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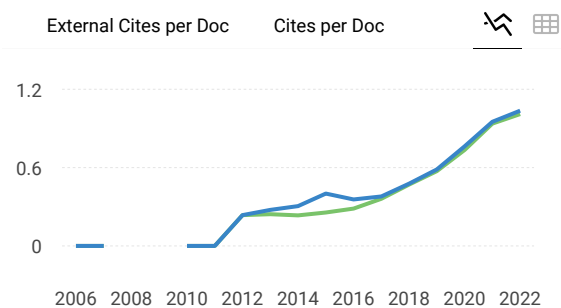
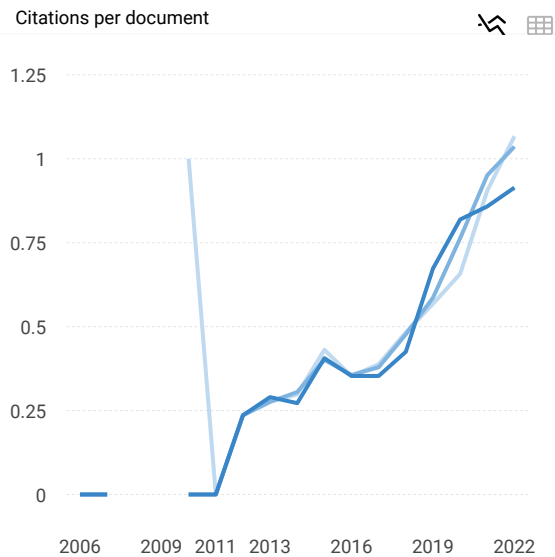
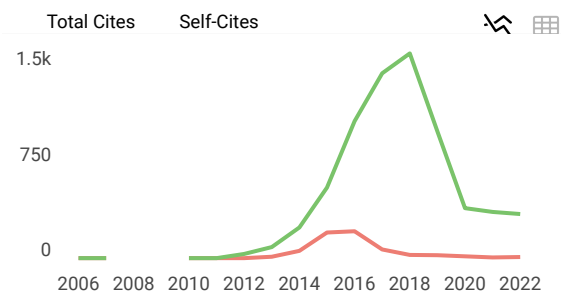
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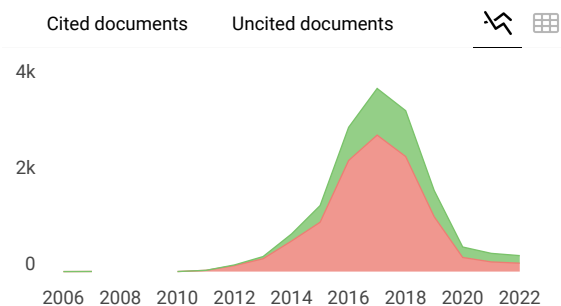
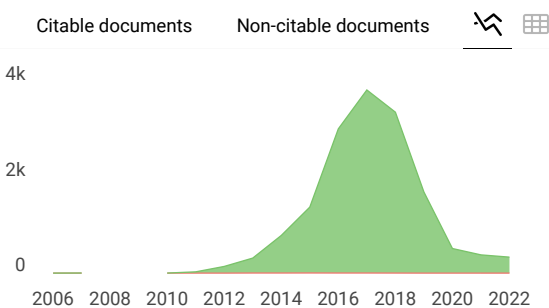
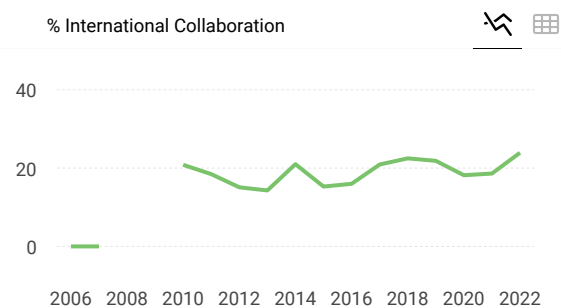
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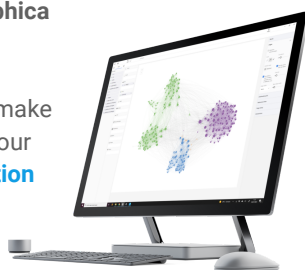
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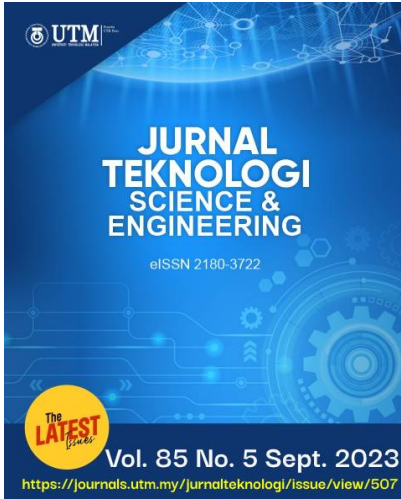
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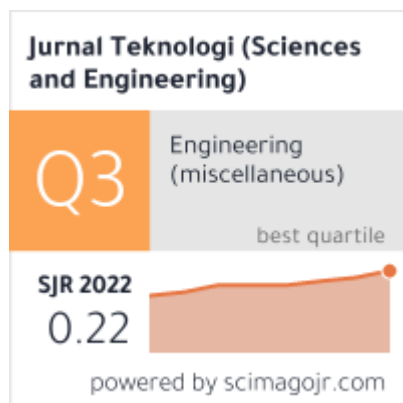
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# IMPROVE THE COMPRESSIVE STRENGTH USING A STRENGTH IMPROVER AGENT (SIA) IN THE CEMENT INDUSTRY IN INDONESIA

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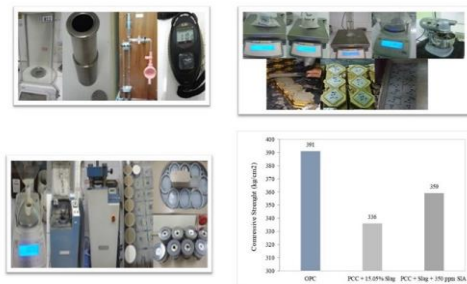
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## Graphical abstract



## Abstract

Greenhouse gas emissions such as CO<sub>2</sub> are released during clinker production through various processes, including the calcination reaction of limestone (CaCO<sub>3</sub>). To reduce CO<sub>2</sub> emissions per ton of cement produced, some studies have explored the use of clinker substitutes. However, a reduction in the amount of clinker can also result in decreased compressive strength of the cement. To address this issue, the addition of a Strength Improver Agent (SIA) can be used to maintain the necessary compressive strength and ensure compliance with all relevant standards. Therefore, This study aimed to determine the optimal amount of SIA required to achieve the desired compressive strength. The study added SIA in varying amounts (100, 150, 200, 250, 300, 350, and 400 ppm), and the compressive strength of cement was measured at 1, 3, 7, and 28 days based on ASTM C 109 and QPT-LAB-SNI-05 standards. Physical tests were also conducted, including 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 showed that the optimum amount of SIA addition is 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

The demand for cement is on the rise in today's modern world, including Indonesia. To meet this growing demand, both the private sector and the government have increased their management of the cement industry [1, 2]. As a result, cement producers are competing fiercely by taking into account several factors, such as production costs, energy efficiency, quality, selling price, and environmental issues [3]. In the kiln, which involves the reaction of calcium carbonate, contributes significantly to the release of greenhouse gas emissions, particularly CO<sub>2</sub> [4, 5]. In other words, an

increase in cement production capacity can potentially contribute to global warming problems if not carefully addressed [5, 6].

Historically, Ordinary Portland Cement (OPC) was typically made by combining clinker and gypsum. However, clinker production involves burning limestone at high temperatures, which requires a significant amount of fuel [7, 8]. This process leads to carbon emissions not only from the calcination reaction but also from the fuel combustion reaction in the kiln [9, 10]. To address his issue, cement producers have been making efforts to reduce clinker by using alternative materials such as trass, slag, fly ash, etc. [11]. Clinker substitution is expected to reduce



combustion energy, which has an impact on reducing CO<sub>2</sub> emissions. However, the reduction of clinker impacts the decrease in 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; this is because the addition of materials such as SIA into the cement mixture can reduce water thereby increasing the strength of the concrete [16]. Furthermore, it is only added in small doses, namely 100 - 400 ppm, during the final grinding of cement [17]. Although SIA is added in small doses, it can significantly improve cement quality and increase the efficiency of 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 determine the optimal amount of SIA added in a way that cement with compressive strength meets all standard criteria but is economical.

Prophycol GX is a dark black liquid material known as a SIA that has the capacity to enhance the compressive strength of cement [19]. By adding SIA during the final grinding stage of cement production, it can effectively reduce the hardness of clinker, resulting in greater efficiency of the grinding machine [20]. This use of SIA has significant implications for reducing energy consumption and greenhouse gas emissions. Some materials commonly used as SIA 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). They were added in low doses ranging from 0.01% to 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]. Subsequently, fine particles from grinding results are positively charged, and some are negative. Positively and negatively charged materials experience electrostatic forces that lead to agglomeration [23, 24]. The presence of SIA, a polar organic compound, can play a role in weakening the electrostatic attraction that arises to prevent agglomeration and minimizing this agglomeration makes it easier to break down the particles 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 Prophycol GX on PCC Slag cement samples available in the market. Variations in the addition of SIA were 100, 150, 200, 250, 300, 350, and 400 ppm. To determine the effect of adding SIA on compressive strength of cement, the ASTM C 109 standard was used.

## 2.0 METHODOLOGY

### Research Tools

In this study, the equipment used 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 15-2049-1994, and XRF Thermo ARL 8480S based on ASTM C 114, ASTM-STP 985 Rapid Methods for Chemical Analysis of Hydraulic Cement.

### Research Materials

In this study, the materials used were blank samples with Clinker composition: 67%, Gypsum: 2.58%, Limestone: 15.37%, and Slag: 15.05%, SIA type PROPHYCOL GX, Ottawa sand, distilled water, NH<sub>4</sub>NO<sub>3</sub> 20%, and 10% NaOH. Meanwhile, the cement used consisted of Portland Composite Cement provided by PT Indocement Tunggal Prakasa Tbk.

### Sample Design

In this study, the comparison sample used is 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 PROPHYCOL GX.

This study started with preparing eight samples consisting of one blank sample (PCC+Slag 15.05%) and seven others, namely blank + SIA with variations of 100, 150, 200, 250, 300, 350, and 400 ppm. The mortar constituents comprised water, cement, Ottawa sand, and SIA-type PROPHYCOL 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 constituent mortar components can be seen in Table 1.

**Table 1** Mortar Composition and Flowability

No.	Sample	Cement	Sand	Flowability	Water	SIA
		g	g	mm	g	ppm
1	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

### Fineness and Residue Test

The tool used in fineness testing is Blaine permeability. The method is carried out by measuring the time taken for the liquid in the manometer to drop from the cement medium (its permeability). The longer it takes to flow, the finer the cement becomes. 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.

### Mortar Preparation and Flowability Test

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

Before being placed in the cube mold, a flowability test was carried out on the mortar sample to determine the workability of the mortar. The test object was poured into the flow table mold about half of the mold and then pounded slowly for  $\pm 20$  times. Furthermore, the rest of the mixture was poured into the mold until it was complete and the collision for  $\pm 20$  more times. After that, the sample was flattened, and the machine was on until the tool tapped the table 24 times. After the machine was turned off, the diameter of the sample formed was measured. 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.



Figure 1 Mortar workability test

### Testing

The tests carried out consisted of physical and chemical tests. Meanwhile, the physical tests include the Blaine test, residue test, flowability test, and compressive strength test. At the same time, the

chemical test tests the insoluble fraction, Loss of Ignition (LOI), and testing the major and minor cement content using XRF, as shown in Table 2. The test equipment and mechanism for XRF are presented in Figures 2 and 3. The test equipment and mechanism for compressive strength are presented in Figures 4 and 5. The mechanism of the Blaine test is presented in Figure 6. The residue test equipment and testing mechanism are shown in Figure 7.



Figure 2 XRF instruments



Figure 3 XRF testing mechanism



Figure 4 Compressive strength test equipment



Figure 5 Compressive strength testing mechanism



Figure 6 Blaine testing mechanism



Figure 7 Residue testing mechanism

**Fineness and Residue Test Results**

The fineness of cement affects compressive strength, normal consistency, and setting time. The finer cement, the greater the specific surface area, and hence, cement is more reactive when reacting with water because the bonding power between the particles will be more substantial, which causes the strength of cement to increase [25,26,27]. 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 test was carried out twice, and the average Blaine value was 3811.85 cm<sup>2</sup>/gr. This result meets the criteria because it is far above the minimum standard

of 2800 cm<sup>2</sup>/gr [28]. In addition, a 45 μm residue test was also carried out on the test sample to determine the particle size uniformity that passed the sieve 45 μm. The test was carried out by spraying 10 ± 1 psi pressurized water toward cement in the sieve. Residues affect cement's homogeneity, and hence, the more significant the deviation obtained from the test, the less homogeneous cement will be. With increasing fineness, the smaller the residue obtained. Subsequently, cement with a low number of residues means that cement has fine particles [29]. The residual value affects the bonding and compressive strength [30,31]. The more residue retained on the sieve, the less binding and compressive strength of the cement; the residual test rate obtained was 11.44%.

**Chemical Test Results**

Chemical analysis of cement oxide was performed to determine Insoluble Residue (IR), LOI, and XRF. IR testing aimed to determine the amount of impurity that does not dissolve when reacted with HCl. The impurity was obtained from clay compounds in gypsum and unbound SiO<sub>2</sub>[32]. The test results provide an IR value of 1.04%, and the LOI test determines moisture, bound water, water from free lime, the amount of carbonate from limestone, and CO<sub>2</sub> in the sample [33]. This content is the total weight of the sample lost after annealing. Specifically, in the case of humidity, a higher LOI value indicates that the water content is too high, causing the cement to agglomerate more efficiently, which reduces the cement's shelf life. The test gives a humidity result of 7.86%, which still meets the requirements of SNI 15-7064-2014.

After the sample meets 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 samples compare to SNI 15-7064-2014

	Major Elements (%)				Minor Elements (%)			
		Max.			Max.			Max.
LOI	7.86	8	SO <sub>3</sub>	1.88	4	TiO <sub>2</sub>	0.34	-
SiO <sub>2</sub>		-	F-		1.5	P <sub>2</sub> O <sub>5</sub>	0.07	-
	18.96		CaO	0.87		SrO	0.24	-
Al <sub>2</sub> O <sub>3</sub>	5.43	6	TA	0.49	-	Mn <sub>2</sub> O <sub>3</sub>	0.1	-
Fe <sub>2</sub> O <sub>3</sub>	2.4	3.17	K <sub>2</sub> O	0.53	0.6	Cr <sub>2</sub> O <sub>3</sub>	0.02	-
CaO	57.74	-	Na <sub>2</sub> O	0.14	0.75	ZnO	0,06	-
MgO	4,25	6	Cl <sup>-</sup>	0,0001	-			

Based on SNI 15-7064-2014, only the SO<sub>3</sub> parameter is the main benchmark for composite Portland cement [35]. The results showed an SO<sub>3</sub> content of 1.88%. This figure still meets the requirements where the

maximum allowed according to the standard is 4%. SO<sub>3</sub> 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 SO<sub>3</sub> content, the greater cement's final compressive strength (28 days) increases.

**Physical Testing Results**

The main physical property of cement is compressive strength [35]. This parameter measures the mortar's ability to withstand compressive loads. Subsequently, 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 selected and put in the mortar using a cube mold with a side measuring ± 2.54 cm to test 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 aimed 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 the 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. 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 ± 2°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 35.2 MPa.

**Table 3** Results of compressive strength measurements

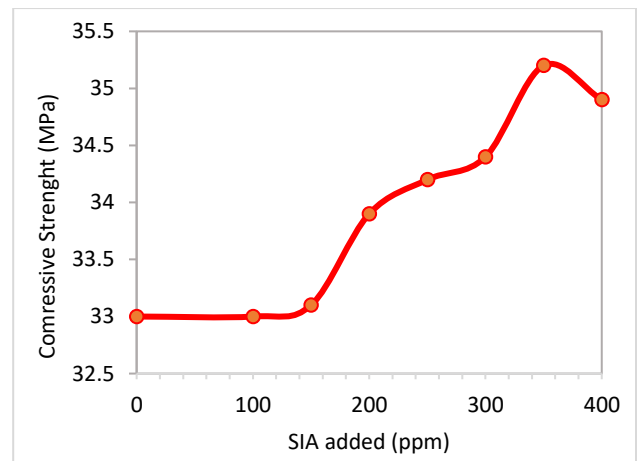
Sample	Compressive Strength (MPa)			
	1 day	3 days	7 days	28 days
QPT-LAB-SNI-05 (SNI-2049, 2015)	-	Min. 12.7	Min. 19.6	Min. 27.5
Blank	8.3	16.0	23.6	33.0
Sample 1	8.4	16.1	23.7	33.0
Sample 2	8.8	16.6	24.0	33.1
Sample 3	9.3	17.2	24.4	33.9
Sample 4	9.7	17.7	24.9	34.2
Sample 5	10.1	18.0	25.3	34.4
Sample 6	10.5	18.6	25.9	35.2
Sample 7	10.1	18.4	25.5	34.9

Figure 8 shows the impact of SIA adding with variations of 100 to 350 ppm on the compressive strength of PCC substituted by 15.05 % slag for curing ages 28 days. It can be seen that there is a significant increase in

compressive strength for the addition of 350 ppm SIA which meets the compressive strength criteria based on ASTM C 109.

The experimental results showed that adding SIA Prophycol GX to PCC Slag cement increased compressive strength, especially the initial compressive strength. At the age 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 high as the initial compressive strength. However, what is interesting here is that 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 9. The figure shows that the addition of 350 ppm SIA slightly reduced the CaO content compared to the blank sample. CaO content can provide an overview of the resulting CO<sub>2</sub> emissions [38,39,40]. From the stoichiometric calcination reaction of Calcium carbonate (CaCO<sub>3</sub>), the number of CaO produced equals the number of moles of CO<sub>2</sub> produced. The results could be more satisfactory; hence, it is necessary to perform an investigation on other types of composite cement.



**Figure 8** Effect of adding SIA on compressive strength at curing age 28 days

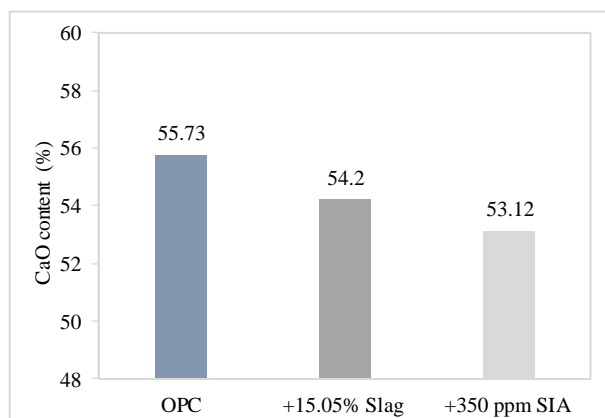


Figure 9 Effect of adding SIA on CaO content in cement

## 4.0 CONCLUSION

Based on a series of studies and observations, it can be concluded that the use of SIA contributes to the increase in compressive strength. Additionally, when SIA is added to PCC Slag cement, it demonstrates greater reactivity during the initial stages of strength development at 1 and 3 days, while its effects slow down at 7 and 28 days of age. The optimum level of the addition of SIA Prophycol GX to PCC Slag cement is 350 ppm, resulting in a compressive strength increase of  $\geq 7\%$  compared to the blank sample.

## Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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