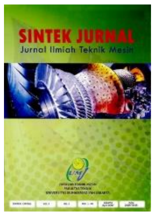
SINTEK JURNAL: Jurnal Ilmiah Teknik Mesin
ISSN: 2088-9038, e-ISSN: 2549-9645
Homepage: http://jurnal.umj.ac.id/index.php/sintek



STUDY EXPERIMENTAL THE EFFECT OF $CaCO_3$ AND Fe_2O_3 MASS COMPOSITION RATIO ON CALCIUM FERRITE PHASE FORMATION BASE ON LOCAL MATERIALS IRON SAND AND LIMESTONE

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Accepted: 11-12-2019 Revised: 10-05-2020 Approved: 01-06-2020

ABSTRACT

Iron sand contains Fe_3O_4 which can be produced Fe_2O_3 and limestone contains dominant $CaCO_3$. Both of these materials are very abundant in Indonesia. Iron sand is generally used as building construction materials, as well as the use of limestone, there has been no development in the management of the combination of the two materials. Because of the abundance of iron sand and limestone which have not yet been developed to the fullest, a study was carried out to manage and develop the product as a follow up to the previous Calcium Ferrite research. This research focuses on analyzing the phase of Calcium Ferrite formed using XRD and SEM-EDX tests. Tests were carried out on samples with a mass ratio of a mixture of Fe_2O_3 and $CaCO_3$ of 1: 4, 1: 6, 1: 8, and 1:12. The XRD test results showed that the phases formed were dominated by the $Ca_2Fe_2O_5$ and $Ca_2Fe_9O_{13}$ phases. And from the SEM-EDX test results, the results indicate the formation of nano-scale Calcium Ferrite with the composition of elements Ca, Fe, O, and Si.

***Keywords**: Calcium Ferrite; Iron Sand; Limestone; Wet Mixing Method; XRD; SEM-EDX.*

1. INTRODUCTION

Iron sand is one of the abundant natural resources in Indonesia. Almost all of the big islands in Indonesia have iron sand with different qualities. There are three types of iron ore including hematite magnetic iron ore, lateritic iron ore, and titan iron ore. In general, iron sand is widely applied as a concrete aggregate in the field of civil construction [1], as a basic material for the steel industry in the industrial field, and as a basic material in the formation of magnets in the field of developing advanced materials [2]. Research on the development of advanced materials that mostly use iron sand is the manufacture of medium and permanent magnets, one of which is a magnet type Barium Ferrite and Strontium Ferrite.

Barium Ferrite can be synthesized by various methods, such as powder metallurgy with milling, annealing, and sintering mechanisms [3] and also by coprecipitation method, by extracting iron sand to be more pure and refined to be easily diffused with Barium or Strontium [4]. Iron sands extracted by coprecipitation method to get Fe_3O_4 which is then sintered into Fe_2O_3 [5]. Barium or Strontium which is diffused that is Barium or Strontium in the form of the carbonate, namely $BaCO_3$ and $SrCO_3$ [4].

In addition to the abundant iron sand, there are also abundant natural resources, namely limestone. Limestone is a sedimentary rock which has a high $CaCO_3$ phase content. In Indonesia, limestone is scattered in various regions namely around 2,156 billion tons, which are spread in Aceh Darussalam (131.12 billion tons (Mt)), North Sumatra (3.24 Mt), West Sumatra (68.1 Mt), Riau (53.2 Million tons (Million)), Bengkulu (137.1 Million), Jambi (157 Million), South Sumatra (294 Million), Lampung (2 Million), Banten (61.6 Million), West Java (660.3 Million) Mt), Central Java (6 Mt), DI Yogya (10 Mt), East Java (3,069 Mt), Bali (154.64 Mt), NTB (1.2 Mt), NTT (132.82 Mt), Central Kalimantan (449 Mt), South Kalimantan (8.33 Mt), East Kalimantan (57 Mt), North Sulawesi (18.8M), Gorontalo (18.5Mt), Central Sulawesi (696Mt), South Sulawesi (31.33Mt), South-East Sulawesi (1,527Mt)), Malut (8.87 Mt), and Papua (2.6 Mt). In general, limestone is applied as a basic material for construction materials, the main material for making portland cement, and as an additive in the metal smelting industry [6]. Limestone which is dominated by the $CaCO_3$ phase is one of the potentials that has not been considered as one of the substitutes of Ba and Sr which both form the carbonate phase.

Ca is one of the elements of the alkaline earth group which is a group with Ba and Sr. The physical and chemical properties of Ca are not much different from Ba and Sr which allows it to form a type of magnet, namely the Calcium Ferrite magnet. Research on Calcium Ferrite has been carried out which produces a type of material known as p-Type thermoelectric properties of the oxygen-deficient perovskite $Ca_2Fe_2O_5$ [7,8]. Considering the abundance of iron sand and limestone which has not yet been developed to the maximum, a study was carried out aimed at analyzing the types of Calcium Ferrite phases formed by synthesizing them based on local natural materials of iron sand and limestone.

2. METHODS

This research was conducted through three stages. The first, synthesis of Fe_2O_3 by extracting iron sand that is iron sand from the Lumajang. The second, synthesis of $CaCO_3$ from limestone kab. Tuban. And the last, synthesis of Calcium Ferrite variations in the mixing ratio of $CaCO_3$ and Fe_2O_3 namely 1:4, 1:6, 1:8, 1:12 with a sintering temperature of 700^0C for 2 hours. Calcium Ferrite samples obtained from the results of the synthesis were characterized by using the XRD test and combined with the SEM-EDX test. XRD data were analyzed by combining two applications namely Match!2 and HighScorePlus to identify and estimate the percentage of phases formed. For more details in terms of the flow of research conducted, it can be seen in figure 1.

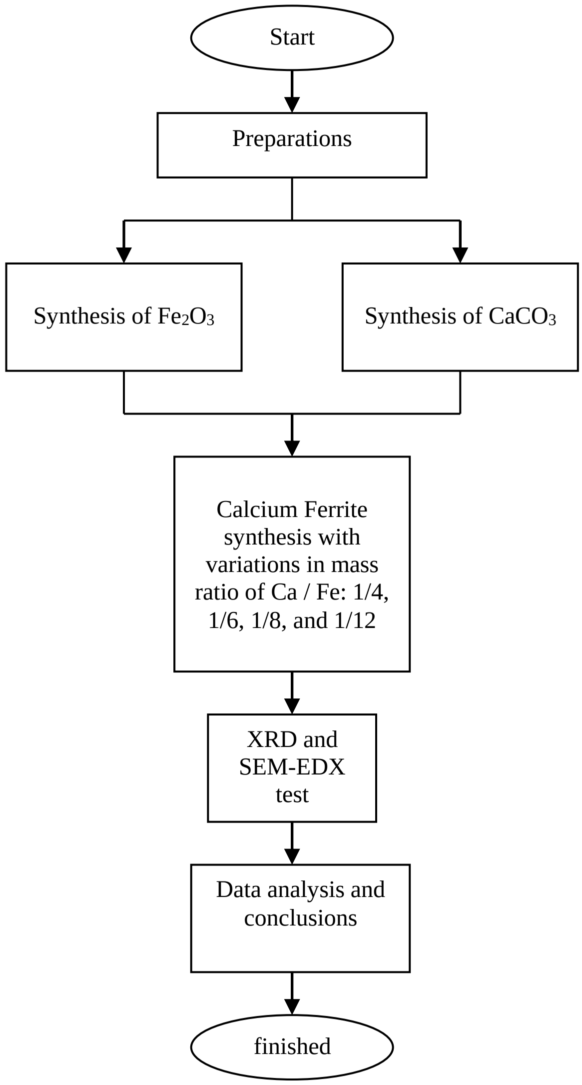

Figure 1. The flow of research methodology

The first stage, that is Fe_2O_3 synthesis, is carried out using the coprecipitation method. This method is done by dissolving iron sand that has been sorted and cleaned into an HCl solution so that a solution of $FeCl_2$ and $FeCl_3$ is found . The resulting solution is then dropped with NH_4OH resulting in precipitate Fe_3O_4. The resulting Fe_3O_4 is sintered at 800^0C for 2 hours to form Fe_2O_3 powder.

The second stage, the $CaCO_3$ synthesis, was carried out using the carbonation method. This method is done by dissolving limestone in distilled water to form a solution of $Ca(OH)_2$. The resulting solution has then flowed with CO_2 gas at a low speed so that precipitate $CaCO_3$ or PCC.

The third stage, that is Calcium Ferrite synthesis, is carried out using the coprecipitation method as well as the Fe_2O_3 synthesis process with the process variables as mentioned above.

3. RESULTS AND DISCUSSION

The formation of Calcium Ferrite phases occurs at temperatures above 500^0C [8]. Fe_2O_3, which is the basic materials of Calcium Ferrite synthesis and also the result of extraction from iron sand, is analyzed for the purity of the phase. This is important as confirmation that the extraction was successful and that Fe_2O_3 was actually obtained, as well as for $CaCO_3$. The results of the XRD test of Fe_2O_3 and $CaCO_3$ are shown in Figure 2 and Figure 3.

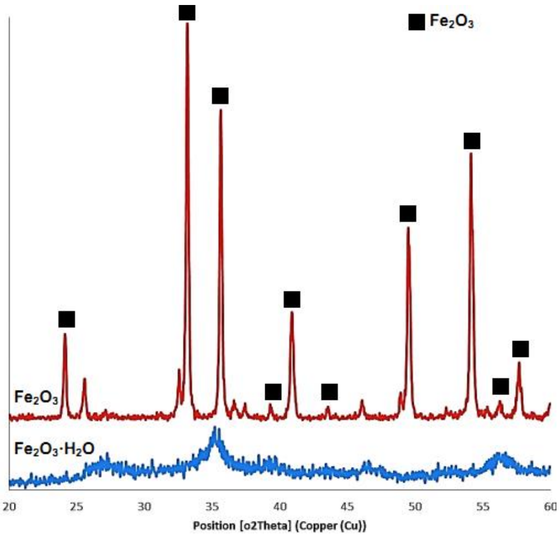

Figure 2. X-ray diffraction pattern of Fe_2O_3 results from the synthesis of iron sand.

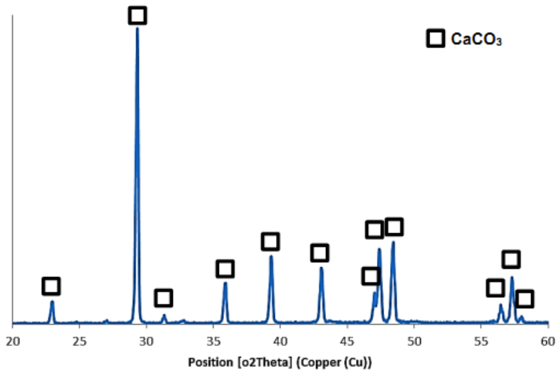

Figure 3. X-ray diffraction pattern of $CaCO_3$ synthesized from limestone.

Figure 2 shows the X-Ray diffraction pattern of Fe_2O_3 synthesized from iron sand. The pattern shows that the synthesis process was successful, that is with the confirmation of the XRD test results, as well as Figure 3 as proof of the successful formation of $CaCO_3$ from the limestone synthesis.

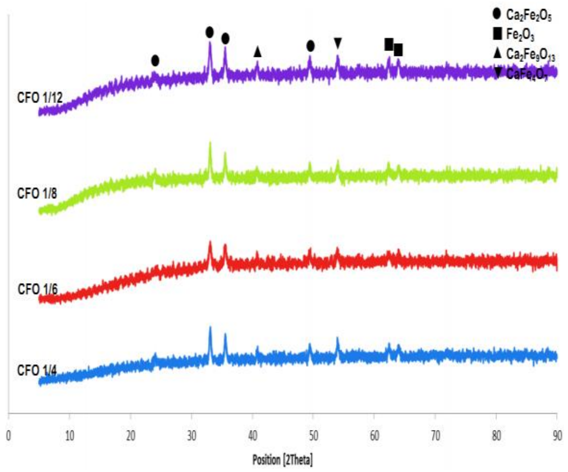

Figure 4. Calcium Ferrite X-Ray diffraction pattern from the results of synthesis by coprecipitation method.

Table 1. Results of analysis of Calcium Ferrite volume fraction calculations with the Match and High Score Plus

Sample Code	Volume Fraction (%)			
	Fe_2O_3	$CaFe_4O_7$	$Ca_2Fe_2O_5$	$Ca_2Fe_9O_{13}$
CFO4	9,56	1,68	36,37	52,39
CFO6	12,64	9,95	44,50	32,91
CFO8	33,24	7,80	9,91	49,05
CFO12	14,43	9,37	47,00	29,20

Figure 4 shows the X-ray diffraction pattern for Calcium Ferrite samples resulting from the coprecipitation method with Fe/Ca 4, 6, 8, and 12. The results of the analysis of the X-ray diffraction pattern data can be seen in Table 1. The XRD test data obtained information that formed three types of Calcium Ferrite phases namely $CaFe_4O_7$, $Ca_2Fe_2O_5$, and $Ca_2Fe_9O_{13}$. Each type of phase formed has different characteristics for each variation of heat treatment. For the $CaFe_4O_7$ phase, this phase has increased along with the increase in the ratio of Fe_2O_3 were given. That is because the phase $CaFe_4O_7$ requires more elements Ca and formed at high temperatures. Whereas for the other phases, namely $Ca_2Fe_2O_5$ and $Ca_2Fe_9O_{13}$, the large pattern of volume fraction values of the formed phase fluctuates and decreases except in comparison 12. This is due to the growth phase of Fe_2O_3 which is formed in the temperature range of 600^0C to 800^0C and for a comparison of 12, it is similar to the behavior hekaferit formation formed by comparison Fe 12. From the overall data, there is still a lot of research to be done for other variations, so that later can get information about the nature of the phase formation and proportions following the desired phase needs.

Information on elemental composition, particle size, and shape of Calcium Ferrite samples were carried out by SEM-EDX testing. SEM-EDX testing done on samples with a ratio of Fe/Ca that is 12 to consider the results of the calculation of the fraction volume of the phase $Ca_2Fe_2O_5$ as a kind Brownmillerite material has attractive electrical properties and also the formation of heksaferit mixing. SEM results with various types of magnifications are shown in Figure 5 and EDX results are shown in Table 2.

Figure 5 shows an indication of the size of the nano-sized particles. Besides, particles also appear agglomerated which indicates the magnetic properties of the particles. This is reasonable considering the basic material of the magnet itself is Fe_3O_4. Table 2 also shows that the constituent elements of the sample are Ca, Fe, O, and Si. These results confirm or explain the roughness formed in the background of the XRD test results data.

Table 2. The results of EDX analysis of Calcium Ferrite in comparison to Fe/Ca 12.

Element	Atomic (wt%)
Ca	3
Fe	36
O	53
Si	8

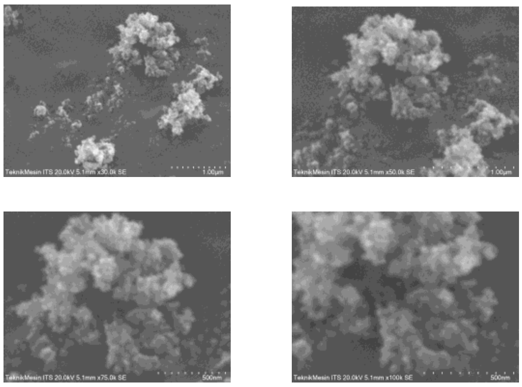

Figure 5. Photograph results of SEM Kalsium Ferit comparison of Fe/Ca 12.

4. CONCLUSION

Calcium Ferrite from the results mixing of Fe_2O_3 extracted from natural iron sand and $CaCO_3$ extracted from natural limestone were successfully synthesized. The results of XRD testing showed that there were three types of calcium ferrite phases, namely $CaFe_4O_7$, $Ca_2Fe_2O_5$, and $Ca_2Fe_9O_{13}$ with $Ca_2Fe_2O_5$, and $Ca_2Fe_9O_{13}$ as the dominant phase. SEM test results show an indication of the size of nano-sized particles and also the particles look agglomerated which indicates the magnetic properties of these particles. The EDAX results show that the elements making up the phase are Ca, Fe, O, and there is a little impurity, Si. The EDAX results confirm or explain the background roughness level from the XRD test results. The SEM and EDAX results also confirm by dominating the composition of the Fe and O elements according to the type of phase formed.

5. ACKNOWLEDGMENT

This research was partly supported by " Hibah Internal PT 2018" provided by 17 Agustus 1945 University Surabaya, Indonesia.

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