INFLUENCE OF SILICA FUME ON THE RHEOLOGICAL PROPERTIES AND AUTOGENOUS SHRINKAGE OF UHPC PASTE

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Abstract- High amount of cementitious material and low water/binder (W/B) ratio increase shrinkage strain which also increases the crack formation risk of ultra-high performance concrete (UHPC). The effects of silica fume replacement ratio on the rheological properties and autogenous shrinkage of UHPC paste were investigated in this study. Four pastes with a very low W/B ratio (0.20) were produced. Besides the rheometer with a ball measuring system, mini slump-flow and V-funnel tests were applied to determine rheological properties. Moreover, autogenous shrinkage was measured by using a laser system at early-age, whereas a mechanical comparator was used for long-term measurements. As a result, the pastes which have SF up to 20% showed dilatant behavior. It was found that early-age shrinkage is much higher than long-term shrinkage. Both early-age and total autogenous deformation was increased as a result of SF replacement. The long-term and total deformations were significantly increased as a result of increasing SF replacement from 20% to 30%.

Index Terms- Rheology, Shrinkage, Silica Fume, UHPC.

I. INTRODUCTION

Ultra High Performance Concrete (UHPC) is a cement-based composite with ultra-high mechanical and durability performance. High ductility in addition to high strength can be obtained by adding metallic fibers. A powerful cementitious matrix with a very low water to binder ratio is essential to reach high-performance. As of late 2010, field-cast UHPC connections between prefabricated bridge components have been implemented in several bridges in Ontario, Canada, and in the United States [1]. Rehabilitation of concrete structures is also possible with using UHPC. However, due to restrained shrinkage of the UHPC bonded to the existing reinforced concrete body, an internal stress state is built up in the composite element including, in particular, tensile stresses in the UHPC layer [2]. These stresses may cause crack formation. Early-age shrinkage properties of UHPC, therefore, should be clarified. In this study, rheological properties and autogenous shrinkage of pastes which have a great influence on the properties of the UHPC were investigated.

II. EXPERIMENTAL

A. Materials and Mix Proportions

Some properties of Portland cement (CEM I 42.5 R) and silica fume (SF) used in this study are presented in Table I. A polycarboxylate based superplasticizer (SP) was used. Paste mixtures with four different SF contents were produced. Mix proportions can be seen in Table II. Note that, superplasticizer was used in different dosages for each paste to reach target spread value (40±1 cm). The mixtures were prepared in a special designed high speed mixer.

Table I. Chemical and physical properties of cement and SF

Chemical Composition (%)	Cement	SF
CaO	61.85	0.49
SiO_2	19.1	92.26
Al_2O_3	4.40	0.89
Fe_2O_3	3.96	1.97
MgO	2.05	0.96
Na_2O	0.27	0.42
K_2O	0.70	1.31
SO_3	3.72	0.33
Cl^-	0.0004	0.09
Loss on Ignition	1.82	-
Free CaO	0.50	-
Physical Properties		
Pozzolanic Activity	-	95
Fineness (m^2/kg)*	369	20000
Specific Gravity	3.12	2.2

*Nitrogen absorption method for SF, Blaine method for Portland cement

Table II. Mix proportions

Components (kg/m^3)	SF Replacement Ratio (%)			
	0	10	20	30
Water	369	357	340	319
Cement	1847	1607	1359	1118
Silica fume	0	179	340	479
SP	18	27	50	85
Design Parameters				
W/B	0.20	0.20	0.20	0.20
W/B*	0.20	0.21	0.21	0.23
Total SP (%)**	1.10	1.65	3.27	5.84
Slump-Flow (cm)	40±1 cm			

*includes additional water from superplasticizer.
**percentage by weight of cement.

B. Test Methods

Slump-flow diameter and V-funnel time of the pastes were measured by mini-apparatuses in conformity with the European Federation of Specialist Construction Chemicals and Concrete Systems [3] standards. The rheological properties of the pastes were measured on the same batch as mini V-funnel and slump-flow tests. Anton Paar Physica MCR 51 rheometer was used. It was equipped with a ball measuring system. The diameter of the ball used in this application was 8 mm. Measurements were conducted at a constant temperature (20±1°C) by way of a water circulation system assembled to the rheometer container. It is possible to obtain any shear rate protocol with using software of rheometer. A flow curve with shear rates up to 14 s^{-1} was recorded. In this study, a five interval shear rate protocol was used. Rheometer and the protocol can be seen in Fig. 1. First interval was applied to obtain the same history for all pastes. Drag resistance of the ball buried in the sample have been recorded as a function of the rotational speed. These data have been converted to shear stress versus shear rate by using empirical conversion constants [4]. The shear stress (Pa) - shear rate (s^{-1}) relationship of the cement pastes have been determined by using the raw data obtained from the 4-5th intervals which reflected negligible hysteresis area. Viscosities at the highest shear rate (14 s^{-1}) and dynamic yield stresses have been determined by using the curves obtained by raw data.

In this research, a relatively new early-age shrinkage test setup has been developed (Fig. 2). A climate controlled cabinet was used with temperature and humidity transmitters, temperature source (lambs), humidity source (ultrasonic steam diffuser). There is a humidity reducer fan to provide low relative humidity.

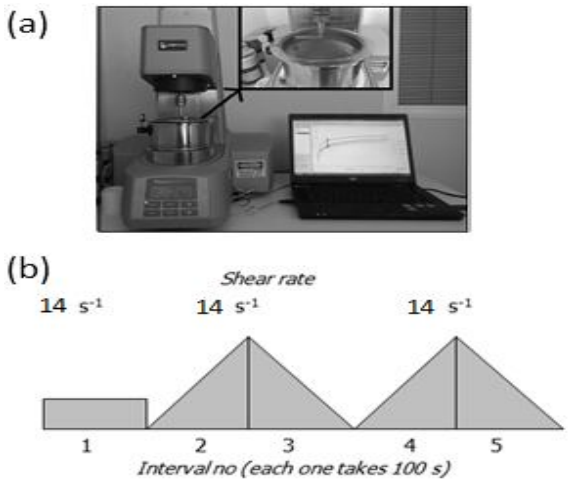


Fig. 1. The rheometer (a) and shear rate protocol (b)

All climatic operations and devices controlled by proportional–integral–derivative (PID) controller. The shrinkage values were measured by five laser distance sensors for each five moulds. The steel moulds with 25×25×290 mm dimensions were covered by plastic sheet and oiled to minimize friction. Temperature, humidity, shrinkage and time data were recorded to the computer simultaneously. The rigid mould system was illustrated in Fig. 2. The steel mould had a fixed-end and a moveable stop-end with steel pins to ensure good contact with the paste. Therefore, one laser sensor which is quite expensive is enough to measure early-age deformation for each mould. The free end of a mould which contains a laser beam hole was closed by a plastic sheet. The plastic sheet was removed at the initial setting time (at the time of self-supporting skeleton starts to form). Thereafter the shrinkage measurement was started. Autogenous shrinkage was measured under conditions of 20±1°C and 97±2% relative humidity (RH) during firs (24 h). In addition, the top surface of the fresh mix was covered with a tight nylon cover in order to avoid moisture loss to the environment. The initial setting time of the mixtures was measured in accordance with TS EN 480-2 [5] by an automatic Vicat apparatus with a temperature-controlled water bath. Note that, a thin layer of paraffin oil was applied to the surface of the fresh mix, for the purpose of avoiding elephant skin formation. Otherwise, it could not be measured correctly.

Long-term autogenous shrinkage was measured by using a mechanical comparator under conditions of 20±1°C and 97±2% relative humidity (RH) in accordance with American Society for Testing and Materials (ASTM) C157 [6]. The moulds with internal dimensions of 25×25×285 mm were immediately covered with a tight nylon cover after casting of paste. The hardened specimens were demoulded and covered by two layers of an aluminum foil tape after 24 h. Two specimens were tested for each mixture.

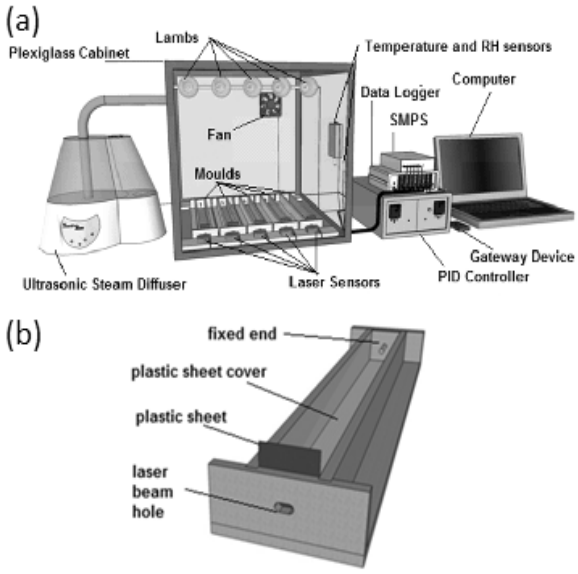


Fig. 2. Shrinkage test setup (a) and the mould design (b)

The compressive strength test was performed on standard (28-day) or steam cured specimens (40×40×160 mm) according to the ASTM C349 [7]. In case of steam curing, six hours after casting, the moulds were put in the steam curing cabin and heating period was started. The temperature of the cabin reached 100°C within six hours and the specimens were kept in this temperature for twelve hours. A gradual cooling period was applied at the end of the curing period. Twelve specimens were tested for each paste mixture.

III. RESULTS AND DISCUSSION

C. Rheological Properties

Superplasticizer demand and mini V-funnel flow time of the cementitious pastes having different SF replacement ratios and the same slump-flow value (40 cm) are presented in Fig. 3. An increase in SF led to an increase in superplasticizer demand and a decrease in V-funnel flow time. The paste without SF showed a flow time of 14 s, whereas the paste with 30% SF exhibited only 6 s. Moreover, the most obvious decrease in flow time was recorded between 20% and 30% SF replacements. Flow and viscosity curves of the pastes obtained by rheometer can be seen in Fig. 4 and Fig.5, respectively. Dynamic yield stresses were very low, between 2 to 5 Pa for all replacement ratios. The apparent viscosity of pastes which have SF replacement up to 20% increased by increasing shear rate so these pastes showed shear thickening (dilatant) behavior.

The dilatant behavior was strongest at control paste without SF. In case of 30% SF replacement, however, apparent viscosity was decreased by increasing shear rate (shear thinning behavior). This can be attributed to the significant rise in effective W/B ratio due to the extreme increase in superplasticizer dosage of SF30 mixture (Table II).

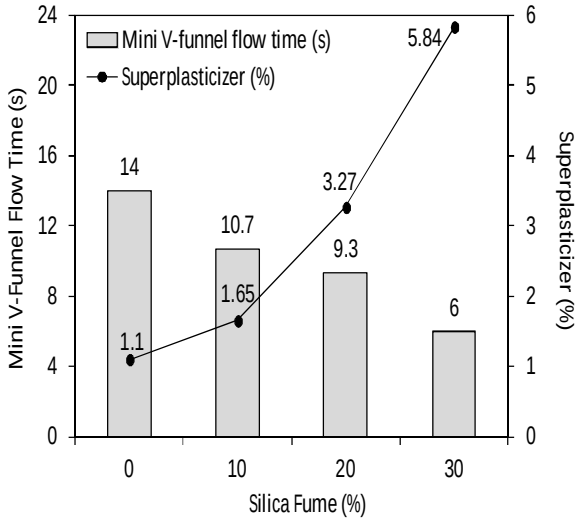


Fig. 3. Flow time and superplasticizer demand of the pastes

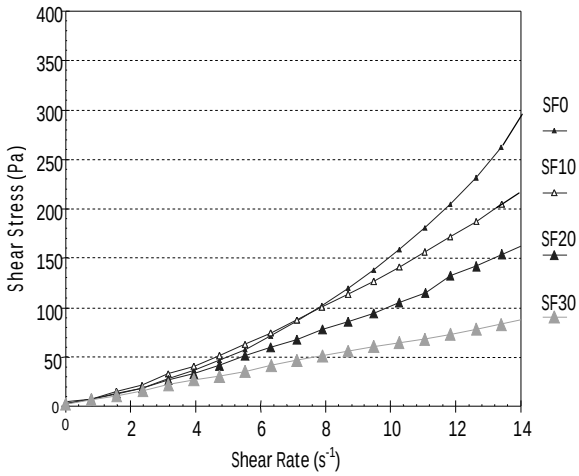


Fig. 4. Shear rate - shear stress curves

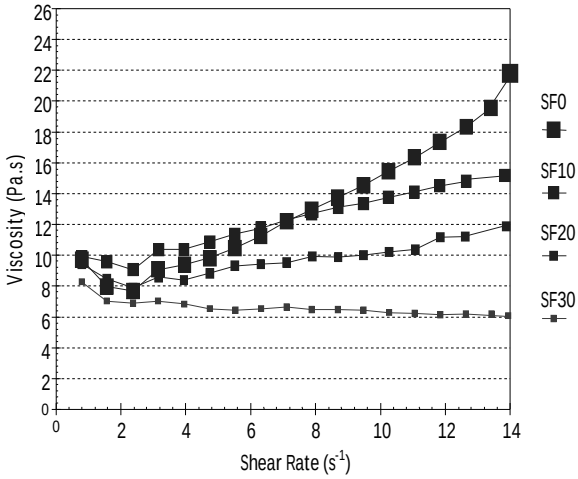


Fig. 5. Shear rate - viscosity curves

Fig. 6 presents the relationship between mini V-funnel flow time and instant apparent viscosity at 14 s^{-1} shear rate. An excellent correlation was obtained. The Marsh funnel is a simple device for measuring viscosity of pastes. However, mini V-funnel apparatus could be also used to assess the viscosity of cement pastes with an extremely low W/B ratio.

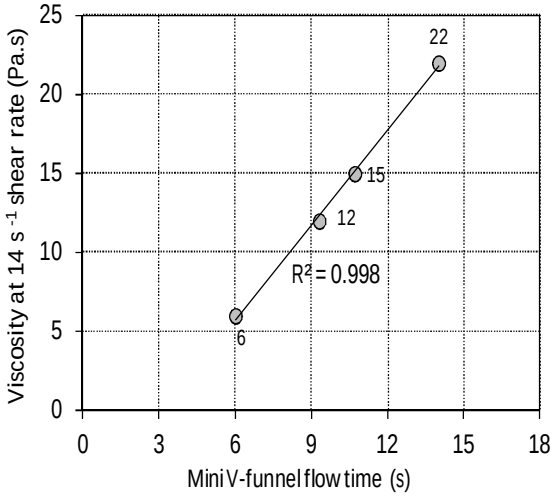


Fig. 6. Flow time vs. viscosity at 14 s^{-1} shear rate

D.Early-age and Long-Term Autogenous Shrinkage

Early-age autogenous deformations of the cementitious pastes measured at 20 °C are plotted as average values of five specimens in Fig. 7. Note that, the shrinkage measurement was started at the initial setting time. Initial setting times were 3.0, 3.45, 5.23, and 6.83 for SF0, SF10, SF20, and SF30 mixtures, respectively. An increase in superplasticizer markedly increased the initial setting times.

It can be seen that most of the autogenous shrinkage occurred in the first few hours after initial set (Fig. 7). After that, the shrinkage of nearly stopped. A swelling in the magnitude of 20 – 100 με was recorded for all pastes after acceleration period. The reasons behind swelling phenomenon are bleed water reabsorption and ettringite as product of early hydration of cement [8]. Swelling at early-ages could also be due to thermal expansion. The maximum temperature increase at the center of the specimens after initial set, however, was only 1.5 °C due to 25×25 mm cross section of the moulds employed in this study. It is obvious that 10% SF replacement reduced the shrinkage importantly. But higher replacement of cement by SF gives higher autogenous shrinkage as a result of greater chemical shrinkage and finer porosity which leads to water meniscus has a greater radius of curvature [9]. The maximum early-age shrinkage (1816 με) was exhibited by SF30 mixture. Moreover, the steepest rise in early-age autogenous shrinkage for 24 h was recorded 320 με by means of increasing SF replacement from 10% to 20%.

Long-term autogenous shrinkage of the cementitious pastes can be seen in Fig. 8. Long-term autogenous shrinkage measurement was started by demoulding of moulds at the end of 24 h in accordance with ASTM C157. The specimens were measured during 90 days. Results showed that increasing SF replacement caused an increase in shrinkage at long-term. The maximum long-term shrinkage (1210 με) was exhibited by SF30 mixture. Steep shrinkage rises were recorded as a result of increasing the SF replacement from 0% to 10%, and from 20% to 30%. It is obvious that early-age shrinkage (Fig.7) is much higher than long-term shrinkage (Fig 8) for all pastes.

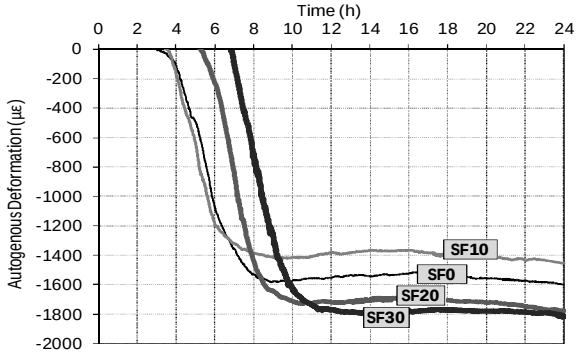


Fig. 7. Early-age autogenous shrinkage

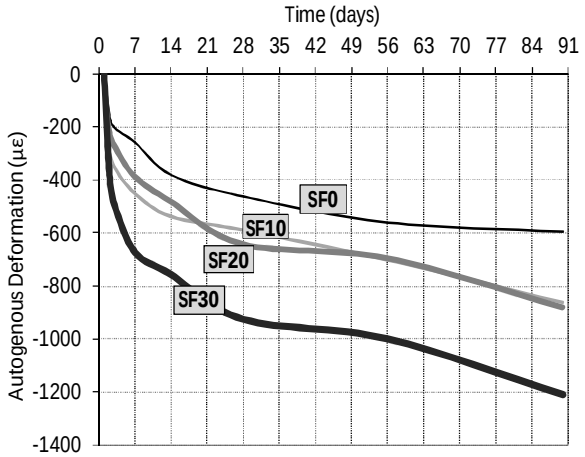


Fig. 8. Long-term autogenous shrinkage

The total autogenous deformation (early-age+ long-term) was significantly increased as a result of increasing SF replacement from 20% to 30%, whereas the magnitude of shrinkage was very close for the SF ratios lower than 20% (Fig. 9). Long-term autogenous shrinkage can be attributed to self-desiccation as the internal pores lose water for continued cement hydration [9]. Note that, internal humidity was not recorded. Hence, further research is needed to clarify the big picture. The maximum total shrinkage (3026 με) was exhibited by SF30 mixture. Moreover, the ratio of early-age to total autogenous shrinkage was slightly decreased by increasing SF replacement.

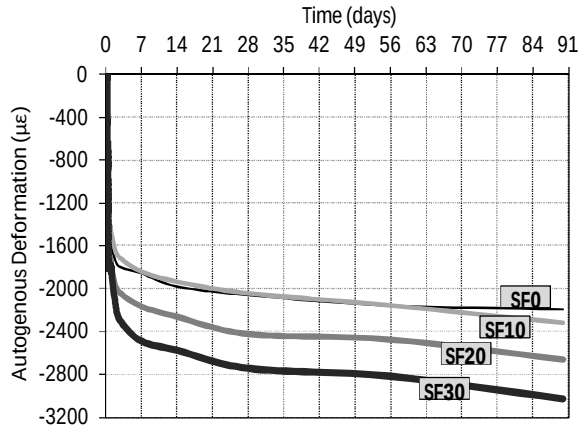


Fig. 9. Total (early-age + long-term) autogenous shrinkage

E. Mechanical Properties

Compressive strengths of the pastes can be seen in Fig. 10. Compressive strengths were in the range of 78 - 116 MPa and 86 - 115 MPa for standard and steam curing, respectively. A higher amount of SF led to lower compressive strengths. The strengths of steam cured specimens were slightly higher than that of standard cured specimens. Strength decrements as a result of SF replacement may be related with lack of dimensional stability and shrinkage induced micro-cracking of the neat paste without any aggregate skeleton. The exact mechanism, however,

was not fully clarified in the scope of this study. Above all, the actual effect of SF ratio of paste phase on the mechanical performance of UHPC is related to interface between fiber (or aggregate) and paste itself.

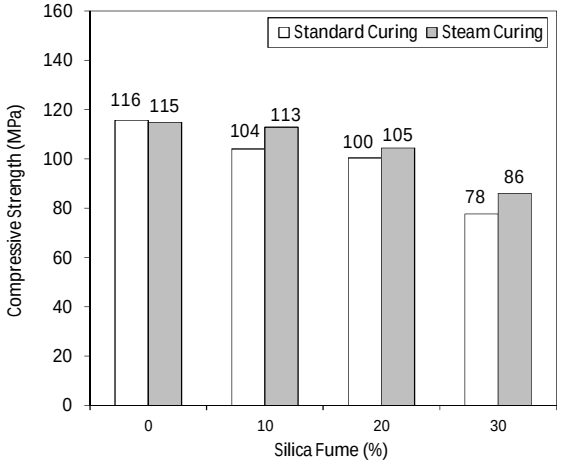


Fig. 10. Compressive strengths of the cementitious pastes

CONCLUSIONS

The following major conclusions can be drawn from the results of this experimental study;

1) An increase in SF led to an increase in superplasticizer demand and a decrease in V-funnel flow time. The most obvious decrease in flow time was recorded between 20% and 30% SF replacements.
2) The apparent viscosity of pastes which have SF replacement up to 20% increased by increasing shear rate and these pastes showed dilatant behavior. In practice, dilatancy of paste phase may reduce pumpability of fresh UHPC.
3) Mini V-funnel apparatus can be used to assess the viscosity of cement pastes with an extremely low W/C ratio. An excellent correlation between flow time and instant apparent viscosity was obtained.
4) 10% SF replacement reduced the early-age autogenous shrinkage importantly. But higher replacement of cement by SF gives higher autogenous shrinkage. Early-age shrinkage is much higher than long-term shrinkage.
5) The long-term and total autogenous deformations were significantly increased as a result of increasing SF replacement from 20% to 30%.
6) There is a high risk of cracking for UHPC, especially at at early-age. Limiting the paste volume may reduce autogenous shrinkage, so the cracking risk of UHPC.

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