International Journal of Engineering & Technology, 10 (1) (2021) 23-27

International Journal of Engineering & Technology

Website: www.sciencepubco.com/index.php/IJET

Research paper

The Effect of Tricalcium Silicate (C_3S) Percentage in Clinkerson the Cement Quality

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Abstract

Composite cement products produced by national cement factories in Indonesia should follow the required quality standards. The quality standard of composite cement refers to the SNI 7064:2014. Some physical parameters of the quality standards set are mortar compressive strength and autoclave expansion. Compressive strength is influenced by C_3S and C_2S in the clinker. The reaction of the formation of mineralogical compounds occurs when clinkers formed. Whereas the expansion by autoclave is influenced by the levels of free lime in the cement. This research was conducted to determine the effect of the percentage of tricalcium silicate (C_3S) on the quality of cement with free lime <2% and free lime > 2% with variations of C_3S in clinkers, namely 55%, 57%, 59%, 61%, 63%, 65%, and 67%. Physical parameters tested in this study are compressive strength of mortar, blaine, and autoclave expansion. While the chemical parameters tested in this study are free lime in cement and SO_3. Based on the research, it was found that if the same percentage of C_3S quality of cement having FCaO <2%, the initial compressive strength results were greater than FCaO> 2%, the ideal condition of the development of compressive strength for FcaO > 2%, 3 to 7 days was at the percentage of C_3S clinker of 63,48%. Whereas the development of ideal compressive strength for 7 to 28 days is at the clinker C_3S percentage of 64,85%. For FCaO <2% the ideal condition 3 days to 7 days is at the percentage of clinker C_3S of 62,79% and the development of compressive strength 7 to 28 days is at the percentage of clinker C_3S of 54.77%. The expansion with autoclave experiencing expansion that does not meet the minimum requirements of SNI 7064:2014 are samples with a percentage of C_3S 54,86% and 61% with FCaO > 2%.

Keywords: *Clinker; C_3S; Free Lime; Cement; Compressive Strength; Autoclave Expansion; Cement Quality.*

1. Introduction

The national cement industry certainly has a reference as a standard for cement quality, thus it has a selling value to face the business competition. As a reference in the standard for determining the quality of cement, the national cement industry uses SNI 2049:2015 for portland cement and SNI 7064:2014 for composite cement [3]

In the standard that has been set as a reference for cement quality, there is a minimum value of mortar compressive strength and a maximum value of expansion with cement paste autoclave that must be achieved, depending on the type of cement to be produced [3]. With the expectation that cement used for road construction projects, buildings and bridges will not experience cracks or collapse when used with heavy loads. Compressive strength is one of the benchmarks to evaluate the ability of mortar or concrete made of cement that is tested against the load it receives. The compressive strength of cement is influenced by the cement hydration process [4]. Compressive strength in cement is influenced by the quality of the clinker used, where the mineralogy in the clinker plays an important role in determining the compressive strength. While expansion by autoclave is influenced by the presence of free lime and magnesium oxide levels in cement. To get the desired compressive strength quality of cement, it is necessary to regulate some of the modulus oxides as the building blocks of cement. Mortar compressive strength has an age of 3, 7, and 28 days. It can be said that at 3 days old the compounds that play an important role are tricalcium silicate (C_3S), meanwhile, at the age of 7 days and 28 days the compound plays this important role is dicalcium silicate (C_2S) [12]. According to Widojoko (2010), C_3S hardens within a few hours and affects the strength of concrete at an early age, particularly in the first 14 days. While the formation of the C_2S compound takes place slowly with slow heat release, this compound influences the process of increasing strength that occurs from 14 days to 28 days.

To increase the compressive strength in the beginning it is necessary to examine the effect of the percentage of C_3S on the cement quality. Tricalcium silicate is influenced by the content of free lime present in cement because the reaction of C_3S formation occurred after C_2S mineral has been formed first with several free limes which are also formed later at a temperature of 1300 °C C_2S will react with free lime to form C_3S. Due to the influence of free lime on the percentage of C_3S, a study was conducted on "The Effect of Tricalcium Silicate (C_3S) Percentage in Clinkers on Composite Cement Quality", hence it is expected to reduce the clinker index used to increase production efficiency and quality of cement fulfilling the 7064:2014 standard for composite cement.

2. Literature review

C_3S is one of the mineralogical constituents of the clinker. The clinker is the main ingredient in cement manufacturing.The problem formulation in this study is “How the same percentage of C_3S (tricalcium silicate) with FCaO content <2% and FcaO> 2% in the clinkers affectsthe cement quality (compressive strength)”, “What is the ideal condition of C_3S on the development of compressive strength on 3, 7, and 28 days”, and “How FcaO influences the influence of FCaO on cement on the expansion by autoclave.

3. Methodology

3.1. Materials and tools

The tools used in this study were an electric jaw crusher, an oven with a temperature of 110°C, ARL 9900 X-Ray, Mini mill, and V-Mixer. The materials used were clinker, limestone, fly ash, and gypsum.

3.2. Work precedur

1) Sample Preparation
   Preparation was conducted by taking a fresh clinker as the outcome of grate output and ana
   lyzing the oxide and FcaO content of the clinker to calculate the C_3S. When the C_3S value of the clinker meet the requirements of predetermined independent variables, the clinker sample was further processed to produce cement.
2) Cement Production
   The proportion of 67% clinker,20% limestone,10% fly ash, and 3% gypsum 3% were mixed using a mini-mill contained ball mill. The sample mixture was grounded until reaching the specified blaine requirements

3.3. Clinker analysis

1) The Cclinker oxide analysis
   Clinker oxides were obtained from the X-ray instrument analysis. The analysis result wasthen used to calculate the C_3S value.
2) FCaO clinker analysis
   FCaO analysis was performed using the wet analysis method that would be used to calculate the C_3S of the clinkers.

3.4. The cement product analysis

1) Fineness test (Blaine)
   The working principle of the blaine is a device that will attract a certain amount of air through a portland cement base (2,9137 grams) with a certain porosity. The air passing through each particle size serves to determine the rate of airflow through the base.
2) Mortar production
   Materials prepared were 500 grams cement samples, 1375 grams Ottawa sand, and 270 ml aquadest. The aquadest was put in the mixer bowl and added the cement sampleMixer was run at the low speed for 30 seconds and slowly added with Ottawa sand for 30 seconds. The stirring speed of the mixer was increased for 30 seconds, then the mixer was turned off. The mixture (cement, Ottawa sand, and aquadest)was once again mixed for 15 seconds and kept for 75 seconds. The mixer was turned on once againfor 1 minute until the mixture was ready to mold.
3) Mortar compressive strength test
   Mortar compressive strength test was performed when the age of mortar according to the guideline of SNI 7064:2014 reached 3, 7, and 28 days. The analysis was done using a compressive strength machine.
4) Sulfur Trioxide (SO_3) content
   1 gram of sample was dissolved in distilled water and followed by the addition of 10 ml of HCl. Stirred, added 50 ml of distilled water and boiled, then filtered with W41 filter paper W41 to obtain a filtrate. The filtrate was boiled and added 10 ml $BaCl_2$, then left for a while. The filtrate was filtered again using W42 filter paper and rinsed with the hot aquadest. The sludge was burned in the furnace at +1000 ºC for 1 hour.

4. Results and conclusion

The C_3S value contained in the clinker was obtained by calculating the oxide. By knowing the oxides contained in the clinker, the C_3S value can be calculated. If the C_3S value in the clinker meets the desired criteria, the clinker sample can be further processed as clinker sampling as an independent variable. While the fixed variable is the proportion (composition) of cement that will become a product, namely clinker 67%, Limestone 20%, Fly Ash 10% and Gypsum 3%. The percentage of C_3S needed to fulfill this independent variable is 55%, 57%, 59%, 61%, 63%, 65%, and 67%. Meanwhile, 2 variations FCaO used are FcaO <2% and FCaO> 2%.
In this study, testing was carried out to determine the quality in the form of physical and chemical parameters of cement with various variations of C_3S, with product quality targets namely: compressive strength of cement mortar and expansion with autoclave following the SNI 7064:2014. The results of the two quality parameters are influenced by the independent variables between C_3S and FCaO in the clinker and made into cement products. The compressive strength standard based on SNI 7064:2014 for compressive strength of 3 days with a minimum of 130 kg/cm^2, 7 days with a minimum of 200 kg/cm^2, and 28 days with a minimum of 280 kg/cm^2. While the expansion with autoclave max 0,8% SNI 7064: 2014.

4.1. Percentage of tricalcium silicate (C_3S) on compressive strength with FCaO> 2%

From the picturegraphic 1. above, it can be seen that the effect of tricalcium silicate on compressive strength of 3 days tends to increase the compressive strength value along with the increase in the percentage of C_3S in the clinker. The C_3S reacts with water to produce amorphous calcium silicate hydrates known as CSH gel (tobermorite gel) which is the main adhesive that binds sand and aggregate particles together in concrete [8]. This can be because C_3S is far more reactive than C_2S and 50% C_3S in cement will be hydrated in 3 days and 80% in 28 days [16]. Following the theory, it is clear that the compressive strength of 3 days will increase along with the increased percentage of C_3S in the clinker. More C_3S content will form the cement with high initial compressive strength and hydration heat. On the other hand, higher C_2S content will form the cement with low initial compressive strength and high chemical attack resistance. The binding of C_3S and water will produce a hardening of the cement paste within a few hours, and then will get most of its strength (around 70%) in the first week after the binding, by releasing heat around 500 joules/gram [11].

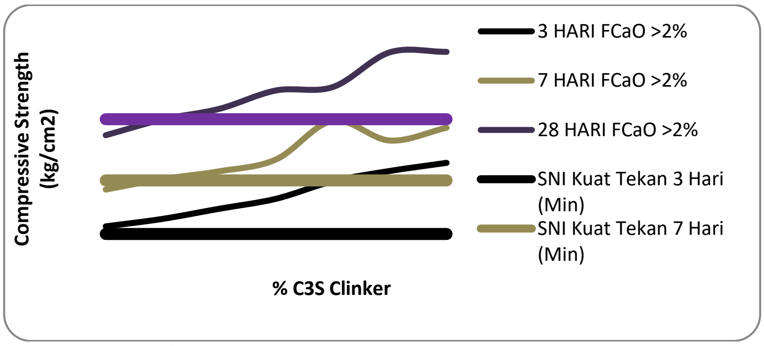

Fig. 1: Percentage of Tricalcium Silicate (C_3S) on Compressive Strength with FCaO> 2%.

At the age of 7 days, C_3S and C_2S played the role. In the graphic above, an upward tendency is presented to reach the optimum value of63% C_3S, while 65% C_3S begins to decrease. This means that the development of compressive strength is no longer ideal. The compressive strength at the age of 28 days is influenced by C_2S in the clinker as it reacts slowly followed with slow heat energy release. The compound influences the development of concrete strength from the age of 14 days onwards. Portland cement with high C_2S has relatively low resistance to the chemical aggression and dry shrinkage which contributes to the durability of the concrete [11]. High FCaO value in the clinker results in total silicate reduction (C_3S and C_2S) on the formation of CSH; CSH is the cement strength forming phase. High FcaO in cement will cause a discontinuity in the cement production, making the cement becomes easily cracked. For 55% C_3S in the clinker, the mortar compressive strength could not obtain the SNI in 7 days with 188,09 kg/cm^2, while the standard in 7 days for the composite cement is 200 kg/cm^2, for compressive strength of 28 days is 259,08 kg/cm^2, and SNI in 28 days for the composite cement is 280 kg/cm^2.

4.2. Tricycumsilicate percentage on the compressive strength with FCaO <2%

Produced at 7 days with 65% C_3S.The mortar compressive strength with FCaO content <2% could obtain the compressive strength required by SNI 7064:2014 standard. The low FCaO value in the clinker results in optimal total silicate (C_3S and C_2S) in the formation of CSH, where CSH is the cement strength forming phase. As for the 28 days compressive strength, the optimum conditions could be obtained.

4.3. Tricycumsilicate percentage on the compressive strength with FCaO <2%

Produced at 7 days with 65% C_3S.The mortar compressive strength with FCaO content <2% could obtain the compressive strength required by SNI 7064:2014 standard. The low FCaO value in the clinker results in optimal total silicate (C_3S and C_2S) in the formation of CSH, where CSH is the cement strength forming phase. As for the 28 days compressive strength, the optimum conditions could be obtained.

4.4. Tricycumsilicate percentage on the compressive strength with FCaO <2%

Produced at 7 days with 65% C_3S.The mortar compressive strength with FCaO content <2% could obtain the compressive strength required by SNI 7064:2014 standard. The low FCaO value in the clinker results in optimal total silicate (C_3S and C_2S) in the formation of CSH, where CSH is the cement strength forming phase. As for the 28 days compressive strength, the optimum conditions could be obtained.[15].

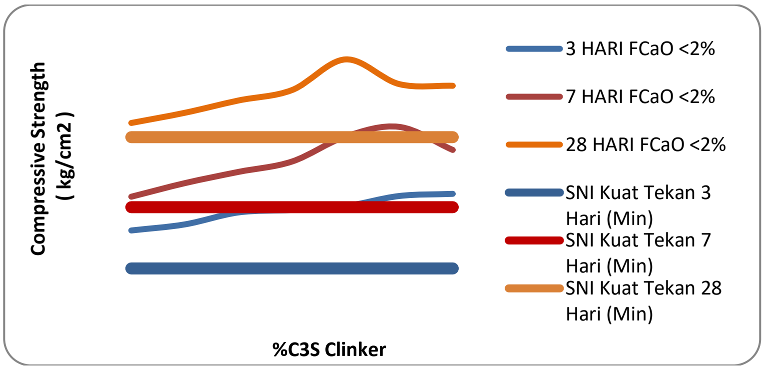

Fig. 2: Tricycumsilicate Percentage on the Compressive Strength with FCaO < 2%.

From the Picture graphic 2 above, the effect of tricalcium silicate on the compressive strength of 3 days has an upward trend with the increased percentage of C_3S in the clinker. This was similar to [18] that the faster C_3S hydration reaction, the larger initial compressive strength of C_3S percentage in the clinker produced. Meanwhile, an optimum value was produced at 7 days with 65% C_3S.The mortar compressive strength with FCaO content <2% could obtain the compressive strength required by SNI 7064:2014 standard. The low FCaO value in the clinker results in optimal total silicate (C_3S and C_2S) in the formation of CSH, where CSH is the cement strength forming phase. As for the 28 days compressive strength, the optimum conditions could be obtained by 61% C_3S because the compound that plays an important role is C_2S, meaning that C_3S did not play any role anymore [9].

4.5. Compressive strength development

Table1: Compressive Strength Development with FCaO > 2%

% C_2S	54,86	57,25	58,70	60,57	63,48	64,85	67,29
Perkembangan 3 ke-7 hari (%)	34,40	34,74	30,04	29,20	39,79	18,73	20,59
Perkembangan 7 ke-28 hari (%)	37,74	38,62	38,50	39,64	15,85	45,71	3,870

Table 2: Compressive Strength Development with FCaO < 2%

% C_2S	54,77	57,44	59,21	60,76	62,79	65,15	66,67
Perkembangan 3 ke-7 hari (%)	22,24	26,13	23,87	28,08	39,55	37,10	23,32
Perkembangan 7 ke-28 hari (%)	39,75	35,34	33,96	32,38	31,40	16,77	27,61

The ideal condition for the development of compressive strength of 3 to 7 days is at the clinker C_3S percentage of 63,48% with FCaO > 2%. This is because of the 3 days compressive strength of 198,90 kg/cm2 and the compressive strength of 7 days increased to 278.05 kg/cm^2, it can be seen that there is an increase in compressive strength of 39,79% and whereas the development of ideal compressive strength for 7 to 28 days occurred in the C_3S percentage of 64,85% with 45,71% development[17].. While for FCaO <2% the ideal conditions 3 days to 7 days are at the C_3S percentage of 62,79%. This is because of the 3 days compressive strength of 201,14 kg/cm^2 and the 7 days compressive strength increased to 280,70 kg/cm^2, there is an increase in compressive strength of 39,55% and the 28 days compressive strength development occurred at C_3S percentage of 54,77% with a compressive strength of 7 days 211,96 kg/cm^2 and 28 days 296,21 kg/cm^2. FCaO relationship with% expansion with autoclave. [7].

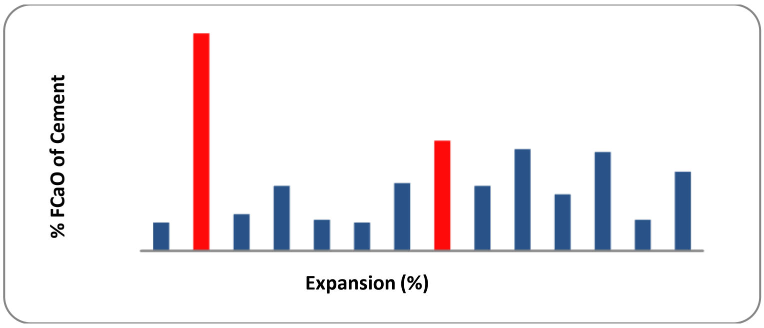

Fig. 3: Percentage (%) FCaO of Cement.

From the graphic picture 3.it can be seen that when FCaO in cement is more than 2,3%, the cement paste will expand. In other words, when the cement paste experiences an expansion, it will exceed the required standard of SNI 7064:2014 which stated thatthe maximal of the autoclave expansion is 0,8%.[7].

5. Conclusion

From this study it can be concluded:

1) Using the same percentage of C_3S, FCaO < 2% obtained higher initial compressive strength than FCaO > 2%.
2) The ideal condition of compressive strength development for FCaO > 2%, at 3 to 7 days occurred while the C_3S percentage of 63,48%. Whereas the development of ideal compressive strength for 7 to 28 days obtained by C_3S percentage of 64.85%. For FCaO <2% the ideal condition of 3 days to 7 days is at the percentage of clinker C_3S at 62,79% and the compressive strength development at 7 to 28 days is at the C_3S percentage of 54,77%.
3) Expansion by autoclave with the expansion that did not meet the minimum requirements of SNI 7064:2014 shown by sample with a C_3S percentage of 54,86% and 61% with FCaO > 2%.

Acknowledgement

1) For further studies, a formula with an increase of 1% C_3S can be used to predict the quality of the cement produced that may become a reference for the cement company.
2) To increase the value of C_3S with FCaO > 2%, it can be done by raw mix design (recalculate) modulus in the raw mill clinker base, by determining the mineralogy of C_3S, C_2S,C_3A and C_4AF, and $c_4$af then the modulus can be determined afterward.

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