

Time-dependent rheological behavior of cement-sand injection grout containing high volume fly ash

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Abstract. Grouting technology is playing an increasingly important role in construction engineering. Various types of grout are available but cement slurry grouts are often selected for injection works due to better workability. Meanwhile, in repair works, cement-sand grout often preferred as the fine aggregates gives better volume stability for large repair area. However, theoretical research on the use of waste material to improve the rheological properties of cement-sand grout is still lacking. For grouts, rheology is a factor of prime importance to the transport, injection and pumping of the material. Cement-sand grouts often have poorer fluidity due to friction created by the sand. Since grouts typically contain cement, therefore fly ash can be a good option to improve the workability of the grout. This paper studies time dependent behaviour of the grout by measuring the flow times and viscosity of cement-sand grout containing high volume fly (HVFA) up to 1 hour from the time of mixing. The result indicates that replacement of HVFA reduces the flow time of the grout and maintained good fluidity up to 1 hour. The viscosity test showed that the grouts, at fresh state, behaved like a Bingham fluid by exhibiting lower viscosities for the mixtures containing fly ash at 0, 60 and 120 minutes. In conclusion, the use of large quantities of fly ash in grouts significantly enhances fluidity of the cement-sand grouts. Methodology comparison also resulted in new knowledge for best method flow test for fly ash grout.

1. Introduction

Grout often used as a repair material for concrete damaged from the fire, attacks from the environment or to increase the strength of concrete which was lower due to some manufacturing problem. It can be said that grout often placed in aggressive environments which concrete has deteriorated, and the grout will act in a structural capacity. Grouting also done in post tensioning cables or bars where complete encapsulation of the reinforcing and protection from corrosion are required. Additionally, grouts can improve fire ratings, acoustic performance, and blast resistance and improve the thermal mass properties of the building elements.

Although many types of grouts are available commercially, cementitious grouts are widely used for injection works. On top of that, cement-sand grout of often used for concrete repair and construction works. In such works, the rheology properties of grout are an important requirement or the measure of the ease of pumping grout. Hence, the most important fluid property of a grout is its ability to flow through voids or spaces [1, 2, 3]. In this sense, it is often that a cement slurry grouts without any sand are selected for injection works. This however not practical and suitable for concrete repair works as it increases the cost and the volume stability of neat cement slurry grout lower compared those filled



with aggregates. Hence, research advancements in grouting can stimulate the development of new materials.

Different to concrete, the potential ways of improving the properties of grout using supplementary cement material is still lacking as only limited amount of literature is available on the use of fly ash in grouts. Furthermore, the amount used in such grouting material is often limited to 30%. The fresh state and rheological properties of grouting materials have been investigated by various researchers. However, there are inadequate reports on the rheological performance of cementitious grouts containing fly ash in the scientific literature [4]. In addition, the existing studies on rheology are mainly focused on cement slurries and pastes while in grout in building repair and construction works are often made with fine fillers like sand.

Fly ash is generally used in concrete manufacturing due to its technical and economic benefits. However, fly ash utilization in technical products such as the cement-sand grout in high volume is still uncommon. Fly ash still available abundantly in every country and being landfilled. The use of large quantities of fly ash as supplementary cementitious material in concrete is beneficial to stakeholders such as the power generators and materials manufacturers in the construction industry. The use of large quantities of fly ash as a construction material can also be considered an ecological disposal approach which reduces environmental degradation. Therefore, numerous socio-economic and environmental benefits can be derived from replacing 50% of the cement with fly ash. In addition, the use of fly ash enhances the durability and the hardening state properties of the cement composites [5, 6].

Fly ash is generally used at replacement levels of 15–25% by mass [7]. The use of 50% fly ash as a replacement in cement, called High Volume Fly Ash (HVFA), has been reported in the literature. Although some studies reported on the use of fly ash in grouts, the application of such grouts are limited to soil and rock grouting. The grouts used in such application are often cement slurry grout without any sand. However, very little literature reported on the use of cement-sand grout with high volume fly ash and used for concrete repair and strengthening, particularly a through rheological behaviour which is a crucial property for injection grouts. Moreover, the use of such grout has been more important as the concrete repair and restoration works are growing rapidly.

The rheological importance of injection grouts was also highlighted by Pusch [8]. The findings indicate that materials with low shear rates can seal cracks through dynamic injection. In general, the rheological behaviour of cementitious grout is complex. Moreover, cementitious materials such as grouts influence the hydration and chemical reaction of the setting process. In addition, this can influence the rheological properties over time [9]. The complex rheological behaviour of cementitious materials is due to their thixotropic properties. This occurs when the viscosity decreases under shear stress due to structural breakdown. Similarly, the cementitious material can restore its rheological behaviour when shearing stops. The study by Yahia and Khayat [10], shows that a grout with low water to cement ratio and incorporated slag can behave differently compared to conventional grout. Use of mineral additives such as limestone can result in the pseudo-plastic behavior of the grout while fly ash reduces the yield stress and plastic viscosity [11].

Considering the importance of rheology of grouts in construction, this paper aims to perform a detailed study on the rheological properties namely; flow times and viscosity of cement-based grouts made from different fly ash contents for a duration of 1 hour.

2. Materials and sample preparation

The grouts were prepared from ordinary Portland cement (OPC) CEM I 52.5N as the control mix. The fly ash grout mixes were made by replacing cement with 10, 20, 30, 40 and 50% fly ash by weight. The water to binder ratio (W/B) was kept constant at 0.40 and all the mixes contained 0.5%

superplasticizer relative to the cement mass. The fly ash used in this study was obtained from a coal-fired power plant located in Peninsular Malaysia. A naphthalene sulphonate based super-plasticizer (SP) with 93% solid content and specific gravity of 0.66 was used in this study. The use of plasticizers is important because it can help to disperse cement particles that are exposed to aggregation due to colloidal interactions. The OPC cement usually composed of 95% of clinker and 5% of gypsum according to BS EN 197 [12]. The physical and chemical properties of the cement and fly ash are shown in Table 1.

Table 1. Physical and chemical properties of OPC and fly ash

	OPC	Fly Ash	ASTM C618, (2012) Requirement (Class F)
Physical Properties			
Blaine Fineness (cm ² /g)	3294	3602	-
Residue 45micron (%)	15.6	20.5	Max 34
Specific Gravity	3.15	2.23	-
Strength Activity Index 7 days (%)	-	76	Min 75
Strength Activity Index 28 days (%)	-	79	Min 75
LOI (%)	1.41	0.59	Max 6
Moisture content (%)	0.2	0.5	Max 3
Chemical Properties			
Silicon dioxide	18.24	49.97	
Aluminium oxide	5.340	28.36	Min Total 70%
Ferric oxide	3.214	7.04	
Calcium Oxide	60.16	5.17	-
Magnesium oxide	0.555	2.07	-
Sulphur trioxide	2.137	0.46	Max 5 %
Sodium oxide	0.039	49.97	-
Potassium oxide	0.5755	28.36	-

All the constituents (cement, fly ash, sand, water, and other additives) were maintained at 25°C to ensure consistent temperature during the final mixing as this could affect the fresh properties. Next, the grouts were mixed with water in a mechanical mixer at 285 rpm. On completion, the rheological tests were immediately performed on the grouts. The result for grout mixture without the fly ash is presented as the control mix. In addition, the water content and superplasticizer (SP) dosage were considered in the selected flow time below 30 seconds as required by ASTM C939 [5]. The required dosage level was selected after conducting the initial flow test with cement grouts containing 0% fly ash and 0.5% of SP. The results showed that the grouts fulfilled the ASTM C939 [5] requirement with flow times below 30 seconds. During each run, all the parameters were kept constant including the water content and superplasticizer in order to understand the effect of fly ash on the rheological properties of the grout.

3. Experimental method

3.1. Particle size distribution

The particle size of the cement and fly ash was determined using a laser diffraction grain analyser (Model: Malvern Mastersizer 2000, Country). The procedure consists of placing 5 g of the test sample into the loading chamber and initiating the testing process. Once the testing was completed, the data was retrieved and plotted.

The particle size distribution of binders such as cement or fly ash greatly influence the fresh and hardening state properties of the material irrespective of the nature of the cementitious material such as concrete or grout [13; 14]. For grouts, in particular, small sized cement particles enhance the timely injection of grouts due to the ease of filling particles inside the voids. In addition, the fine particles of fly ash used with cement-based grout act as micro-filter which improve the grout's properties. The benefit of micro-filler properties to the strength of the material is not limited to fly ash, but also can be seen in other materials such as palm oil fuel ash with the correct fineness as reported by Hafizah [15].

As observed in Figure 1, the particle size distribution of fly ash is comparable to the OPC, which has an average particle size (D50) of 30 μm . Furthermore, the OPC and fly ash have 5.9% and 9.2% of particles above 90 μm , respectively. About 8% of the fly ash particles are coarser than 100 μm while Portland cement has around 5% coarser particle. This is generally not a concern as large particles that do not participate in the pozzolanic reaction, can still act as micro-aggregates in the system resulting in a denser microstructure. However, when the fly ash particle is fine-grained, the reaction with $\text{Ca}(\text{OH})_2$ is greatly enhanced.

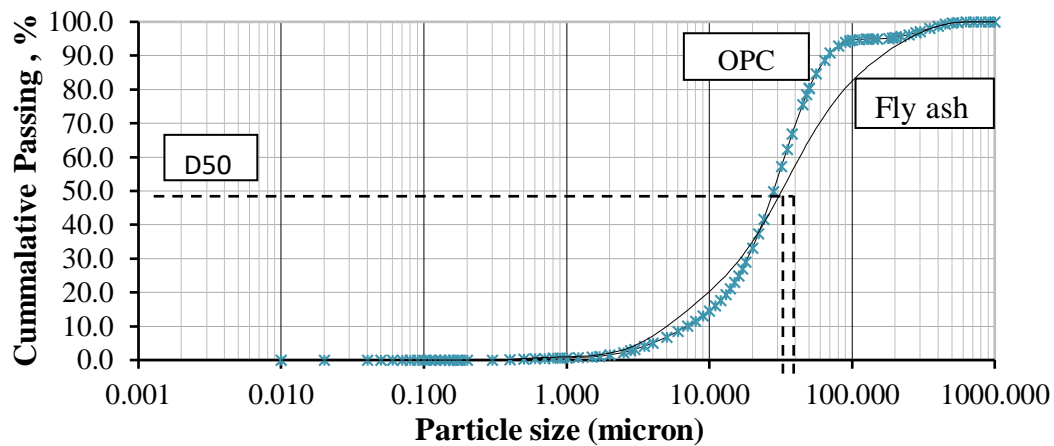


Figure 1. Particle size distribution of fly ash and OPC

3.2. Microstructure of fly ash and cement particles

The microstructural analysis of the fly ash and cement particles was performed using a scanning electron microscope (SEM Model: Hitachi Model S-3400N). The SEM magnification used to examine the morphology was $\times 5000$.

3.2.1. Morphology of the cement and fly ash grain

One important physical property of a binder is its morphology [16; 17]. The grain shapes, as analysed in Figure 2, show that the fly ash used in the study is spherical shaped whereas the cement grain is angular. The shape of particles plays an important role in the rheological behaviour of the grout. In general, the more rounded the shape of the particle, the lower the friction between the particles. Therefore, it is expected that the introduction of fly ash in the grout significantly affects the fresh state properties of grout.

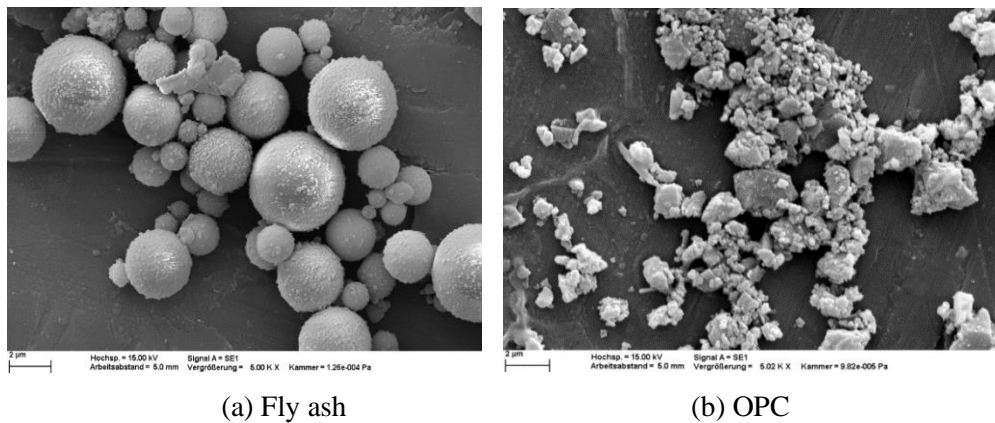


Figure 2. Morphology image of (a) fly ash and (b) OPC particles.

3.3. Fresh state properties

In this study, three different rheological properties (flow time and plastic viscosity) were examined namely for six (6) mixes containing fly ash contents of 0, 10, 20, 30, 40, and 50%. The measurements were performed instantly after the mixing to avoid temperature changes due to cement reactions.

3.3.1. Setting times

The setting time of the grouts was examined using an automated Vicat apparatus. The ring was filled with the grout to flush its top within 2 mins after complete mixing. The timely completion of moulding was recorded as a measurement of time set and the specimen was allowed to remain undisturbed until the test was completed. Next, the 1 mm needle penetration commenced at 15-m intervals until a penetration of 25 mm or less was obtained. This is denoted as the initial setting time whereas the final setting is when the needle can no longer sink into the mix.

3.3.2. Expansion and bleeding

The fresh state expansion and the bleeding of the grout were examined by ASTM C940 [18]. Controlled expansion is important for grout to, ensuring positive surface contact between the grout and the parent material. About 800 ml of the grout was placed into a 1000-ml graduated cylinder and covered to prevent evaporation. Next, the upper surface of the grout and the bleed water were recorded at 15 mins intervals for the first hour, and hourly after bleeding stops. The expansion was decanted as a percentage of the original sample volume whereas bleeding is the volume of the water level above the grout that passed through the expansion.

3.3.3. Flow time

The flow of the grouts was measured according to ASTM C939 [5]. The selected method has a 1700 ml volume capacity. In general, the fluidity of grout, expressed in seconds (s), is measured as the time taken for the filled quantity of grout to completely pass through the orifice of the cone. The flow times of about 2000 ml of grout material from each mixture was tested and the measurements repeated twice to ensure repeatability of the results. The efflux time should be less than 35 seconds according to ASTM C939 [5]. For comparison, it was determined that 1 L of water requires 10 seconds to flow through the cone. The time required for the grout to flow through the cone is related to its viscosity and the flow time increases with increasing viscosity. It is also known that the grouts' ability penetrate obstacles in the flow path without particle blockage is called "filtration tendency". This is typically influenced by the distribution of particle size [19]. Different method of flow cone used to determine the suitability of the test method in the grout measurement.

3.4. Viscosity

For years, it was assumed that cementitious materials such as mortar and concrete are categorized as Bingham materials which can be characterized by viscosity and shear yield measurements. In the

Bingham model [20], the relationship between applied shear stress and rate of shear strain is near-linear with a positive intercept on the stress axis (Eq. 1).

$$\tau = \tau_0 + \mu \dot{\gamma} \quad (1)$$

where τ is shear stress, $\dot{\gamma}$ is shear rate and τ_0 and μ is the Bingham constants of shear yield stress and plastic viscosity. In the event the force brings enough shear stress τ to overcome the shear yield stress τ_0 , Bingham flow will occur and the rate of the shear strain in the flow is proportional to $\tau - \tau_0$. The Bingham equation shows that the apparent viscosity is dependent on the shear rate. However, the existence of yield stress can affect the flowability of non-Newtonian cementitious grout especially for mixtures without vibration. Therefore, the yield stress can be used as an indicator for determining the self-levelling properties of grouts without vibration.

Cementitious materials can also behave like Newtonian fluids depending on the composition of the materials such as the cement and water reducing chemicals [21; 22]. For a Newtonian fluid, the shear stress, τ , is proportional to the shear rate, where the proportionality constant, μ , is the viscosity of the fluid (μ is dependent only on temperature and pressure). The relationship is shown in Eq. 2.

$$\tau = \mu \dot{\gamma} \quad (2)$$

In general, the viscosity is measured using viscometers although some researchers including Pitt [23] Roy and Roussel [24] and Nguyen [25] have investigated the possibility of using a Marsh funnel. This method is frequently used to measure the yield stress at different shear rates.

In the current study, the viscosity of grout was determined using a rotating co-axial cylinder viscometer from Anton Paar. It was used to determine the apparent viscosity at different shear rates. The test was contained in the annular space inside a rotational cylinder of radius 18.42 mm and height 38.00 mm. The rotational cylinder was placed inside a cup containing 350 ml of grout. During each test, the viscosity reading is displayed when the cylinder, rotating at a known speed, exerts a viscous drag on the fluid. The drag creates a torque on the cylinder, which is transmitted to instrument's measurement system to determine the shear stress. The measurement was made at the different shear rates of 1/10, 1/20, 1/30, 1/40, 1/50, 1/60, 1/70, 1/80, 1/90 and 1/100 S. The shear rate was gradually increased and the reading from the viscometer was recorded with increasing rotational speeds. The shear stress reading was recorded 30 s after the change in shear rate.

4. Results and discussion

4.1. Setting times

The measured initial and final setting times, for the six grout mixtures, are presented in Table 2. It can be seen that fly ash grout mixes exhibit longer initial and final set times compared to the OPC mixture. The increase in the setting time increased with higher fly ash content. The control grout without fly ash had an initial setting time of 4 h and final setting time of 5 h 30 m. The setting times for the initial and final increased by 5 h 45 m and 8 hours 30 m for the grout with 50% of fly ash. Although the initial setting time does not change much with the increase in fly ash, the final setting time of the grout increased by around 3 hours compared to the control mix. As observed, the grout with 50% fly ash produced a setting that allowed extra time for transportation and injection of the grout. This can be advantageous in some cases, particularly in hot climates. Similarly, Bentz [26] reported a longer initial setting time for a cement paste mixture containing 60% FA as cement replacement compared to the control. From the above-mentioned studies in this section, it is safe to conclude that the inclusion of high volume fly ash (HVFA) in the mixture prolonged the initial and final setting time. Hence, a fly ash grout will potentially exhibit lower workability losses. This is critical in the area of underground injection where extended working with the grout is required. Currently, the industry uses retarders to extend the working hours which is expansive move to after uses retarders.

Table 2. Setting times of grout with various level of fly ash.

Materials	Fly ash content (%)					
	0%	10%	20%	30%	40%	50%
Initial Setting Time (hours : minutes)	4:00	4:00	4:15	4:30	5:00	5:45
Final Setting time (hours : minutes)	5:30	6:00	6:30	7:00	7:45	8:30

4.2. Expansion and bleeding

Another important parameter of a grout is expansion. This allows enhanced bonding between the substances and removal of voids [27]. In the tests performed, it was observed that the increase in the fly ash content marginally reduced the expansion. This is due to the fact that the expansion agent used in the study is aluminium based which reacts with the cement to release gaseous pockets or air that increase the volume. Lower cement content due to replacement with fly ash accounts for the marginally lower expansion.

As shown in Table 3, the bleeding was found to increase at higher fly ash content. It is generally agreed that fly ash reduces bleeding in concrete whereas the case is reversed in grouts. Nevertheless, the grout with 50% fly ash did not exceed the EN 447 [28] requirement of 2% bleeding. Furthermore, the bleeding did not increase for fly ash content up to 20%. When the fly ash content was between 30% and 40%, the bleeding increased by 1.33%. The minor increase in bleeding is related to the self-consolidating properties of grouts with fly ash, which drives the excess water to the surface. This presents the opportunity to lower the water of the grout resulting in improved mechanical and durability properties.

Table 3. Expansion and bleeding of grout with various level of fly ash

Materials	Fly ash content (%)					
	0%	10%	20%	30%	40%	50%
Expansion (%)	1.33	1.33	1.33	1.33	0.67	0.67
Bleeding (%)	0.67	0.67	0.67	1.33	1.33	2.00

4.3. Flow times

The addition of fly ash reduces the time required for the grout to flow for both cones used. However, the reduction of flow time is not significant until the replacement level was as high as 30%. This scenario is applicable to both types of flow tests. The flow time was around 25 s for the mixes with 0, 10 and 20% of fly ash. But, when the replacement content was at 30, 40, and 50%, the flow time reduced significantly to 22, 21 and 20 s, respectively as shown in Figure 3. In addition, a reduction up to 5 s was noticed on grout containing 50% of fly ash.

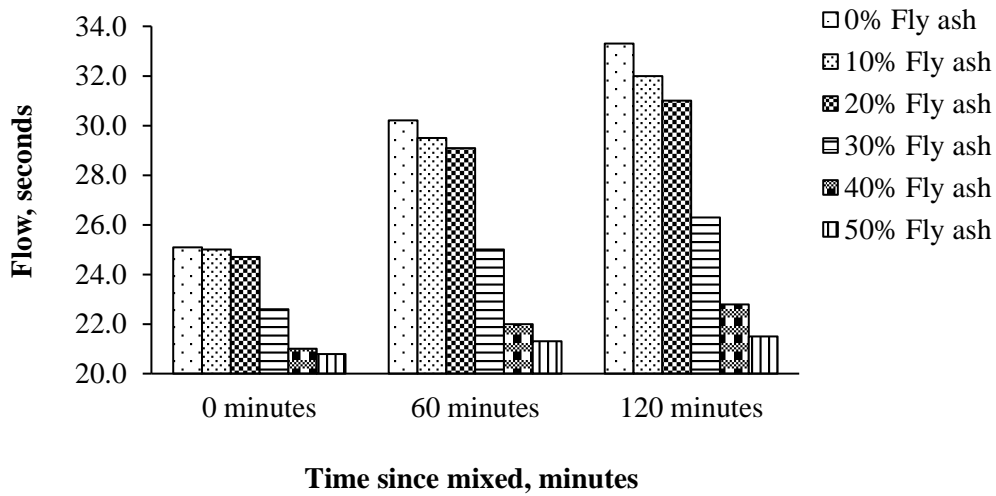


Figure 3. Flow time of grouts at different duration

The increase in flow is in some way interesting as the flow of the control specimen was mainly attributed to the use of a plasticizer. However, without using any additional SP, the flow time can be also reduced by using fly ash. This behaviour was expected due to the morphology of the spherically shaped fly ash which acts as a ball-bearing between particles resulting in faster flow through the cone. Therefore, the use of fly ash not provides technical benefit but also lower the use of SP in the grout. The high dosage requirements and cost of SP can affect the desired level of workability.

4.4. Viscosity

The shear stress diagram is shown in Figure 4. At 0 mins, it can be seen that all the grout mixes behaved like a Bingham fluid. At the low shear rate of 1/10s, there was no marked difference between the grouts shear stress. However, when the shear rate increased, the mix with higher fly ash content, exhibited lower resistance to shear. Hence, the mixes with fly ash showed lower shear stress proportionally to the fly ash content. The viscosity (plastic) obtained from the curve’s slope are summarized in Figure 5. It appears that at a low shear rate, the viscosity is significantly different between the grouts containing 0% and 10% of fly ash compared to the blends with 20, 30, 40, and 50% content. However, as the shear rate increases, although the grout with fly ash has lower viscosity, the differences diminished.

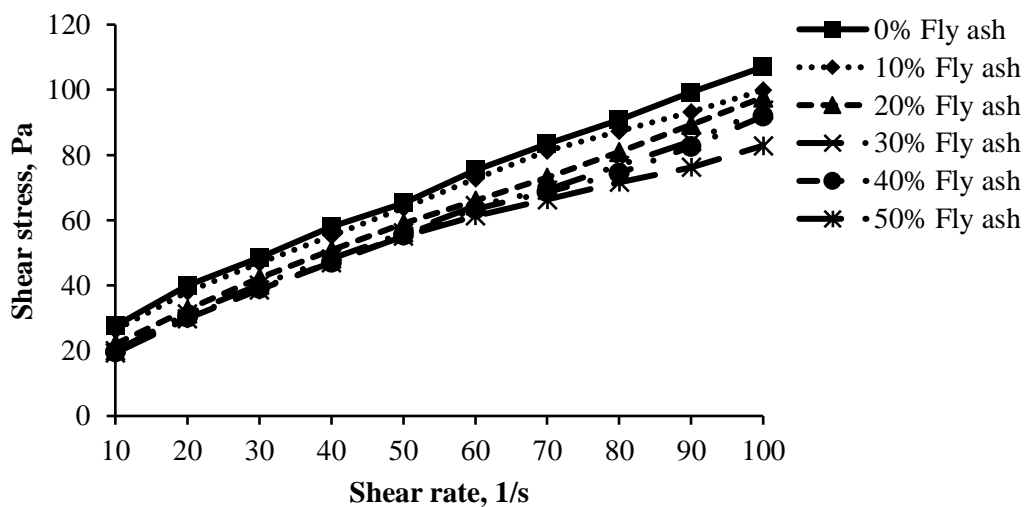


Figure 4. Rheogram of grout with different level of fly ash at 0 mins elapsed time.

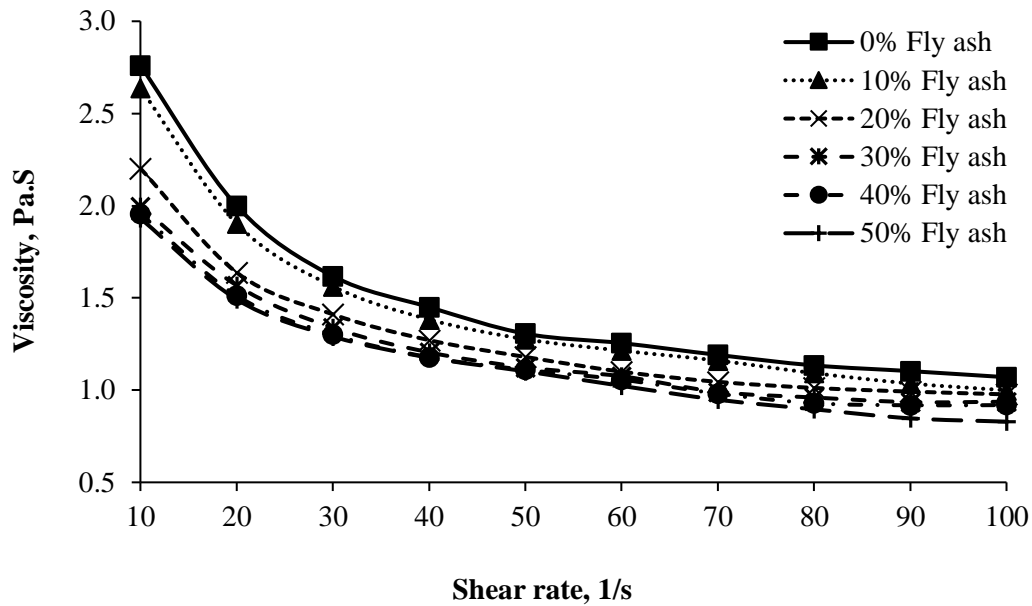


Figure 5. Viscosity of grouts with various level of fly ash at 0 mins elapsed time.

As shown in Figure 6, the shear stress of all the grouts increased at 60 mins. This is due to the increase in rigidity of the grout over time. The increase is due to the bond formation resulting from cement hydration. However, even at this stage, all the mixes still behaved like a Bingham fluid. Over time, the mixes' shear stress becomes more significant compared to grout tested at 0 mins. In general, it can be observed that the grout without fly ash has shown a steeper increase in shear stress compared to the mixes with fly ash. As shown in Figure 7, the mixes with 0% and 10% fly ash have higher viscosity compared to mixes with 20% to 50% content.

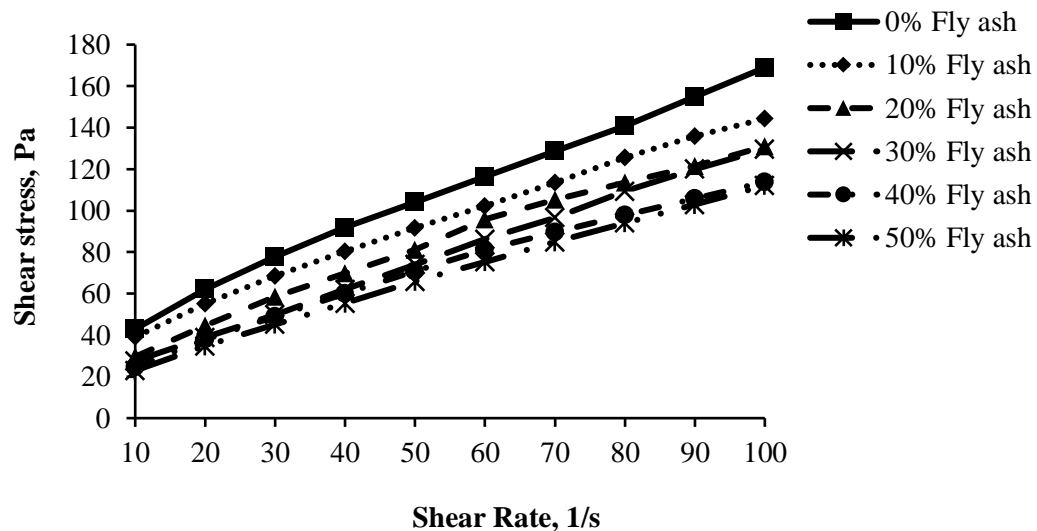


Figure 6. Rheogram of grout with different level of fly ash at 60 m elapsed time.

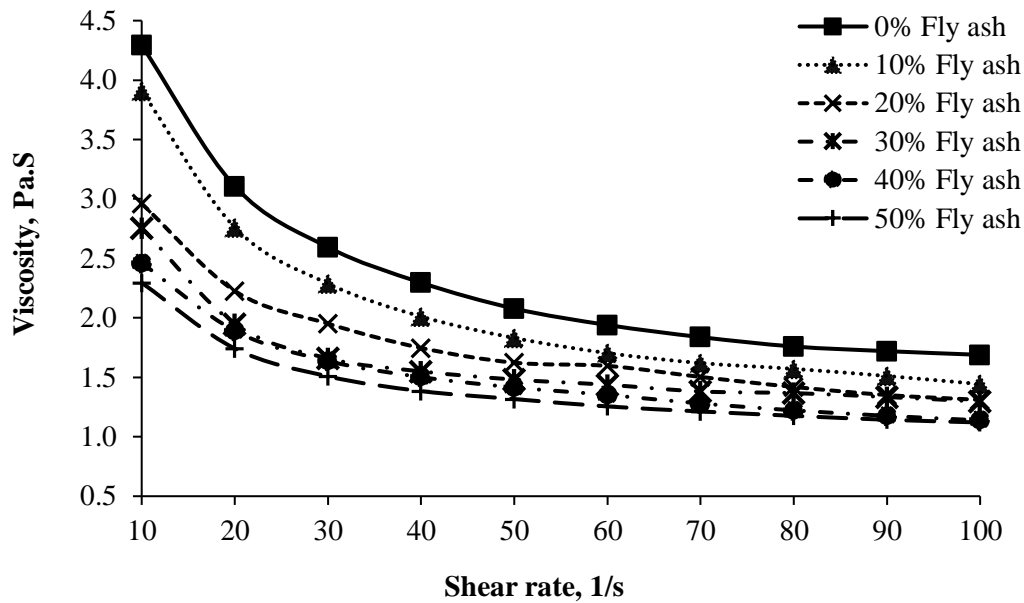


Figure 7. Viscosity of grouts with various level of fly ash at 60 m elapsed time.

The grout viscosity tested at 120 mins showed a different behaviour compared to other fly ash mixes. It was found that the grout without fly ash and grout with 10% fly ash, no longer behaved like a Bingham fluid (Figure. 8), due to the absence of a linear relationship between shear stress and shear rate. This behaviour is known as pseudo-plastic which is typically observed for time-dependent cementitious grouts. This can be observed in the behaviour of the grout with lower apparent viscosity when the shear rate increases (Figure. 9). Brookfield [28] highlighted that for pseudoplastic fluids, the rotation of the spindle will temporarily change the structural molecules of the liquid. This will result in the formation of the molecules parallel to the spindle surface. Therefore, slowing down the spindle rotation will decrease the change in the molecular structure. When the spindle spins faster, a higher fraction of the molecular structure is destroyed resulting in lower molecular coherence and lower viscosity.

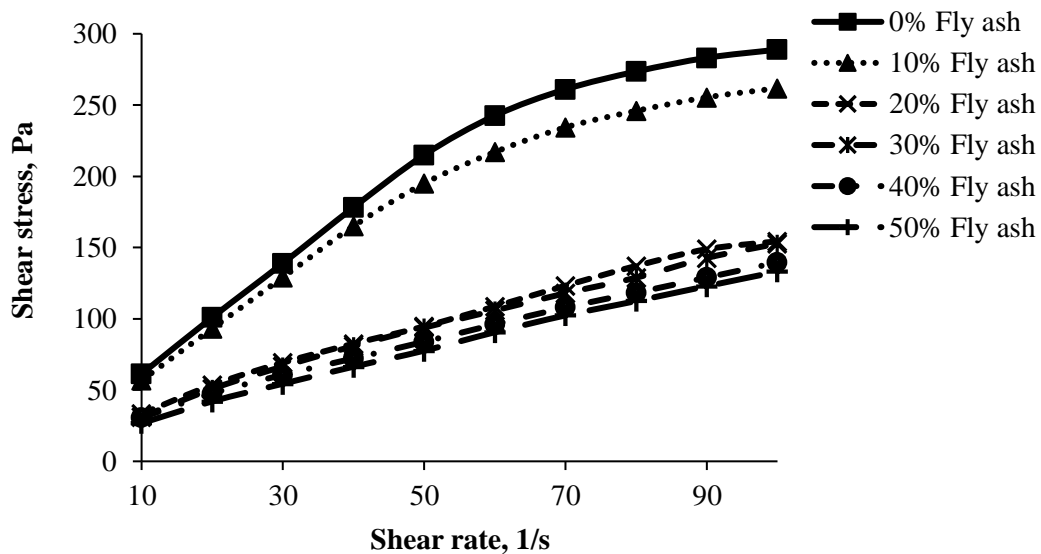


Figure 8. Rheogram of grout with different level of fly ash at 120 m elapsed time.

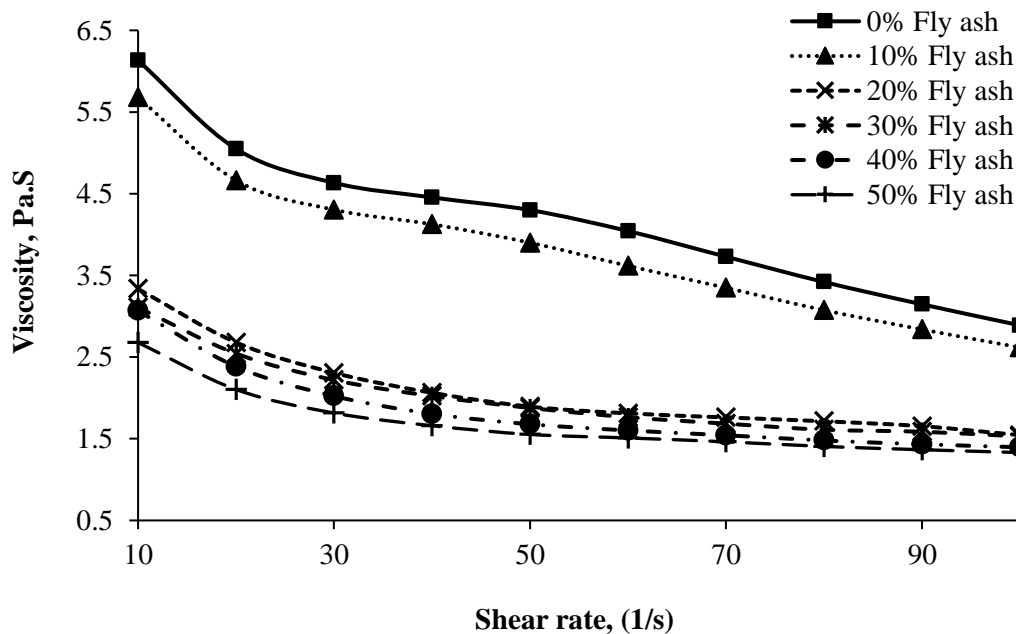


Figure 9. Viscosity of grouts with various level of fly ash at 120 m elapsed time.

However, it is interesting to note that, all the grout with over 20% fly ash, behaved like a Bingham fluid even after 2 h. This finding demonstrates that grout containing HVFA maintained the behavioural attributes of a Bingham fluid. More interestingly, this occurred without structural breakdown over the range of shear rates used in the test after prolonged mixing. The viscosity of grout is time-dependent and directly related to the variation of the flow-strength behaviour with time. Thus, while developing the grout mix, change of viscosity with time must be balanced with stability for efficient functioning. This is because grouts with low viscosity are often preferred for penetrating fine fissures or long distances.

5. Conclusion

It was found that the use of fly ash, particularly in high volume significantly affects the rheological properties of grouts. Hence, the following conclusions can be made:

- The introduction of fly ash reduces the flow time of the grout for all grout mixes.
- The significant reduction in the flow time occurred at fly ash of 30% and above.
- The flow test demonstrated a reduction in flow time. However, the differences were only evident when the replacement level was at 30% or higher. Hence, the proposed method is suitable to test to check the flow of fly ash grouts with high efflux.
- The viscosity of the fly ash grouts showed that the shear stress for all mixes reduced over time. The grouts with HVFA showed lower viscosity compared to OPC grouts.
- The grouts made with OPC and 10% of FA stiffened showing a pseudo-plastic behaviour. However, the grouts with 20% or higher FA maintained the Bingham fluid behaviour. In addition, the

rigidity of grout increased over time. The increase is due to the formation of bonds inside the mixture. However, the introduction of fly ash resulted in changes in viscosity behaviour of the grout which is useful for efficient operation.

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