



THE EFFECT OF TEMPERATURE ON CEMENT CLICKER CHARACTERIZATION WITH A 10% SUBSTITUTION OF BASALT STONE ON LIMESTONE MASS

EL EFECTO DE LA TEMPERATURA EN LA CARACTERIZACIÓN DE CLICKER DE CEMENTO CON UNA SUSTITUCIÓN DEL 10% DE LA PIEDRA DE BASALTO EN LA MASA DE PIEDRA CALIZA

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ABSTRACT

This research was conducted to determine the effect of temperature on the chemical content and crystal structure of the cement clinker with 10% substitution of basalt on the mass of limestone. The cement clinker is made from the composition of limestone, basalt stone, clay, silica stone and iron sand. Basalt stone used came from the East Lampung area. The basalt stone has fulfilled ASTM C618 standard with a total compound of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ of 79.2%. The raw material was then mixed, milled, formed into pellets with a size of 0.8 - 1.4 cm and continued with combustion at calcination temperatures of 1000 °C, 1100 °C and 1200 with a holding time of 2 hours. Based on the results of the XRF test (X-Ray Fluorecence) of the three cement clinker samples with variations in temperature used, the sample is closer to the standard at a temperature of 1000 °C. For the XRD (X-Ray Diffraction) test results, the three samples have tetragonal, monoclinic, rhombohedral, and orthorhombic crystal structures. As for the MO test results, there was a strong binding between iron sand, limestone, clay, and silica sand characterized by a smooth surface while basalt stones that look like black holes were not completely bound.

Keywords: Basalt Stone; Limestone Mass; Cement; Temperature.

RESUMEN

Esta investigación se realizó para determinar el efecto de la temperatura sobre el contenido químico y la estructura cristalina del clínker de cemento con un 10% de sustitución de basalto sobre la masa de piedra caliza. El clínker de cemento está hecho de la composición de piedra caliza, piedra de basalto, arcilla, piedra de sílice y arena de hierro. La piedra de basalto utilizada vino del área de East Lampung. La piedra de basalto

ha cumplido el estándar ASTM C618 con un compuesto total de $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ del 79,2%. La materia prima se mezcló, se molió, se formó en gránulos con un tamaño de 0,8 a 1,4 cm y se continuó con la combustión a temperaturas de calcinación de 1000 °C, 1100 °C y 1200 con un tiempo de retención de 2 horas. Según los resultados de la prueba XRF (fluorescencia de rayos X) de las tres muestras de clínker de cemento con variaciones de temperatura utilizadas, la muestra está más cerca del estándar a una temperatura de 1000 °C. Para los resultados de la prueba de XRD (difracción de rayos X), las tres muestras tienen estructuras cristalinas tetragonales, monoclinicas, romboédricas y ortorrómbicas. En cuanto a los resultados de la prueba de MO, hubo una fuerte unión entre arena de hierro, piedra caliza, arcilla y arena de sílice caracterizada por una superficie lisa, mientras que las piedras de basalto que parecen agujeros negros no estaban completamente unidas.

Palabras Clave: Piedra de basalto; Masa de piedra caliza; Cemento; Temperatura.

1. INTRODUCTION

Cement is an important construction material, which is widely used in civil engineering throughout the world (Laibao *et al.*, 2013; Marzuki, 2009). As infrastructure development in Indonesia increases, so does the demand for the cement industry. The need of cement causes the increase of cement consumption level in Indonesia increase by year (Putri & Ramadhani, 2016). In order to improve the quality of cement, building construction continues to be improved including the quality of materials used (Waani & Elisabeth, 2017). According to experts working on observations on global warming, 7% of the production of CO₂ emissions in nature is derived from cement production. It is also known that about 1 ton of cement produces 1 ton of CO₂ (Uzal, B., Turanli & dan Mehta, 2007), so this encourages experts in the field of building construction engineering to look for alternative materials to replace or substitute cement. In the manufacture of cement preceded in the manufacture of clinker that is the process of changing the physical and chemical raw materials into clinker, where the clinker has undergone a cooling process and then grinding (grinding process). Clinkers are "semi-finished" materials during cement production, produced by burning limestone and alumina-silicate-containing materials such as clay (Rahmawatie & Damayanti, 2017). In making cement, the readiness of the main raw materials such as limestone is very important, where limestone in the formation of cement requires a composition of 80%. So that if their needs are not met, the results will unmet demand and the cessation of the production process (Fitriadi & Utama, 2014; Ishlah, 2008; Kahn, 2002). Therefore an alternative material is needed to prevent the scarcity of raw materials, but it has a composition that is similar to limestone, so it does not affect the quality of cement produced. Pozzolan material such as basalt is a material from the coal industry that can be used as a substitute material in cement (Waani & Elisabeth, 2017). The amount of basalt stone material reserves in Lampung Province is 318,480,000 tons and has not been explored optimally. Based on the analysis of the chemical composition of scoria basal stone material from Labuhan Maringgai, East Lampung, Indonesia, namely $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ is 78.66%. Basuhan scoria Labuhan Maringgai East Lampung, Indonesia fulfills ASTM C618 (American Society for Testing and Materials C168) requiring that the chemical component is a $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ minimum of 70% (Ningsih *et al.*, 2012; Oktamuliani *et al.*, 2015; Patty, 2013). So many reserves have only been used as a foundation for housing construction. By optimizing basalt minerals, the economic value will increase, for this reason it is necessary to conduct research on the characteristics of basalt minerals as an alternative raw material for cement clinkers (Amin, 2017). When basalt reacts with other chemicals it does not produce reactions that are harmful to the environment. Therefore basalt is suitable for applications, especially in the field of ceramics (Alisjahbana *et al.*, 2018; Saraya, 2014). Basalt is chemically rich in magnesium oxide, calcium, sodium, potassium, silicon and iron (Benmohamed *et al.*, 2016; Chirikure *et al.*, 2010; Dhand *et al.*, 2014). Basalt stone can be used as an alternative raw material for Portland cement (Andrade *et al.*, 2010), especially as a substitute for clay raw materials in certain areas where clay resources have begun to be scarce. It is recommended that basalt stones can replace clay in 1: 1 proportions (Andrade *et al.*, 2010; Ardiantono *et al.*, 2018). According to research by El-Desoky *et al.*, 2017,

clinker furnaces with temperatures around 1200-1450 ° C show good combustion (El-Desoky et al., 2017). All samples that have been sintered at 1450 ° C have a unique clinker structure. The dominant mineralogical phases such as alite, belite, calcium aluminate, ferrite and lime crystallized to produce a peak of 2 Theta (2θ). The levels of various types of reactions in clinkers according to El-Hafiz et al., 1971, the levels of various types of reactions in clinkers that are affected by temperature are as follows (El-Hafiz et al., 2015):

100-500 °C	The clinker is dried and the water content evaporates.
500-600 °C	Dehydroxylation from clay $2\text{SiO}_2\text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O} \rightarrow 2\text{SiO}_2 + \text{Al}_2\text{O}_3 + 2\text{H}_2\text{O} \uparrow$
700 °C	Silicate activation and water evaporation
700-900 °C	Decarbation of calcium carbonate with the main combination of alumina, ferric oxide
900-1200 °C	Belite formation from $2\text{CaCO}_3 + \text{SiO}_2 \rightarrow \text{C}_2\text{S} + \text{CO}_2 \uparrow$
>1250 °C	Liquid phase formation (aluminate and ferrite)
1300 °C	The liquid phase appears and the C ₂ S reaction with CaO forms to form C ₃ S
1450 °C	As the reaction is complete, alite and belite increase scattered in several small sizes of limestone

Based on the problems and solutions above, a study was carried out on the effect of temperature in the processing of cement clinkers by substituting (10%) of basalt against the mass of limestone in the process of making cement clinkers. Cement clinker was made with the composition of substitution limestone basalt, clay, silica stone, iron sand.

2. RESEARCH METHODOLOGY

Sample preparation (Pellet making), sample preparation was carried out in the following steps:

Raw materials such as limestone, basalt, clay, silica sand and iron sand were prepared with a percentage of mass in 2 kg, namely basalt (8% = 160 g), limestone (72% = 1440 g), clay (9 % = 180 g), silica as much (10% = 200 g), and iron sand as much (1% = 20 g). The composition of the raw material was prepared and then ground for 5-6 hours in a ball mill until it becomes a powder mixture. The powder was filtered using 270 mesh sieve, clinker pellet formation using a pelletizing machine with water added to the powder mixture that has been ground with a ball mill and formed into small granules with a size of 0.8-1.4 cm, pellets were dried for 1- 2 days at room temperature, the pellet was heated for 5-6 hours at 100 °C (Suryanarayana, 1998).

The reduction process (heat treatment), the reduction process was carried out with the following stages:

12 dried pellets as many as 12 grains on a gravitational disc were inserted into the muffle furnace with room temperature as the initial temperature, the temperature was raised slowly on the muffle furnace until it reaches the specified temperature that was at a temperature of 1000°C, 1100°C, 1200°C, the process of raising the temperature was stopped when the furnace reached the temperature specified number, sintering carried out for 2 hours, the sample was left at temperature room until the temperature in the sample was equivalent to room temperature (Widyawati, 2012).

3. RESULTS AND DISCUSSION

The raw material used in making cement clinker was characterized by using the XRF method, the results of the XRF analysis on the raw material for making cement clinker are shown in Table 1.

Table 1. Results of XRF analysis of raw materials for cement clinker

Component	Basalt Stone	Lime Stone	Silica Stone	Iron-sand	Clay	Unit
SiO ₂	47,3	4,036	95,2	18,7	53,8	%
Al ₂ O ₃	18,9	1,653	3,1	6,3	27,4	%
Fe ₂ O ₃	12,9	0,552	0,4	58,0	15,5	%

CaO	10,9	92,973	0,8	3,1	0,2	%
MgO	4,4	0,399	-	4,8	0,3	%
TiO ₂	1,3	0,105	0,1	6,7	1,3	%
K ₂ O	0,5	0,104	-	0,2	0,8	%
Na ₂ O	2,8	-	-	-	-	%
MnO	0,1	-	-	0,5	-	%
SO ₃	-	-	-	0,5	-	%
V ₂ O ₅	-	-	-	0,3	-	%
Cr ₂ O ₃	-	-	-	0,1	-	%
Eu ₂ O ₃	-	-	-	0,2	-	%

Based on the XRF analysis data in Table 1, it shows that the chemical content in limestone was dominated by CaO compounds as much as 92.9%, while the remaining compounds were SiO₂, Al₂O₃, MgO, K₂O, TiO₂, and Fe₂O₃. For silica stones, the chemical content was dominated by SiO₂ compounds of 95.2% and the rest were Al₂O₃, CaO, TiO₂, and Fe₂O₃ compounds. For iron sand, the highest chemical content was found in Fe₂O₃ compound at 58.0%, while the rest were MgO, Al₂O₃, SiO₂, SO₃, K₂O, CaO, TiO₂, V₂O₅, Cr₂O₃, MnO, and Eu₂O₃. For the biggest clay compounds, SiO₂ was 53.8% and Al₂O₃ was 27.4%, while the rest were MgO, Fe₂O₃, K₂O, CaO and TiO₂ compounds. As for the chemical content in basaltic rocks, the SiO₂ compound was dominated by 47.3%, Al₂O₃ is 18.9%, and Fe₂O₃ is 12.9%. Based on ASTM C168 pozzolan requirements, the minimum SiO₂ + Al₂O₃ + Fe₂O₃ compound content was $\pm 70\%$. While basalt stone had all three compounds of 79.2%. Thus basalt had fulfilled the requirements as a raw material for pozzolan for making cement clinker.

Furthermore, an analysis of the cement clinker results from combustion, XRF analysis results of the chemical content of the cement clinker sample to the combustion temperature variation that was sintered for 2 hours with a substitution of 10% basalt stone to the mass of limestone is shown in Table 2.

Table 2. Data analysis of XRF analysis of raw materials for cement clinker

Compound	Combustion Temperature Variations				Unit
	1000°C	1100°C	1200°C	ASTM C150	
CaO	60,93	55,64	55,75	65,5 - 66,2	%
SiO ₂	21,18	25,38	26,36	21 - 22	%
Fe ₂ O ₃	6,84	7,11	7,45	4 - 4,5	%
Al ₂ O ₃	8,22	7,57	7,08	5 - 5,5	%
MgO	1,31	1,48	1,86	Max 1,5	%
SO ₃	0,27	1,39	-	Max 1	%
TiO ₂	0,64	0,69	0,67	-	%
MnO	0,12	0,27	0,13	-	%
K ₂ O	0,28	0,26	0,39	-	%

Based on the results of the XRF analysis in Table 5.2, the cement clinker sample powder with the treatment temperature closest to the ASTM C150 standard for CaO content was at 1000 °C, SiO₂ was at 1000 °C, Fe₂O₃ was at 1000 °C, Al₂O₃ was at 1200 °C MgO was at 1000 °C and SO₃ was at 1000 °C. So that it can be said that the cement clinker content with temperature variations closer to ASTM C150 was at a temperature of 1000 °C (ASTM C618, 2008; Bendersky, 2001).

Characterization of the crystal structure of cement clinker samples in powder form was carried out using PANalytical XRD. The cement clinker sample was inserted into the holder then an XRD analysis was performed to see the compounds arranged in the cement clinker sample after the reduction process. The process of reducing the cement clinker was done by heating it in a furnace. In this test using three samples

of cement clinker namely cement clinker heated at temperatures of 1000 °C, 1100 °C, and 1200 °C. Each sample was heated for 2 hours. The results of the cement clinker characterization can be seen in Figure 1, 2 and 3.

XRD test results of cement clinker samples at a temperature of 1000 °C, for cement clinker samples carried out by heating at a temperature of 1000 °C with a holding time of 2 hours and XRD test results obtained as in Figure 1

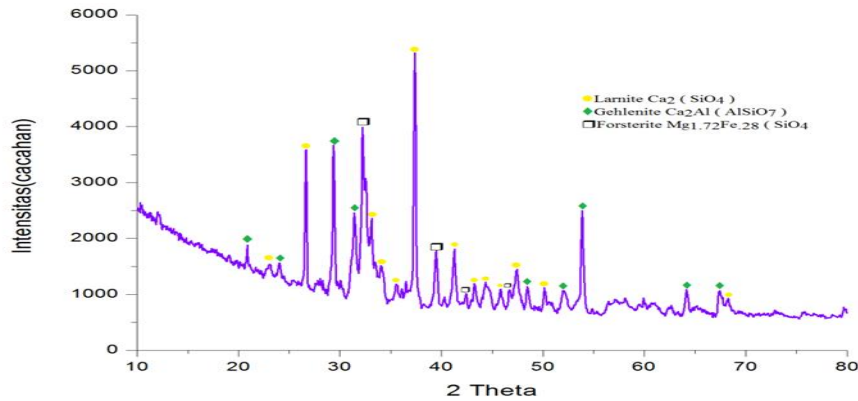


Figure 1. The compounds are arranged in a cement clinker sample at a temperature of 1000 °C with a holding time of 2 hours.

The graph in Figure 1 shows the crystalline phase of the cement clinker sample at 1000 °C with a holding time of 2 hours. The formation of monoclinic crystalline structure with the highest peak of Ca at an angle of 37,3784° with the Larnite phase with chemical compound $\text{Ca}_2(\text{SiO}_4)$. In addition to the position 37.3784° other peaks were at position 2θ (23.1018°, 26.6473°, 28.2071°, 32.5529°, 33.1172°, 34.1503°, 41.2621°, 42.4464°, 43.2717°, 44,3655°, 45,818°, 47,3915°, 50,1739°, 58,1511°, and 68,2573°). In addition to the Larnite phase the Gehlenite compound with the $\text{Ca}_2\text{Al}(\text{AlSiO}_7)$ chemical compound formed a tetragonal crystal structure. In the tetragonal crystal structure with the highest peak was formed with an angle position of 29.33873°. Other peaks are also in the position of 2θ (20.8718°, 24.0519°, 31.4276°, 48.4707°, 52.0544°, 53.8732°, 61.0135°, 64, 1404° and 67.3556°). Then orthorhombic crystal structure formed with the mineral Forsterite with the chemical compound $\text{Mg}_{1.72}\text{Fe}_{.28}(\text{SiO}_4)$ with a peak of 2θ at 32.2177°, and the other peak was also at position 2θ (35.5826°, 36.5026°, 39.4885°, 40.2782°, 46.6843°, 56.581° and 62.6267°).

Cement clinker sample at a temperature of 1100 °C, the cement clinker samples were heated at a temperature of 1100 °C with a holding time of 2 hours and XRD test results are obtained as in Figure 2.

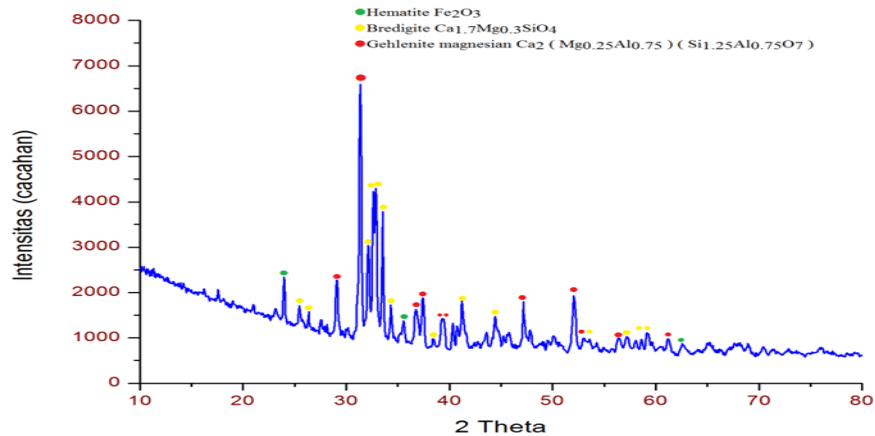


Figure 2. The compounds arranged in a cement clinker sample at 1100 ° C with a holding time of 2 hours

The graph in Figure 2 shows the highest crystalline phase that was a cement clinker sample with temperature of 1000 °C and with a holding time of 2 hours. The formation of a tetragonal crystalline structure with the highest peak belonging to Ca at position 270 31.3703o of magnesian gehlenite phase with chemical compounds that was $\text{Ca}_2(\text{Mg}_{0.25}\text{Al}_{0.75})(\text{Si}_{1.25}\text{Al}_{0.75}\text{O}_7)$. In addition to the 31.3703o position, the other peaks were also in the position of 2θ (23.18181, 29.0868o, 36.6901 °, 37.431o, 39.2519o, 39.4928o, 45.8021o, 47.1941o, 50.1135o, 52.0715o, 53.2772o, 56.33382o, 61.1777o, 65.1016o, 67.949o, and 76.0103o). In addition to the magnesian gehlenite phase, Bredigite compounds with the chemical compound $\text{Ca}_{1.7}\text{Mg}_{0.3}\text{SiO}_4$ were also formed, orthorhombic crystal structures. In this crystal structure the highest peak was formed with an angle position of 32.624o. Other peaks were also in position 2θ (25.4684o, 26.3603o, 27.5439o, 30.1388o, 32.1386o, 32.9119o, 33.5333o, 34, 3317o, 38.4329o, 40.3272 o, 40.7599o, 41.2256o, 43.6143o, 44.4113o, 45.2331o, 47.8456o, 49.5264o, 54.2678o, 57.1423o, 58.6335o, 59.1218o, 68.9647o and 70.4678o). Then formed a rhombohedral crystal structure such as the mineral Hematite with a chemical compound Fe_2O_3 had a peak with a position of 2θ at 23.9873o, with another peak at position 2θ (35.5688o, 62.589o).

Sampel klinker semen pada suhu 1200°C, pada sampel klinker semen ini dilakukan pemanasan *furnace* pada suhu 1200°C dengan waktu tahan selama 2 jam dan diperoleh hasil pengujian XRD seperti pada Figure 3.

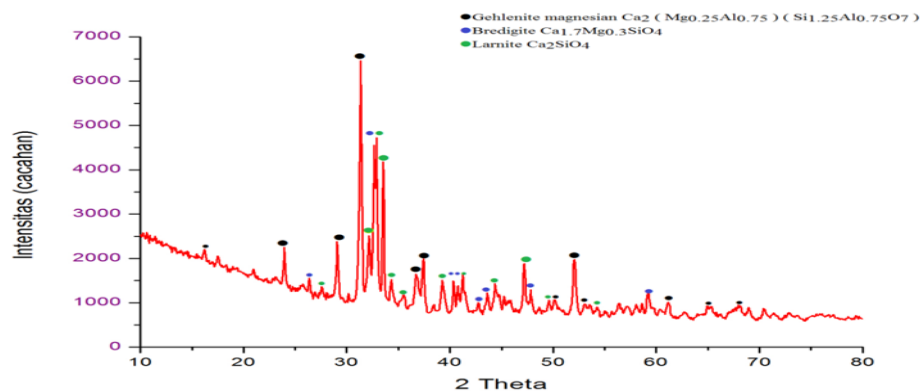


Figure 3. The compounds arranged in a cement clinker sample at 1200 ° C with a holding time of 2 hours

The graph in Figure 3 shows the highest crystalline phase in a cement clinker sample at 1200 °C with a holding time of 2 hours. The formation of a tetragonal crystalline structure with the highest peak of Ca at

an angle of 31.3624° phase of the magnesian Gehlenite phase with chemical compounds was (Mg_{0.25}Al_{0.75}) (Si_{1.25}Al_{0.75}O₇). In addition to the 31.33624° position, the other peaks were also in the 2θ position (16.1989°, 23.9696°, 29.0925°, 36.6928°, 37.4274°, 50.1295°, 52.0412°, 53.0452°, 61.2588°, 62.7966°, 65.0009° and 68.0865°). In addition to the magnesian Gehlenite phase formed Bredigite compounds also had a chemical compound Ca_{1.7} Mg_{0.3} SiO₄ formed orthorhombic crystal structure. In the orthorhombic crystal structure the highest peak was formed with an angle position of 32.6371°. Other peaks were also in the position of 2θ (26.3765°, 33.5475°, 40.3495°, 40.7646°, 42.7593°, 43.6319°, 45.2609°, 47°, 2165°, 47.8648°, 59.1833°, 68.961°, 70.4598° and 72.8017°). Then the Larnite Ca₂SiO₄ mineral formed with monoclinic crystal structure has a score of 33 with the highest peak of 32.9118°. In addition, Larnite minerals had other peaks at position 2θ (27.5745°, 32.1555°, 34.2929°, 35.5609°, 39.2466°, 41.2265°, 44.33657°, 45.6496°, 49.5934°, 54.2816°, 56.4078°, 57.2068°, and 58.6243°).

4. CONCLUSION

Based on research that has been done, it can be concluded that the variation of combustion temperature can affect the crystal structure and chemical content of the cement clinker. The element content obtained from the XRF test was dominated by CaO, SiO₂, Fe₂O₃, and Al₂O₃. Based on ASTM C150 results from the three temperature variations used, the cement clinker sample is closer to the standard at 1000 °C. Based on the XRD test results, the three samples had tetragonal, monoclinic, rhombohedral, and orthorhombic crystal structures. The highest peaks were at 1100 °C and 1200 °C with tetragonal crystalline phase. As for the MO test results, there was a strong binding between iron sand, limestone, clay, and silica sand characterized by a smooth surface while basalt stones that look like black holes were not completely bound.

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