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To cite this article: Herliati *et al* 2021 *IOP Conf. Ser.: Mater. Sci. Eng.* **1053** 012116

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Optimization of Gypsum Composition Against Setting Time And Compressive Strength In Clinker For PCC (Portland Composite Cement)

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Abstract. Gypsum is one of the additive raw materials added to the clinker grinding process that regulate the binding time of cement or commonly known as a retarder. This research aims to observe the effect of gypsum added variation on the compressive strength of mortar and the whole quality of Portland Composite Cement at plant 14 P.T Indocement Tunggal Prakasa Tbk. The PCC compositions did prepare with a variation of the addition of 0 - 5% gypsum where the clinker ratio did fix at 75%, and two other additives, namely limestone, and trass did add with a fixed ratio of 1.2: 1. The effect of adding gypsum to clinker measured by performing a mortar compressive strength test and the binding time at the ages of 1, 3, 7, and 28 days. The results of series observation and analysis show that composite Portland cement has the most optimal quality in the addition of gypsum as much as 3.5%, where SO₃ content is 1.7%, the initial setting time is ≥ 90 , and the final setting time is maximum at 375 minutes.

Keywords: additives, mortar, XRF

1. Introduction

Construction in Indonesia is growing, along with increasing human needs. Indonesia is a developing country that continues to carry out developments in various fields such as housing, toll roads, office buildings, hospitals, and others. Therefore, the use of cement in the country continues to increase. Ministry of Industry data shows that the total national cement consumption in 2017 reached 102 million tons of the total production capacity of 70 million tons per year. Therefore, the cement industry must meet these needs both in terms of quality and quantity [1].

Gypsum is one of the additives added to the cement clinker to match the required specifications. This compound can affect the setting time and affect the compressive strength, both the initial compressive strength value and the compressive strength value during use [2]. The amount of gypsum added to the clinker grinding process does control by the content of sulfur trioxide (SO₃) in the cement produced. The higher SO₃ content in cement indicates that the gypsum composition is also high. However, the content of SO₃ compounds in cement according to the SNI 15-7064-04 standard should be ≤ 4.0 [3]

Although it provides positive effects such as controlling the plasticity of cement dough, if the amount exceeds the maximum standard at five weight percent (5%), gypsum can negatively affect, which causes expansion of the cement during use [4]. Therefore, the use of gypsum does strictly controlled.



Investigated clinker characteristics by adding some gypsum already done by several studies [4]. However, the effect of SO_3 content caused by gypsum added still needs more observation.

Before being used, the raw materials used must be analyzed to guarantee the quality of the product. X-ray fluorescence spectrometry (XRF) did use as an instrument of qualitative and quantitative analysis. This method is a non-destructive method of analysis used to identify and determine the concentration of compounds present in solids. The XRF used to determine the PCC's chemical content in this study is a type of Energy-dispersive X-ray Fluorescence (EDXRF).

Portland Composite Cement (PCC) is cement from grinding clinker with a cement content composition of 60% -70%. Not only gypsum, but other additives that do also added during grinding are also slag, trass, and limestone. PCC can be used in concrete construction, masonry, plastering, and paving blocks. This study aims to observe the effect of adding gypsum on setting time and compressive strength of mortar with a limit of SO_3 content in cement ranging from 1.5% to 1.9%. These research results are expected to provide information on the optimum value of gypsum content added to the PCC at plant 14 P.T Indocement Tbk to be a reference in the supply of cement with quality market needs. [5]. Also, the use of optimal gypsum levels can reduce the cost of additive raw materials.

2. Research Methods

2.1. Tools and Materials

The equipment used in this study consisted of a scale, oven, Herzog grinding machine, Vessel Disk, ACMEL machine, Mixers 300/260 L, Vicat E055N, measuring cup with a capacity of 200 mL and 250 mL. Meanwhile, the raw materials used are cement, distilled water, gypsum, limestone, and trass. The setting time testing was carried out with the Vicat tool manually and automatically. Meanwhile, the compressive strength test of hydraulic cement mortar did carry out using a 50 mm side cube mold.

2.2. Procedure

First, the test object is prepared, namely cement paste, which is made by mixing 650 g of cement with a certain amount of water until the dough has the appropriate thickness. The mixture was put in a mixer and stirred at a low speed of $140 \pm$ five revolutions per minute for 30 seconds. Furthermore, PCC is produced by mixing cement paste with gypsum, limestone, and trass with various ratios with a target of $3800\text{-}4000 \text{ cm}^2 / \text{g}$ of Blaine. The test object is formed into a ball with both hands (wearing rubber gloves) and then inserted into a large hole in the Vicat tool, namely a cone ring with a height of 40 ± 1 mm and a flat plate as can be seen in Figure 1.a. The surface of the test object has a minimum diameter of $60 \text{ mm} \pm 3$ mm. The samples are then stored in a humid room for 20-24 hours with the top surface in contact with humid air but must be avoided from water droplets.

2.3. SO_3 Measurement

The SO_3 composition is determined according to SNI 15-7064-04. The samples were prepared and fused automatically by the machine, calibrated by NIST standards. The samples were then analyzed by Energy-dispersive X-ray Fluorescence (EDXRF).

2.4. Fineness Measurement

PCC with a particular composition, then milled using a 5 kg mini tube mill capacity. The material mixture is ground for 65 ± 5 minutes to achieve a fineness (Blaine) $3850 \pm 50 \text{ cm}^2 / \text{g}$. After the material mixture's smoothness meets the requirements, the material is fed into the tubular for 55 ± 5 minutes to the homogenization process.

2.5. Setting Time and Compressive Strength Testing

The test object as prepared was left in a damp room for 30 minutes after printing, then penetrated with a 1 mm diameter needle every 15 minutes. The distance between the penetration points is not less than 9.5 mm from the inner mold wall, and the distance between the two penetration points is not less than

6.4 mm. The test results were then made to interpolate to determine the initial binding-time where the penetration of 25 mm was obtained.

For the compressive strength testing, the test object that has been prepared is then left in the mold in a humid room for one day, as shown in Figure 1b. Then, the test sample is immersed in water containing lime until the time of the test begins. The method of testing the compressive strength of cement mortar was carried out using a 50 mm cube mold. The goal to be achieved is to find out whether the cement has met specifications or not.

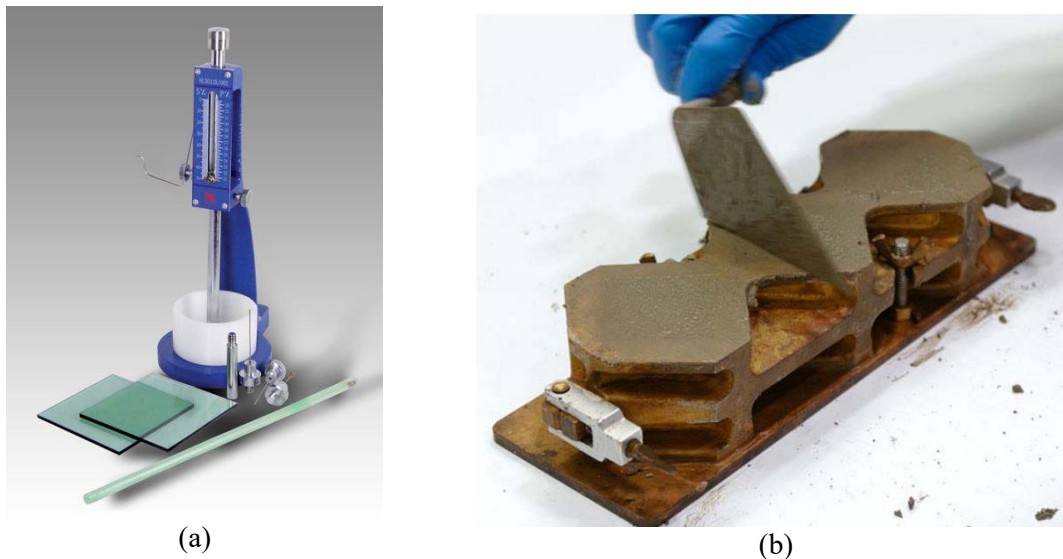


Figure 1a. Testing tool setting time and 1b. Compressive Strength Testing Tool

3. Result and Discussion

3.1. PPC Cement Preparation

In this research, a total mass of 3.500 g PCC (Portland Pozzolan Composite) is made by grinding clinker, gypsum, limestone, and trass together to produce the desired smoothness. The PCC composition was prepared with a variation of the addition of 0 - 5% gypsum, where the clinker ratio was fixed at 75 wt% while limestone to trass at weight ratio 1.2. All sum up, the percentage of clinker, gypsum, limestone, and trass are at 100 percent. The composition of PCC-with eleven variations in the amount of gypsum can be seen in Table 1

Table 1. The Composition of PCC

NO	Clinker		Gypsum		Limestone		Trass	
	%	Gram	%	Gram	%	Gram	%	Gram
1	75	2625	0.0	0	13.64	477.3	11.36	397.7
2	75	2625	0.5	17.5	13.37	467.8	11.13	389.7
3	75	2625	1.0	35	13.09	458.2	10.91	381.8
4	75	2625	1.5	52.5	12.82	448.7	10.68	373.8
5	75	2625	2.0	70	12.55	439.1	10.45	365.9
6	75	2625	2.5	87.5	12.27	429.6	10.23	357.9
7	75	2625	3.0	105	12.00	420	10.00	350
8	75	2625	3.5	122.5	11.73	410.5	9.77	342
9	75	2625	4.0	140	11.45	400.9	9.55	334.1

NO	Clinker		Gypsum		Limestone		Trass	
	%	Gram	%	Gram	%	Gram	%	Gram
10	75	2625	4.5	157.5	11.18	391.4	9.32	326.1
11	75	2625	5.0	175	10.91	381.9	9.09	318.1

3.2. Chemical Analysis of SO_3

The cement products' chemicals affect physical properties, such as compressive strength and setting time [6]. All samples follow the SNI standard for PCC, as mentioned above, for all variations of gypsum additions, based on sulfur oxide content. The analysis showed that the highest SO_3 content was in the cement sample PCC 11, namely 2.48%, where the highest gypsum composition was 5%. This statement is possible because gypsum with the chemical formula $CaSO_4 \cdot 2H_2O$ is the primary source of SO_3 [3]. Therefore, the higher the gypsum used, the higher the SO_3 content in the PCC-produced, as shown in Table 2 and Figure 2.

Table 2. SO_3 content in PCC and Gypsum Cement

Code	Gypsum added (%)	SO_3 content (%)
PCC 01	0.0	0.34
PCC 02	0.5	0.55
PCC 03	1.0	0.74
PCC 04	1.5	0.95
PCC 05	2.0	1.19
PCC 06	2.5	1.36
PCC 07	3.0	1.60
PCC 08	3.5	1.79
PCC 09	4.0	2.02
PCC 10	4.5	2.23
PCC 11	5.0	2.48

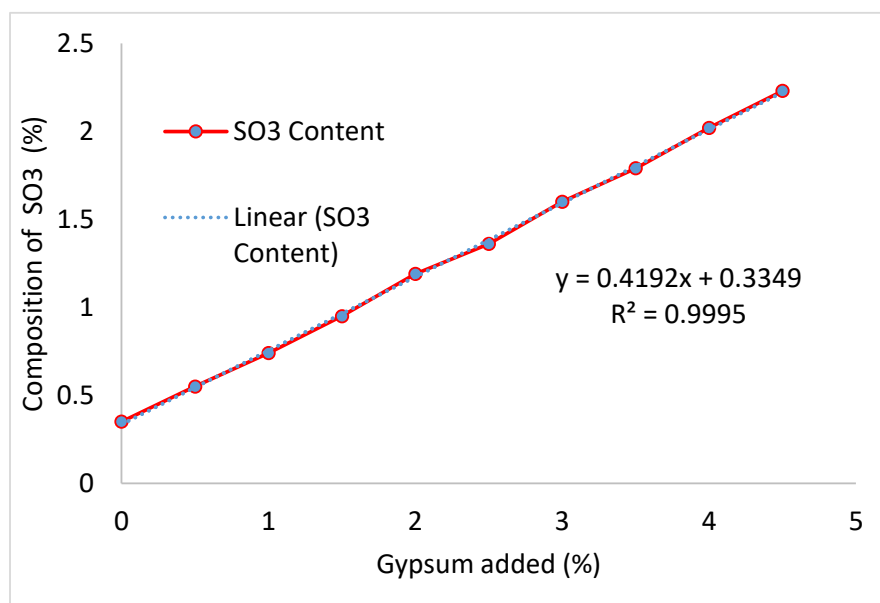


Figure 2. Effect gypsum on the levels of SO_3

Figure 1 shows that the relationship between the increase in gypsum composition and the increase in SO_3 content is directly proportional. High SO_3 levels are an indication that the use of gypsum is too high in cement production. However, for all variation gypsum added, the SO_3 content meets the requirement of less than 3.5%. The resulting linear regression curve has a high correlation, namely 0.9995.

3.3. Fineness analysis

The cement's fineness was analyzed using the $45\mu\text{m}$ sieve method based on ASTM C340, while the Blaine specific surface was measured based on ASTM C204. The fineness of the cement affects the bonds that will form between the particles contained in the cement. The smoother the cement, the larger the surface area. The surface area of the cement is what affects the reactivity of the cement with water [7]

Since a controlled variable, the fineness factor is kept in the range of $3850 \pm 50 \text{ cm}^2/\text{gr}$, as can be seen in Table 3. This value is under the standard specifications of PCC-according to SNI standards. Table 3 shows that all the fineness of samples had met the specific Blaine surface of P.T Indocement for PCC, namely $3800\text{-}4000 \text{ cm}^2/\text{gr}$ [8].

Table 3. Fineness analysis

Sample	Target Blaine 3800- 4000(cm^2/g)	Setting Time (minute)		Compressive Strength (kg/cm^2)			
		Original	Final	Day 1	Day 3	Day 7	Day 28
PCC 1	3824	5	10	31	145	202	262
PCC 2	3838	90	180	46	155	211	273
PCC 3	3890	100	190	61	183	243	289
PCC 4	3828	110	200	74	194	249	309
PCC 5	3862	120	210	75	201	255	316
PCC 6	3891	130	220	80	216	267	324
PCC 7	3811	140	230	89	224	279	333
PCC 8	3854	145	235	98	233	291	348
PCC 9	3866	150	240	95	230	280	346
PCC 10	3829	155	245	91	229	287	345
PCC 11	3866	160	250	81	203	259	331

Gypsum in cement significantly affects setting time, compressive strength, and heat of hydration. From the results of previous studies [9], it is known that the addition of gypsum to the cement clinker aims to slow down the initial hardening of the cement and the final hardening of the cement. Besides gypsum, it also affects the setting time of cement is the content of C3A (tricalcium alumina). In cement, C3A will react the fastest to produce CAH (calcium aluminate hydrate) in the form of a gel and is rigid. Fortunately, with gypsum, CAH will react to form ettringite, which will wrap the surface of CAH and C3A so that it prevents the C3A reaction from forming a gel, and the setting process will be prevented. However, due to the osmosis phenomenon, the ettringite layer will break so that the C3A hydration reaction will occur again, but soon a new ettringite will also be formed. This process will produce a setting time. The more ettringite that is formed, the longer the setting time will be, and the gypsum's presence obtained this. The effect of adding gypsum on setting time can be seen in Figure 3.

SNI 15-7064-2004 requires that the initial setting time for PCC is ≥ 90 minutes, and the final setting time ≤ 375 minutes. In this study, all of the cement added with gypsum still met the specifications required by the SNI. In other words, the cement which was not added with gypsum did not fulfill it because there was no set time.

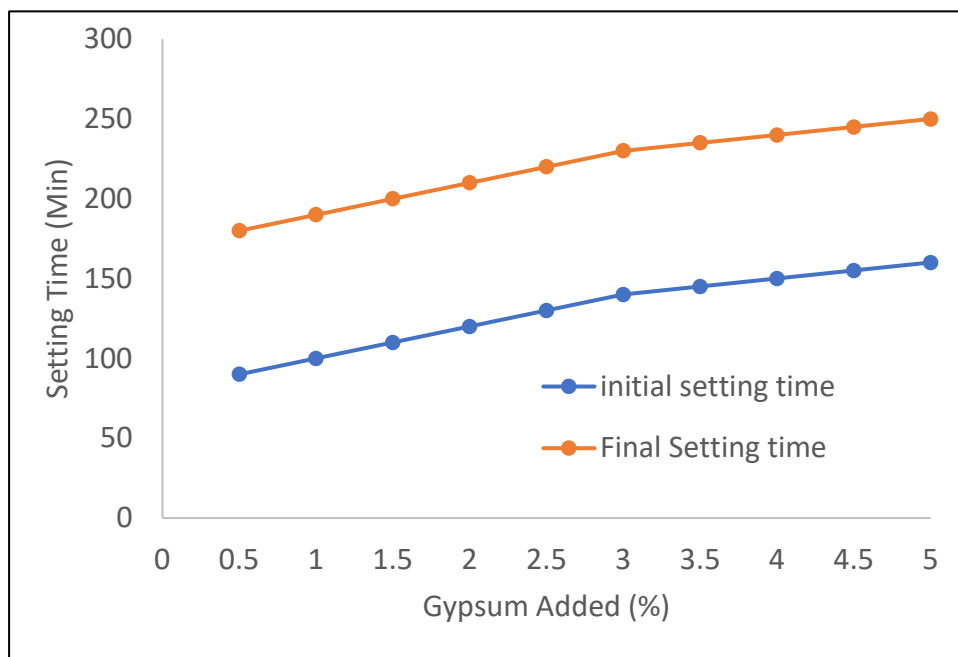


Figure 3. Effect of gypsum on the setting time

3.4. Compressive Strength

Compressive strength is the ability to withstand a pressure load. Compressive strength is an essential property in cement quality criteria. Compressive strength is generally used in mortar and concrete. Mortar is a mixture of cement, water, and sand in a specific ratio. In this study, the mortar's compressive strength was analyzed at one day, three days, seven days, and 28 days of mortar age.

Gypsum affects cement's compressive strength because it can regulate the hydration reaction speed so that the compressive strength obtained can be optimal. If the composition of gypsum in cement is less than standard, it can cause excess C3A. This situation can increase heat during the cement hydration process so that the cement breaks easily during use. Meanwhile, if the gypsum content in cement is excess, it can cause several things such as cracking, slow hardening time, and waste of additives. The gypsum content used in this study is by the company standards of P.T Indocement Tbk, which is in the 3-5% range [9]. The compressive strength of cement mortar PCC 1- PCC 11 can be seen in Figure 4.

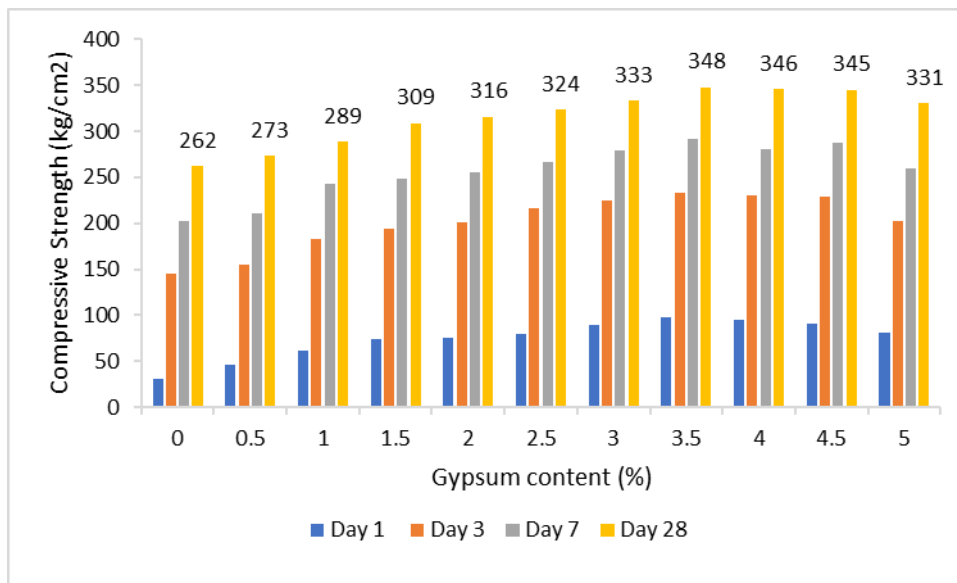


Figure 4 Effect of gypsum on the compressive strength

Figure 4 shows that the variation in gypsum content in composite cement affects the compressive strength's physical properties. The higher the gypsum content, the higher the compressive strength of PCC. However, if the gypsum is added more than 3.5 percent, the compressive strength will gradually decrease [10].

4. Conclusion

The results of observations on variations in the levels of gypsum added to clinker in this study obtained the following conclusions:

1. The addition of gypsum affects the SO_3 levels in cement. The higher the gypsum content added, the higher the SO_3 content in cement.
2. Variations in gypsum content in composite cement affect physical properties, namely setting time. The higher the gypsum content, the longer the cement setting time will be.
3. Variations in gypsum content in composite cement also affect physical properties, namely compressive strength. The higher the gypsum content, the higher the compressive strength of PCC. However, if gypsum added $> 3.5\%$, the compressive strength will gradually decrease.

Acknowledgment

Thanks to the P.T Indocement Tunggul Prakasa Tbk. which has provided support for laboratory and library facilities. Also, thanks to the Faculty of Industrial Technology, Jayabaya University, for the funding provided.

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