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IMPROVE THE COMPRESSIVE STRENGTH USING A STRENGTH IMPROVER AGENT (SIA) IN THE GREEN CEMENT INDUSTRY IN INDONESIA

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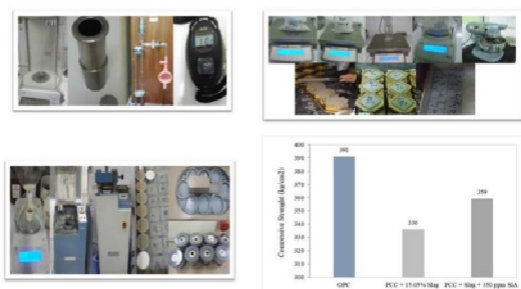
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Graphical abstract



Abstract

Greenhouse gas emissions such as CO₂ are produced from the calcination reaction of limestone (CaCO₃) in the manufacture of clinker. This research considers the use of clinker substitutes so that every ton of cement produced uses less clinker. As a consequence, it can reduce CO₂ emissions. However, the reduction in clinker is directly proportional to the decrease in the compressive strength of the cement. An additive called Strength Improver Agent (SIA) can be used to maintain the compressive strength to meet all applicable standards. This research aims to determine the optimal amount of SIA used to obtain a compressive strength that meets the standards. SIA was added in this study in several variations: 100, 150, 200, 250, 300, 350, and 400 ppm. The compressive strength of cement was tested at the ages of 1, 3, 7, and 28 days to see the effect of the addition of SIA on the quality of the mortar, based on ASTM C 109 and QPT-LAB-SNI-05. As a comparison, the test sample used was a cement blank in the form of PCC slag, which is on the market with a composition of 67% Clinker, 2.58% Gypsum, 15.37% Limestone, and 15.05% Slag. Besides that, physics tests were also carried out, such as Blaine, 325 mesh residue, Insoluble Residue (IR), Loss on Ignition (LOI), and XRF based on ASTM C 114, ASTM-STP 985, XRF Thermo ARL 8480S. The observations and analysis show that the optimum amount of SIA addition is at 350 ppm, where the resulting compressive strength increases at least 7% compared to blanks.

Keywords: Clinker, carbon capture, compressive strength, improver, slag

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1.0 INTRODUCTION

In today's modern world, the need for cement is increasing, and Indonesia is no exception. This fact was followed by an increase in the cement industry, managed by the private sector and the government [1, 2]. Inevitably, cement producers compete strictly by considering various aspects such as quality, production costs, energy efficiency, selling price, and environmental issues [3]. The calcination process in the kiln contributes significantly to releasing CO₂ greenhouse gas emissions due to the calcium carbonate calcination reaction [4, 5]. In other words, an increase in cement production capacity can potentially contribute to global warming problems if not adequately anticipated [5, 6].

Historically, cement is a mixture of clinker and gypsum known as Ordinary Portland Cement (OPC). Limestone combustion occurs at high temperatures and requires a large amount of fuel [7, 8]. This condition implies that carbon emissions occur not only due to the calcination reaction, as mentioned above, but the combustion process in the kiln also contributes significant emissions due to the fuel combustion reaction [9, 10]. Cement producers then make efforts to reduce clinker by using alternative materials such as trass, slag, fly ash, and others [1, 11]. Clinker substitution is expected to reduce combustion energy which has an impact on reducing CO₂ emissions. Nevertheless, the reduction of clinker impacts the

decrease in the compressive strength of the mortar [12, 13]. For this reason, an effort that can be made to maintain the compressive strength of cement that meets the standards is to add additives such as Strength Improver Agent (SIA) [14, 15].

SIA is known to increase compressive strength, but it can also reduce energy in the final cement grinding process [16]. SIA is only added in small doses, namely 100 – 400 ppm at the final grinding of cement [17]. Although SIA is added in small doses, it can significantly improve cement quality and increase efficiency in final cement grinding [18]. Another advantage of the addition of SIA is that it can increase cement's fineness, which is closely related to compressive strength. However, it is necessary to observe to determine the optimal amount of SIA added, so that cement with compressive strength meets all standard criteria but is economical.

Strength Improver Agent (SIA) type Propylcol GX is a material in the form of a dark black liquid that is known to have the ability to increase the compressive strength of cement [19]. SIA, which is added during the final grinding of cement, can reduce the level of clinker hardness so that it can increase the efficiency of the grinding machine [20]. This objective has implications for reducing energy consumption, reducing greenhouse gas emissions. Some materials commonly used as SIAs are tri ethanol amine (TEA), mono- and diethylene glycol (DEG), oleic acid, sodium oleate, sulfite waste liquid, dodecylbenzene sulfonic acid, and sodium lignosulfonate (from the paper industry). This organic material is added in low doses ranging from 0.01-0.05% [21].

Based on the theory, SIA can perfectly remove the ball coating to disperse the milled material. Ball coating can occur due to several factors, namely surface energy, electrostatic forces, adsorption, and mechanical impact [21, 22]. Fine particles from grinding results are positively charged, and some are negative. Materials with positive and negative charges experience electrostatic forces resulting in agglomeration [23, 24]. The presence of SIA, which is a polar organic compound, can play a role in weakening the electrostatic attraction that arises to prevent agglomeration. Minimizing this agglomeration causes the particles to be easier to crush into smaller sizes. In the end, using SIA can provide technical benefits such as

increasing mill productivity or production capacity and providing economic benefits, namely reducing energy costs.

This study used SIA Propylcol GX on PCC Slag cement samples available on the market. Variations in the addition of SIA were 30, 150, 200, 250, 300, 350, and 400 ppm. To determine the effect of adding SIA on the compressive strength of cement, refer to the ASTM C 109 standard.

2.0 METHODOLOGY

Research tools

The equipment used in this study is the Blaine test tool based on ASTM C 204-00, the residue test tool based on ASTM C 430-08, the compressive strength test tool based on ASTM C 109 and QPT-LAB-SNI-05, and the cement chemical test kit namely tests I.R., LOI based on SNI 15-2049-115, and XRF Thermo ARL 8480S based on ASTM C 114, ASTM-STP 985 Rapid Methods for Chemical Analysis of Hydraulic Cement.

Research Materials

The materials used in this study were: blank samples with Clinker composition: 67%, Gypsum: 2.58%, Limestone: 15.37%, and Slag: 15.05%, SIA type PROPYCOL GX, Ottawa sand, NH_4NO_3 20%, and 10% NaOH.

Sample Design

The comparison sample used in this study was called a blank, namely PCC slag with the following percentages: Clinker: 67%, Gypsum: 2.58%, Limestone: 15.37%, and Slag: 15.05%. The type of SIA used is PROPYCOL GX.

The study was started by preparing eight samples consisting of one blank sample (PCC+Slag 15.05%) and seven other samples, namely blank + SIA with variations of 100, 150, 200, 250, 300, 350, and 400 ppm. The mortar constituents in the study consisted of water, cement, Ottawa sand, and SIA-type PROPYCOL GX. The amount of cement and sand was kept constant, namely 740 g and 2035 g, while the amount of water added followed the flowability target of 110 ± 5 mm. The mortar constituent components can be seen in Table 1.



Figure 1 Mortar Workability Test

Table 1 Mortar Composition and flowability

No.	Sample	Cement	Sand	Flowability	Water	SIA
		g	g	mm	g	ppm
6	BLANK	740	2035	109.0	357	-
2	Sample 1	740	2035	107.0	355	100
3	Sample 2	740	2035	108.0	352	150
4	Sample 3	740	2035	108.5	352	200
5	Sample 3	740	2035	111.5	352	250
6	Sample 4	740	2035	113.0	350	300
7	Sample 5	740	2035	115.0	350	350
8	Sample 6	740	2035	108.0	345	400

Mortar preparation and flowability

First, prepare a mortar dough of cement, sand, and water with a ratio of 1: 2.75: 0.485. Eight specimens were prepared by weighing 740 grams of cement, 2035 g of Ottawa sand, water, and SIA. The mixture was stirred at a low speed of 140 ± 5 rpm for 30 seconds. The sand material was added until it finished, increasing the speed to 285 ± 10 rpm and mixing for another 30 seconds. Then, the mixer was stopped and allowed to stand for 90 seconds. Mix for another 60 seconds at the same speed and then put it into the cube mold.

Before being put into the cube mold, a flowability test was carried out on the mortar sample to determine the workability of the mortar. Pour the test object into the flow table mold about half of the mold, then pound it slowly for ± 20 times. Then pour the rest of the mixture into the mold until it is complete and do the collision for ± 20 more times. After that, flatten the sample, turn on the machine until the tool taps the table 24 times, then turn off the machine and measure the diameter of the sample formed. If the results of the flowability test meet the requirements, then the mortar mix can be directly fed into the cube molds, but if the results are not in accordance, then a new mortar mix is made again with the same procedure. The mortar workability testing mechanism can be seen in Figure 1.

Testing

The tests carried out consisted of physical and chemical tests. Physical tests such as the Blaine test, residue test, flowability test, and compressive strength test. While the chemical test tests the insoluble fraction, the loss of glow (LOI), and testing the major and minor content of cement using XRF.

3.0 RESULTS AND DISCUSSION

Fineness and Residue Test Results

The fineness of the cement affects the compressive strength, normal consistency, and setting time. The finer the cement, the greater the specific surface area so that the cement is more reactive when it reacts with water because the bonding power between the particles will be more substantial, which causes the strength of the cement to increase [25–27]. If the cement produced is too coarse, its plasticity, strength, and consistency will be reduced, and at the same time, the separation between the particles will be more visible.

The tool used in fineness testing is Blaine permeability. The method is carried out by measuring the time it takes for the liquid in the manometer to drop from the cement medium (its permeability). The longer it takes to flow, the finer the cement means. The liquid in the manometer used is Dibutyl Phthalate because it meets the criteria such as non-volatile, non-hygroscopic, and has low viscosity and density. The test was carried out twice, and the average Blaine value was $3811.85 \text{ cm}^2/\text{gr}$. This result meets the criteria because it is far above the minimum standard of $2800 \text{ cm}^2/\text{gr}$ [29]. The mechanism of the Blaine test is presented in Figure 2. In addition, a $45 \mu\text{m}$ residue test was also carried out on the test sample. The residue test was carried out to determine the particle size uniformity that passed the sieve $45 \mu\text{m}$. The test was carried out by spraying 10 ± 1 psi pressurized water toward the cement in the sieve. Residues affect the cement's homogeneity, so the more significant the deviation obtained from the test, the less homogeneous the cement will be. With increasing fineness, the smaller the residue obtained. Cement with a slight residue number means that the cement has fine particles[30]. The residual value affects the bonding and compressive strength [31]. The more residue retained on the sieve, the less binding and compressive strength of the cement. The residue rate obtained was 11.44%. The residue test equipment and testing mechanism are shown in Figure 3 and Figure 4.



Figure 2 Blaine Testing Mechanism



Figure 3 Cylindrical sieve with $45\mu\text{m}$ pore diameter



Figure 4 Residue Testing Mechanism

Chemical Test Results

Cement oxide chemical analysis was conducted to determine the insoluble residue (IR), Loss of Ignition (LOI), and XRF. Insoluble residue (IR) testing is intended to determine the amount of impurity that does not dissolve when reacted with HCl. The impurity came from clay compounds in gypsum and unbound SiO_2 [32]. The test results provide an IR value of 1.04%.

The Loss on Ignition (LOI) test determines moisture, bound water, water from free lime, the amount of carbonate from limestone, and CO_2 in the sample [33]. This content is the total weight of the sample lost after annealing. Specifically for humidity, the greater the LOI level, it indicates that the water content is too much, which tends to make cement agglomerate more efficiently, resulting in a shorter storage life for cement. The test gives a humidity result of 7.86%, which still meets the requirements of SNI SNI 15-2049-1994.

After the sample meets the LOI testing requirements, its chemical content can be measured using an X-Ray Fluorescence (XRF) instrument [34]. Chemical testing is intended to determine the content of major and minor oxide elements in cement using the XRF tool, as shown in Table 2.

Table 2 Results of cement oxide test for blank samples

Major Elements (%)			Minor Elements (%)		
LOI	7.86	SO ₃	1.88	TiO ₂	0.34
SiO ₂	18.96	F-CaO	0.87	P ₂ O ₅	0.07
Al ₂ O ₃	5.43	TA	0.49	SrO	0.24
Fe ₂ O ₃	2.4	K ₂ O	0.53	Mn ₂ O ₃	0.1
CaO	57.74	Na ₂ O	0.14	Cr ₂ O ₃	0.02
MgO	4,25	Cl ⁻	0,0001	ZnO	0,06

Based on SNI 15-7064-2014, only the SO₃ parameter is the main benchmark for composite Portland cement [35]. The results of this study showed an SO₃ content of 1.88%. This figure still meets the requirements where the maximum allowed according to the standard is 4%. SO₃ levels affect the formation of C2S and C3S compounds during the combustion process in the kiln [36]. C3S contributes to the initial compressive strength of cement, while C2S content contributes to cement strength at a longer life and is a determinant of the final compressive strength of cement [37]. The higher the SO₃ content, the greater the cement's final compressive strength (28 days) increases. The test equipment and mechanism for XRF are presented in Figures 5 and 6.

**Figure 5** XRF Instruments**Figure 6** XRF Testing Mechanism

Physics Testing Results

The main physical property of cement is compressive strength [35]. This parameter measures the mortar's ability to withstand compressive loads. Mortar compressive strength measurements were carried out from curing age to 1, 3, 7, and 28 days according to ASTM C 109 and QPT-LAB-SNI-05 standards. The greater the compressive strength, the better the quality of the mortar.

The sample was first made into the mortar using a cube mold with a side measuring ± 2.54 cm for testing the compressive strength. However, before that, it is necessary to determine the workability of the mortar using a flow table. Flowability testing using a flowable tool aims to estimate the amount of water used in making mortar because the total

water used affects the resulting compressive strength [38]. The addition of SIA to the sample affects the need for added water in the mortar. Whereas more SIA levels are added, the water requirement decreases. The allowable flowability value is 110 ± 5 mm. The results of the flowability test can be seen in Table 1, which meets the criteria for all test samples.

**Figure 7** Compressive Strength Test Equipment**Figure 8** Compressive Strength Testing Mechanism

The specimens are stored in a humid room for 20-24 hours but must be protected from dripping water. The storage room is made of non-rusting material and contains saturated lime water where the room temperature is maintained at $21 \pm 2^\circ\text{C}$. The compressive strength test method follows ASTM C 109 and QPT-LAB-SNI-05. The effect of adding SIA on compressive strength can be seen in Table 3, where sample 5, namely the addition of 350 ppm SIA, showed the best compressive strength of 359 kg/cm^2 . The compressive strength test equipment and mechanism can be seen in Figures 7 and 8.

Table 3 Results of compressive strength measurements

Compressive Strength (kg/cm ²)	1 day	3 days	7 days	28 days
Blank	85	163	241	336
Sample 1	86	164	242	337
Sample 2	90	169	245	338
Sample 3	95	175	249	346
Sample 3	99	180	254	349
Sample 4	103	184	258	351
Sample 5	107	190	264	359
Sample 6	103	188	260	356

Figure 9 compares the compressive strengths of OPC, PCC substituted by 15.05 % slag, and PCC substituted by 15.05 %

slag + SIA 350 ppm for curing ages 28 days. It can be seen that there is a significant decrease in compressive strength with some of the clinkers are replaced by slag. However, the compressive strength has increased with the addition of 350 ppm SIA to meet the compressive strength criteria, even though it is still below the OPC compressive strength.

The experimental results showed that adding SIA (Strength Improver Agent) Propylcol GX to PCC Slag cement increased the compressive strength, especially the initial compressive strength. At the ages of 1 and 3 days, there was an increase in compressive strength of 25.53%, while at the ages of 7 and 28 days, there was still an increase in compressive strength, although not as big as the initial compressive strength. Nevertheless, what is interesting here is that the compressive strength began to decrease with the addition of 400 ppm SIA.

Other researchers reported that adding SIA with a varied range of 250 – 300 ppm gave a compressive strength that met the standard requirements. It is also essential to consider that adding SIA is associated with additional costs, so the optimum point between quality and cost must be determined.

The effect of the addition of SIA on the CaO content can be seen in Figure 10. The figure shows that adding 350 ppm, SIA slightly reduced the CaO content compared to the blank. CaO content can provide an overview of the resulting CO₂ emissions [38–40]. From the stoichiometric calculation reaction of Calcium carbonate (CaCO₃), the number of moles of CaO produced equals the number of moles of CO₂ produced. The results are certainly not very satisfactory, so it is necessary to research other types of composite cement.

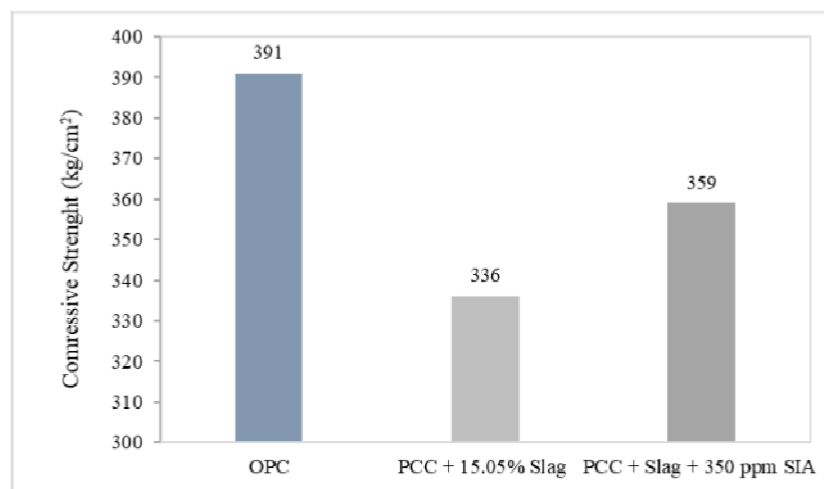


Figure 9 Effect of adding SIA on compressive strength at curing age 28 days

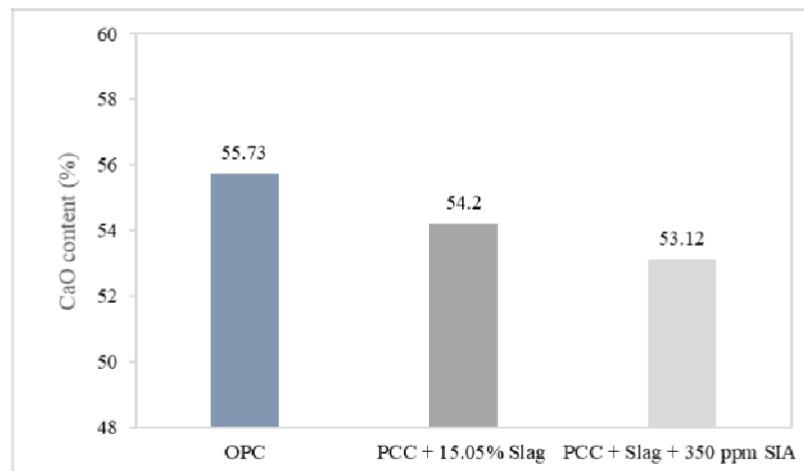


Figure 10 Effect of SIA addition on CaO content in cement

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4.0 CONCLUSION

From a series of studies and observations, it can be concluded that: use of SIA (Strength Improver Agent) contributes to the increase in compressive strength. Furthermore, the addition of SIA to PCC Slag cement appeared to be more reactive at 1 and 3 days of early strength and slowed down at 7 and 28 days of age. The optimum level of adding SIA Prophycol GX to PCC Slag cement is 350 ppm, increasing the compressive strength $\geq 7\%$ against blank.

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