The Effect of Addition Basalt Stone and Coal as Substitution Material for Producing Cement Clinker

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ABSTRACT

National infrastructure development in Indonesia requires supporting materials, one of which is cement. This increases cement clinker production. The composition of limestone as the main raw material required in the manufacture of clinker is 81%. Basalt is used as an alternative raw material because it contains SiO_2, Al_2O_3, and Fe_2O_3 with a total of >70%. It meets the chemical requirements of ASTM C618 as a substitute material for cement making. This study aims to determine the effect of adding 10% basalt rock and coal variations as a limestone substitution in cement clinker manufacture. Coal is obtained from the Tanggamus district with the variations used, namely 5, 10, 15, and 20% outside the total mass. Raw materials such as limestone, silica stone, clay, basalt stone, coal, and iron sand are ground with a *ball mill* and formed into *pellets* followed by burning at sintering temperatures of 1100, 1200, and 1300 °C with a holding time of 2 h. The clinker was analyzed using XRF (X-Ray Fluorescence), XRD (X-Ray Diffraction), and OM (Optical Microscope). This study aims to know the effect of coal on the cement clinker. The results showed that the manufacture of cement clinker at 15% substitution of coal at 1300 °C obtained a CaO compound of 66.384% and SiO_2 of 22.747% with sample results close to ASTM requirements (C150-99). The phases formed are alite, belite, aluminate, and ferrite. From microanalysis with a 100 µm magnification, it can be seen that the phases alite and belite.

Keywords: alite; basalt stone; belite; cement clinker; coal.

1 Introduction

Clinker is the main ingredient in cement manufacturing, where 70% to 95% is produced from the combustion process in a thermal chamber, which contains limestone, silica sand, iron sand, and clay, which form granules [1]. The results of this clinker can form cement by grinding the clinker with the addition of gypsum and pozzolan, which is carried out in a cement grinding machine that contains iron balls so that the clinker becomes powder. Semen is a mixture of calcium silicate and a small amount of calcium aluminate [2]. The main compounds needed in the manufacture of cement are lime oxide ($CaCO_3$), silica oxide (SiO_2), alumina oxide (Al_2O_3), and iron oxide (Fe_2O_3) [3]. In essence, cement making is taking the cement raw material's oxides, which will eventually form new minerals to form the cement composition.

Indonesia has abundant natural material wealth, which has the potential as a material for cement production. Until now, the use of this material has not been optimal. One of these materials is Scoria Basalt rock, which until now has not been fully explored. According to [4], Scoria Basalt stone's availability in Lampung Province reaches 338 million/m^3, which are scattered in various districts and are used by residents as a foundation for housing. According to the Energy and Mineral Resources Ministry's Energy and Mineral Resources Information Data Center in 2011, Indonesia's number of basalt reserves amounted to 5,571,251,000 tons. While according to the Lampung Mining and Energy Office in 2013, the amount of basalt material resources in Lampung Province spread across districts totaled 318,480,000 tons. And so far, it has only been used as a building foundation. The added value of basalt material needs to be optimized to become the raw material for lime substitution in cement manufacturing to improve the economy for the surrounding community basalt mineral becomes a more highly economic value.

One of the third raw materials for PCC type cement is basalt. Basalt is a mineral whose chemical composition is dominated by silica, iron, lime, and alumina. PCC cement must meet the requirements required by ASTM C618 to become the third raw material for cement type. The material must have properties pozzolan with chemical components SiO_2 + Al_2O_3 + Fe_2O_3 remains at > 70-80% total weight—the pozzolan reaction against the lime component will produce hydration in cement [5]. The criteria for active pozzolan are components of the chemical composition of pozzolan SiO_2 + Al_2O_3 + Fe_2O_3, which will affect the compressive strength of cement mortar. PCC with ASTM C618 standards is a minimum of 75% on the pozzolan index [6].

Based on the analysis of the basalt material's chemical composition from Labuhan Maringgai, East Lampung, are SiO_2: 55.10% + Al_2O_3: 17.95% + Fe_2O_3: 5.61% total: 78.66% and other chemical components [4], for the physical requirements of a material used as a pozzolan, it must meet a pozzolanic activity index of at least 75%. In comparison, basalt can meet a pozzolan index's requirements when mixed with cement as much as 10%. Thus the basalt material originating from the Maringgai port of East Lampung meets the criteria required in ASTM C618 [7] so that the chemical components have pozzolanic properties which are SiO_2 + Al_2O_3 + Fe_2O_3 at least 70%, which can be used as the third raw material for this type of cement. PCC. The chemical components in the raw materials are tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A), and tetra calcium alumina ferrite (C_4AF). The compounds forming cement compounds are C_2S, C_3S, C_3A, and C_4AF [2].

Coal is a fossil fuel formed from organic sediment, mainly plant remains and formed through coalification. Its main elements consist of carbon, hydrogen, and oxygen. Coal is also an organic rock with complex physical and chemical properties found in various forms. Coal, which is usually only used as fuel, but coal will be mixed into the composition in making cement clinker in this study. The external combustion process accelerates the combustion of the cement clinker. This study aims to know the effect of coal on the cement clinker.

2. Material and Methods

2.1 Material

Limestone comes from Batu Putu, Natar District, South Lampung Regency, silica stone comes from Pagelaran District, Tanggamus district, clay comes from Rejosari Village, Natar District, South Lampung, iron sand comes from Liwa, West Lampung, basalt stone comes from Labuhan Maringgai District, East Lampung, coal comes from Tanggamus district.

2.2 Methods

Raw materials are limestone, basalt, clay, silica sand, iron sand, and coal with a mass percentage in 200 g, namely limestone (71%), clay (9%), silica sand (9%), and iron sand (1%) and mixed coal with variations of 5, 10, 15, and 20% as a reducing agent. Each raw material is ground using a grinding ball for 7 h until it becomes powder, then the milled material before use is sieved with a size of 200 mesh. The powder is weighed and mixed with water to form a paste, then formed into small granules or pellets [8] with a size of 0.8-1.4 cm and dried for 1-2 days at room temperature oven-dried for 4 h at 100 ^oC.

2.3 Characterization of Cement Clinker Samples

Clinker samples were characterized using X-Ray Diffraction (XRD) to see the crystal structure and phase. X-Ray Fluorescence (XRF) was determined to see the chemical composition of cement clinker samples, and Optical Microscopy to see the structure on the sample's surface. It is seen from the value of LSF (Lime Saturation Factor), SM (Silica Modulus), AM (Alumina Modulus), to get good clinker as required in ASTM C150.

3. Result and Discussion

3.1 Results of Characterization of Clinker Raw Materials using XRF

XRF analysis carried out in this study was characterized using XRF PANalytical Epsilon3XLE Bench Top. XRF analysis showed that the chemical content of raw materials and cement clinker samples with temperature variations of 1100, 1200, and 1300 ^oC. This study's raw materials include basalt, limestone, silica stone, clay, iron sand, and coal. The XRF analysis results on the raw material for making cement clinker can be seen in Table 1. Table 1 shows the results of XRF characterization. The CaO value in limestone is quite high, namely 92.973%, so that it can be used for making clinkers. Limestone is needed to manufacture cement clinker because it plays a role in hydration reactions and as the main compounds forming C_3S and C_2S [9]. Silica stone shows that the highest percentage is the oxide compound SiO_2 of 95.289%. The amount of SiO_2 more than 90% is perfect for use as raw material for making clinker. In cement, SiO_2 always binds to calcium oxide, either as C_3S or as C_2S. The amount of SiO_2 will affect the value of *silica modulus* (SM). The *silica modulus* low value will affect the short cement binding time and low initial compressive cement strength at 3-7 days. Table 1 also shows the presence of SO_3 that appears but only in raw materials for iron sand and coal. A high level of SO_3 in the clinker is needed because, in cement, the SO_3 is obtained from *gypsum*.

Table 1 also shows that XRF characterization results obtained the amount of SiO_2, Al_2O_3, and Fe_2O_3 as the raw material for basalt rock of 79.293%. It can be said that it has met the chemical requirements as a substitute material for cement or pozzolan based on ASTM C-618, where the requirements for natural pozzolanic or type N contain SiO_2, Al_2O_3, and Fe_2O_3 at least 70%. This is the following research [10] that the pozzolanic content with the total percentage of SiO_2, Al_2O_3, and Fe_2O_3, which is more than 50%. It generally produces good pozzolanic material and high pozzolanic activity to be used as a material for adding cement**.**

3.2 Results of Analysis on Semen Clinker Samples

The chemical composition of the clinker in ASTM C150 used to make Portland cement is shown in Table 2. In contrast, the XRF analysis results of cement clinker samples' chemical content against heating temperature variations for 2 h with substitution of 5, 10, 15, and 20% coal are shown in Table 3. Table 3 shows the effect of variations in coal composition, basalt rock, and temperature sintering on the cement clinker formation process, which can be seen from the XRF results. XRF characterization results on cement clinker samples with coal variations, namely 5, 10, 15, and 20%, and temperature variations of sintering 1100, 1200, and 1300 °C with a holding time of 2 h were characterized using XRF PANAnalytical Epsilon3XLE Bench Top.

The sample with the highest CaO compound of 67.934% was found in the specimen sintered at 1100 °C with a composition of 5% coal, while the lowest was in the clinker sample with 20% coal concentration at 1300 °C, which was 67.382%. The CaO content is still very high at a temperature of 1100 °C, with a mass concentration of 5% coal. This is because limestone has a melting point at 1100 °C. At a temperature of 1100 °C it is still high in CaO content, resulting in cement clinker being easily destroyed and easily destroyed. Cracks when used in cement making. The clinker sample owned the SiO_2 highest at 1300 °C with 20% coal, 22.868%, while the lowest clinker sample was at 1100 oC with a composition of 5% coal was 21.042. XRF results on cement clinker showed that the higher the temperature and the increase in coal's mass concentration, the higher the SiO_2 content. The increasing of the temperature sintering and the concentration of coal mass, which is included in the cement clinker composition of the limestone, will increase the SiO_2 compound while the CaO compound content decreases. This is because coal has high SiO_2, so that when the coal is used, the greater the concentration added to the clinker sample can increase the SiO_2 compound and reduce the CaO compound. Based on XRF analysis results, the cement clinker obtained in this study contained compounds that mostly met the requirements [11], such as SiO_2, SO_3, MgO, and some have not fully met the ASTM requirements [11]. This is because the composition of limestone used is still quite a lot. The basalt stone used as a substitution also needs to be recalculated so that the chemical composition in the clinker meets the ASTM standard requirements [11].

3.3 Results Calculations of LSF, SM and AM on Cement Clinker

LSF is the ratio between the maximum amount of CaO needed to react with other oxides. The LSF in clinker ranges from 94-98% [13]. *Silica Modulus* (SM) is the ratio between silica oxide with several alumina oxides and iron oxide proportions. SM on clinker ranges between 2-2.4%. *Alumina Modulus* (AM) is the ratio between alumina oxide and iron oxide. AM will affect the color of clinker and cement. So that after the calcination process, the color of the clinker changes color. This can also be caused by the influence of the clinker raw material. The higher the AM, the brighter the color of the cement. AM usually ranges from 1.4-1.8%. The results of LSF, SM, and AM calculations can be seen in Table 4. Table 4 shows that cement clinker from the raw material substitution of 5, 10, 15, and 20% coal *sintered* at 1100, 1200, and 1300 °C within 2 h has met the requirements. The comparison between all CaO in the mixture of raw materials with several other oxide proportions must produce an LSF value of around up to 94% to 98%, as has been done in research [13] in Table 3. It's just that at a temperature of 1200 °C with 20% coal variation resulted in an LSF of 89.232%, which did not meet the cement clinker manufacture requirements. If the resulting LSF value is 94%-98%, this research is according to the research conducted by [13]. His research also explains that LSF shows the maximum amount of CaO needed to react with other oxides so that there is no *free lime* excessively the clinker. Increasing levels of LSF and SM resulted in a reduction in the combustion temperature used [14]. The LSF value is influenced by the levels of CaO, SiO_2, Al_2O_3, and Fe_2O_3. The higher the CaO content, the higher the LSF, and vice versa. Furthermore, it fulfills the requirements, namely the ratio between silica oxide with several proportions of alumina oxide and iron oxide to produce an SM value of about 2-2.4%, as has been done in studies [15] and [16]. Table 3 shows that the fulfillment is at the time of calcination with a temperature of 1100 °C, respectively 2.2959%, 2.3017%, 2.3250%, and 2.2939%. At a temperature of 1200 °C, respectively 2.3939%, 2.3702%, 2.3303% and 2.3043. At a temperature of 1300 °C, the value of SM increased, namely 2.5557%, 2.4861%, 2.5333%, and 2.5200%, meaning that these values were by what was done in research [14] and [15] achieve a good clinker target, namely that to meet the requirements the SM value ranges from 2-2.4%. The value of SM was influenced by the levels of SiO_2, Al_2O_3, and Fe_2O_3. The higher the SiO_2, the higher the SM, and vice versa. Then the AM value of cement clinker from coal raw materials at each value almost meets the requirements; namely, the ratio between alumina oxide and iron oxide produces a good AM value, which is around 1.4-1.8%. as in studies [17] and [16]. AM value will affect the resistance of cement to sulfate. If the AM value is higher than the standard, it indicates that the cement is susceptible to sulfate [18].

3.4 XRD Characterization

Cement clinker samples used in this study were characterized using XRD PANalytical X'Pert3 Powder. This characterization aims to determine the phase formed in the sample. A qualitative analysis was carried out on the XRD results using the search match analysis method or matching the data obtained with the Powder Diffraction File database (PDF) database to determine the degree formed. The sample diffract program brought were then analyzed using the High Score Plus (HSP) version 3.0e (3.0.5) application, then the HSP output results were used as input to create graphics with the Matlab software. Analysis of these cement clinker samples was heated at 1100, 1200, and 1300 °C for 2 h, respectively. The data obtained is in the form of a diffractogram of the relationship between intensity with an angle of 2θ. On the diffractogram, there will be specific peaks that can determine the crystal phase. All cement clinker samples produced crystalline phases such as C_3S (Ca_3SiO_5), according to ref. ICDD code 00-016-0406 [19] with the mineral name is Hatrurite. C_2S (Ca_2SiO_4) corresponding to ref. Code in ICDD 01-070-0388 [20] with the mineral name is Larnite. Crystal phase C_3A (Ca_3AlO_6), according to ref. ICDD code 00-006-0495 [21] with Calcium Aluminum Oxide. The crystal phase of C_4AF ($Ca_4Al_2Fe_2O_{10}$) is according to ref. ICDD code 00-011-0124 [22] with the mineral name is Brownmillerite. The clinker sample diffractogram using Matlab software is shown in Figures 3, 4, and 5. The graph shows that the values of 2θ, d spacing, and intensity are obtained and from the research data, then compared with the HSP data so that the difference between d spacing (Δd) and the formed phase is obtained. The Δd value from the XRD results of the clinker sample at temperatures of 1100 and 1200 °C with variations of 5, 10, 15, and 20% of coal at the base HSP data is less than or equal to 0.01 Å. With results dominated by the Belite phase.

In contrast, the Δd from the XRD results of the clinker sample at 1300 °C with variations of 5, 10, 15, and 20% of coal in the HSP database is less than or equal 0.01 Å. The Alite phase dominates the results. Based on the results of XRD analysis on cement clinker samples using variations in the composition of coal 5, 10, 15, and 20% with sintering temperatures of 1100, 1200, and 1300 °C, it shows that the formation of crystalline phases refers to the results of the analysis using research [23], where the main component clinker constituents such as C_3S, C_2S, C_3A, and C_4AF. In this data, the phases that appear mostly at diffraction peaks are Belite (C_2S) and Alite (C_3S), which influence the quality of cement clinker produced, especially on the compressive strength when the cement clinker is turned into a cement product. At the same time, the Aluminate phase (C_3A) and Ferrite (C_4AF) influence cement clinker color [24]. At each peak temperature, the Belite phase (C_2S) is almost dominated, in which the Belite (C_2S) phase is formed from CaO compounds. It is obtained from limestone raw materials containing $CaCO_3$ when undergoing a high-temperature process decomposes into CaO when the clinker is burned at 1000 until 1200 °C. The limestone has just begun to experience a melting point process so that at this temperature, the CaO content is still high. At a temperature of 1300 °C, the highest peak is dominated by the Alite (C_3S) phase formed from the SiO_2 compound obtained from the raw materials used in making clinker samples from basalt rock and coal variations, which affect the appearance of the Alite (C_3S) phase. So the number of clinker phases formed depends on the temperature and composition of the coal used.

3.5 Results of Micro Structure

Observation of microstructure aims to determine the effect of temperature and variations in coal composition of 5, 10, 15, and 20% on cement clinker. The instrument used in this study was the optical microscope Nikon Inverted Metallurgical. Before the microstructure observation, each clinker sample has gone through a process of grinding on the sample's surface. The surface is visible on the optical microscope presented in Figure 4, Figure 5, and Figure 6. The figures have shown the results of the structural test analysis. Micro using an optical microscope with a magnification of 100 times. This data is indicated by a white circle, which is an area containing crystals Belite and a red process, which is an area containing crystals Alite. It can be seen that there is a strong bond between the raw materials on the Alite, which is characterized by a smooth surface. Whereas Belite, which looks like black holes, is not perfectly bound. The black hole is formed after going through the sintering process to affect the cement clinker results' compressive strength. Crystals Alite obtained show a dense structure. The solid phase of the phase is Alite due to the raw mixture rich in lime. The dimensions of the crystals Alite in the clinker get more significant as the heating temperature increases.

In contrast, the dimensions of the crystal Belite in the clinker get smaller with increasing heating temperature. The result of [17] structure Alite of clay clinker is more precise than clay clinker; this indicates that the burning of clay clinker is better than clay clinker. It can be seen that clinker with a temperature of 1300 °C has minerals Belite in groups compared to clinker by heating at 1100 and 1200 °C.

4. Conclusion

The conclusions obtained from the results of the research that have been carried out are from the addition of 10% basalt rock with variations of coal 5, 10, 15, and 20% with sintering temperatures of 1100, 1200, and 1300 °C, there is clinker formation that is closer to the standard, namely at a temperature of 1300 °C with the addition of 15% coal. The SiO_2 compound was 22.747%, and the CaO compound was 66.384%. This is because coal has a high content of SiO_2, so that when the coal is added to the clinker, the greater the concentration, it increases the SiO_2 compound and reduces the CaO compound. The resulting phases in the XRD characterization of each coal and 10% basalt rock variation were dominated by alite, belite, aluminate, and ferrite phases. The microstructure analysis results showed that clinker with a temperature of 1300 °C had minerals Belite in groups compared to clinker with heating at a temperature of 1100 and 1200 °C.

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Appndix:

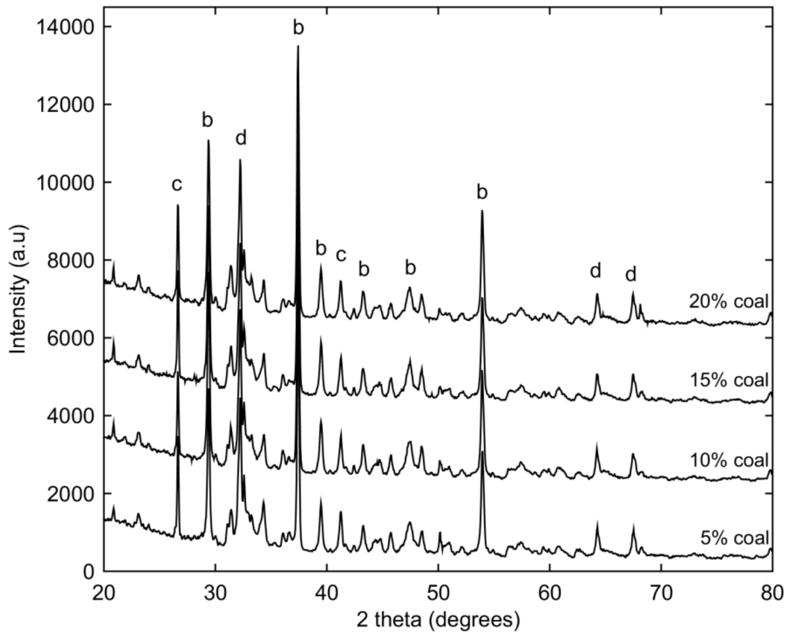

Fig 1. XRD diffractogram of clinker samples at 1100°C with variations coal (a) 5, (b) 10, (c) 15, (d) 20% (a = Alite, b = Belite, c = Aluminate and d = Ferrite).

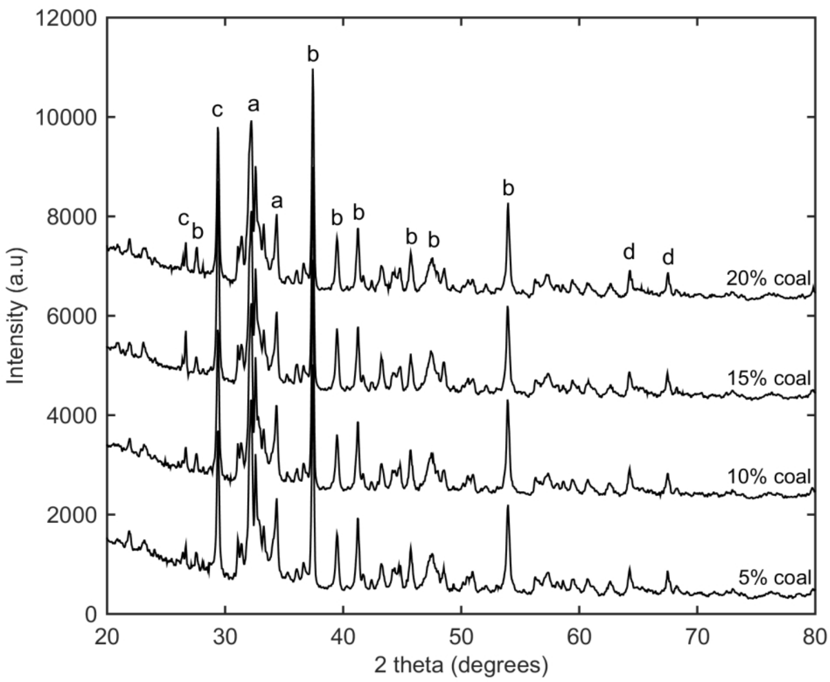

Fig 2. XRD diffractogram of clinker samples at 1200°C with variations coal (a) 5, (b) 10, (c) 15, (d) 20% (a = Alite, b = Belite, c = Aluminate and d = Ferrite).

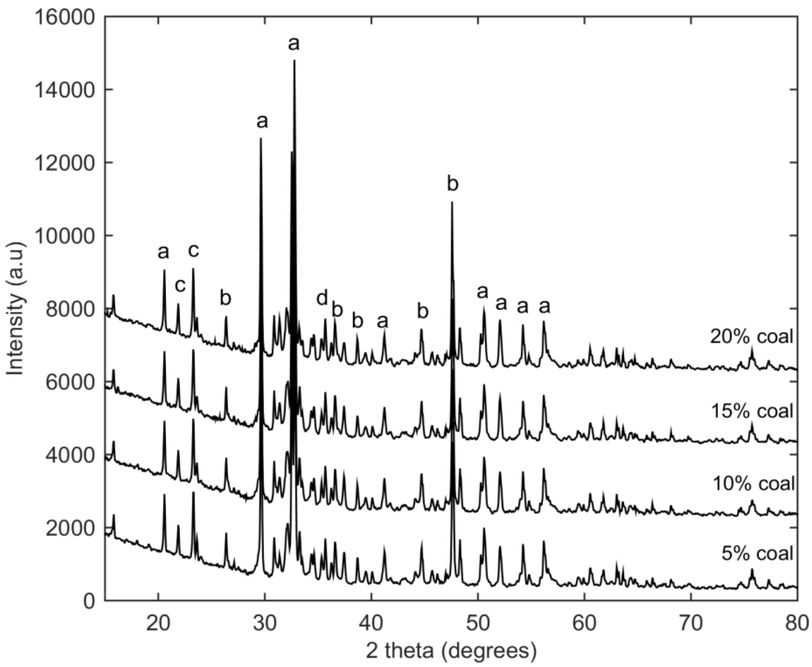

Fig 3. XRD diffractogram of clinker samples at 1300°C with variations coal (a) 5, (b) 10, (c) 15, (d) 20% (a = Alite, b = Belite, c = Aluminate and d = Ferrite).

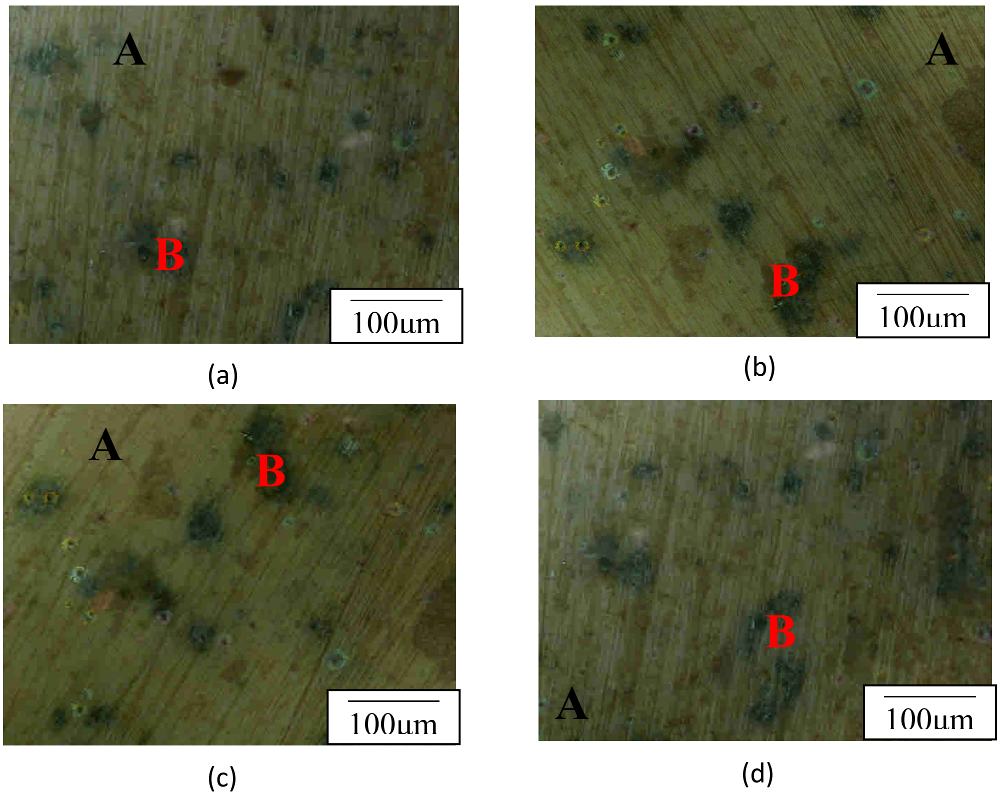

Fig 4. Micro structure of cement clinker at temperature of *sintering* 1100 °C with variations of coal (a) 5, (b) 10, (c) 15, (d) 20% (A = Alite dan B = Belite).

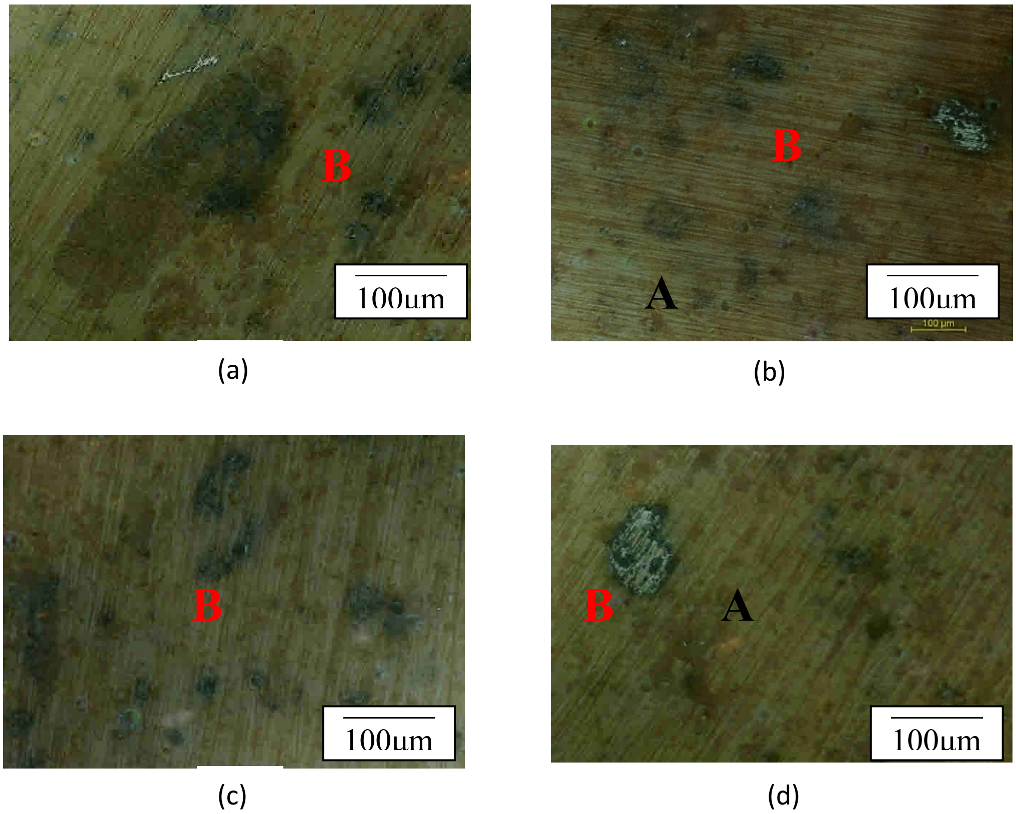

Fig 5. Micro structure of cement clinker at temperature of *sintering* 1200 °C with variations of coal (a) 5, (b) 10, (c) 15, (d) 20% (A = Alite and B = Belite).

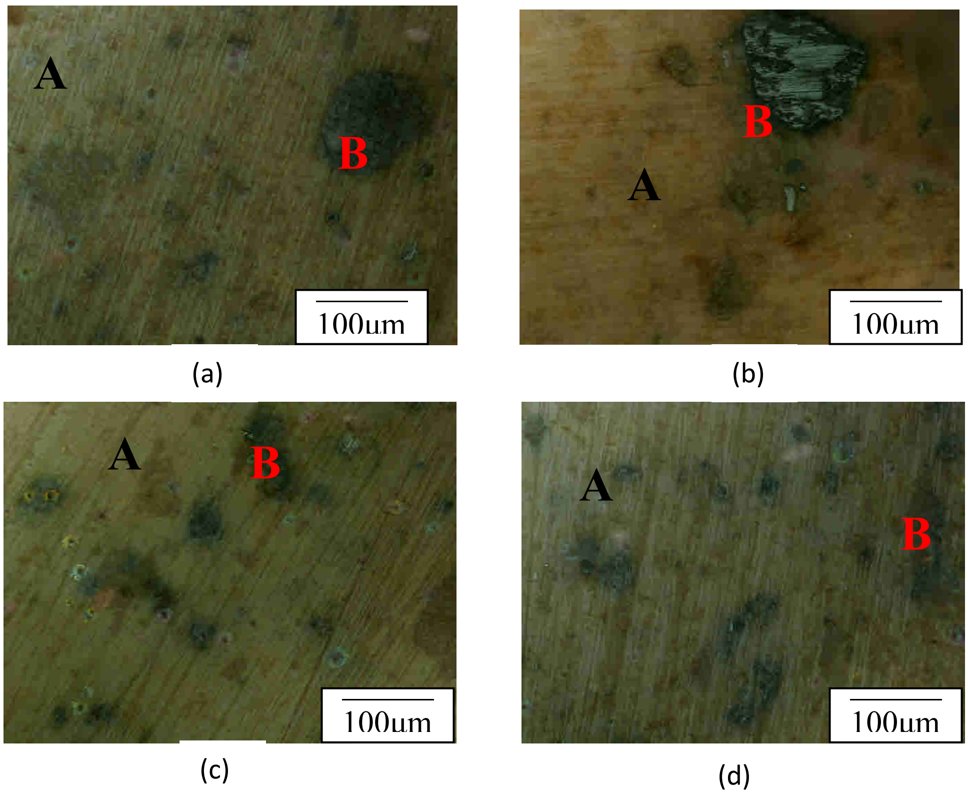

Fig 6. Micro structure of cement clinker at temperature of *sintering* 1300 °C with variations of coal (a) 5, (b) 10, (c) 15, (d) 20% (A = Alite and B = Belite).

Table 1. XRF analysis results for cement clinker raw materials

Compounds (%)	Percent (%)					
	limestone	Silica Stone	Clay	Iron Sand	Coal	Basalt Rock
SiO_2	4.036	95.289	53.812	18.777	15.555	47.376
Al_2O_3	1.653	3.169	27.489	6.340	13.970	18.924
Fe_2O_3	0.552	0.422	15.597	58.026	12.643	12.993
CaO	92.973	0.841	0.220	3.126	1.141	10.977
MgO	0.399	-	0.369	4.814	-	4.452
Na_2O	-	-	-	-	-	2.866
TiO_2	0.105	0.126	1.385	6.701	0.879	1.354
K_2O	0.104	-	0.879	0.266	0.278	0.587
MnO	-	-	-	0.511	-	-
SO_3	-	-	-	0.557	54.871	-

Table 2. Chemical composition of clinker in ASTM C150

Compounds (%)	Percentage (%)
CaO	65.5-66.2
SiO_2	21-22
Al_2O_3	5-5.5
Fe_2O_3	4-4.5
MgO	Max 1.5
SO_3	Max 1
LSF	94-98
SM	2-2.4
AM	1.4-1.8
C3S	55-60
C2S	10-14
C3A	8-12
C4AF	1-12

Table 3. Results of XRF analysis on cement clinker samples with substitution of 5, 0, 15 and 20% coal against sintering temperature variations for 2 h

Coal (%)	Compounds (%)	Variations of heating temperature			ASTM C150-99
		1100(°C)	1200(°C)	1300(°C)	
	CaO	67.934	67.106	66.907	65.5 - 662%
	SiO_2	21.042	22.389	22.518	21 - 22%
5	Al_2O_3	4.996	5.272	4.838	5 – 5.5%
	Fe_2O_3	4.169	4.081	3.738	4 – 4.5%
	SO_3	0	0	0	Max 1%
	MgO	0.358	0.324	0.393	Max 1.5%
	CaO	67.,926	66.967	66.424	65.5 - 662%
	SiO_2	21.344	22.418	22.741	21 - 22%
10	Al_2O_3	5.168	5.284	4.908	5 – 5.5%
	Fe_2O_3	4.105	4.132	3.837	4 – 4.5%
	SO_3	0	0.318	0	Max 1%
	MgO	0.34	0.308	0.381	Max 1.5%
	CaO	67.731	66.136	66.384	65.5 - 662%
	SiO_2	21.829	22.877	22.747	21 - 22%
15	Al_2O_3	5.249	5.247	4.916	5 – 5.5%
	Fe_2O_3	4.139	4.141	3.865	4 – 4.5%
	SO_3	0.382	0.31	0	Max 1%
	MgO	0.367	0.131	0.441	Max 1.5%
	CaO	67.382	65.082	66.386	65.5 - 662%
	SiO_2	21.926	22.995	22.866	21 - 22%
20	Al_2O_3	5.374	5.638	4.987	5 – 5.5%
	Fe_2O_3	4.097	4.211	3.77	4 – 4.5%
	SO_3	0.354	0.43	0.191	Max 1%
	MgO	0.395	0.381	0.441	Max 1.5%

Table 4. Calculation Results of LSF, SM and AM Cement Clinker with Variations Composition of Coal

Calculating Result	Temperature of sintering (°C)	Coal Variation				ASTM C150-99
		5%	10%	15%	20%	
	1100 °C	99.276	97.806	96.180	98.974	
LSF	1200 °C	93.772	93.776	95.719	89.232	94-98%
	1300 °C	96.257	96.044	94.599	95.382	
	1100 °C	2.2959	2.3017	2.3250	2.2939	
SM	1200 °C	2.3938	2.3702	2.3303	2.3043	2-2.4%
	1300 °C	2.5557	2.4861	2.5333	2.5200	
	1100 °C	1.1984	1.2590	1.2682	1.3117	
AM	1200 °C	1.2918	1.2788	1.2671	1.3389	1.4-18%
	1300 °C	1.2943	1.2791	1.2719	1.3228	