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INFLUENCE OF CEMENT FINENESS AND LIMESTONE CONTENT ON STRENGTH DEVELOPMENT AND DRYING SHRINKAGE OF PORTLAND LIMESTONE CEMENT

Nguyen Nhu Quy[1]

[1]Associate Prof., Dept. of Building Materials Technology, The National Univ. of Civil Engineering, 55 Giai Phong Rd., Hai Ba Trung Dist., Hanoi, Vietnam.

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**Corresponding Author,*
Email address: nnquy@hotmail.com

ABSTRACT :

The production of Limestone Portland Cement was proved to be an effective approach to limit the carbon dioxide emission associated with cement clinker firing promoting sustainability development. The addition of limestone to some extent reduces the cement strength so the cement manufacturers try to increase as high as possible the cement fineness to accommodate more limestone content while maintaining its target strength at desired level. Some problems may arise in the concrete made from such a kind of cement, namely the threat of concrete cracks due to shrinkage when it is made from cement having too fine and too fast in rate of hardening. There are not many research publications available dealing with the strength development and shrinkage of the Portland Limestone Cement with varying fineness and different limestone content. This paper reports the influence of the Portland Limestone Cement fineness and limestone content on its strength development and shrinkage. Hence these research results could help to set the limits of Portland Limestone Cement fineness for corresponding limestone content to minimize the threat of the concrete cracks which may harm its durability in use.

KEYWORDS: Portland Limestone Cement cements fineness, cement strength development, and cement mortar shrinkage.

1. Introduction

In order for promoting the sustainability development the great quantity of carbon dioxide emission associated with cement clinker firing must be reduce. The production of composite cements using various mineral additives is said to be an effective approach to reduce output of cement clinker keeping cement production volume unchanged. These mineral additives may like fly ash, burned shale, blast-furnace slag, limestone, etc. The production of Portland Limestone Cement (PLC) becomes more and more popular. In a country like Thailand the share of PLC production volume in Siam Cement Company alone is reached at 29 percent. The technical requirements for PLC are produced in many standards like BS EN 197-1:2011, [1]. In Vietnam an extensive research work was done to promote the PLC production at Hoang Thach Cement Company for which second grade limestone with high Magnesium content was used, [2]. The content of limestone used ranged from 6 to 20 percent with the the cement fineness between 3000 ± 50 cm2/gr and 5200 ± 50 cm2/gr. It was found that the higher content of limestone the lower cement strength. Moreover the presence of limestone somehow increases cement early strength but consequently reduces the 28-day strength. What happens if someone tries to maintain PLC strength by increase its fineness and what kind of influence on the concrete quality when it is made from such an excessive fine PLC. In 2015 a research program at The National University of Civil Engineering was set to investigate the influence of cement fineness and limestone content on the strength development and dry shrinkage of Portland Limestone Cement. The purpose of this research work is to set a limit for fineness and corresponding limestone content for PLC in order for ensuring concrete durability.

2. Test materials and methodology

2.1 Test materials

- Clinker: Portland cement clinker produced by But Son Cement Company complying with Vietnamese standard TCVN 7024 : 15 was used. The main characteristics of this clinker are shown in Tables 1 and 2.
- Gypsum: Gypsum stone was imported from Laos. The chemical composition of the gypsum is shown in Table 3.
- Limestone: Limestone was taken from Kien Khe quarry. The chemical composition of the limestone is shown in Table 4.

2.2 Methodology

For making cement a laboratory ball mill with a capacity of 10 kg per batch was used. For control Portland cement total weight of clinker is 96%, remaining 4 % went to gypsum. For making PLC the content of gypsum is 4 % of calculated clinker content. The maximum limestone content in PLC was set based on BS EN 197-1:2011, [1] ranges from 8 to 20 percent. The fineness of control PC, PLC and corresponding limestone content were set according to Central Composite Design (CCM) method which is known as a method of statistic design of experiments, [3] as seen in the experimental plan in Table 5. Making cement with fixed Blaine fineness is proved to be a laborious and requires experience. By the use of trail and errors method with frequent checking cement fineness during grinding the author could successfully control cement fineness with accuracy range of about 50 cm2/gr. For the test on cement standard consistency, setting times, ement strength, Vietnam standards TCVN 6016 : 1996 and TCVN 6017 : 2011 were used. For the test on cement mortar drying shrinkage, ASTM C596-07 [4] was used.

3. Results and discussion

3.1 Results of test on control cement

Tests on control cement strength were carried out at 3 days, 7 days, and 28 days. The results of these tests are shown in Table 6.

Tests on control cement strength show that from the same type of cement clinker by changing cement fineness one can produce various cements with various strength classes. One thing must be noted that strength of cement may not only depend on its fineness but also on the cement particle size distribution and the last one may also be affected by method of grinding. In this research the laboratory mill was use so there may be some deviation in cement particle size distribution when compared to that of cement ground in industrial mills.

Table 1 Oxide content of clinker

Oxide content, %									
SiO_2	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**SO_3**	**CaO_{free}**	**K_2O**	**Na_2O**	**LOI**
21.19	5.36	3.29	65.76	1.88	0.26	1.43	0.55	0.20	0.33

Table 2 Chemical composition of clinker

Chemical content, %			
C_3S	**C_2S**	**C_3A**	**C_4AF**
65.13	11.59	8.64	10.00

Table 3 Chemical composition of gypsum

Chemical content, %			
CaO	**SO_3**	**H_2O_{bound}**	**$CaSO_42H_2O$**
33.5	42.23	18.94	90.49

Table 4 Chemical composition of limestone

Oxide content, %									
SiO_2	**Al_2O_3**	**Fe_2O_3**	**$CaCO_3$**	**$MgCO_3$**	**SO_3**	**K_2O**	**Na_2O**	**P_2O_5**	**Total**
-	0.02	-	99.22	0.61	0.04	-	0.10	0.02	100

Table 5 Experimental plan according to statistic design of experiments, [3]

Run	Designated PLC	Coded factors		Real factors	
		X_1	X_2	Limestone content, %	PLC fineness, cm^2/gr
1	PLC-01	-1	-1	8.05	3150
2	PLC-02	+1	-1	17.95	3150
3	PLC-03	-1	+1	8.05	4850
4	PLC-04	+1	+1	17.95	4850
5	PLC-05	-1.414	0	6.00	4000
6	PLC-06	+1.414	0	20.00	4000
7	PLC-07	0	-1.414	13.00	2800
8	PLC-08	0	+1.414	13.00	5200
9	PLC-09	0	0	13.00	4000
10	PLC-10	0	0	13.00	4000
11	PLC-11	0	0	13.00	4000
12	PLC-12	0	0	13.00	4000
13	PLC-13	0	0	13.00	4000

The curves shown in Fig.1 clearly show that cements with higher fineness have higher rate of strength development especially at the early ages, and for cements of high fineness strength development seems not to obey logarithmic law of time. In practice the excessive heat of hydration released at early age of high strength class cement may cause thermal cracking to concrete made from this cement. Moreover, as it was proved the hydration of C3S and C3A minerals are activated in presence of CaCO3. Therefore the threat of thermal cracks in concrete may become more burdened when PLC with high fineness is used. This requires future studies.

Tests on control cement drying shrinkage were carried out at 28 days, 60 days, and 90 days. The results of these tests are shown in Table 7.

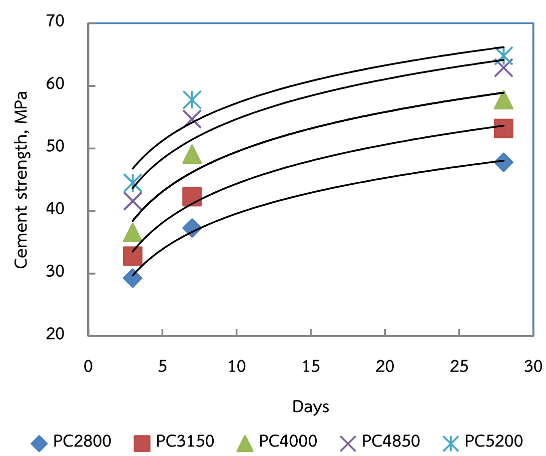


Figure 1 Strength development of laboratory prepared control cement with varied fineness

Table 6 Strength of laboratory prepared control cement with varying fineness

No.	Designated PC	Cement fineness, cm^2/gr	Cement strength, MPa.		
			3 days	7 days	28 days
1	PC2800	2800	29.3	37.3	47.3
2	PC3150	3150	32.8	42.3	53.3
3	PC4000	4000	36.6	49.1	57.8
4	PC4850	4850	41.6	54.7	62.9
5	PC5200	5200	44.5	57.8	64.8

Table 7 Drying shrinkage of laboratory prepared control cement with varied fineness

No.	Designated PC	Cement fineness, cm^2/gr	PC Drying shrinkage $x10^{-6}$		
			At 28 days	At 60 days	At 90 days
1	PC2800	2800	1092.4	1216.1	1243.9
2	PC3150	3150	1146.2	1284.7	1323.3
3	PC4000	4000	1187.5	1315.0	1342.0
4	PC4850	4850	1219.9	1337.5	1373.3
5	PC5200	5200	1252.6	1382.2	1412.0

Tests on control cement drying shrinkage show that cement fineness greatly affect cement mortar drying shrinkage. When cement fineness increases from 2800 cm^2/gr to 5200 cm^2/gr the cement mortar drying shrinkage at 60 days increases more than 13 percent. Data also reveal that cement mortar drying shrinkage development rate before 60 day is greater but after 60 days cement mortar drying shrinkage development rate slows down as shown in Fig. 2.

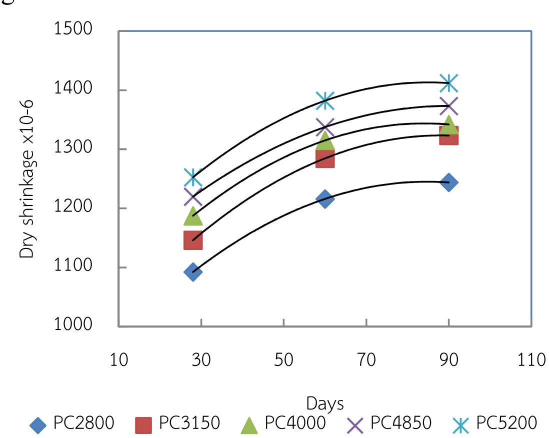


Figure 2 Drying shrinkage of laboratory prepared control cement with varied fineness

3.2 Results of test on PLC

Tests on strength of laboratory prepared PLC with fineness and limestone content varied according to Central Composite Design method were carried out at 3 days, 7 days, and 28 days. The results of these tests are shown in Table 8.

By employing Design Expert software, a regression function represented in coded factor for 28-day cement strength was derived as shown below:

$$f_{\text{cement}} = 46.61 - 2.41X_1 + 3.92X_2$$

Where: f cement is 28-day cement strength, X1 is the coded factor representing limestone content, and X2 is the coded factor representing cement fineness.

Table 8 Strength of laboratory prepared PLC with varied fineness and limestone content

Run	Designated PLC	Coded factors		Real factors		Cement strength, MPa		
		X_1	X_2	Limestone content, %	PLC fineness, cm^2/gr	3 days	7 days	28 days
1	PLC-01	-1	-1	8.05	3150	30.2	41.0	46.8
2	PLC-02	+1	-1	17.95	3150	27.2	36.6	43.2
3	PLC-03	-1	+1	8.05	4850	35.0	45.1	51.7
4	PLC-04	+1	+1	17.95	4850	30.5	39.9	48.9
5	PLC-05	-1.414	0	6.00	4000	29.6	40.1	49.4
6	PLC-06	+1.414	0	20.00	4000	24.9	35.1	40.3
7	PLC-07	0	-1.414	13.00	2800	21.5	30.8	39.2
8	PLC-08	0	+1.414	13.00	5200	35.6	45.4	53.9
9	PLC-09	0	0	13.00	4000	29.3	39.0	47.8
10	PLC-10	0	0	13.00	4000	27.8	39.0	45.6
11	PLC-11	0	0	13.00	4000	28.4	38.	46.7
12	PLC-12	0	0	13.00	4000	28.4	39.5	46.3
13	PLC-13	0	0	13.00	4000	28.9	39.9	46.1

The 3D and contour plots for this regression function are shown in Fig.3. The results show that both cement fineness and limestone content strongly affect cement strength. The finer cement, the higher its strength and the more limestone content, the lower cement strength. The addition of limestone strongly reduces 28-day strength of cement. For instance, in the case of cement fineness is 4000 cm2/gr, and the limestone content is 13 percent, the reduction in cement strength at 28 days is about 20 percent when compared to cement-only mixture. Nevertheless, limestone doesn't affect very much the 3-day and 7-day strength. The mechanism of these phenomena was also studied by the author, [2,5,6,7].

Tests on drying shrinkage of laboratory prepared PLC with fineness and limestone content varied according to Central Composite Design method were carried out at 28 days, 60 days, and 90 days. The results of these tests are shown in Table 9.

A regression function represented in coded factors for 60-day cement drying shrinkage was also derived as shown below:

$$Sh_{cement} = 999.58 + 17.15X_1 + 95.82X_2 - 15.87X_1X_2 - 17.76X_1^2 - 60.29X_2^2$$

Where: Sh cement is 60-day cement drying shrinkage, X1 is the coded factor representing limestone content, and X2 is the coded factor representing cement fineness.

The 3D and contour plots for this regression function are shown in Fig.4. The results show that both cement fineness and limestone content affect cement drying shrinkage. The finer cement, the higher cement drying shrinkage, but the addition of limestone in the range from 8 to 18 percent shows no significant effect on cement drying shrinkage.

From the above discussions, for the benefits of particular strength class of PLC with as high as possible content of limestone, lower cement mortar drying shrinkage, and low heat of cement hydration due to low cement fineness, it is advisable to select mixture PLC07 for PLC 30 (Portland Limestone Cement with 28-day strength not less than 30 MPa) and mixture PLC01 for PLC 40 (Portland Limestone Cement with 28-day strength not less than 40 MPa) according to Vietnamese Standard on PLC.

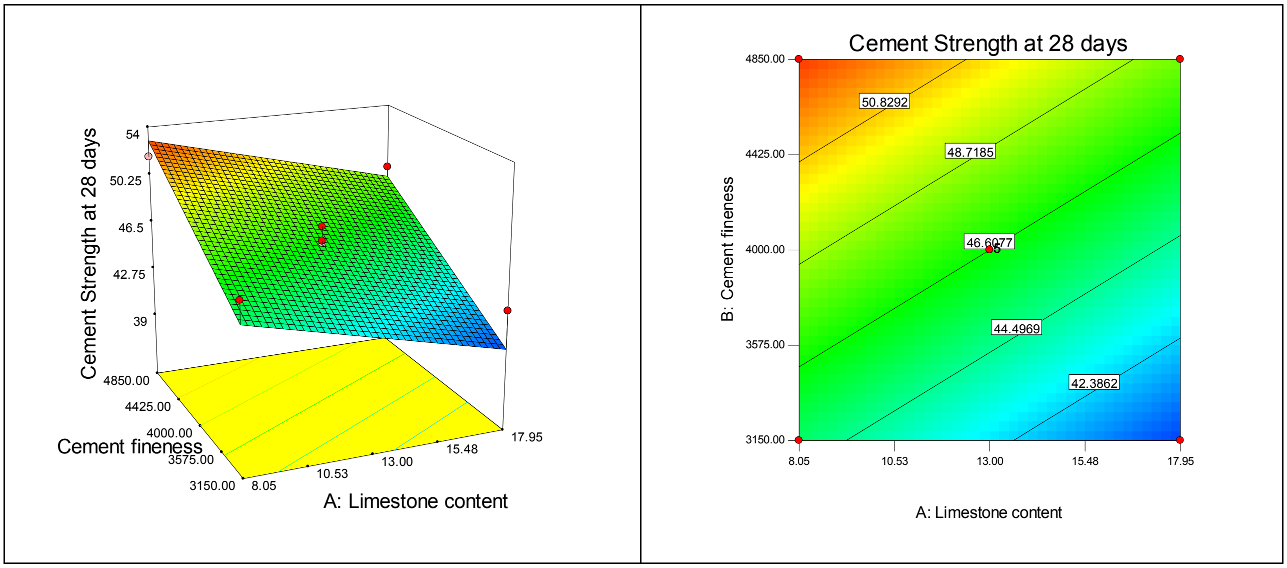
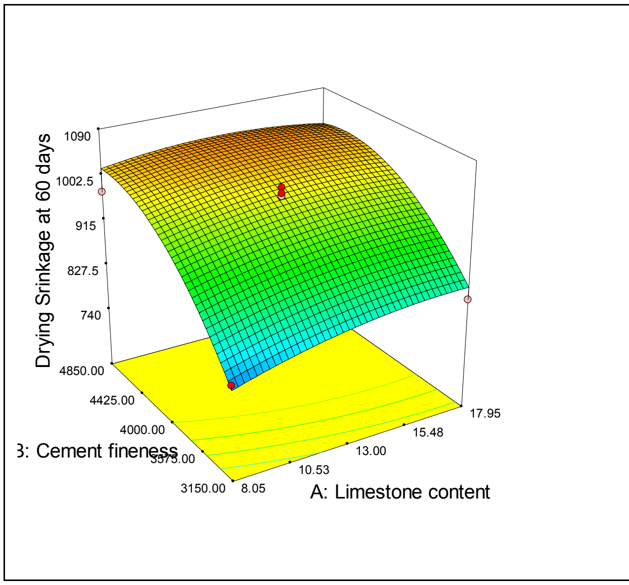


Figure 3 3-D and counter plots of regression function representing the 28-day strength of cements with varied fineness and limestone content

Table 9 Drying shrinkage of laboratory prepared PLC with varied fineness and limestone content

Run	Designated PLC	Coded factors		Real factors		PLC Drying shrinkage x10^{-6}		
		X_1	X_2	Limestone content, %	PLC fineness, cm^2/gr	After 28 days	After 60 days	After 90 days
1	PLC-01	-1	-1	8.05	3150	669.0	801.4	878.1
2	PLC-02	+1	-1	17.95	3150	730.5	834.9	911.4
3	PLC-03	-1	+1	8.05	4850	798.1	971.6	940.3
4	PLC-04	+1	+1	17.95	4850	809.7	941.2	1017.3
5	PLC-05	-1.414	0	6.00	4000	826.0	950.9	964.8
6	PLC-06	+1.414	0	20.00	4000	906.2	1045.7	1108.4
7	PLC-07	0	-1.414	13.00	2800	726.1	740.0	774.6
8	PLC-08	0	+1.414	13.00	5200	920.4	1086.5	1121.1
9	PLC-09	0	0	13.00	4000	852.6	999.3	1034.2
10	PLC-10	0	0	13.00	4000	840.3	1000.0	1027.8
11	PLC-11	0	0	13.00	4000	804.2	993.0	1028.0
12	PLC-12	0	0	13.00	4000	815.9	1011.2	1032.1
13	PLC-13	0	0	13.00	4000	841.5	994.4	1050.1



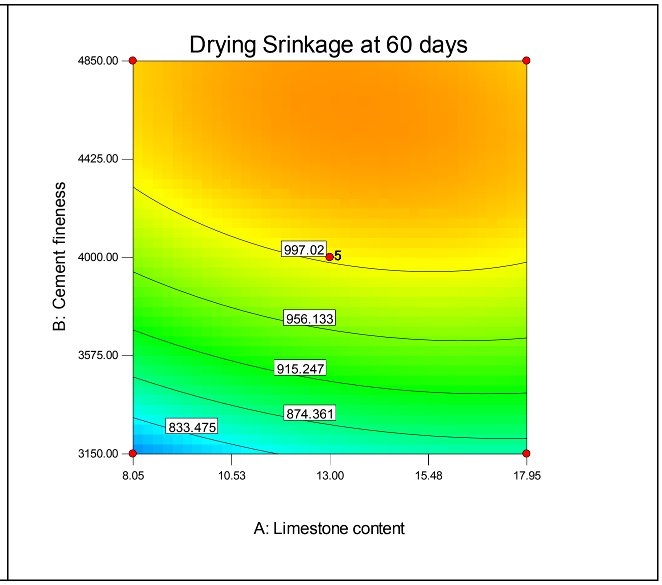


Figure 4 3-D and counter plots of regression function representing the 60-day drying shrinkage of cements with varied fineness and limestone content

4. Concluding remarks

Based on the tested materials and test mothods used in the research, the following conclussions can be drawn:

1) Research shows that the cement fineness greatly affects cement strength at all ages for the control cements as well as for laboratory manufactured Portland Limestone Cements, and the finer cement, the higher strength.
2) Research shows that the content of limestone in Portland Limestone Cement as mineral additive significantly reduces cement strenght especially at 28-day cement strength compared to that of control cement, and the more limestone content, the lower cement strength.
3) Research shows that the fineness of cements both control and the laboratory manufactured PLC greatly increases cement drying shrinkage, this may lead to an excesive drying shrinkage of concrete made from these cements potentially causing concrete cracks.
4) Research shows that the increase in limestone content in PLC doesn't affect much PLC drying shrinkage.

5. References

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