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## Communication

# The effect of ultra-fine admixture on the rheological property of cement paste

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#### **Abstract**

The effect of ultra-fine admixtures (UA), such as blast furnace slag (BFS), silica fume (SF), fly ash (FA), limestone (LS) and anhydrous gypsum (AG), upon the rheological property of cement paste is researched. The results show that the yield stress value of cement paste is generally decreased with the increase of the UA quantity added, but the viscosity varies greatly with different types and quantity of UA. Ultra-fine LS, SF, FA and slag can decrease the viscosity of cement paste, whereas AG can increase it when the UA quantity is smaller than or equal to 15%. The viscosity and the yield stress of cement paste are decreased obviously with the increase of the UA quantity added, in which the effect of ultra-fine slag is the most significant when the UA quantity is more than 15%. The viscosity and the yield stress can be decreased by a respective addition of 10% ultra-fine LS, SF, FA or a single addition of 35% ultra-fine slag. The acting effect is ranged as: slag>LS>SF >FA. The viscosity and the yield stress of cement paste can be raised significantly by a single addition of 10% ultra-fine AG. © 2000 Elsevier Science Ltd. All rights reserved.

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#### 1. Introduction

The demand for high performance concrete (HPC) is rising increasingly along with the development of modern construction particularly for high-rise buildings, large-span, underground and marine structure. At present, the general understanding of HPC in the world is that the concrete could possess super strength, high mobility, and high durability. The current technical practice of processing HPC internationally is ordinary as Portland cement with high performance addition agent with ultra-fine admixtures (UA) [1]. The rheological property of HPC must satisfy the needs of high mobility and non-segregation simultaneously. Comparing with that of ordinary concrete, the rheological property of HPC is characterized by a smaller value of the yield stress and a higher plastic viscosity [2]. As the sixth component of HPC, UA is a kind of micro-powder with a

### 2. Experimental

#### 2.1. Raw materials

The raw materials used in the present paper are listed in Table 1.

An ordinary Portland cement (OPC) grade 525 from Japan Cement Association is used as basic cementitious material. Blast furnace slag (BFS), SF, FA, LS, AG from Japan are used as UA after grinding to desired fineness. The main mineral component of AG is anhydrite. A high efficiency water reducer (named super-200) also from Japan is used as high performance addition agent, which is a liquid solution containing 18% solid.

large specific surface area, which has a considerable effect upon the rheological property of the fresh concrete. In order to control and improve the rheological property of the fresh HPC, the effect of UA, such as slag, silica fume (SF), fly ash (FA), limestone (LS) and anhydrous gypsum (AG), on the rheological property of cement paste is studied in the paper.

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Table 1 Raw materials

Raw	Density	Specific surface area (m <sup>2</sup> /kg)	Average grain size	Chemic	Chemical composition (%)								
material	$(g/cm^3)$		(μm)	Loss	SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	$SO_3$			
Cement	3.17	327	14.0	0.60	21.30	5.30	2.60	64.40	2.20	1.90			
Slag	2.89	846	_	0.40	33.10	15.40	0.50	41.00	7.50	_			
Silica fume (SF)	2.28	2030(BET) <sup>a</sup>	0.5	_	93.00	0.54	0.25	0.20	_	_			
Anhydrous gypsum (AG)	2.96	2621(BET) <sup>a</sup>	_	_	_	_	_	_	_	_			
Fly ash (FA)			2.9	1.76	67.0	20.90	2.04	1.72	0.81	_			
Limestone (LS)	2.69	763	4.5	43.2	0.7	0.20	0.10	55.20	0.40	_			

<sup>&</sup>lt;sup>a</sup> Measured by BET method.

### 2.2. Preparation of samples

In order to investigate the effect of different kinds and quantity of UA on the rheological property of cement paste, a series of samples are designed and their compositions are listed in Table 2. The water—cement ratio is 0.25 and the amount of super-200 added is 5%.

#### 2.3. Test apparatus

A rotary duplex cylinder viscometer (from K.K. CODLX) consisting of sample apparatus, stress control device, and data processing computer is used to test the yield stress, apparent viscosity, and the rheological curve of fresh cement paste. The sample apparatus contains an internal rotator and an external sleeve. The diameter of the internal rotator is 18.0 mm and the diameter of the sleeve is 19.5 mm in the present research and different dimensional combinations of the rotator and sleeve can be chosen according to the test requirements. The stress control device consists of a drive balance system and a temperature control system, which is used to control the rotator drive and the temperature of the samples. The attached computer with special software controls the stress domain for the determination and the measurement process, which is also used to store the data, calculate the rheological parameter, and control the rheological curve. The measurement process is LOG STEP and the range of shear stress is 0-50 Pa in this research.

#### 3. Results and discussion

### 3.1. The quasi-conjugation of the rheological property

The data determined by the computer attached to the rotary duplex cylinder viscometer are listed in Table 3. The results show that the rheological properties of cement pastes with all types of UA basically belong to Casson fluid. The model equation of Casson fluid is as follows in Eq. (1):

$$\tau^{0.5} = A + B\gamma^{0.5} \tag{1}$$

where A, B is the apparatus constant,  $\tau$  is the yield stress (Pa),  $\gamma$  is the apparent viscosity (Pa s).

The rheological curves of cement pastes are quasi-conjugated with the Casson model equation, the correlation coefficient of the quasi-conjugation equation is  $\geq 0.8$ , the standard mean square error is  $\leq 0.46$ .

# 3.2. The orthogonal analysis of the effect of the UA quantity on the rheological property of cement paste

According to the orthogonal experimental principle, the results of the relationship among the apparent viscosity, the yield stress, and the admixture composition of cement pastes (sample  $X_1$ – $X_9$ ) are summed up in Table 4 and Table 5. It shows that all the AEC of the yield stress of cement pastes with all types of UA trend to be significantly decreased with the increase of the level of the UA quantity added. Among them, the difference value for BFS is the largest. The AEC

Table 2
The composition of samples (wt.%)

I		I	,											
Sample	$X_1$	$X_2$	$X_3$	$X_4$	$X_5$	$X_6$	$X_7$	$X_8$	$X_9$	$X_{10}$	$X_{11}$	X <sub>12</sub>	X <sub>13</sub>	$X_{14}$
BFS	0	10	25	0	10	25	0	10	25	10	0	0	0	0
LS	0	5	10	5	10	0	10	0	5	0	10	0	0	0
AG	0	5	10	10	0	5	5	10	0	10	0	10	0	0
OPC	100	80	55	85	80	70	85	80	70	80	90	90	90	90
SF	0	0	0	0	0	0	0	0	0	0	0	0	0	10
EA	0	0	0	0	0	0	0	0	0	0	0	0	10	0

Table 3
The rheological property data of the samples

Sample	$X_1$	$X_2$	$X_3$	$X_4$	$X_5$	$X_6$	$X_7$	$X_8$	$X_9$	$X_{10}$	$X_{11}$	$X_{12}$	$X_{13}$	$X_{14}$
Apparent viscosity (Pa s)	4.45	25.62	0.55	3.54	0.92	0.51	20.86	1.18	1.27	1.14	2.52	3539.40	3.17	3.54
Yield stress (Pa)	8.31	5.20	0.01	1.89	0.04	0.03	0.85	1.33	0.68	0.004	1.60	15.04	1.79	2.26
A	2.88	2.28	0.11	1.37	0.19	0.16	-0.92	1.15	0.82	0.06	1.27	-3.88	1.34	1.50
В	2.13	5.63	0.74	1.88	0.96	0.17	4.57	1.08	1.13	1.06	1.58	59.49	1.78	1.88
Standard mean (Square error)	0.12	0.46	0.25	1.15	0.45	0.26	0.36	0.31	1.03	0.42	0.61	0.02	0.38	0.13
Correlation coefficient	0.99	0.85	0.99	0.80	0.97	0.99	0.82	0.98	0.87	0.98	0.91	0.88	0.94	0.99

of the yield stress is  $44.3 \times 10^{-2}$  when slag quantity added is at level 1 (0%) and that is decreased to  $26.4 \times 10^{-2}$  at level 2 (10%) and to  $2.9 \times 10^{-2}$  at level 3 (25%) respectively, which indicates that slag can significantly decrease the yield stress of cement paste. The effect of LS on the yield stress is smaller than that of slag, and the effect of AG is relatively small. Accordingly, we could believe that the increase of the UA quantity added may also decrease the viscosity of cement paste, but the variation of the AEC value of viscosity differs greatly with the type and quantity of UA. For example, when the UA quantity added is within a lower level, the AEC of viscosity for slag at level 1 and level 2 are  $37.6 \times 10^{-2}$  and  $36.1 \times 10^{-2}$ , respectively. The small difference between them indicates that the slag quantity added affects the viscosity of cement paste infinitesimally. The AEC of viscosity are  $8.1 \times 10^{-2}$  and  $39.6 \times 10^{-2}$  $10^{-2}$  when the quantity of ultra-fine LS added is at level 1 and at level 2, respectively, which shows the viscosity of cement paste can be increased in a lower extent of the LS quantity added. It is noted that when the quantity of ultrafine AG added is at level 1 and 2, respectively, the AEC are  $8.8 \times 10^{-2}$  and  $61.1 \times 10^{-2}$ . The difference between these two AEC value is up to  $52.3 \times 10^{-2}$ , which indicates that ultra-fine AG can dramatically increase the viscosity of cement paste in the lower level of its quantity added. On the other hand, the AEC values of viscosity for the above three types of UA are significantly decreased when the quantity of UA added is within a higher level. For example, the AEC of the viscosity are  $1.5 \times 10^{-2}$  for slag,  $6.8 \times 10^{-2}$ for AG and 29.0  $\times$  10<sup>-2</sup> for LS, respectively when the quantity added is at level 3, which demonstrates that all

these UA have significant effect on the decrease of the viscosity (and also the yield stress) of cement paste when the UA quantity added is at a higher level, in which the effect of slag is most obvious.

# 3.3. The effect of UA types on the rheological properties of cement pastes

 $X_{11}$ - $X_{14}$  are the samples, in which the same amount (10%) of LS, AG, SF and FA are added, respectively. The results in Table 3 show that the viscosity of  $X_{12}$  with ultra-fine AG is as high as 3539.40 Pa s, and its yield stress is up to 15.04 Pa, which indicate that AG has significant effect on the viscosity and the yield stress of cement paste. The viscosity and the yield stress of  $X_{11}$  added with ultra-fine LS are 2.52 Pa s and 1.60 Pa, respectively. Both of them are lower compared with those of samples  $X_1$  (neat OPC paste), especially the yield stress value is 6.71 Pa lower than that (8.31 Pa) of  $X_1$ . Compared with those of  $X_1$ , the viscosity and the yield value of  $X_{13}$  and  $X_{14}$ , which are the samples added with SF and FA are also significantly lower, but not as small as that of sample  $X_{11}$ . The viscosity values of  $X_{13}$ and  $X_{14}$  are quite approximate (3.17 and 3.54 Pa s), while the yield stress value of  $X_{14}$  is 2.26 Pa, rather larger than that of  $X_{13}$  (1.79 Pa).  $X_{10}$  is the sample admixed only with 35% slag. Its yield stress value is 0.004 Pa, the viscosity is 1.14 Pa s, both of them are noticeably less than those of neat OPC paste.

The above test results show that a respective addition of 10% ultra-fine LS, SF, and FA can decrease the viscosity and the yield stress of cement paste, the acting

Orthogonal analysis of the effect of UA quantity on the rheological properties of cement paste

	Level of U	A quantity added	(%)	Viscosity (Pa s)	Yield stress	Effect coefficient			
Sample	BFS	LS	AGS		(Pa)	Viscosity	Yield stress		
$\overline{X_1}$	1 (0)	1 (0)	1 (0)	4.54	8.31	$17.7 \times 10^{-2}$	$100.0 \times 10^{-2}$		
$X_2$	2 (10)	2 (5)	2 (5)	25.63	5.20	$100.0 \times 10^{-2}$	$62.6 \times 10^{-2}$		
$X_3$	3 (25)	3 (10)	3 (10)	0.55	0.01	$2.1 \times 10^{-2}$	$0.1 \times 10^{-2}$		
$X_4$	1	2	3	3.54	1.89	$13.8 \times 10^{-2}$	$22.7 \times 10^{-2}$		
$X_5$	2	3	1	0.92	0.04	$3.6 \times 10^{-2}$	$0.5 \times 10^{-2}$		
$X_6$	3	1	2	0.51	0.03	$2.0 \times 10^{-2}$	$0.4 \times 10^{-2}$		
$X_7$	1	3	2	20.86	0.85	$81.4 \times 10^{-2}$	$10.2 \times 10^{-2}$		
$X_8$	2	1	3	1.18	1.33	$4.6 \times 10^{-2}$	$16.0 \times 10^{-2}$		
$X_9$	3	2	1	0.68	1.27	$5.0 \times 10^{-2}$	$8.2 \times 10^{-2}$		

Table 5
The average of effect coefficient (AEC)

Factor	AEC o	of viscos	ity 10 <sup>-2</sup>	AEC of yield stress 10 <sup>-2</sup>					
level	BFS	LS	AGS	BFS	LS	AGS			
1	37.6	8.1	8.8	44.3	38.4	36.2			
2	36.1	39.6	61.1	26.4	31.2	24.4			
3	1.5	29.0	6.8	2.9	3.6	12.9			
Difference value	36.1	31.5	54.3	41.4	34.8	23.3			

effect is ranged as ultra-fine LS>SF>FA. A single addition of 10% ultra-fine AG can significantly increase the viscosity and the yield stress. A single addition of 35% ultra-fine slag can noticeably decrease the viscosity and the yield stress. This feature of slag is particularly of benefit in producing HPC with excellent rheological properties, and the test results are comparable to those reported in Xuequan and Roy's paper [3].

#### 4. Conclusions

(1) The yield stress value of cement paste is decreased generally with the increase of the UA quantity added. However, the viscosity varies appreciably with different types of UA and its quantity.

- (2) When the UA quantity added is smaller than or equal to 15%, the ultra-fine SF, FA, and LS can decrease the viscosity of cement paste, remarkably, the effect of ultra-fine slag is not noticeable. However, ultra-fine AG can significantly increase the viscosity of cement paste. When the UA quantity added is more than 15%, the values of the viscosity (and also the yield stress) of cement paste are significantly decreased with the increase of the UA quantity added and especially, the effect of ultra-fine slag is the most significant.
- (3) A respective addition of 10% ultra-fine LS, SF or FA can decrease the viscosity and the yield stress of cement paste. The acting effect is ranged as LS>SF>FA. A single addition of 10% ultra-fine AG will significantly increase the viscosity and the yield stress of cement paste. A single addition of 35% ultra-fine slag can significantly decrease the viscosity and the yield stress of cement paste.

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