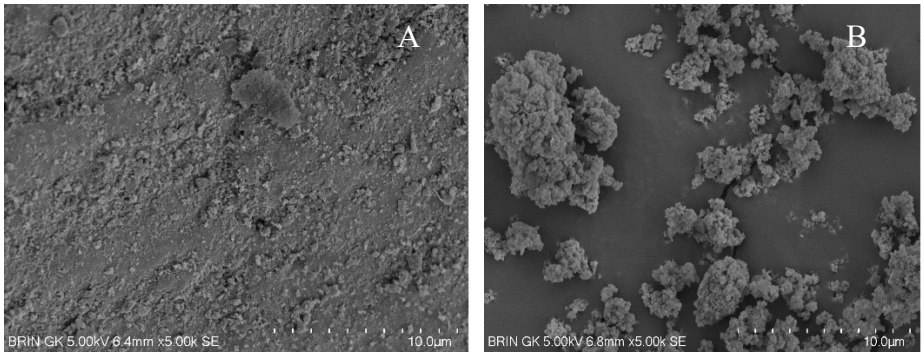

Fig.-7: SEM image of ilmenite feed (A) and (B) after reaction (2)

In Fig.-7, it can be shown that the surface image before and after reaction (1) where the images (A) before reaction or as ilmenite feed and (B) after which are residues are observed at 10 µm each. The difference between the two lies in the density of the spherical aggregate particles, where before or feed (A) in the form of a separate solid. While in the residue of the reaction (1), in the form of a formless clumped solid.

Qualitative and Quantitative Product of Reaction (2)

The product of reaction (2) qualitatively or quantitatively will depend on the temperature and contact time of the reaction with conditions of 16 M or more H_2SO_4.[17] Furthermore, after the third layer is separated with a pipette, the results and results are carried out.

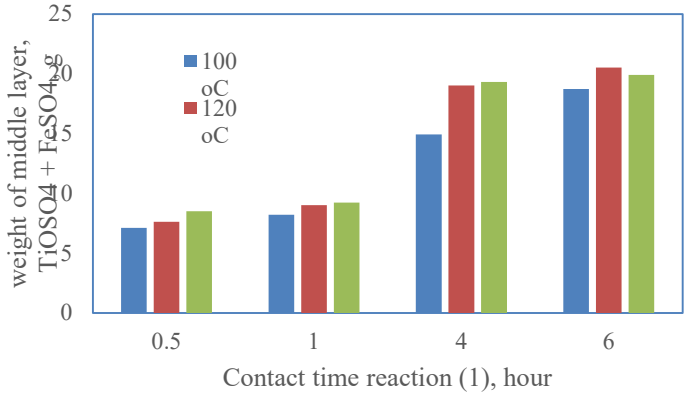

Fig.-8: The Weight Result of Phase 3 from after Reaction (1) is Formed

From Fig.-8, it can be shown that the weight of the 3rd fraction containing a mixture of $Ti(SO_4)_2$ and $FeSO_4$, turns out to take at least 4 or 6 hours with a temperature of 120℃ with a maximum weight of 19 g - 20.5 g. The temperature was chosen at 120℃ because it was in accordance with the results of FTIR; at the temperature of 140℃ is estimated that different species compounds have occurred in both $Ti(SO_4)_2$ and $FeSO_4$ (Fig.-9 and Fig.-10). Furthermore, after separation based on solubility using methyl alcohol, according to Han, the difference in the qualitative image of the resulting compound between $FeSO_4$ and $Ti(SO_4)_2$ with FTIR is shown in Fig.-9 and Fig.-10.[17]

In Fig.-9 it can be seen that the effect of temperature application on reaction (2), where 100℃ and 120℃ have an image pattern similar to each other. Furthermore, based on research, that in Fig.-9 shows bands 1178.7 cm^{-1} and 1056 cm^{-1} which indicate the presence of $V_3(SO_4)$ antisymmetric stretching and/ or Fe-OH that has been deformed. While the band at 995.5 cm^{-1} is estimated to be a band of $V_1(SO_4)$ symmetric stretching and 765 cm^{-1}, 612 cm^{-1}, 580.1 cm^{-1}, and 552 cm^{-1} in the form of $V_4(SO_4)$ antisymmetric or Fe-O stretching. [26] While the band at 433.2 cm^{-1} is estimated to be $V_2(SO_4)$. [28]

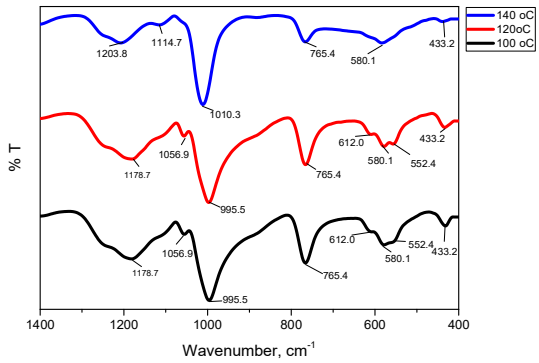

Fig.-9: FTIR Image of $FeSO_4$ Isolation After Separation from H_2SO_4-$FeSO_4$-$Ti(SO_4)_2$-H_2O with Methyl Alcohol

Furthermore, when reaction (2) is heated to 140°C shown in Figure-8, there will be a new band at 1203.8 cm^{-1}, and there has been a shift at 1114.7 cm^{-1} and 1010.3 cm^{-1} in the form of $V_3(SO_4)$ antisymmetric stretching and or Fe-OH deformed. [26] But bands at 765 cm^{-1}, 580.1 cm^{-1} and 433.2 cm^{-1} remain as they do at heating 100°C and 120°C. Thus, it can be predicted that the result of the reaction (1), when heated at 140°C, the chemical molecular structure has undergone changes or is a different species from $FeSO_4$.

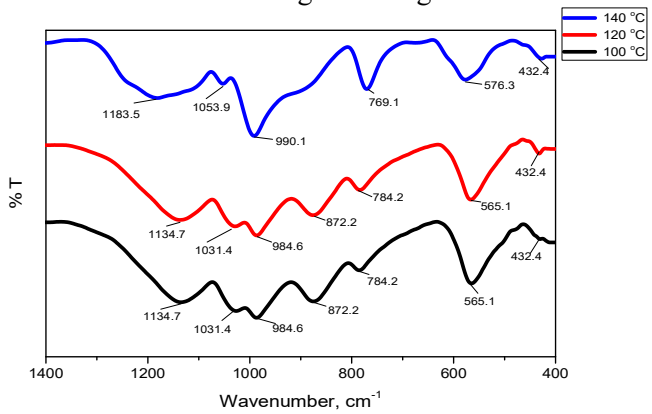

Fig.-10: FTIR image of $Ti(SO_4)_2$ Isolation After Separation from H_2SO_4-$FeSO_4$-$Ti(SO_4)_2$-H_2O with Methyl Alcohol

In Fig.-10, it can be shown that the isolation results of $Ti(SO_4)_2$ from reaction (2) heated at 100°C and 120°C have similarities and are identical both in the FTIR image and the number of bands. Seen in bands 1134.7 cm^{-1} and 1031.4 cm^{-1} are bands of $V_3(SO_4)$ antisymmetric stretching, but bands 984.6 cm^{-1} are $V_1(SO_4)$ symmetric stretching. While 872.2 cm^{-1} and 784.2 cm^{-1} are bands of V(Ti-O) or V(Ti-O) stretching.[28] Furthermore, the band at 565.1 cm^{-1} is $V_4(SO_4)$ anti-symmetric and the band at 432.4 cm^{-1} is the bonding model of φTiO_6. [26,28] In Fig.-10, the result of isolation of $Ti(SO_4)_2$ from reaction (2) when heated at 140°C has an FTIR image with different bands with 100°C and 120°C. The difference is marked by the shifting of the bands at 1183.5 cm^{-1} and 1053.9 cm^{-1} as $V_3(SO_4)$ antisymmetric stretching, while 990.1 cm^{-1} represents $V_1(SO_4)$ symmetric stretching. [26] Furthermore, 769.1 cm^{-1} is a band of V(Ti-O) or V(Ti-O)s stretching but like the band at heating 100°C and 120°C, the band 565.1 cm^{-1} is $V_4(SO_4)$ antisymmetric and the band at 432.4 cm^{-1} is a bonding model of φTiO_6.[26,28] Thus it can be predicted that the result of the reaction (2), when heated at 140°C, the structure of the chemical molecule has undergone changes or is a different species from $Ti(SO_4)_2$.

In Fig.-11, it can be shown that the % recovery $Ti(SO_4)_2$ will increase with the increase in contact time and temperature. Contact time is chosen at 4 hours or 6 hours, but the temperature should still be maintained at 120°C. This is by FTIR data (Fig.-10) so that there are no different species of $Ti(SO_4)_2$ compound from the desired product. Meanwhile, the influence of contact times and temperature on $FeSO_4$ isolation results can be seen in Fig--12, and it turns out that the %recovery of $FeSO_4$ in selected 4 hours with a temperature of 120°C is only 9.2%, compared to $Ti(SO_4)_2$ which is 20.6% under the same conditions. It is estimated that there is still a lot of $FeSO_4$ carried in washing with ethyl alcohol.

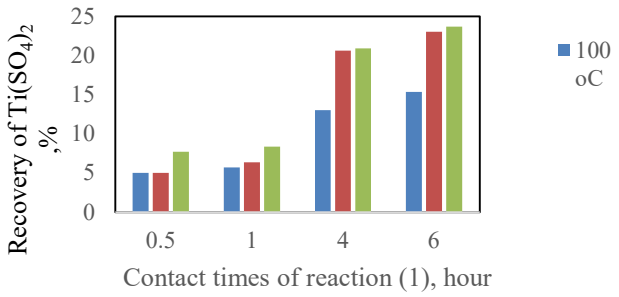

Fig.-11: Effect of Contact Times and Temperature on % Recovery $Ti(SO_4)_2$

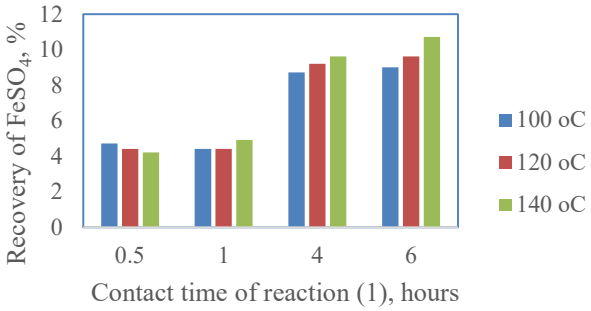

Fig.-12: Effect of Times Contact and Temperature on %Recovery $FeSO_4$

CONCLUSION

Simple separation of $Ti(SO_4)_2$ and $FeSO_4$ from H_2SO_4-$FeSO_4$-$Ti(SO_4)_2$-H_2O was found to be possible based on solubility in methyl alcohols. The experimental results showed that the qualitative residual results were different from feeds using FTIR. The residual content contains 9.6% ilmenite residue and 33.3% ilmenite-hematite mixture as well as several other minerals such as rutile, tistarite, hematite, xenotime, monazite, zircon, and quartz. The product loses weight of reaction (2) highest residues at 4 hours or 6 hours and surface image between before and after reaction (2). The maximum weight of the middle layer results in 6 hours of contact time. FTIR images between $FeSO_4$ and $Ti(SO_4)_2$ show different functional groups and %recovery $Ti(SO_4)_2$ is optimal at the temperature of 140°C with 23.67% weight while $FeSO_4$ is only 10.7%.

ACKNOWLEDGMENTS

With the completion of this paper, the authors would like to thank Dr. Susilo Widodo and Ir. Herry Poernomo, who helped a lot in terms of funding through DIPA 2018 with SP number 080.01.1.017290/2018. The author was also very grateful to Ulul Duwi Yulianda and Triyono ST, who have helped a lot with the laboratory work.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID IDs, given below:

Muzakky https://orcid.org/0000-0003-3401-661X
Suyanti https://orcid.org/0009-0002-1019-7276
Kharistya Rozana https://orcid.org/0000-0002-4726-1166
M. A. Charisma Putri https://orcid.org/0009-0005-3333-9224

REFERENCES

1. C. S. Pomelli, C. Chiappe, F. Barontini, M. Puccini, M. Seggiani, and S. Vitolo, *Clean Technol Environ Policy*, **19**, 259(2017), https://doi.org/10.1007/s10098-016-1207-y
2. M. Crudu *et al.*, In ICAMS 2010-3rd International Conference on Advance Material and Systems, pp. 27-32 (2010)
3. M. Seggiani, M. Puccini, S. Vitolo, C. Chiappe, C. S. Pomelli, and D. Castiello, *Clean Technol Environ Policy*, **16**, 1795(2014), https://doi.org/10.1007/s10098-014-0722-y
4. B. Peng, B. Shi, D. Sun, Y. Chen, and D. C. Shelly, *Ultrason Sonochem*, **14**, 305(2007), https://doi.org/10.1016/j.ultsonch.2006.07.001
5. M. Crudu, V. Deselnicu, D. C. Deselnicu, and L. Albu, *Waste Management*, **34**, 1806(2014), https://doi.org/10.1016/j.wasman.2013.12.015
6. G. D. Yadav and J. J. Nair, *Microporous and Mesoporous Materials*, **33**, 1(1999), https://doi.org/10.1016/S1387-1811(99)00147-X
7. J. Grabis, G. Heidemane, and D. Rašmane, *Mater. Sci,* **14**, 292 (2008)
8. Y. Dong, H. Yang, R. Rao, and A. Zhang, *Journal of Nanoscience and Nanotechnology*, **9**, 4774(2009), https://doi.org/10.1166/jnn.2009.1083
9. L. Yue *et al.*, *Journal of Alloys and Compounds*, **765**, 1263(2018), https://doi.org/10.1016/j.jallcom.2018.06.283
10. E. Al-Sarray, *Nuclear Science* **3**, 16(2018), https://doi.org/10.11648/j.ns.20180301.13
11. E. Yustanti, A. Rosani, and L. H. Lalasari, *Teknika: Jurnal Sains dan Teknologi*, **17**, 21(2021), https://doi.org/10.36055/tjst.v17i1.10789
12. L. Gao, B. Rao, H. Dai, H. Xie, P. Wang, and F. Ma, *Physicochemical Problems of Mineral Processing*, **55**, 467(2019), https://doi.org/10.5277/ppmp18159
13. L. H. Lalasari, R. Subagja, A. H. Yuwono, F. Firdiyono, S. Harjanto, and B. Suharno, *Advanced Materials Research*, **789**, 522(2013), https://doi.org/10.4028/www.scientific.net/AMR.789.522
14. A. v. Dubenko, M. v. Nikolenko, A. Kostyniuk, and B. Likozar, *Minerals*, **10**, 1(2020), https://doi.org/10.3390/min10060538
15. S. Zhang and M. J. Nicol, *Hydrometallurgy*, **103**, 196(2010), https://doi.org/10.1016/j.hydromet.2010.03.019
16. Y. Zhang, J. Zhou, and Z. Li, *Journal of Chemical and Engineering Data*, **62**, 973(2017), https://doi.org/10.1021/acs.jced.6b00783
17. Y. Zhang, Z. Li, and E. Asselin, *The Journal of Chemical Thermodynamics*, **102**, 219(2016), https://doi.org/10.1016/j.jct.2016.07.006
18. B. Liang, C. Li, C. Zhang, and Y. Zhang, *Hydrometallurgy*, **76**, 173(2005), https://doi.org/10.1016/j.hydromet.2004.10.006
19. K. N. Han, T. Rubcumintara, and M. C. Fuerstenau, *Metallurgical Transactions B*, **18**, 325(1987), https://doi.org/10.1007/BF02656150
20. Y. Zhang, Z. Li, and E. Asselin, The *Journal of Chemical Thermodynamics* **102**, 219(2016), https://doi.org/10.1016/j.jct.2016.07.006
21. E. Königsberger, L. C. Königsberger, I. Szilágyi, and P. M. May, *Journal of Chemical Engineering and Data*, **54**, 520(2009), https://doi.org/10.1021/je800484m
22. M. Nurdin, A. H. Watoni, N. Abdillah, and D. Wibowo, *ChemTech*, **9**, 483(2016)
23. W. Bullough, *J. Appl. Chem*, **2**, 703 (1952), https://doi.org/10.1021/j150310a002
24. M. Kakihana, M. Kobayashi, K. Tomita, and V. Petrykin, *Bulletin of the Chemical Society of Japan*, **83**, 1285(2010), https://doi.org/10.1246/bcsj.20100103
25. X. Tang and K. Hu, *Journal of Materials Science*, **41**, 8025(2006), https://doi.org/10.1007/s10853-006-0908-8

26. F. Cheng, Q. Cao, Y. Guan, H. Cheng, X. Wang, and J. D. Miller, *International Journal of Mineral Processing*, **122**, 36(2013), https://doi.org/10.1016/j.minpro.2013.04.007
27. V. Rouchon *et al.*, *Journal of Raman Spectroscopy*, **43** (2012), https://doi.org/10.1002/jrs.4041ï
28. F. Ahangaran, A. Hassanzadeh, and S. Nouri, *Inter. Nano Lett*, **3**, 23 (2013)
29. L. K. Noda, F. R. Sensato, and N. S. Gonçalves, *Quim Nova*, **42**, 1112(2019), https://doi.org/10.21577/0100-4042.20170427
30. L. Gao, B. Rao, H. Dai, H. Xie, P. Wang, and F. Ma, *Physicochemical Problems of Mineral Processing*, **55**, 467(2019), https://doi.org/10.5277/ppmp18159
31. H. A. Khan, S. Kim, and K. D. Jung, *Catalysis Today*, **352**, 316(2020), https://doi.org/10.1016/j.cattod.2019.10.037
32. A. V. Dubenko, M. v. Nikolenko, E. v. Aksenenko, A. Kostyniuk, and B. Likozar, *Processes*, **8(6)**, 640(2020), https://doi.org/10.3390/pr8060640
33. H. Liu, V. G. Papangelakis, M. S. Alam, and G. Singh, *Canadian Metallurgical Quarterly*, **42**, 199 (2003), https://doi.org/10.1179/cmq.2003.42.2.199
34. P. Vehmaanperä, R. Salmimies, and A. Häkkinen, *Mining Metallurgy & Exploration*, **38**, 69(2021), https://doi.org/10.1007/s42461-020-00308-4
35. D. Qi, *Hydrometallurgy of Rare Earths*, **2018**, 1(2018), https://doi.org/10.1016/b978-0-12-813920-2.00001-5
36. L. Berry, V. Agarwal, J. Galvin, and M. S. Safarzadeh, *Canadian Metallurgical Quarterly*, **57**, 422(2018), https://doi.org/10.1080/00084433.2018.1478490
37. A. I. L. Abd El Fatah, in *IOP Conference Series: Materials Science and Engineering*, pp. 1-12 (2020), https://doi.org/10.1088/1757-899X/975/1/012022
38. C. S. Romanek, C. Jiménez-López, A. R. Navarro, M. Sánchez-Román, N. Sahai, and M. Coleman, *Geochimica et Cosmochimica Acta*, **73(18)**, 5361(2009), https://doi.org/10.1016/j.gca.2009.05.065
39. L. Jia *et al.*, *Hydromet*, **150**, 92(2014), https://doi.org/10.1016/j.hydromet.2014.09.016
40. Purwani, MV and Suyanti, *Prosiding Pertemuan dan Presentasi Ilmiah-Penelitian Dasar Ilmu Pengetahuan dan Teknologi Nuklir*, pp. 145-150 (2014)

[RJC- 8741/2023]