Time milling influence on the size of the Lemabang iron sand powder synthesized by using high energy milling method

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Abstract. Has been synthesized iron sand from the Lemabang-Sumsel Area by using High Energy Milling Method. Before synthesized, extraction of Lemabang iron sand in advance by using permanent magnets and Methanol-Soap Bathed Method in order to separated iron sand and impurities. After it, iron sand milled by using High Energy Milling with variations in milling time 2 hours, 4 hours, and 6 hours. Milling time optimization done in order to see the effect of time milling on powder size and surface morphology. Then Iron sand already in milling characterized by using XRD to see crystal structure and crystal size, and SEM-EDS used to see surface morphology and composer elements. XRD's result show that, the longer of the milling time resulting the shorter of the powder size. Whereas the results of the SEM-EDS's photo, its seem that the grain morphology of the iron sand powder after milling is smoother and more homogeneous be compared before milling

1. Introduction

Iron Sand is one of the abundant natural resources found in Indonesia. There are many uses or applications for this material, starting from magnetic based materials to high quality steel materials. In the application, iron sand has been used in a variety of length scale, ranging from the millimeter-down to the nanometer length scale. The main problem in the synthesis and processing of nanoparticle iron sand, is the lengthy time it takes to finish the process. Nanoparticle synthesis variously has been done but it was not efficient in the conventional scale. By using High Energy Milling especially Shaker Mill PPF-UG, iron sand nanoparticle could be synthesized with a higher efficiency as far the duration of processing time is concerned. The rotational speed of this apparatus is up to 800 rpm. The synthesis of iron sand nanoparticle then takes up only a few hours.

Iron sand is a mineral containing various oxide iron compounds such as magnetite, ilmenit, hematite, and also other minerals (but in lesser quantities), such as silica and titania with varying concentration depending upon the location Zulfalina et al.[1]. Experiment about magnetization still exist at synthesize. Its cause more benefit and it have nice prospect by various sector. In Indonesia several research groups are involved in the expeimental work of iron sand synthesis. Presently iron sand synthesis technology has become more rapidly developed Zaehir et al.[2].

High Energy Milling (HEM) is a method of synthesis using an apparatus which gives priority to transfer high mechanical energy to the material. HEM using collision of balls in a vial to crush the material until the smallest size material is obtained. This type of apparatus does not need high temperature smelting. This process produces smooth nanoparticle powder by maximizing operation,

by Wank et al.[3].Theoretically, nanocrystalline metals didn't deform at high temperaturesby Darling et al.[4].

Several ways done to achieve nano-scale, such as a nanoparticle self-stabilization mechanism in molten metal by Chen et al.[5], layer-by-layer assembly of two-dimensional buildingblocks under vacuum by Kang et al.[6],a molecular-level liquid–liquidmixing/doping technique by Liu et al.[7].Complicated way to synthesize nano-scale material.

Innovative tools and technology made their effort go furtherby Pain et al.[8].Several conventional synthesis have reached certain limits in further improving the propertiesof metals by Nie et al.[9].

Shaker Mill PPF-UG is a new innovation in milling system which was developed by the HEM-E3D system milling company (previous product). This apparatus has a rotational speed in the range of 700 rpm to 800 rpm, by Sukarto et al.[10]. It has been the authors'experience, that the milling's duration time parameter is also important in this case. High energy milling is a very suitable method to minimize particles size in a sample and then observe the physical changes occuring in the milled sample.

By virtue of XRD reflection intensity data, the particle size could be obtained by calculating the FWHM (Full Width Half Maximum). Particle sized be found by using the *Scherrer Formula*, by Hadiati et al.[11],

$$D = \frac{0.9\,\lambda}{B\cos\theta} \quad (1)$$

With D is the particle size, B is the *Full Width Half Maximum*, $\lambda$is the X-Ray wave length, and $2\theta$is the Bragg diffraction angle. This method is an accurate technique to calculatethe particle size in nanoparticles but it is not suitable to calculate particle size in bulk material. Comparator parameter has been calculated by observing the SEM data

2. Experimental Method

The sand material has been obtained from a location in Lemabang-South Sumatera, Indonesia. This sample was rinsed with water and and then dried. First extraction of iron sand has been carried out repeatedly forthirty-five times using a permanent magnet to separate it from the main impurity. And then the sample was milled by mortar in order to separate it from its sticky impurity, followed by the second extraction by a permanent magnet to extricate the iron sand. By assuming that the process has been going smoothly as planned, the authors have expected to be able to obtain least 48 grams of ironsand sample. The sample is then rinsed with 25 ml water and 1 mg detergent, and then stirred until it turns foamy. Twenty-five ml of technical methanol is then added to the sample and the stirring continues until all foam has been cleansed from the sample. The iron sand is then collected using a permanent magnet device. After dehydrating the collected ironsand sample, it isthen divided into four equal parts; the first part of the sample is destined for comparator parameter (0 hours milling), thesecond sample for 2 hours milling, the third sample for 4 hours milling, and the fourth sample for 6 hours milling. The next step is characterization of the first sample or the comparator parameter by using SEM-EDS. Followed by characterization of the second, the third, and the fourth sample by using XRD method. Finally analyzing the collected experimental SEM-EDS data in order to get particle size, bulk material by observed data, nanomaterial by observed XRD data. XRD tabulation has done by using Match Application.

3. Results and Discussion

3.1. Particle Size Calculation of 0 hours milling sample

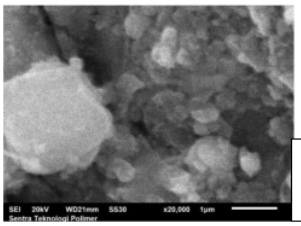

Figure 1. SEM-EDS surface area for 0 hours milling

By comparator line, be obtained iron sand particle size for about 0.5 µm. Minority particle more than 1 µm but majority particle has 0.5 µm.

3.2. Calculation Particle Size of 2 hours milling sample

Before execution of particle size calculation by way of the Scherrer Formula, XRD data must first be refined. Refining has been done by using the Match! Application. And then the corresponding FWHM value was computed for each of the XRD reflection peak. The XRD refinement reflection intensity (two- hours milled sample) are shown in Figure 2 below.

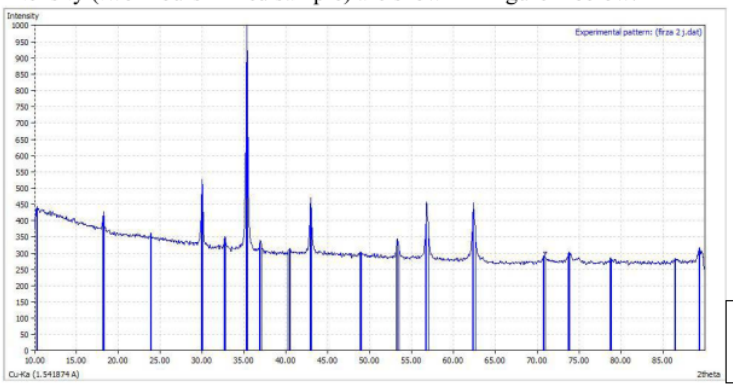

Figure 2. XRD of 2 hours milling after refine

The experimental XRD results was used to calculate the particle size by using Scherrer Formula. Particle Size Calculation results for the two hours milling sample are shown in Table 1.

Table 1. Particle Size Calculation of 2 hours milling

2θ	B_{FWHM} (Degree)	B_{FWHM} (Radian)	D (particle size), nm
10.3100	0.0900	0.001570796	88.70164659
18.2100	0.1800	0.003141593	44.73509856
23.8700	0.2300	0.004014257	35.33274379
29.9800	0.1200	0.002094395	68.5912472
32.7000	0.2000	0.003490659	41.42971027
35.3300	0.2300	0.004014257	36.27964325
36.9300	0.2300	0.004014257	36.44524975
40.4300	0.2600	0.004537856	32.58753274
42.9500	0.2400	0.00418879	35.60002363
48.9300	0.1200	0.002094395	72.7929078
53.3000	0.3300	0.005759587	26.95737754

56.8000	0.3200	0.005585054	28.24586286
62.3800	0.3500	0.006108652	26.5551639
70.8400	0.3100	0.005410521	31.47272919
73.7900	0.1200	0.002094395	82.84867431
78.7700	0.1400	0.002443461	73.47893311
86.4900	0.0800	0.001396263	136.4381314
89.3900	0.1600	0.002792527	69.90519402
Particle Size Average			53.79988166

The average value of particle size after two hours of milling is found to be 53.79988166 nm, and the round-off value is 53.8 nm. It is amazing to get nanoparticles after milling two hours only from the bulk material.

3.3.Calculation Particle Size of 4 hours milling sample.
Before execution of particle size calculation by way of the Scherrer Formula, XRD data must first be refined. Refining has been done by using the Match!Application. And then the corresponding FWHM value was computed for each of the XRD reflection peak. The XRD refinementreflection intensity (4 hours of milling sample) are shown in Figure 3 below.

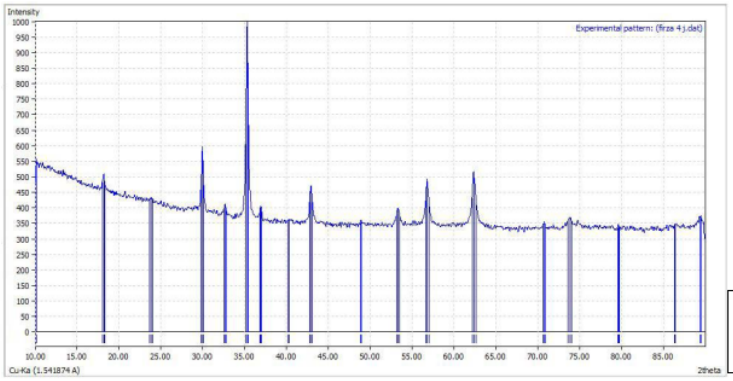

Figure 3. XRD of 4 hours milling after refine

By this result, the angular position of the diffraction peaks was then obtained. This data was then used to calculate the particle size by using the Scherrer Formula, and the results for the sample milled for four consecutive hours.are shown in Table 2.

Table 2. Particle Size Calculation of 4 hours milling

2θ	B_{FWHM} (Degree)	B_{FWHM} (Radian)	D (particle size), nm
10.1300	0.0600	0.001047198	133.0337818
18.1700	0.2600	0.004537856	30.96872227
23.8300	0.3300	0.005759587	24.62403641
29.9600	0.2400	0.00418879	34.29402145
32.6600	0.1900	0.003316126	43.60575856
35.3000	0.2800	0.004886922	29.7986521

36.9300	0.1900	0.003316126	44.1179339
40.3000	0.1600	0.002792527	52.93266274
42.9200	0.3200	0.005585054	26.69726899
48.8900	0.1200	0.002094395	72.78135303
53.2900	0.3700	0.006457718	24.04201363
56.7700	0.3800	0.006632251	23.78262406
62.3400	0.4300	0.007504916	21.61010299
70.8200	0.2800	0.004886922	34.84048325
73.9000	0.4400	0.007679449	22.61139731
79.6500	0.1700	0.00296706	60.89779093
86.4200	0.0800	0.001396263	136.3598121
89.4700	0.0800	0.001396263	139.9070613
Particle Size Average			53. 16141538

The average particle size after four hours of milling time is 53.16141538 nm., rounded-off to the nearest decimal point to 53.2 nm. The average particle size of 4 hours milled sample is smaller than the value of the average particle size in the two-hours milled sample, which is about 0.6 nm smaller.

3.4.Calculation Particle Size of 6 hours milling sample

Before execution of particle size calculation by way of the Scherrer Formula, XRD data must first be refined. Refining has been done by using the Match! Application. And then the corresponding FWHM value was computed for each of the XRD reflection peak. The XRD refinement reflection intensity (six hours of milling sample) are shown in Figure 4 below.

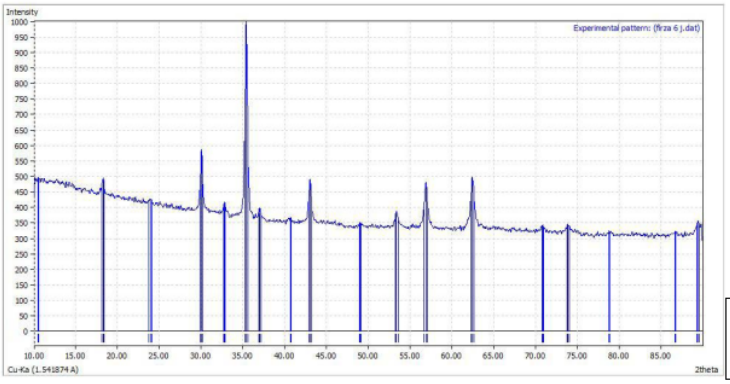

Figure 4. XRD of 6 hours milling after refine

By this result, has been obtained data of diffraction angle. This data was using to calculate particle size by using Scherrer Formula. Here table for 6 hours milling.

Table 3. Particle Size Calculation of 6 hours milling

2θ	B_{FWHM} (Degree)	B_{FWHM} (Radian)	D (particle size), nm
10.5	0.0700	0.00122173	114.0621927
18.2800	0.2300	0.004014257	35.01351147
23.9700	0.3700	0.006457718	21.96765793

30.0600	0.2800	0.004886922	29.40175213
32.8000	0.2300	0.004014257	36.03507415
35.3900	0.3200	0.005585054	26.08034604
36.9800	0.2700	0.004712389	31.05048045
40.7500	0.1000	0.001745329	84.8151294
43.0300	0.3400	0.005934119	25.13633825
49.0200	0.1400	0.002443461	62.4162445
53.3500	0.3400	0.005934119	26.17024663
56.9000	0.3800	0.006632251	23.79722752
62.4400	0.4000	0.006981317	23.24313916
70.8800	0.1000	0.001745329	97.58969325
73.8400	0.3000	0.005235988	33.15033121
78.8200	0.1000	0.001745329	102.9073792
86.7300	0.1000	0.001745329	109.3661815
89.4600	0.2500	0.004363323	44.76638915
Particle Size Average			51. 49829526

Particle size average after 6 hours milling is 51.49829526 nm. The round-off value is 51.5 nm. Particle size of 6 hours milling smaller than 4 hours milling, it is for about 1.7 nm smaller.

4. Conclusion

A longer milling time would result in the smaller particle size and by a significant disparity, as shown at table 4.

Table 4. Particle Size of Iron Sand

No.	Milling Time	Particle Size
1.	0 Jam	0.5 µm
2.	2 Jam	53.8 nm
3.	4 Jam	53.2 nm
4.	6 Jam	51.5 nm

After a milling time of only two-hours, the authors are able to obtain iron sand nanoparticle, furthermoreit is observed that there is a big alteration in size. However by consecutively increasing the milling-time by two-hours for each sample,no big alteration in size has been observed. Only changes ranging from 0.6 nm up to 1.7 nm has been found in this experiment. Therefore based upon this data the authors make the assessment that after 2 hours of milling no further minimization with respect to the particle size would take place, but only a better and more homogenous distribution of particles in the sample.

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