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Magnetic susceptibility, composition, and morphology of iron oxide particles resulting from ball milling of natural sand in the Rokan River

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ABSTRACT

The magnetic susceptibility value and alterations in the composition and dimensions of iron oxide particles in the natural sand from the Rokan River, which were prepared using ball milling (BM), have been measured. Prior to undergoing the BM procedure, the sample's magnetic and non-magnetic particles are initially separated using an iron sand separator and a neodymium iron boron magnet. Subsequently, a 120-gram sample was obtained and subjected to the initial stage of BM for a duration of 80 hours, resulting in the formation of a product referred to as BM1. Next, product BM1 undergoes a second stage of BM with an extended duration of 30, 40, and 50 hours. The resulting products are termed BM2A, BM2B, and BM2C, respectively. The milled balls utilised were a total of 16 combined iron balls with a diameter of 2 cm, 32 combined iron balls with a diameter of 1.5 cm, and 64 combined iron balls with a diameter of 0.7 cm. The findings indicated a positive correlation between the duration of BM rotation and the magnetic susceptibility value. Specifically, the value increased from 11,361.6 in BM1 to 12,398.7 in BM2A, 13,383.4 in BMB, and 14,541.2 in BM2C. The XRF test findings also indicated an increase in the fraction of the magnetic element, Fe, from 38.113% in BM1 to 40.133% in BMA, 41.629% in BM2B, and 42.478% in BM2C. The SEM test findings indicated a decrease in the average particle size of the samples from 696 nm to 401, 356, and 288 nm.

Keywords: Ball milling; magnetic susceptibility; natural sand; NdFeB; SEM; X-ray flourescence

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INTRODUCTION

Indonesia is a country that has an abundance of natural resources, one of the riches that Indonesia itself has is iron sand. We can find iron sand in sand in coastal areas and sand around rivers. Indonesia, with its very unique geological, is home to iron sand deposits. Starting from Banda Aceh, at the northern tip of Sumatra, to Sarmi on the north coast of Papua. There are at least iron sand resources in Indonesia, namely 4 billion tons, while iron sand reserves are 897 million tons [1].

Iron sand contains quite strong magnetic compounds, this is because the number of Fe atoms in iron sand is greater than in other compounds. Apart from that, iron sand also contains Fe_3O_4 compounds and small amounts of titanium, silica, manganese, calcium, and vanadium. As has been reviewed by [2], the magnetic compound most often found in coastal areas or rivers is the Fe_3O_4 compound. In general, iron sand is dark gray or black. With characteristics like these, iron sand can be easily distinguished. with other sand.

The Rokan River is in Ujung Batu City, Riau Province. The distance from Pekanbaru to the Rokan River is 194.5 km or 3 hours 30 minutes by car. It is suspected that Rokan River sand has better quality iron sand compared to other rivers. This can be seen from the black and shiny color of the iron sand. Because of its superiority and uniqueness, this was the reason researchers took samples from the Rokan River.

With these abundant natural resources, we can make the best use of them. According to Yulianto and Cholil in [2], permanent magnet and thin film manufacturing, electronic engineering, and the steel industry are examples of magnetic applications in the industrial sector. Apart from that, according to opinion [1] sandy deposits, especially iron sand, are considered very economical. This is because the minerals it contains can be used in various applications, ranging from magnetic which can be used for making steel which can be used as raw material for titanium. However, unfortunately, the use of iron sand in Indonesia is not good enough.

Particle size greatly influences the quality of a material. The flow properties and compactness of a material can be seen from the size of the powder particles. Larger-sized particles allow it to flow more easily, while smaller-sized particles will form a thicker suspension more easily and quickly [3-5]. Apart from that, [6] adds that when the size of the magnetic particle is reduced to nanometer size, it will have superparamagnetic properties.

Getting the right particle size that matches the target is a challenge for researchers. Priyadarshana [7] said that magnetite nanoparticles can be prepared using top-down and bottom-up mechanisms. Top-down mechanisms are usually created mechanically, namely by crushing magnetite en masse. Natural sand can be reduced from millimeter to micrometer to nanometer size using a tool called ball milling (BM) [8-10]. Meanwhile, the bottom-up method is a way of arranging atoms or molecules and combining them through chemical reactions to form nanostructures, namely using sol-gel techniques, chemical precipitation, and phase agglomeration.

BM is a tool or machine that can be used to smooth or even crush materials to be smaller than their original size which is widely used in the fields of biopharmaceuticals, construction, and other industries [11-13]. The BM machine is in the form of a tube that will continue to rotate at a time and speed set by the operator. By utilizing balls from various materials and various sizes, BM machines can easily do their job. According to [6], several things can influence the results of BM, including the length of milling time, the size of the BM used, and the speed of the BM. These differences can be shown by analyzing the particle size, morphology, and magnetic susceptibility.

Many studies have been carried out using the BM method, one of which is research conducted by [6] who conducted research using a combination of 0.5 cm BM; 0.7 cm and 1.5 cm, and using natural sand originating from the Suci Bengkulu River beach. As well as research in year [8] which used a combination of 60 hours and 100 hours. However, this research is different from previous research. This research uses an iron ball with a BM variation of 0.7 cm; 1.5 cm and 2 cm and the ball is milled in two stages. The first stage for 80 hours is named product BM1 and the second stage with additional time for 30, 40, and 50 hours is named BM2A, BM2B, and BM2C respectively.

It is hoped that this research will be useful for producing even smaller particle sizes, especially in crushing iron sand to nanometer sizes. This research sample was synthesized using the BM method and then characterized using the scanning electron microscope (SEM) to determine its size and the X-ray fluorescence (XRF) to determine its composition.

RESEARCH METHODS

Tools and Materials

The tools and materials used in this research are BM used as a sample crusher, power supply used as an electric voltage source, connecting cable used as a circuit connector, PS-2162 Pasco probe used as a magnetic field meter, solenoid used as a tool to measure magnetic induction, neodymium iron boron (NdFeB) magnets are used as non-magnetic and magnetic particle separators after BM, laptops are used as data storage and processing, XRF is used as a sample particle size identifier and SEM is used as a sample morphology identifier.

Determination of Magnetic Susceptibility Values

A magnetic induction value is required before a magnetic susceptibility assessment is carried out. A magnetic coil of 2500 turns is needed with a length of 10 cm and a diameter of 3 cm. The solenoid is used to measure magnetic induction as a function of current and distance using a PASCO PS magnetic probe and is connected to a laptop running the data studio program. Calculations were carried out with a magnetic induction distance to the electromagnet of 1 mm and determined the current to be 200, 400, 600, 800, and 1000 mA.

There are three magnetic induction measurements, namely as follows:

1. Without natural river sand to obtain the magnetic induction value without a core (B_0).
2. With intact natural sand samples and natural sand samples that have been separated using the iron sand separator (ISS) method, they are inserted into the solenoid alternately and the Bs value and BISS value are obtained.
3. Natural sand samples that have been separated using the ISS method and have been subjected to BM stages 1 and 2.

Determination of magnetic susceptibility (χ_m) is obtained using the formula for the magnetic induction value B0 and with a core (B_T) as stated in (1):

$$\chi_m = \frac{B_T - B_0}{B_0} \tag{1}$$

Sample Separation Process with ISS and NdFeB Magnets

The process of separating iron sand samples from ordinary sand is carried out using the ISS or ISS method. This is a method that separates iron particles containing magnetic and non-magnetic compounds. Then the iron sand samples were weighed as much as the research target, namely 120 g. The sand that has been in BM stages 1 and 2 is then separated again using a NdFeB magnet. This aims to ensure that the sample to be tested only contains magnetic particles.

BM Process

The sand that has been weighed is then put into the BM machine. Here researchers carried out 2 stages of the grinding process. In the BM process, researchers used 16 combined iron balls with a diameter of 2 cm; 32 pieces of 1.5 cm, and 16 pieces of 0.7 cm, 64 pieces. Then the first stage of BM was carried out for 80 hours. Meanwhile, the second stage of the BM process is carried out with an additional time of 30, 40, and 50 hours.

XRF Test

Natural sand samples that had been separated using the ISS method and had been subjected to BM stages 1 and 2 were sent to the Padang State University FMIPA Laboratory to carry out XRF tests. The XRF test was carried out to determine the composition contained in the natural sand samples.

SEM Test

Natural sand samples that had been separated using the ISS method and had been ball milled in stages 1 and 2 were sent to the Diponegoro University FMIPA Laboratory to carry out SEM tests. The SEM test was carried out to determine the size of the particles that had been ball-milled.

RESULTS AND DISCUSSION

Magnetic Susceptibility

Table 1 shows the measurement data for the magnetic susceptibility value of natural sand, ISS products, BM1 for 80 hours, and BM2 (A, B, and C) for 30, 40, and 50 hours.

From Figure 1, it can be seen that the magnetic susceptibility value is increasing. The magnetic susceptibility value of natural sand is 2635.2, an increase in the ISS product of 4993.9. The cause is the separation of magnetic and non-magnetic particles using an ISS machine so that the magnetic content contained in the particles is more than natural sand. The magnetic susceptibility value continues to increase in BM products, namely at BM1 it is 11,361.6, BM2A of 12,398.7, BM2B is 13,383.4, and BM2C of 14,541.2. The preparation and separation process in BM causes the magnetic concentration of BM products (1, 2A, 2B, and 2C) to be higher compared to ISS products.

Table 1. Magnetic susceptibility values (χm) of natural sand, ISS products, BM1 for 80 hours, BM2 (A, B, and C) for 30, 40, and 50 hours.

Product	B_0 (mT)	B_T (mT)	χ_m (10^{-5})
Iron sand	11,574	11,879	2,635.2
ISS	11,574	12,152	4,993.9
BM1	11,574	12,889	11,361.6
BM2A	11,574	13,009	12,398.7
BM2B	11,574	13,123	13,383.4
BM2C	11,574	13,257	14,541.2

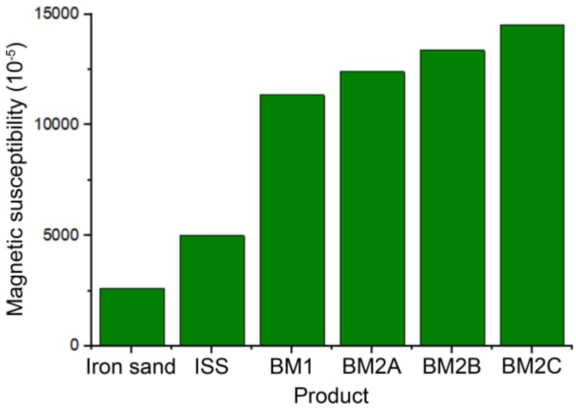


Figure 1. Graph of magnetic susceptibility value of natural sand, ISS products, BM1 for 80 hours, BM2 (A, B, and C) for 30, 40, and 50 hours

Composition of Magnetic Nanoparticles

Identification using XRF of the natural sand of the Rokan River aims to determine the elements contained in the natural sand of the Rokan River which has been in BM stage 1 (BM1) for 80 hours and BM stage 2 (BM2A, BM2B and BM2C) for 30, 40 and 50 hours.

In the first stage of BM for 80 hours, silicon (Si) has a percentage of 32.244%. Meanwhile, in the second stage of BM, Si experienced a percentage decrease of 30.159% in an additional 30 hours; 29.068% in 40 hours of additional time, and 28.564% in 50 hours of additional time. Other non-magnetic elements also experienced a decrease in percentage. Meanwhile, the iron (Fe) element in the first stage of BM for 80 hours had a percentage of 38.113%, while in the second stage of BM, the percentage increased, namely 40.133% in an additional 30 hours; 41.629% in 40 hours of additional time and 42.478% in 50 hours of additional time. And other magnetic elements also experienced a percentage increase.

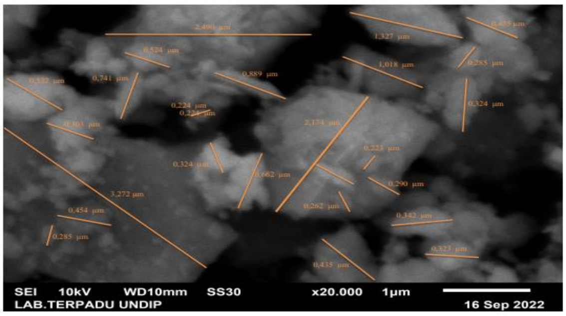
Table 2. Data on Element Identification of Rokan River Natural Sand after BM Stages 1 and 2.

Element	Composition (%)			
	80 hours	110 hours	120 hours	130 hours
Al	7.814	8.167	8.634	9.379
Si	32.244	30.159	29.068	28.564
P	2.394	2.303	1.762	1.439
K	4.335	4.112	3.952	3.267
Ca	10.427	10.384	10.083	9.888
Ti	2.316	2.428	2.502	2.519
V	0.071	0.078	0.079	0.085
Cr	0.061	0.065	0.072	0.079
Mn	0.441	0.297	0.193	0.156
Fe	38.113	40.133	41.629	42.478
Ni	0.024	0.02	0.018	0.009
Cu	0.03	0.03	0.027	0.011
Zn	0.078	0.057	0.042	0.022
Pb	0.024	0.023	0.02	0.005
Other	1.629	1.744	1.919	2.099

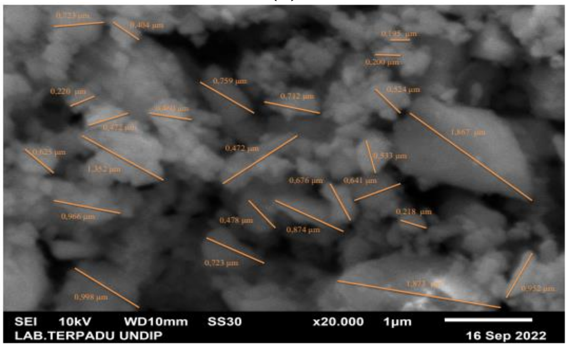
The cause of increasing and decreasing the percentage of each element is influenced by the length of BM time. The longer the grinding time, the smaller the particle size will be, causing the collision process between the walls of the milling tube and the iron balls to become larger so that the size becomes smaller, where the non-magnetic elements that were originally still together will be split so that when they are separated with a magnet Strong NdFeB magnetic particles will be attracted more. Magnetic particles that experience separation will experience an increase in the percentage value, while non-magnetic particles will experience a decrease in the percentage value.

Morphology of Magnetic Nanoparticles

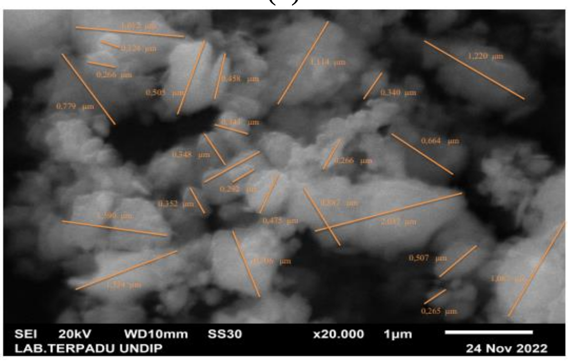
The purpose of testing using a SEM is to determine changes in particle size after BM stages 1 and 2. The results of SEM testing are images with a magnification of 20,000× and can be processed using ImageJ software to obtain the average size of the particles. the. As seen in Figure 2 below.



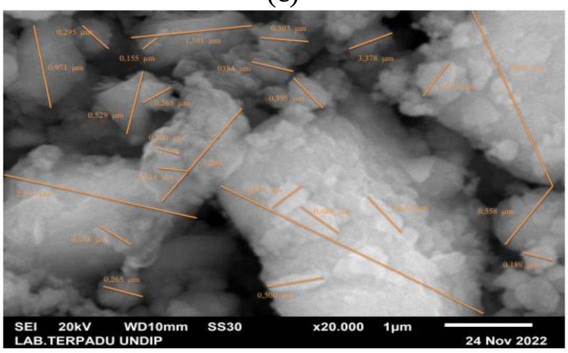
(a)



(b)



(c)



(d)

Figure 2. Size of natural sand particles in Rokan River after BM stages 1 and 2: (a) BM product 1; (b) BM product 2A; (c) BM product 2B; and (d) BM product 2C.

As shown in Figure 2 (a – d) above, the surface shape of the natural sand of the Rokan River which has been in BM stages 1 and 2 tends to be irregular. If you look closely, several nanoparticles have a round and elongated shape. In BM stage 1, the average particle size is 685 nm. Meanwhile, in BM stage 2, the average particle size is 656 nm for 30 hours; 630 nm for 40 hours; and 630 nm for 50 hours. For the average size of the BM product stage 2, namely at an additional time of 40 and 50 hours, the average size of the nanoparticles is the same. This is caused by several particles accumulating. This occurs due to the sample clumping when tested, making it a little difficult to measure the size of the sample nanoparticles.

The results of measurement tests using a SEM show that the longer the BM time, the smaller the particle size produced. Apart from that, this happens according to the physics formula, namely the momentum formula. Where the greater the mass is given, the greater the momentum that occurs. By using combined and varied milling balls, the sand particles and milling balls collide with each other and have great momentum. So the resulting BM product will be smoother.

CONCLUSION

Based on the research data obtained, it can be concluded that the magnetic susceptibility value of Rokan River natural sand which has gone through the BM process has increased along with the increase in BM rotation time of 11361.6 at BM1; 12398.7 on BM2A; 13383.4 on BM2B and 14541.2 on BM2C. The XRF identification results show that the percentage of magnetic element composition increases with increasing BM rotation time, such as Fe, namely 38.113% at BM1; 40.133% in BM2A; 41.629% on BM2B, and 42.478% on BM2C. SEM identification results show that the particle size of the natural sand of the Rokan River becomes smaller as the BM rotation time increases, namely 685 nm at BM1; 656 nm on BM2A; 630 nm on BM2B, and 630 nm on BM2C.

REFERENCES

1. Satria, B., Masrurah, Z. & Fajar, S. J. (2021). Magnetic susceptibility and grain size distribution as prospective tools for selective exploration and provenance study of iron sand deposits: A case study from Aceh, Indonesia. *Heliyon*, **7**(12).
2. Yulianto, Y. W. & Cholil, M. (2024). Landslide susceptibility levels analysis based on geographic information system (GIS) in Simo District, Boyolali Regency. *IOP Conference Series: Earth and Environmental Science*, **1357**(1), 012044.
3. Santoso, U. T., Rodiansono, R., Junaidi, A. B., Ariyanti, C., Oktari, R., Nopitasari, P. & Hasanah, H. (2019). Pengaruh penyaringan dan pengeringan terhadap ukuran partikel oksida besi: Tinjauan karakterisasi kualitatif menggunakan mikroskop optik. *Jurnal Fisika Flux*, **1**(1).
4. Sinuraya, S., Amiruddin, E., Nurrohmah, D. & Wulandari, T. (2021). Analisa perubahan suseptibilitas magnetik dan komposisi partikel pasir alam sungai rokan sebagai fungsi kecepatan putar tabung ball milling. *Komunikasi Fisika Indonesia*, **18**(3), 225–229.
5. Alisna, S. & Sinuraya, S. (2021). Pemetaan suseptibilitas magnetik dan penentuan kandungan logam pada air gambut di Kelurahan Tuah Madani Kecamatan Tampan Pekanbaru. *Komunikasi Fisika Indonesia*, **18**(1), 12–17.
6. Laia, S. & Erwin. (2021). Suseptibilitas magnetik dan morfologi nanopartikel oksida besi dipreparasi menggunakan ball milling melalui varias bola milling. *Seminar Nasional Fisika Universitas Riau VI (SNFUR-6)*, **6**(1), 1004.
7. Priyadarshana, G., Kottegoda, N., Senaratne, A., de Alwis, A. & Karunaratne, V. (2015). Synthesis of magnetite nanoparticles by top-down approach from a high purity ore. *Journal of Nanomaterials*, **2015**(1), 317312.
8. Sinuraya, S., Amiruddin, E., Wahyuni, L. & Jesika, N. (2021). Analysis of the size and composition of natural sand particles in the Rokan River Riau Province as a function of ball milling time. *Journal of Aceh Physics Society*, **10**(3), 80–83.
9. Sirait, R. A., Salomo, S., Muhammad, J. & Taer, E. (2022). Sintesis dan karakterisasi nanopartikel oksida besi menggunakan metode ball milling dan kopresipitasi. *Indonesian Physics Communication*, 19(2), 91–98.
10. Kurniawan, R., Salomo, S., Erwin, E. & Defrianto, D. (2022). Pengaruh doping mangan terhadap komposisi dan sifat kristalinitas partikel oksida besi pasir alam Sungai Rokan dipreparasi dengan metode ball milling. *Indonesian Physics Communication*, **19**(2), 113–118.
11. Li, Z., Wang, Y., Li, K., Lin, W. & Tong, X. (2018). Study on the performance of ball mill with liner structure based on DEM. *Journal of Engineering & Technological Sciences*, **50**(2).
12. Salomo, S. & Yosefi, N. R. (2022). Pengaruh doping tembaga terhadap suseptibilitas magnetik dan komposisi serta sifat kristalinitas pasir alam sungai rokan dipreparasi dengan metode ball milling. *Indonesian Physics Communication*, **19**(3), 154–160.
13. Nurhidayah, I., Sinuraya, S., Amiruddin, E. & Setiadi, R. N. (2023). Analisa perubahan suseptibilitas dan komposisi serta ukuran partikel oksida besi sebagai fungsi kecepatan putaran tabung ball milling. *Indonesian Physics Communication*, **20**(1), 75–82.