論文 Effect of Limestone Powder on Rheological Behavior of Highly-flowable Mortar

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ABSTRACT: The effect of replacement dosage and specific surface area of limestone powder and their interactions with various mixture parameters on the rheological behavior of highly-flowable mortar was investigated using a statistical experimental design. The derived mathematical models indicate that the slump flow, V-funnel flow time, yield stress, and plastic viscosity are highly influenced by the replacement dosage and specific surface area of limestone, the water-to-cementitious materials ratio, the high-range water-reducer percentage, the sand volume, and various interactions of these parameters. Comparisons between predicted and measured properties confirm the good accuracy of the established models.

KEYWORDS: Highly-flowable mortar, limestone powder, rheology, specific surface area, statistical experimental design

1. INTRODUCTION

The use of mineral admixtures such as pulverized limestone powder (LP), fly ash (FA), and blast-furnace slag (BFS) as partial replacement for cement in highly-flowable concrete (HFC) is actually a common practice. HFC containing such supplementary mineral components can potentially provides higher rheological and mechanical properties and good durability.

The main objective of this study is to evaluate the effect of the replacement dosage and specific surface area (SSA) of LP and their interactions with various mortar parameters, such as the water-to-cementitious materials ratio (W/CM), high-range water-reducer (HRWR) dosage, and sand volume on the rheological properties of highly-flowable mortar (HFM) using statistical design approach [1, 2]. The established models can identify parameters and the two-way interactions that have significant effect on slump flow, V-funnel flow time, yield stress, and plastic viscosity of HFM. On the other hand, the use of these models can facilitate the test protocol required to optimize suitable mixtures to proportion HFC [1, 2].

The evaluation of the effect of such supplementary materials on the rheological behavior of HFM is important in several ways. HFC is a suspension of inert coarse aggregates in mortar. The evaluation of the rheological behavior of mortar may, therefore, provide relevant information on that of concrete. Furthermore, such evaluation is often achieved by carrying out consistency assessments using simple empirical test methods and, therefore, the effect of these supplementary admixtures on the yield stress value and plastic viscosity of HFM is not well established.

2. EXPERIMENTAL PROGRAM

The experimental program consisted of 30 mixtures carried out to design a composite fractional factorial plan and evaluate the primary effect and the two-way interaction of the various parameters on slump flow, V-funnel flow time, yield stress, and plastic viscosity of HFM. The targeted mortar interest for this investigation is a HFM containing LP that can be used to proportion HFC. Such mixture must achieve required fresh properties, such as relative funnel speed and flow area values of 1 ± 0.1 and 5 ± 0.5, respectively [3].

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2.1 STATISTICAL DESIGN METHOD

Parameters that could influence the above relevant properties of HFM were carefully selected to establish statistical models for slump flow, V-funnel flow time, yield stress, and plastic viscosity. The parameters included the W/CM ratio, HRWR dosage, sand volume, replacement dosage and SSA of LP. Initial level and expanded values considered in the factorial design are summarized in **Table 1**.

Table 1. Coded and absolute values for the investigated parameters

Coded	-2	-1	0	1	2
Factors	Absolute values				
W/CM	0.695	0.83	0.965	1.1	1.23
HRWR	-	2.2	3.0	3.8	-
LP	0	15	30	45	60
Sand	35	40	45	50	55
SSA	-	5000	7500	10000	-

Coded W/CM = (abs. W/CM– 0.965)/0.135
Coded HRWR = (abs. HRWR – 3.0)/0.8
Coded LP = (abs. LP – 30)/15
Coded Sand = (abs. Sand – 45)/5
Coded SSA = (abs. SSA – 7500)/2500

It's important to mention that the replacement dosage of LP, W/CM ratio, sand volume, and HRWR dosage are all on volume basis.

2.2 MATERIAL PROPERTIES

The mortar used to establish statistical models was systematically proportioned using an ordinary portland cement with a Blaine fineness of 3460 cm^2/g, a well-graded sand with a fineness modulus of 2.7, a specific gravity corresponding to the saturated and surface dry state of 2.6, and an absorption value of 1.21%. A polycarboxylic acid-based polymer with a specific gravity of 1.09 and a solid content of 26.5% was used for the HRWR. All the investigated mixtures incorporated 1% air-deforming agent, by HRWR volume. This dosage was fixed after some trial batches and found to be sufficient to ensure minimum entrapped air in mixtures (less than 0.6%). A pulverized LP with three different SSA of 5000, 7500, and 10000 cm^2/g was used. It's specific gravity ranges between 2.71 and 2.75. The initial replacement dosage of LP was varied between 15 and 45%, by volume of cementitious material (CM).

2.3 TEST PROCEDURES

All mixtures were prepared in 2.5 L batches using a Hobart mixer type similar to that specified by JIS R 5201 standard. All the materials were prepared and kept at a constant temperature of 20°C 1 day before testing. The used sand was set at a constant humidity corresponding to the saturated and dry surface state. The mixing sequence consisted of homogenizing the sand and CM for 15 seconds, then the HRWR diluted with the mixing water were added over 30 s, while the mixer was turned on. The mixture was then mixed for 225 seconds. After 1 minute of rest, the mixing was resumed for an additional 60 seconds. Following the end of mixing, all mixtures had constant temperatures of 24±2°C.

For each mixture, the slump flow, V-funnel flow time, yield stress, and plastic viscosity were determined after 6 and 90 min of age. The slump flow value was determined using a flow cone similar to that specified by JIS R 5201 standard. The V-funnel flow test for mortar is similar to that used by Okamura et al. [3]. Rheological measurements were performed using a coaxial cylinder viscometer with 10 mm gap size. The viscometer was calibrated using JS14000 standard solution complying with JIS Z 8809 specifications before carrying measurements on mortar. The mortar sample was sheared for 30 s at 100 rpm, corresponding to shear rate of 41.4 s^{-1}, to ensure an equilibrium state (breakdown of the structure). Rheological profile was then obtained by increasing the rotational velocities from 5 to 100 rpm. Rheological parameters were established using a non-linear model similar to that used by De Kee et al. [4, 5].

The mixture corresponding to the central point was prepared several times to investigate the reproducibility of slump flow, V-funnel flow time, yield stress, and plastic viscosity tests. Measurements were assessed in the same sequence after 6 and 90 min of age by three different operators in order to take into consideration the inherent error due to the operator. **Table 2** summarizes the statistical characteristics identifying the reproducibility of the four measured properties (responses).

Table 2. Reproducibility results of various measured responses

	Slump flow (cm)		V-Funnel (s)		Yield stress (Pa)		P.viscosity (Pa.s)	
Age of testing	6 min	90 min	6 min	90 min	6 min	90 min	6 min	90 min
Repeated tests	9	9	9	9	8	9	8	9
Mean value	31.7	29.2	3.95	6.48	8.2	15.6	3.5	5.4
S. deviation	0.5	1.1	0.14	0.48	3.0	1.0	0.45	0.51
± 95% C. limit	0.3	0.6	0.1	0.3	1.7	0.6	0.26	0.29

3. TEST RESULTS AND DISCUSSION

The derived statistical models for the slump flow, V-funnel flow time, yield stress, and plastic viscosity are summarized in **Table 3**.

Table 3. Parameter estimates of the established models for various responses

	Slump flow (cm)		V-F flow time (s)		Yield stress (Pa)		P.Viscosity (Pa.s)	
Parameters	6 min	90 min	6 min	90 min	6 min	90 min	6 min	90 min
Mean value	31.3	30.4	3.9	6.3	10.6	15.4	4.0	5.1
W/CM	5.9	8.0	-4.1	-6.9	-5.8	-12.0	-3.9	-5.0
HRWR	3.7	7.1	-0.5	-0.7	-3.4	-9.6	NS	NS
% LP	6.0	6.8	-2.5	-3.9	-4.2	-3.3	-2.2	-1.8
SAND	-3.9	-5.7	0.7	2.9	2.7	NS	1.2	1.8
SSA	-1.1	NS	NS	-1.1	NS	NS	1.6	0.9
W/CM. W/CM	-2.3	-1.8	1.5	2.3	2.4	20.4	1.7	6.8
%LP. W/CM	-2.0	-2.2	1.8	2.9	3.6	9.8	1.5	NS
%LP.HRWR	1.1	0.5	1.8	0.5	NS	8.8	-1.3	NS
%LP.%LP	-1.4	-1.7	0.9	1.1	-2.4	-2.4	NS	NS
SAND. W/CM	-1.1	NS	0.4	-1.0	NS	-6.1	NS	-3.5
SAND.HRWR	-0.8	NS	NS	NS	-1.8	-7.1	0.7	-3.6
SAND.%LP	NS	NS	1.2	-0.6	-1.6	NS	0.7	NS
SAND.SAND	-1.5	-1.6	NS	0.6	2.2	-3.8	NS	NS
SSA. W/CM	NS	-2.7	NS	1.2	NS	-8.3	-1.6	NS
SSA.HRWR	NS	-1.6	-1.5	NS	NS	-10.3	-1.0	NS
SSA.%LP	-0.9	-2.4	NS	NS	NS	-3.7	-0.8	NS
SSA.SAND	-1.0	NS	NS	NS	NS	NS	1.4	NS
HRWR.W/CM	NS	NS	0.3	NS	2.0	15.4	NS	2.4
HRWR.HRWR	NS	-1.8	0.9	1.4	3.5	NS	NS	NS

NS: Not significant

For example, the slump flow model can be written as given in **Eq. 1**

SLUMP FLOW (cm) = **31.3** + **5.9** W/CM + **3.7** HRWR + **6.0** %LP – **3.9** SAND – **1.1** SSA – **2.3** W/CM.W/CM – **2.0** LP.W/CM + **1.1** %LP.HRWR – **1.4** %LP.%LP – **1.1** SAND. W/CM - **0.8** SAND.HRWR – **1.5** SAND.SAND – **0.9** SSA.%LP – **1.0** SSA.SAND **(1)**

The estimate for each factor refers to the effect of that parameter on the considered response. These estimates were found by least squares and have a significant effect with 95% confidence limit. These models were established using coded values for the investigated factors (**Table 1**). A negative estimate value means that the measured response decrease when increasing that parameter level. For any given measured property (response), the presence of interaction with coupled terms (HRWR.HRWR or W/CM.W/CM) indicates that the influence of the parameter on that response is quadratic.

3.1 ACCURACY OF THE ESTABLISHED MODELS

The accuracy of each of the proposed models was determined by comparing predicted-to-measured values obtained with mixtures prepared at the center of the experimental domain. The predicted-to-measured ratio for slump flow, V-funnel flow time, yield stress, and plastic viscosity are summarized in **Table 4**.

Table 4. Predicted and measured ratios for mixtures for established models

	Predicted/Measured							
	Slump flow		V-Flow time		Yield stress		P. viscosity	
Age (min)	6	90	6	90	6	90	6	90
5 points SSA = 7500)	0.97	1.04	0.98	1.06	1.02	0.90	0.86	0.85
1 point (SSA = 5000)	1.04	1.01	0.87	-	0.89	0.85	0.91	0.84
1 point (SSA =10000)	0.98	-	0.93	-	0.86	0.92	0.83	0.84

The ratio between predicted and various measured properties ranges between 0.83 and 1.04, thus indicating good accuracy for the established models to predict slump flow, V-funnel flow time, yield value, and plastic viscosity for HFM. The proposed statistical models can therefore be used to evaluate the effect of a group of variables on the rheological properties of HFM. For example, the effect of replacement dosage of limestone powder vs. that of increasing HRWR dosage can be evaluated for mixtures prepared with a constant W/CM. Also, for a given fluidity level, we can use these models to establish the trade-off between increasing the replacement dosage of limestone and decreasing the W/CM to achieve suitable mechanical properties. This can reduce the effort often required in carrying out trial batches to optimize suitable mixture proportion.

3.2 TRADE-OFF BETWEEN LIMESTONE POWDER CONTENT, W/CM AND HRWR DOSAGE

The impact of reducing the W/CM on the required HRWR dosage to maintain a given fluidity for mixtures containing 40% sand volume and various content of limestone is presented in **Fig. 1**.

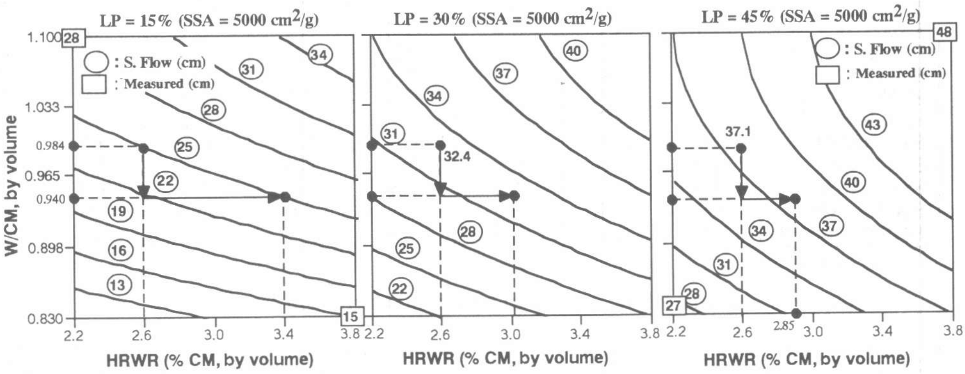

Fig. 1. Iso-predicted slump flow values for mixtures made with 40% sand

As can be seen from **Fig. 1**, for mixtures made with 15% limestone, 0.984 W/CM, and 2.6% HRWR, a slump value of 25 cm can be expected. Increasing the replacement dosage of limestone to 30 and 45% resulted in higher slump flow value of 32.4 and 37.1 cm, respectively. On the other hand, it can be seen (**Fig. 1**) that many combinations of W/CM and HRWR can be used to ensure a given slump flow value. It is therefore necessary to determine an optimal balance between the W/CM and HRWR dosages as well as the content of LP to develop high-performance mixture with targeted properties.

The W/CM can be lowered to achieve high resistance to segregation or to enhance the mechanical properties and durability. However, in such case an increase in the HRWR dosage is necessary to maintain the initial fluidity level. The required amount of HRWR depends on the initial slump flow and the mixture parameters. For example, for mixtures containing 15% LP, the decrease of W/CM from 0.984 to 0.94 necessitates an increase of the amount of HRWR from 2.6 to 3.4% (an increase of 0.8%) to maintain a SF value of 25 cm. However, for mixtures containing higher dosages of LP corresponding to 30 and 45%, the required HRWR content ranges, respectively, from 2.6 to 3.0 (an increase of 0.40%) and 2.6 to 2.85% (an increase of 0.25%) to maintain greater slump flow values (32.4 and 37.1 cm).

4. EFFECT OF LIMESTONE POWDER CONTENT ON THE RHEOLOGICAL PARAMETERS OF MORTAR

The variation of the experimental yield stress and plastic viscosity values for mixtures prepared with 0.83 W/CM, 40% sand and different dosages of limestone powder is shown in **Fig. 2.**

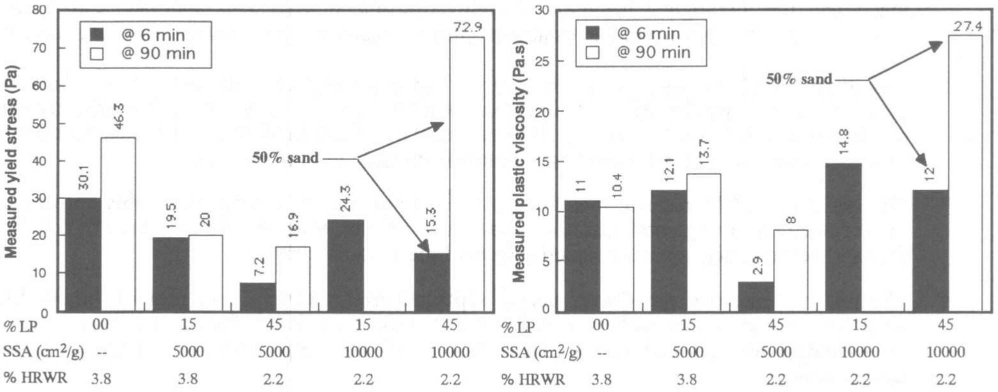

Fig. 2. Yield stress and plastic viscosity values for mixture made with 0.83 W/CM

Regardless of the HRWR dosage and the SSA of LP, replacing a part of cement by LP resulted in a decrease in the yield stress value and an improvement in plastic viscosity, thus secures better spreading, deformability, and resistance to segregation for the mixture. For example, after 6 min of age, mixture made with neat cement and contained 3.8% HRWR showed a yield stress and plastic viscosity of 30.1 Pa and 11 Pa.s, respectively. When replacing 15% of cement by LP with 5000 cm^2/g SSA, the yield stress decreased to 19.5 Pa and the plastic viscosity increased to 12.1 Pa.s. Increasing the LP content from 15 to 45% resulted in a further reduction in the yield stress (7.2 Pa), even for a lower HRWR dosage of 2.2%. This can be due to a better grain size distribution of the powder that can result in a liberation of some water otherwise entrapped between cement particles. This physical filler effect results therefore in greater contribution of the mixing water, thus enhance rheological properties of the mixture. On the other hand, replacing a part of cement by LP which is less reactive, may lead to a decrease in the hydration rate and therefore a reduction in the inter-particles friction that takes place between into the hydration products network.

Compared with mixture prepared with neat cement, test results showed that the incorporation of LP with 10000 cm^2/g SSA combined with a lower HRWR amount of 2.2% resulted generally in a decrease in the yield stress and an improvement in the plastic viscosity after 6 min of age.

However, a substantial increase in the yield stress after 90 min of age is observed. After 6 min of age the use of 15% limestone powder with higher SSA of 10000 cm^2/g and 2.2% HRWR resulted in a decrease in the yield stress from 30.1 to 24.3 Pa and an increase in the plastic viscosity to 14.8 Pa.s. Increasing the replacement dosage from 15 to 45%, resulted in further decrease in the yield stress to 15.3 Pa, even for mixture made with higher volume of sand corresponding to 50%.

However, in such case a substantial increase in the yield stress and the plastic viscosity was observed after 90 min of age. Compared with 5000 cm^2/g SSA, the use of higher SSA of 10000 cm^2/g resulted in relatively higher yield stress and plastic viscosity, regardless the age of testing. Therefore, in such case (higher SSA) it's necessary to increase the HRWR dosage or the W/CM ratio to achieve suitable rheological properties and prevent the fluidity loss observed after 90 min of hydration.

5. CONCLUSIONS

Based on the results presented in this paper, the following conclusions can be warranted:

1. Statistical design approach can provide useful information concerning the various parameters that affect the rheological behavior of highly-flowable mortar. This approach can reduce the number of trial batches needed to establish optimum balance among various mortar parameters.

2. Statistical models are established for a given set and range of materials and are shown to be accurate to evaluate the effect of limestone powder and its interaction with mortar parameters on rheological behavior of highly-flowable mortar. Such models can be used to establish a trade-off between mortar parameters to optimize suitable mixture proportion.

3. The derived models indicate that the measured properties of mortar are highly influenced by the replacement dosage and specific surface area of limestone, the W/CM, the HRWR dosage, the sand volume, and several coupled effects of these parameters.

4. The use of limestone powder as partial replacement for cement can provide higher fluidity level for a given W/CM and HRWR dosage. On the other hand, for a fixed fluidity level, the use of limestone results in a reduction in the W/CM, thus improving the mechanical properties and durability.

5. The use of limestone powder results in a reduction in the yield stress and an improvement in the plastic viscosity of highly-flowable mortar.

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