

Statistical Analysis of the Relationship between the Sintering Process and Magnetic Energy on the Improvement of the Magnetic Properties of the Permanent Magnet BaFe₁₂O₁₉

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Abstract

Permanent magnets are one of the supporting materials for components in electronic equipment. In particular, NdFeB magnets are usually used in low-speed generators. Another alternative that can be used is BaFe₁₂O₁₉. A study has been carried out using the sintering process to obtain the value of the magnetic energy of BaFe₁₂O₁₉. The magnetic energy data obtained were processed using Multiple Linear Regression Analysis to examine the relationship between the sintering process and magnetic energy in increasing the magnetic properties of BaFe₁₂O₁₉. The predictor variables are temperature, T (deg C) and time, t (hours), and response variable is magnetic energy. The temperature levels are 750 deg C, 800 deg C and 850 deg C and the time levels are 8 hours, 10 hours and 12 hours. It was found that there was a very significant relationship between the sintering process and magnetic energy in increasing the magnetic properties of the permanent magnet BaFe₁₂O₁₉ (p-value > 0.01) and R² = 83.61%. The resulting model can be used to predict the average value of magnetic energy if the sintering process is carried out at a certain temperature and time. If the temperature is 900 deg C and the time 10 hours, the predicted mean value of magnetic energy is 113.611 T.kA/m.

Keywords

Permanent magnet, sintering process, magnetic energy, multiple linear regression and significant.

1. Introduction

Permanent magnets are one of the supporting materials for components in electronic equipment. In particular, NdFeB magnets are usually used in low-speed generators (Prasetijo et al. 2012). Another alternative that can be used is a ferrite-based multiferoic material $\text{BaFe}_{12}\text{O}_{19}$ (barium hexaferrite) (Andreas et al. 2018).

$\text{BaFe}_{12}\text{O}_{19}$ is known to have good magnetic properties and is widely used in magnetic devices to store energy in static magnetic fields. This can be seen from the magnetic energy value of the material, which will determine the quality of the magnet and will subsequently affect the electric power (Andreas et al. 2018).

Purnama et al. (2015) have conducted research on the synthesis of $\text{BaFe}_{12}\text{O}_{19}$ material to determine the magnetic and electrical properties of $\text{BaFe}_{12}\text{O}_{19}$ material. From this research, it was found that temperature affects the electrical properties of $\text{BaFe}_{12}\text{O}_{19}$ material, especially when the temperature is above 500°C .

Widanarto et al. (2015) have conducted research to improve the magnetic properties of ferrite magnets, one of which is by controlling the temperature of the sintering process. Barium hexaferrite was synthesized by the solid reaction method at a sintering process temperature of 1100°C . From this study, it was found that the magnetic properties increased significantly at 1100°C .

Suastiyanti (2018) has conducted research to improve the magnoelectric properties of ferrite-based ceramic materials (BiFeO_3). It is known that if this material is subjected to the effect of an external magnetic field it will produce an electric voltage response. This study aims to synthesize BiFeO_3 which produces the highest voltage response. The method used is the sol-gel method and material characterization by X-Ray Diffraction test. The calcination process was carried out for 4 hours at a temperature of 210°C and 230°C , while the sintering process was carried out at a temperature of 560°C for 4, 6 and 8 hours. From this study, it was found that the highest electrical voltage response was approximately 220 volts, obtained from powders that were treated with a calcination temperature of 230°C with a sintering process temperature of 560°C for 8 hours.

Andreas et al. (2018) have conducted research to obtain the highest magnetic properties of $\text{BaFe}_{12}\text{O}_{19}$ through variations in the sintering process treatment. The research sintering process variables were the sintering process temperature (750°C , 800°C and 850°C) and the sintering process (8 hours, 10 hours and 12 hours). The magnetic properties studied were remanent properties, coercivity properties and saturation properties. The research was conducted using the Thermo Gravimetric Analyzer/ Differential Thermal Analysis (TGA/DTA) method. From the data of the three magnetic properties of materials obtained data of magnetic energy. Based on the results of Andreas, K. et al's research, it was found that the highest magnetic energy was when the sintering temperature was 850°C and the sintering time was 12 hours.

Suastiyanti, D., et al. (2019) conducted a study to improve the magnetic properties of a solid solution of BiFeO_3 - $\text{BaFe}_{12}\text{O}_{19}$. BiFeO_3 - $\text{BaFe}_{12}\text{O}_{19}$ was synthesized using the sol-gel method, with a weight ratio of 1: 1. Magnetic properties were tested using a permagraph test. The sintering process was carried out at temperatures of 750°C , 800°C and 850°C , while the sintering time was 8, 10 and 12 hours. From this study, it was found that the highest magnetic energy of 10,716 GkA/m was obtained from powders that were treated with a sintering temperature of 850°C and a sintering time of 12 hours.

One of the materials synthesis techniques is the solid-state reaction technique. This technique is a method of heating at high temperatures and in a relatively long time to form solid materials. This is one of the nanotechnology in material synthesis that has developed recently which is useful for increasing battery efficiency (Cho 2012).

Haque et al. (2014) has conducted research by synthesizing spinel type ferrite material $\text{Mg}_{0.35}\text{Cu}_{0.20}\text{Zn}_{0.45}\text{Fe}_{1.94}\text{O}_4$ using the solid-state reaction technique. According to Haque et al. (2014) sintering time can affect the dielectric behavior of ferrite $\text{Mg}_{0.35}\text{Cu}_{0.20}\text{Zn}_{0.45}\text{Fe}_{1.94}\text{O}_4$.

Another study using the solid-state reaction technique was conducted by Zabotto et al. (2012) for the preparation of nickel ferrite (NiFe_2O_4) material. This study aims to evaluate the structure, microstructure, electrical properties and magnetic properties of nickel ferrite material (NiFe_2O_4). From this study, it was found that high temperatures increase the saturation properties.

Huang et al. (2014) has conducted research on the microstructure and electrical properties of ferrite-based magnets,

BiFeO₃ (bismuth ferrite). BiFeO₃ synthesis was carried out using a sol gel. Then the sintering process was carried out at temperatures of 800°C, 820°C, 850°C, 880°C and 900°C to see its effect on the microstructure and electrical properties of BiFeO₃.

Zhang et al. (2019) has conducted a study to analyze the effect of the sintering process on the magnetic and electrical properties of the multiferroic barium hexaferrites material. The study observed differences in the microstructure and electrical properties of sintered barium hexaferrites in different N₂ and air atmospheres.

According to Ismail et al. (2017) there is an influence of the microstructure of magnetic materials on magnetic properties, especially in ferrite-based multiferroic materials. Ismail, I. et al. conducted research on several ferrite-based multiferroic materials. The sintering process is carried out in the form of single-sample sintering (SSS) and multi-sample sintering (MSS). With the sintering process at various temperatures, it is found that there is a change in the microstructure of the material under study and ultimately affects the magnetic properties of the material. The higher the temperature, the higher the saturation properties. In contrast, the coercivity property initially increased, but then decreased.

Samosir and Simamora (2020) have performed statistical analysis of the above data to statistically examine the effect of the sintering process on the magnetic properties of ferrite-based multiferroic materials. From the results of the analysis, it was found that for the Ba : Fe = 1 : 11.5 ratio and with a significance level of = 0.07, there was a significant relationship between the sintering process and the magnetic properties of ferrite-based multiferroic materials (barium hexaferrite). Data analysis was carried out using the Multiple Linear Regression Model and data processing was carried out using the Minitab 19 program.

In the regression model, two variables are defined, namely the predictor variable or independent variable written with the notation x_1, x_2, \dots, x_k , and the response variable or dependent variable written with the notation y . In general, we want to know how changes in the predictor variables affect the value of the response variable (Sembiring 1995, Mendenhall and Sincich 2012).

In this study, a statistical analysis of the relationship between the sintering process and magnetic energy was carried out to increase the magnetic properties of the BaFe₁₂O₁₉ permanent magnet. The predictor variables of the study

2. Data

2.1. Material Magnetic Properties Data

The data in this study are secondary data from the research of Andreas et al. (2018). From the results of the analysis carried out using the permagraph test equipment, data on the magnetic properties of the material were obtained, namely remanent properties (T), coesivity properties (kA/m) and saturation properties (T). The data are presented in Tables 1, 2 and 3.

Table 1. Remanent Properties

Temperature, T (°C)	Sintering Time, t (hours)		
	8	10	12
750	0.097	0.10	0.109
800	0.101	0.128	0.187
850	0.121	0.156	0.199

Table 2. Coercivity Properties

Temperature, T (°C)	Sintering Time, t (hours)		
	8	10	12
750	201.4	200.1	211.3
800	226.5	356.4	389.4
850	298.9	402.9	400.9

Table 3. Saturation Properties

Temperatur (°C)	Sintering Time, t (hours)		
	8	10	12
750	0.10	0.13	0.13
800	0.15	0.23	0.28
850	0.22	0.31	0.33

2.2. Magnetic Energy Value

The value of magnetic energy (EM) is calculated using the formula: $EM = R \times K$ where R is the remanent value and K is the coercivity value. By using this formula for the combination of the sintering process temperature values (T) 750°C, 800°C, 850°C and the sintering process time (t) 8 hours, 10 hours and 12 hours, the value of Magnetic Energy (EM) is obtained as presented in Table 4.

Table 4. Magnetic Energy

Temperature, T (°C)	Time, t (hours)		
	8	10	12
750	19.5358	20.01	23.0317
800	22.8765	45.6192	72.8178
850	36.1669	62.8524	79.7791

3. Methodological Approach

Multiple Linear Regression Model (Sembiring 1995, Mendenhall and Sincich 2012) for the data in Table 4 is

$$y = E(y) + \varepsilon \quad (1)$$

This model consists of two components, namely the deterministic component, $E(y)$; read “y mean”, and the random error component. The deterministic component is given in Equation 2 below.

$$E(y) = \beta_0 + \beta_1 x_1 + \beta_2 x_2 \quad (2)$$

where y is the value of Magnetic Energy (ME) in T.kA/m, x_1 is the sintering process temperature, T (°C) and x_2 is the sintering process time, t (hours). So the model in Equation 1 can be written as

$$y = \beta_0 + \beta_1 T + \beta_2 t + \varepsilon \quad (3)$$

$$H_0 : \beta_{1,0} = \beta_{2,0} = 0 \text{ with alternative hypothesis } H_1 : \text{at least one beta} \neq 0$$

The rejection criteria for H_0 can be seen from the p-value in the coefficient table at the output of data processing. If the p-value is smaller than the significance level criterion $\alpha = 0.05$, then H_0 is rejected, and the test is said to be significant, which means that there is a significant relationship between the sintering process and magnetic energy in increasing the magnetic properties of the $\text{BaFe}_{12}\text{O}_{19}$ permanent magnet. If the p-value is less than $\alpha = 0.01$, the test is said to be very significant, which means that there is a very significant relationship between the sintering process and magnetic energy in increasing the magnetic properties of the $\text{BaFe}_{12}\text{O}_{19}$ permanent magnet. If

the p-value is greater than the criterion $\alpha = 0.05$, then H_0 is accepted that there is no relationship between the sintering process and magnetic energy in increasing the magnetic properties of the $\text{BaFe}_{12}\text{O}_{19}$ permanent magnet.

If the null hypothesis is rejected, further analysis is carried out on the resulting regression model. The accuracy of the model can be seen from the analysis of variance table (ANOVA). Inference to the accuracy of the model using the F test statistic:

$$F = \frac{\text{MSR}}{\text{MSE}} = \frac{\text{SSR} / \text{df}(\text{SSR})}{\text{SSE} / \text{df}(\text{SSE})} \quad (5)$$

If the value of $F > F_{\alpha, 1, (n-2)}$ then the model is suitable for observational data. This can be seen from the p- value in the ANOVA table, i.e. if the p-value $< \alpha$, the model is suitable to express the relationship between the sintering process and magnetic energy in increasing the magnetic properties of the $\text{BaFe}_{12}\text{O}_{19}$ permanent magnet.

Another criterion used is the value of R^2 (%). The best criterion is 100%. In this study, the results of the analysis are said to be good, in the sense that the suitability of the regression model used to express the functional relationship between the sintering process and magnetic energy in increasing the magnetic properties of the $\text{BaFe}_{12}\text{O}_{19}$ permanent magnet is appropriate or suitable, if the value of $R^2 \geq 85$.

If the model is suitable, in the end analyze the magnitude of the influence of the two sintering process variables on the average value of magnetic energy which can be interpreted through the estimated values of β_1 and

4. Result and Discussion

$\text{BaFe}_{12}\text{O}_{19}$ (barium hexaferrite) is a ferrite-based multiferroic material that has good magnetic properties which in turn has good electrical properties especially if synthesized at temperatures above 500°C (Purnama et al. 2015). Widanarto et al. (2015) conducted a study to improve the magnetic properties of barium hexaferrite synthesized by the solid reaction method. Magnetic properties increase significantly if the sintering process temperature is 1100°C . In research by Suastiyanti (2018), a ferrite-based ceramic material (BiFeO_3) was synthesized using the sol- gel method. From this study, it was found that the highest electrical voltage response was approximately 220 volts, obtained from powders that were treated with a calcination temperature of 230°C with a sintering process temperature of 560°C for 8 hours.

In research by Andreas et al. (2018), it was found that the magnetic energy of $\text{BaFe}_{12}\text{O}_{19}$ ferrite-based material can be increased by the sintering process. From this research, it was found that with the sintering process the highest magnetic energy occurred at a temperature of 850°C and a sintering process time of 12 hours.

Suastiyanti et al. (2019) conducted a study to improve the magnetic properties of a solid solution of BiFeO_3 - $\text{BaFe}_{12}\text{O}_{19}$ and found that the highest magnetic energy of 10,716 GkA/m was obtained from powders that were treated with a sintering temperature of 850°C and a sintering process time of 12 hours.

From several searches, it can be seen that there are still few research results that analyze how much influence the sintering process temperature and sintering time have on the magnetic properties of ferrite-based multiferroic materials, using statistical analysis. Therefore Samosir and Simamora (2020) analyzed the Andreas et al. (2018) data using the regression method to see the relationship between the sintering process and the magnetic properties of ferrite-based multiferroic materials. From the results of statistical analysis, it was found that there was a significant relationship between the sintering process and the remanence, coercivity and saturation properties.

This research continues the results of Samosir and Simamora's research (Samosir and Simamora 2020), namely to analyze the relationship between the sintering process and magnetic energy on increasing the magnetic properties of

The results of hypothesis testing for the parameters of the multiple linear regression model (β_0 , β_1 and β_2) are given in Table 5 below.

Table 5. Hypothesis Testing Model Parameters

Term	Coef	SE Coef	T-Value	p-value
Constant	-348.3	76.6	-4.55	0.004
Temperature	0.3874	0.0912	4.25	0.005
Time	8.09	2.28	3.55	0.012

with the value of R^2 is 83.61%. From Table 5 above it can be seen that:

$$\hat{\beta}_1 = 0,3874 \text{ dan } \hat{\beta}_2 = 8,09$$

This means that for every 1°C increase in sintering temperature, the average value of magnetic energy (ME) will increase by 0.3874 T.kA/m and for every 1 hour increase in sintering time, the average value of magnetic energy (ME) will increase by 8.09 T.kA/m.

Figure 1 is gives a surface graph depicting a linear relationship between the sintering process and magnetic energy on the improvement of the magnetic properties of the permanent magnet $BaFe_{12}O_{19}$ with a mole ratio of Ba: Fe = 1 : 11.5. This graph shows that there is a trend of increasing the average value of magnetic energy along with the increasing temperature of the sintering process and the time of the sintering.

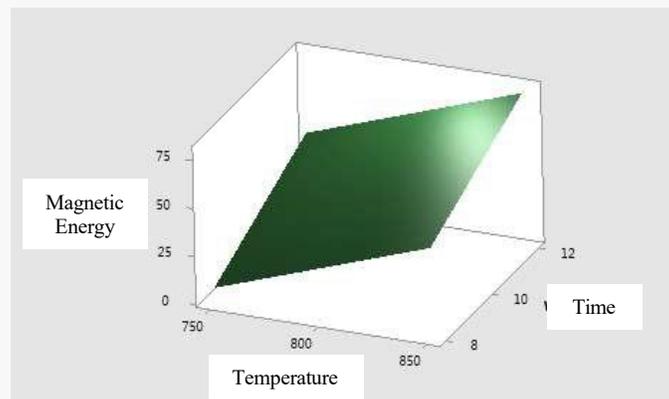


Figure 1. Surface graph of the sintering process with magnetic energy

The ANOVA table for the data in Table 4 is given in Table 6.

Table 6. Analysis of Variance (ANOVA)

Source	df	Sum of Squares	Mean Square	F-value	p-value
Regression	2	3821.0	1910.5	15.30	0.004
Temperature	1	2251.2	2251.2	18.03	0.005
Time	1	1569.8	1569.8	12.57	0.012
Error	6	749.2	124.9		
Total	8	4570.2			

Based on the ANOVA table above, the F value in Equation 5 is obtained as follows:

$$F = \frac{MSR}{MSE} = \frac{SSR / df(SSR)}{SSE / df(SSE)} = \frac{3821/2}{749.2/6} = \frac{1910.5}{124.9} = 15.3$$

F-value = 15.3 with p-value = 0.004 < α = 0.05. So it can be said that the model is very adequate to describe the relationship between the sintering process and magnetic energy in increasing the magnetic properties of $BaFe_{12}O_{19}$ permanent magnets with a mole ratio of Ba: Fe = 1:11.5. Thus, we can use the model to make predictions

and confidence intervals for the average magnetic energy value for a certain value of temperature and time for the sintering process as follows.

1. Temperature 750°C and time 12 hours.

Table 7. Settings T = 750°C, t = 12 hours

Variable	Setting
Temperature	750
Time	12

Table 8. Prediction

Fit	95% Confidence Interval	95% Prediction Interval
39.3258	(21.0970, 57.5546)	(6.46341, 72.1882)

2. Temperature 850°C and time 12 hours.

Table 9. Settings T = 850°C, t = 12 hours

Variable	Setting
Temperature	850
Time	12

Table 10. Prediction

Fit	SE Fit	95% Confidence Interval	95% Prediction Interval
78.0661	7.44971	(59.8373, 96.2949)	(45.2037, 110.928)

3. Temperature 900°C and time 14 hours.

Table 11. Settings T = 900°C, t = 14 hours

Variable	Setting
Temperature	900
Time	14

Table 12. Prediction

Fit	SE Fit	95% Confidence Interval	95% Prediction Interval	
113.611	13.4301	(80.7488, 146.474)	(70.8609, 156.361)	X

X denotes an unusual point relative to predictor levels used to fit the model.

In the third prediction, the data used are the temperature and time values of the sintering process, not the data used to construct the model. This can be seen in the prediction output (highlighted in yellow). Using this model, we can predict that if we take a temperature of 900°C and a time of 14 hours, we get an average value of magnetic energy is 113.611 T.kA/m.

5. Conclusion

The regression equation for the relationship between the sintering process and magnetic energy on increasing the magnetic properties of the permanent magnet BaFe₁₂O₁₉ with a ratio of Ba : Fe = 1:11.5 is given as follows: with the value of R² is 83.61%.

This means that for every 1°C increase in sintering temperature, the average value of magnetic energy (ME) will increase by 0.3874 T.kA/m and for every 1 hour increase in sintering time, the average value of magnetic energy (ME) will increase by 8.09 T.kA/m.

F-value = 15.3 with p-value = $0.004 < \alpha = 0.05$. So it can be said that the model is very adequate to describe the relationship between the sintering process and magnetic energy in increasing the magnetic properties of $\text{BaFe}_{12}\text{O}_{19}$ permanent magnets with a mole ratio of Ba: Fe = 1:11.5. Thus, we can use the model to make predictions and confidence intervals for the average mechanical energy value for a certain value of temperature and time for the sintering process. By using this model we can predict that if the sintering temperature is 900°C and the sintering time is 14 hours, the average value of magnetic energy is 113.611 T.kA/m.

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