Rheology of binary cement paste system blended with silica fume and alccofine

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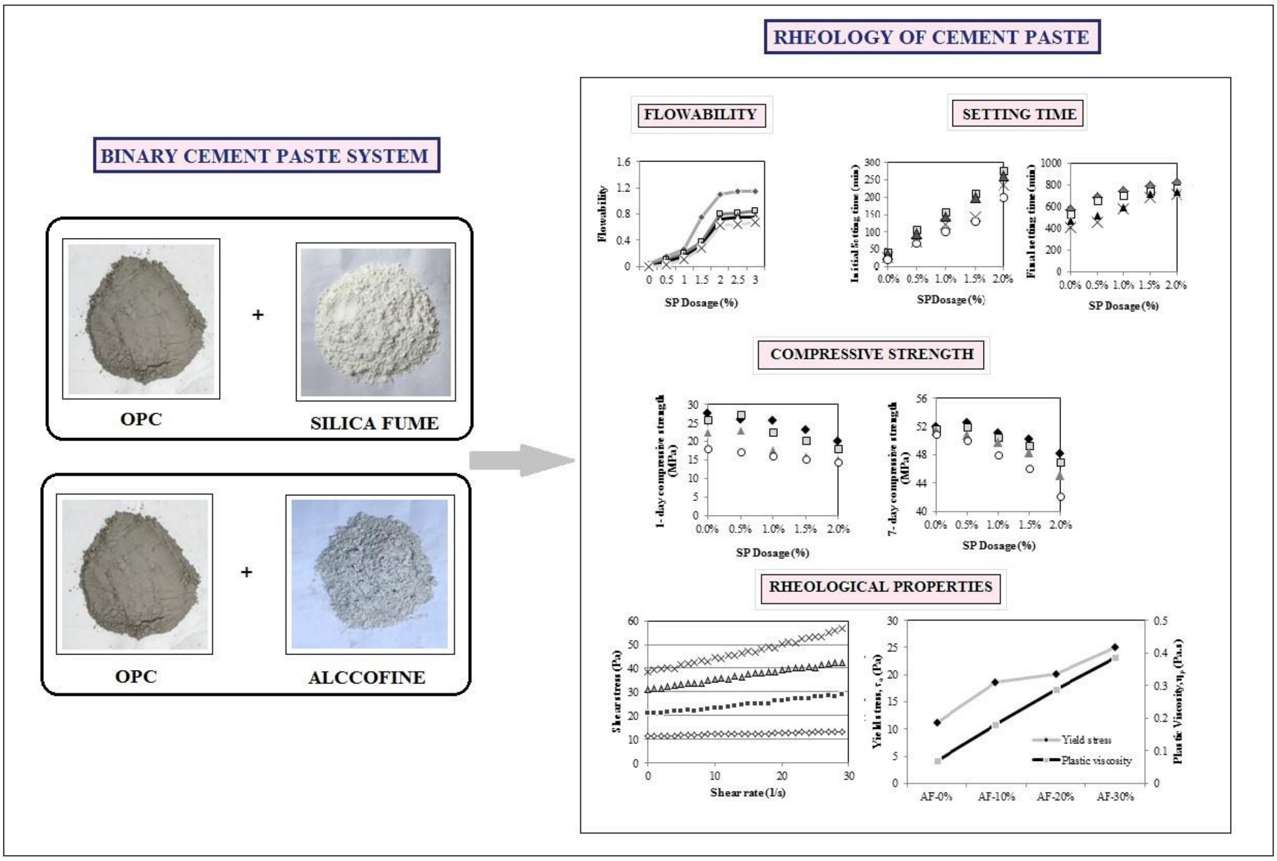
GRAPHICAL ABSTRACT



ABSTRACT

The flow properties of the binary cement paste system added with supplementary cementitious materials, the effect of polycarboxylate-based superplasticizer dosage and their influence on the yield stress and the plastic viscosity were studied. The cementitious materials under the study are silica fume and alccofine. Both the plain cement and the binary cement paste system were considered and tested. A comparative study between the plain cement paste without any additions, silica fume blended cement paste and alccofine blended cement paste was performed. The addition of cementitious materials varies in the range of 10%, 20% and 30% and the superplasticizer dosage varies in the range of 0.5%, 1.0%, 1.5%, 2%, 2.5% and 3% in the mini-slump flow test and the saturation point was determined to be 2% in both the cases of silica fume and alccofine. The setting time and compressive strength testing on the cement paste considered the superplasticizer dosage up to saturation point. The flow characteristics are broadly the same among the binary paste systems. The setting time results indicated the increase in setting time with an increase in cementitious additions and with an increase in superplasticizer dosage. The higher percentage substitution of cement with silica fume and alccofine resulted in a decrease in compressive strength due to the simultaneous acceleratory and retarding effects of cementitious additions and the superplasticizer. The results of the rheological measurements revealed that silica fume possesses greater elasticity than that of alccofine which possesses greater yield stress and plastic viscosity.

Keywords: Flowability, setting time, compressive strength and rheological properties.

1. Introduction

Green concrete experienced rapid growth all around the world as a part of the circular economy, which involves obtaining resources from waste materials through recycling and reuse. Especially the rotational utilization of supplementary cementitious materials as a blending material in the manufacturing of cement as well as the mineral admixtures in concrete production increases day to day [Liu et al 2018]. The over consumption of cement in concrete industries also increases the threat to the bio-diversity by means of CO_2 emission which globally affects the environment. This in turn draws the attention of the researchers to find a potential and alternative solutions replacing the cement usage. Following this, the exploration on supplementary cementitious materials is also growing in its line day to day. A detailed investigation on the incorporation of SCM in cement and concrete matrix is of primary importance. Rheological study on the cement mortar estimates the flow behavior and helps in assessing the pumpability of concrete. The addition of particles with high fineness improves the matrix arrangement by modifying the granular structure and the frictional phase in the solid-liquid interface. The voids in the mortar particles are filled by the finer grains which in turn results in increased compactness of the mortar by releasing the entrapped water in the pores, thereby decreasing the water demand [Mokhtaria Benkaddour et al 2023]. The introduction of ultra-fine powder in concrete manufacturing not only has an influence on the particle size and morphology but also on the workability and strength of the cementitious materials [Niu et al 2002, Liu et al 2018]. Ground granulated blast furnace slag (GGBFS) is one of the potential cementitious materials and their reactivity is not only depends on the chemical composition but also the fineness [Pal et al 2003]. Furthermore, many studies were carried out with silica fume as the cement replacing material and demonstrated its beneficial effect on the strength and durability properties of the concrete [Mazloom et al 2004, Saad et al 1996 and Koksal et al 2002]. A research study on the incorporation of nano-slag demonstrated the modified microstructure and enhanced strength of the concrete [Lim et al 2008]. The ultra-fine GGBS (UFS) known as alccofine and the silica fume are the supplementary cementitious materials (SCM) considered in the study. The filler effect of the SCM greatly contributes to the particle packing and densification whichever ensures the homogeneity resulting in higher strength concrete. Besides the enhanced strength and performance of concrete by employing a low water-binder ratio and higher dose of SCM, it also imposed poor workability [Min Wang et al 2021]. In addition, increased fineness of the binding materials accelerates setting and lowers the workability of the cement paste or concrete [Collins et al 1999]. The past studies on the rheological properties of cement paste blended with the UFS [Luo Ting et al 2019] and silica fume [Weina Meng et al 2019] demonstrated the beneficial effects of the addition of admixtures like superplasticizer which helps in achieving satisfactory workability [Abdulkareem et al 2018, Yu et al 2015, Boukendakdji et al 2012, Drissa Ouattara et al 2017]. Furthermore, the recent studies on the rheology of cement paste blended with ashes obtained from the calcination of sugarcane bagasse and rice straw [Athira et al 2023] and on the rheological, mechanical and durability performance of commercial binary and ternary blended cements [Mokhtaria Benkaddour et al 2023] showed the revolution and innovation in research studies on the cement paste system.

The microstructure of the cement paste associated with the hydration process has a great influence on the development of the rheological properties of the cement paste system. To reduce the agglomeration effect of the SCM particles and to improve the workability of the fresh cement mixture, polycarboxylate-based superplasticizers are used as the dispersing agents. Many studies have explained the role of superplasticizers in dispersing the flocculated particles, improving the packing density and the thickness of the water film in the cement paste thereby reducing the viscosity of the fresh cement paste. This in turn possesses a decelerating effect on the hydration and the adverse effects could be further alleviated by the addition of finer SCM particles [Drissa Ouattara et al 2017, Flatt et al 1998, Diamond et al 2004]. Poor rheological properties may adversely affect the strength properties in the hardened state of concrete, which could result in a construction material with compromise in quality [Qing et al 2009, Mitchell et al 1998]. Therefore, it seems prominent to study the effect of varying doses of SCM and SP on the rheology of the cement paste and to formulate the optimum proportions of SCM and SP to produce ultra-high-performance concrete. The well-known and commonly used methods to describe the rheology of cementitious materials are Bingham fluid model and Herschel-Bulkley model [Xiaodi Dai et al 2022]. However, Bingham fluid model is a simpler and more familiar model which describes cement paste as the Non-Newtonian plastic fluids [Juan Manuel Beltran et al 2023]. For better understanding on the rheological behavior, the yield stress and plastic viscosity of the cement paste is evaluated which can be characterized and defined by Bingham fluid model. Certainly, incorporating the Bingham fluid model in the present analysis on the rheological behaviour of cement paste offers valuable insights into its flow properties under varying conditions.

The present study experimentally investigated the rheological properties of the plain and binary cement paste system added with supplementary cementitious materials namely silica fume and alccofine. The novel approach in the study concentrates on examining the combined effect of increasing contents of SCM (0% - 30%) and the different doses of SP (0% - 2% by weight of cement) on the rheology of the cement paste system. The experimental study on the cement paste system includes determination of flowability, initial and final setting time, compressive strength (1 and 7 days), the rheological flow curve measurements and determination of yield stress and plastic viscosity. A comparative study on the rheological behaviour of the plain and two different binary cement paste systems was presented.

2. Materials and methods

2.1. Materials

Ordinary Portland cement (OPC -53 grade), silica fume and alccofine are the materials used. The chemical composition of the materials was obtained from the X-ray fluorescence (XRF) analysis and the results are listed in Table 1. The mean diameter and D_{50} (μm) of the materials obtained from the particle size analysis are mentioned in Table 2. Polycarboxylate-based superplasticizer namely sika viscocrete was used. The water-cement ratio was considered as 0.3 and was constant throughout the study. The superplasticizer was added to the water at different dosages (0%, 0.5%, 1%, 1.5% and 2% by weight of the cement). The upper limit of the superplasticizer dosage was considered as 2% which was the saturation point determined from the mini-slump flow test

Table 1. Chemical composition of cementitious materials

Constituent	Cement	Silica fume	Alccofine
SiO_2	22.4	93.10	40.83
Al_2O_3	5.10	0.90	-
Fe_2O_3	3.60	1.86	0.92
CaO	63.10	0.46	56.80
MgO	2.0	0.94	-
SO_3	2.45	1.29	-
Na_2O	0.15	0.42	-
K_2O	1.2	0.31	0.65
TiO_2	-	-	0.80
LOI	0.65	1.2	0.1

Table 2. Particle size distribution of the materials

Materials	Mean Diameter (μm)	D_{50} (μm)
Ordinary Portland Cement	0.331	0.344

Silica fume	0.272	0.263
Alccofine	0.279	0.277

2.2. Methods

The experimental investigation on the plain and binary cement paste system comprises of mini-slump flow test, initial and final setting time, and compressive strength (1 and 7 days). Furthermore, the yield stress and plastic viscosity of the cement pastes were also determined by the Bingham fluid model. The cement paste samples were prepared with a constant water binder ratio of 0.3 added with SP increasing in the range of 0.5% (0-2%). The effect of varying contents of SCM (0-30%) and varying SP dosages on the flow and the rheological behaviour of the cement paste system were studied. The experiments were carried out at the room temperature of 27±2℃ and the relative humidity ranging from 38% to 52%.

The mini-slump flow test was performed in accordance with ASTM C 230/C 230 M. This simple and convenient test indirectly measures the yield shear stress and plastic viscosity of the cement paste. The experimental procedure for the test is as follows. (1) The cement paste to be tested is filled into the mini-slump cone with a bottom diameter of 100 mm and, a top diameter of 70 mm allowing the cement paste to undergo self-consolidation; (2) The mini-slump cone is then lifted and the paste is allowed to spread; (3) the maximum spread diameter of the cement paste is measured in two perpendicular directions. The measured spread diameter (S) in mm is used to determine the relative mini-slump flow from the formula (S-100)/100. The initial and final setting times of the cement pastes were determined by using the Vicat apparatus in accordance with ASTM C191. The setting time of the cement pastes prepared with a constant water-cement ratio of 0.3 was tested for the increasing dosages of SCM and the SP. The compressive strength (1 and 7 days) testing was performed on the 50 mm cubes of plain and binary cement paste specimens in accordance with ASTM C 109. The rheological properties of the cement paste system were determined by using Brookfield rotor rheometers. The flow curve measurement which includes shear rate (s^{-1}) versus shear stress (Pa) was made by increasing the shear rate from 0 s^{-1} to 30 s^{-1} over 120 s followed by a decrease from 30 s^{-1} to 0 s^{-1} over the next 120 s [Luo Ting et al 2019]. Using the Bingham fluid model, from the slope and intercept of the linear relationship between the shear stress and shear rate, the rheological parameters such as the yield stress (τ_o) and plastic viscosity (η_p) were calculated from equation (1) expressed as follows.

$$\tau = \eta_p . \gamma + \tau_o \qquad \text{Eqn. (1)}$$

where γ denotes the shear rate (s^{-1}). Three samples per test were replicated and the coefficient of variation was found to be in the range of ±5%. Every cement paste mix was prepared and tested at room temperature (27±2°C)

3. Results and Discussion

3.1. Mini-Slump flow

The flowability of the cement paste added with silica fume and alccofine are represented in Figure 1(a) and (b) respectively. The relative slump flow of the cement paste increases with an increase in SP dosage till it reaches the dosage of 2% by weight of cement. Beyond this limit of SP dosage (2%), no significant change in the flowability occurs in both cases of cementitious additions. This critical limit denotes the saturation point of the cement paste mixture [Weina Meng et al 2019]. This saturation point is considered as the upper limit of SP dosage for every cement paste mix whereas lower limits correlate with 25%, 50% and 75% of the saturation point dosage of SP and the homogeneity of the cement paste mixes were ensured. The binary cement paste blended with silica fume and alccofine both exhibited similar saturation points. This reveals that the influence of the cementitious additions with the incorporated SP on the flowability characteristics of the cement paste was almost similar.

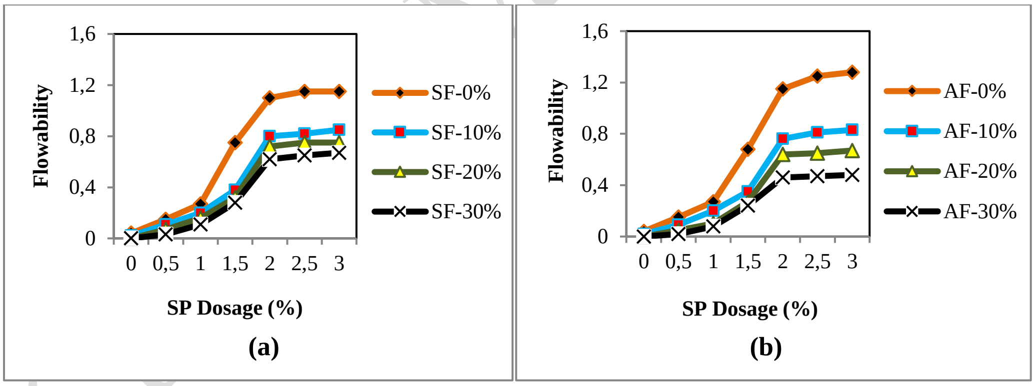


Figure 1. Flowability of cement paste added with: (a) Silica fume and (b) Alccofine

The addition of fine powders in the cement paste system results in reduced workability which in turn increases the water demand. However, the morphological characteristics of the cement substitutes especially the spherical grains with smooth texture provides a lubricating effect and minimize the internal friction between particles. In addition, the role of SP in the binary cement paste system is highly appreciable in reducing the water requirement.

3.2. Initial and final setting time

The initial and final setting time of the cement paste system blended with silica fume and alccofine determined for varying SP dosages are given in the table 3 and graphically represented in the Figure 2 and Figure 3 respectively. In both the cases of the cementitious additions, the initial and final setting time decreased and increased linearly to the increasing dosages of SCM and the SP respectively. The finer particles of the silica fume and alccofine possess larger surface area which in turn enhances the nucleation and precipitation activities of the hydration reaction, which simultaneously develops the connectivity of the hydrates and the micro-structure. Hence, the addition of SCM shortened the setting times of the pastes. On the other hand, the increasing SP addition to the cement paste retards the hydration process and affects the connectivity of the hydrates, which in turn extended the setting times [Weina Meng et al 2019]. The combined and alternate effect of SCM and SP dosage results in a composite behaviour of the cement paste system.

Table 3. Initial and final setting time of cement paste

SP(%)	*Initial setting time (min)*					*Final setting time (min)*				
	0%	**0.50%**	**1%**	**1.50%**	**2%**	**0%**	**0.50%**	**1%**	**1.50%**	**2%**
SF-0%	40	105	160	210	275	570	680	740	790	815
SF-10%	35	95	145	200	260	550	665	720	755	790
SF-20%	25	70	120	145	235	495	535	615	725	760
SF-30%	20	65	100	130	200	435	470	590	700	745
AF-0%	40	105	160	210	275	570	680	740	790	815
AF-10%	30	85	135	185	205	535	650	705	740	765
AF-20%	20	60	110	140	185	470	520	600	720	740
AF-30%	10	55	90	115	150	410	455	575	685	715

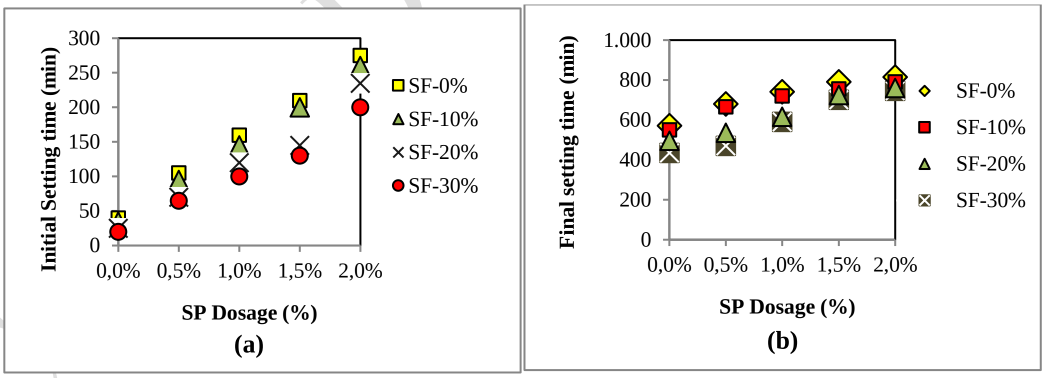


Figure 2. (a) Initial setting time and (b) final setting time of silica fume blended cement paste

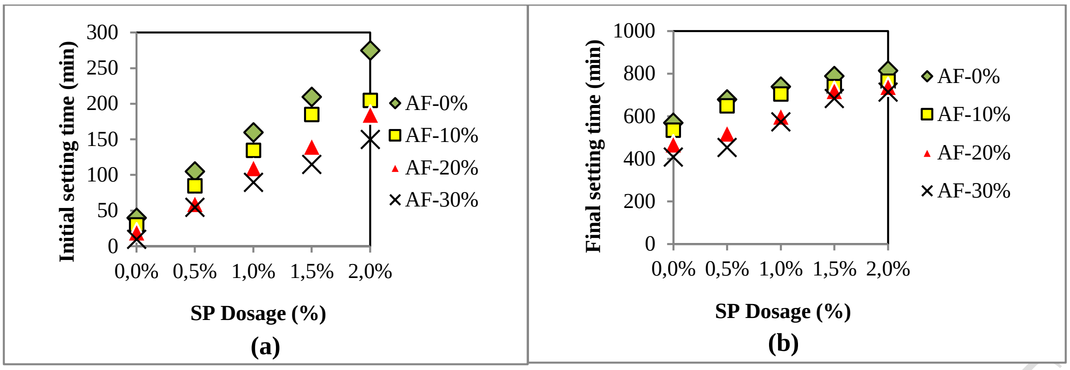


Figure 3. (a) Initial setting time and (b) final setting time of alccofine blended cement paste

The initial setting time of the plain cement paste not added with SP and the final setting time of the binary cement paste with a maximum SP dosage show least variation in the setting times when compared with that of other combinations. Contrarily, the upper and lower limit of SP dosage possesses distinguishable effects on the initial setting time and final setting time respectively. With the increase in silica fume content from 0% to 30%, the initial setting time was reduced from 40 min to 20 min and the final setting time was reduced from 570 min to 435 min. In the case of increasing contents of alccofine from 0% to 30% decreased the initial setting time from 40 to 10 min and the final setting time from 570 min to 410 min. The similarity in the variation of setting time with the change in SP dosage occurs between both cases of SCM. However, the initial and final setting time of silica fume blended cement paste is comparatively greater than alccofine blended cement paste. This indicates the competing effect of alccofine in accelerating hydration and decreasing retardation due to the increasing SP dosage when compared with that of silica fume.

3.3 Compressive strength

The compressive strengths of plain and silica fume blended cement paste at 1 and 7 days are given in the table 4 and graphically shown in the figure 4 (a) and 4 (b) respectively and 1 and 7 days compressive strength of plain and alccofine blended cement paste are shown in the figure 5 (a) and 5 (b). On comparative analysis of the influence of SCM in the strength gain of early and later age compressive strength of the cement paste system, an almost similar response was encountered. However, a slight difference in the 1 day and 7 days compressive strength of cement paste system under study was observed.

Table 4. Compressive strength of cement paste system

SP(%)	*1-day compressive strength (MPa)*					7-day compressive strength *(MPa)*				
	0%	**0.50%**	**1%**	**1.50%**	**2%**	**0%**	**0.50%**	**1%**	**1.50%**	**2%**
SF-0%	27.5	26	25.5	23	20	52	52.5	51	50.2	48.1
SF-10%	26	27.25	22.5	20.25	18.15	51.75	51.9	50.6	49.1	46.15
SF-20%	22.5	23	17.95	16.1	15.25	51.2	52	49.1	47.2	42.15

SF-30%	18	17.2	16.1	15.2	14.6	50.8	50.65	47.3	44.15	40.7
AF-0%	27.5	26	25.5	23	20	52	52.5	51	50.2	48.1
AF-10%	27.2	27.45	23.65	21.7	19	51.6	51.9	50.45	49.25	46.95
AF-20%	25.35	25.25	19.15	17.9	15.05	51.35	50.8	49.8	48.25	45.1
AF-30%	19.15	18.2	17.2	16.8	13.9	50.95	50.1	48	46.1	42.2

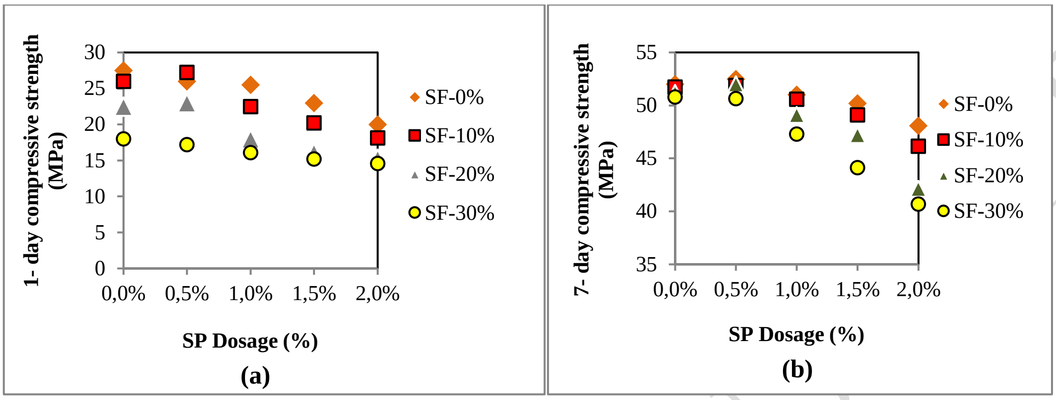


Figure 4. (a) 1-day compressive strength and (b) 7-days compressive strength of silica fume blended cement paste

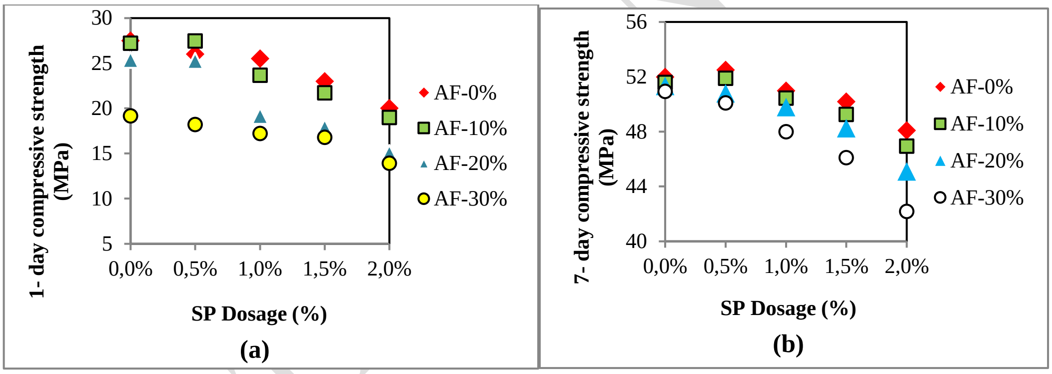


Figure 5. (a) 1-day compressive strength and (b) 7-days compressive strength of alccofine blended cement paste

The results denoted the decrease in the compressive strength of the cement pastes with increasing SP dosage and SCM contents. It was known that this was primarily due to the increasing contents of SCM as a substitute of cement, which reduces the cement content in the cement paste. This in turn results in a reduction of the strength gain by decreasing the C-S-H formation and increasing the porosity [Weina Meng et al 2019]. However, a sufficient proportion of cement and SCM when combined with a suitable dosage of SP helps in enhancing the early strength and the pozzolanic activity. This was noted in both the case of SCM addition, especially in the 1-day compressive strength of the binary cement paste added with 10% SCM and 0.5% SP dosage gained greater strength than that of the plain cement paste, which was less pronounced at the age of 7 days. The greater SP dosage (i.e., > 0.5%) and greater substitution of SCM (i.e., > 10%) adversely affect the formation of C-S-H and the pozzolanic activity. Beyond this limit, no such significant response in the early and later age compressive strengths was observed. Furthermore, the similarity in the strength development by the silica fume and alccofine reveals the competent pozzolanic effect of the SCM particles.

3.4 Rheological performance

The rheological properties of the plain and binary cement paste added with the SCM were studied by the flow curve measurements by addition of the resulted optimum SP dosage of 0.5%. The resulting flow curves obtained by plotting the shear rate (1/s) versus shear stress (Pa) are shown in figure 6 and figure 7 for the cement paste blended with silica fume and alccofine respectively. Both the shear rate and the shear stress increased with the increase in the contents of silica fume and alccofine in each case. The yield stress and plastic viscosity of the cement paste added with silica fume and alccofine obtained using Bingham's fluid model are presented in figure 8 and figure 9 respectively.

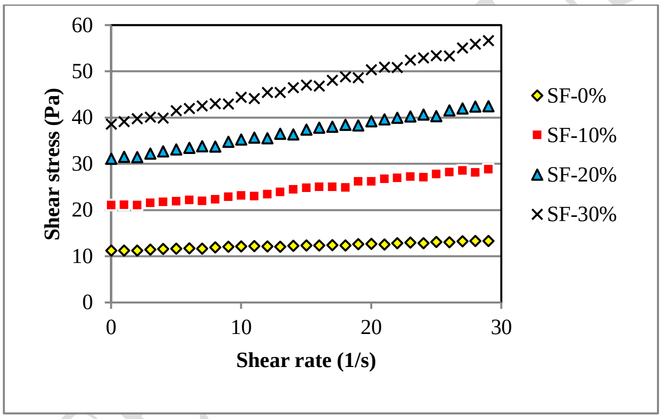


Figure 6. Shear rate vs shear stress for silica fume blended cement paste

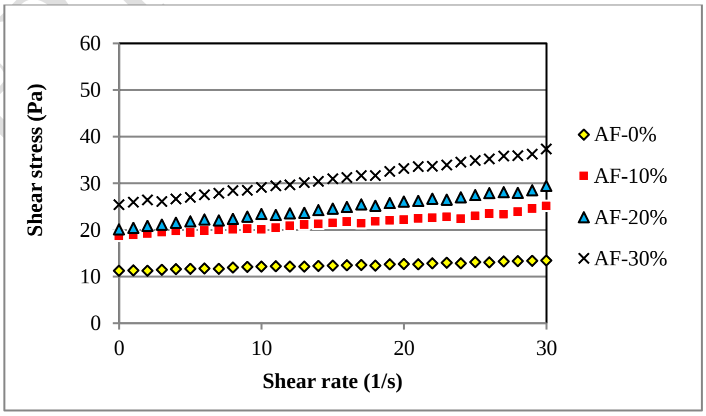


Figure 7. Shear rate vs shear stress for alccofine blended cement paste

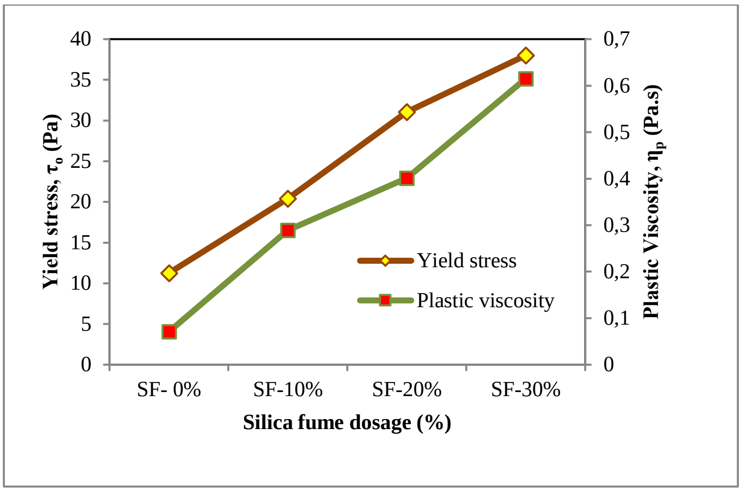


Figure 8. Yield stress and plastic velocity for cement paste added with silica fume

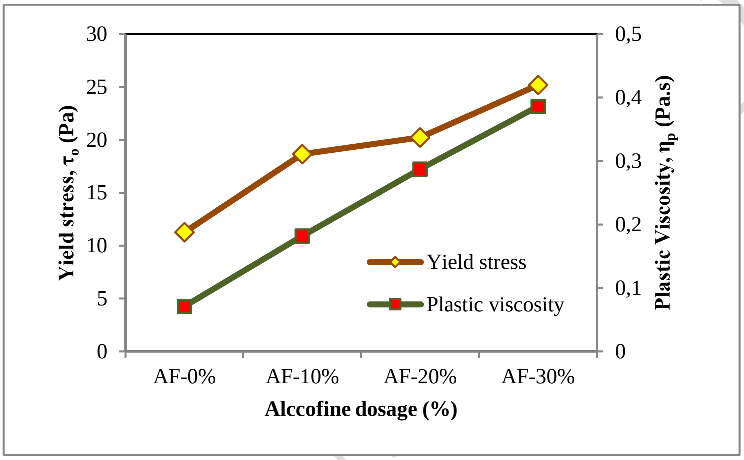


Figure 9. Yield stress and plastic velocity for cement paste added with alccofine

The transition of cement paste from a fluid state to a solid state governs the rheology of the cement paste. The cement paste with higher stress values greater than the yield stress possess the ability to flow. Furthermore, the shape and gradation of the particles in the cement mixture greatly influence the rheological behavior of the cement paste [Gaurav Sant et al 2008]. Both the yield stress (τ_o) and the plastic viscosity (η_p) of the cement paste increased with increase in the silica fume and alccofine contents in the cement paste. This explains the force of attraction between the SCM particles and the water molecules which gets enhanced with increasing specific surface area which increases with SCM content. As a result of this, the thickness of the lubricating film decreases and increases the friction between the materials thereby increasing the shear resistance [Luo Ting et al 2019]. Comparing the results obtained for the silica fume blended cement paste and alccofine blended cement paste, τ_o and η_p of the cement paste added with silica fume are higher than that of the cement paste added with alccofine. This is primarily due to the difference in the specific surface area of the SCM. Since the specific surface area associated with the lower particle size of the silica fume is higher than the alccofine, this results in increased yield stress and plastic viscosity. The demonstrated flowability characteristics determined from the rheological parameters helps in designing a economically feasible and pumpable concrete design.

The microstructure of the cement paste is highly influenced by the rheology of the fresh cement paste system. The hydration products are the major contributors of the microstructure development in the cement paste [Drissa Ouattara et al 2017]. The reaction between the water and the cement particles gets retarded at higher replacements of cement by either silica fume or alccofine. This was mainly because of the water demand associated with the decrease in the workability of the fresh cement paste mix. Therefore, an optimum dosage of SP helps in reducing the shear stress and increases the workability of the binary cement pastes incorporated with silica fume or alccofine. Furthermore, imprecise understanding on the pumbability and shootability of the fresh concrete especially in case of shotcrete applications can be significantly avoided by the rheological study on the cement paste.

4. Conclusion

Based on the rheological study of the cement paste system considered in the present study, the following conclusions can be drawn:

(1) The mini-slump flow test carried out on the cement paste system revealed that the silica fume-based cement paste and alccofine-based cement paste possess almost similar flowability characteristics. The flowability increased with the increase in SP dosage and decreased with increasing SCM contents. The saturation point was determined to be 2% SP dosage beyond which no significant change in the flowability occurs in both the cases of SCM.

(2) Both the initial and final setting time of the cement paste system increased and decreased with the increase in SP dosage and increase in SCM contents respectively almost in a linear manner. However the cement paste blended with silica fume exhibits comparatively greater setting time than that of alccofine based cement paste which revealed that the alccofine is highly competent in accelerating the hydration reaction and in resisting the retardation of hydration due to the increasing SP dosage.

(3) The binary cement paste with silica fume performed similar to the binary cement paste with alccofine in case of compressive strength test. The ability of the strength gain by the cement paste decreases with higher level substitution of SCM and higher SP dosages. This was mainly due to the suppressed pozzolanic activity resulted from high SCM content. However, the compressive strength of the cement paste at 1 day and 7 days is found to be maximum for the binary cement paste with 10% SCM content at 0.5% SP dosage. This

indicates the synergistic effect of the SCM and SP blended in the cement paste on the strength development when added in an optimum proportion.

(4) The shear stress of the plain and binary cement paste system increased with increasing shear rate and decreased with increasing doses of the SCM. Both the silica fume and alccofine showed similar variation in the rheological properties, however, the increment and decrement range of the shear stress values for the alccofine blended cement paste system is significantly lower when compared with that of the silica fume. Both the yield stress and the plastic viscosity increased with increase in SCM in the cement paste. Furthermore, the binary cement paste with silica fume possesses higher yield stress and plastic viscosity than the alccofine blended cement paste. This indicated the increased specific surface area of the silica fume particles which lowers solid-liquid interconnectivity and the workability of the fresh cement paste mix. However, the combination of SCM and SP imparts both accelerating and decelerating effect in the rheological behaviour of the cement paste system.

(5) The high performance of cement paste was found to be at the optimum proportion of 10% silica fume content and alccofine content with 0.5% superplasticizer dosage under the considerations and conditions in the present study. This in turn supports the decision on trial mixes and certainly reduces the resources and raw materials.

(6) The binary cement paste system with silica fume and alccofine possesses potential rheological properties and helps in boosting both ecological and economical sustainability. The use of silica fume and alccofine as the cement substitute proved to be a better alternative avenue for reducing cement consumption in concrete production units thereby decreasing the rate of carbon dioxide emission.

Recommendations

Future research should prioritize to study on the influence of significant factors like temperature, humidity, etc. on the rheological and fresh properties of the cement paste system. Furthermore, the effect of particle size and fineness of the supplementary cementitious materials in the hydration process are recommended to be investigated in future.

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