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Rheological Behavior of Cement Paste: An Experimental Study

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***Abstract*—This paper reports an experimental study into the rheological behavior of cement paste. The investigation aimed at quantifying the impact of the varying amounts of supplementary cementitious materials on the rheology of cement paste without any chemical admixture. Apart from the ordinary Portland cement (OPC), the cementitious materials such as fly ash (FA), ground granulated blast furnace slag (GGBS) and micro-silica (MS) were used in different percentages keeping the mix paste volume constant while testing samples at ambient temperature (22° to 30° C). The rheological properties of cement paste were investigated using an Brook-field Rotational Viscometer RV DV 2+PRO.In this experimental study water/binder ratio: 0.45 was used with cement paste samples. Also time dependency of the rheological properties of cement paste investigated. The instrument Brook-field Viscometer DV 2+PRO employed in present study for evaluating the rheological behavior of cement paste to detect systematic changes in flow curve with cementitious materials to calculate yield stress and plastic viscosity. It can be concluded that the rheology is effected by w/b ratio and time duration after mixing of cement samples for predicting the flow behavior of cement paste made with different cementitious materials.The cement paste rheological data also compared using simple test, the mini slump. The conclusion is that mini slump test is reliable for certain cases to co-relate slump flow with yield stress. The results for optimum material quantities could be used as a guide for initial trial mixes, minimizing the time and material wastage.**

Keywords*—Cement Paste(OPC); rheology; viscosity; ground granulated blast-furnace slag (GGBS); fly ash (FA); micro silica (MS); Brook-field Viscometer DV 2+PRO.

I. INTRODUCTION

Fresh cement pastes are highly concentrated suspensions; their rheological behavior is generally very complex and is dependent on several factors of different nature, such as:(i)physical factors (the water/binder ratio,the cement grain shape and size, etc.),(ii)chemical and mineralogical factors (the cement composition and its structural modification due to hydration processes, etc.),(iii)mixing conditions (stirrer type and rate, the stirring time, etc.),(iv)measurement conditions (the measuring instruments and the experimental procedures, etc.),(v)presence of additives (water reducing agents, superplasticizers).The science focusing on the deformation and flow of matter, and the emphasis on flow means that it is concerned with the relationships between time, strain, stress and rate strain is referred to Rheology [1].Therefore, studies based on rheology are much more important to evaluate the functionality of cement paste. The rheology is important because of the scope

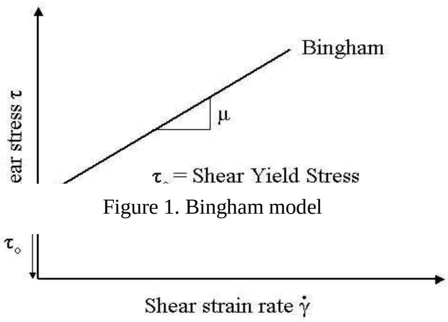

Figure 1. Bingham model

It offers for characterizing fresh cement paste, and for understanding as to how they perform in practical applications. Rheology is dominated by the structure that exists in the cement paste, which is the only binding matrix in concrete system. The rheological behavior of cement paste is characterized by parameter τ_0 and μ as defined by Bingham material model which shows good correlation with experimental data and is defined by equation[1]:

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

where τ is the shear stress applied to the material(in Pa),τ_0 is the yield stress(in Pa),μ is the plastic viscosity(in pa-s) and γ is the shear strain rate(in s^{-1})[2].Fig. 1 elaborates the Bingham model, which gives the simplest relationship of all. Rheological properties of a paste could be simply defined using the Bingham constants; Yield shear stress and Plastic viscosity.

II. REVIEW OF LITERATURE

There have been many research works involving the studies on the rheological behavior of cement paste but effect of using SCM on the thixotropic properties were discussed only in limited number of studies. Moreover, the potential benefit of using various amounts of SCM in binary combinations with OPC on rheological and thixotropic properties of cement paste is not well documented. Thus, the effects of different blends of SCM on rheological properties as well as flow ability of cement pastes are studied. Tattersall and Banfill [3] investigated the structural breakdown as follows: When cement particles and water come together, a hydrate membrane immediately covers and links the particles. If the cement paste is sheared, the linkages between them may be broken, separating the particles. The breaking of linkages was considered to be an irreversible process and thus non-thixotropic [4]. There are several ways of studying the thixotropy and structural breakdown. By gradually increasing and then decreasing the angular velocity of the viscometer spindle, the corresponding torque values will form a hysteresis loop if the mixture is thixotropic. The area between the up and down curves can quantify the thixotropy [3,5,6]. Wallevik [7] studied, by both his model and experimental work, that the time-dependent behavior is governed by both thixotropy (combination of coagulation, dispersion and re-coagulation of the cement particles) and structural breakdown (breaking of chemically formed linkages between the particles). Barnes [8] studied Thixotropy as a gradual decrease of the viscosity under shear stress followed by a gradual recovery of structure when the stress is removed. Geiker [9] researched that suspensions of cement in water exhibits dispersion or coagulation at changing shear rate. The composition of cement paste and total content of water were found to affect duration of relaxation period in mix. Total time at each shear rate should be long enough to obtain steady state, but as short as possible to limit the segregation. BML Viscometer was used to study torque curve. Wallevik [10] reported that Particle Flow Interaction theory had the potential to produce the rheological response of cement paste under more complex shear rate condition. Three concepts thixotropic behavior, thixotropic breakdown and structural breakdown were investigated.

Ferris et al [11] reported that,if the volume concentration of a cement paste is held constant, the addition of mineral admixtures improves cement matrix performance but reduces workability. The most common reason for poor workability is that the addition of fine powder will increase the water demand due to the increase in surface area. Ahari [12] investigated that class F fly ash has a spherical morphology and a smooth surface texture of grains, leading to a higher ball bearing effect to reduce inter-particle friction. Therefore, it reduces the plastic viscosity and enhances the workability. Other than that, low porosity of particles and a lower fineness has contributed to the low HRWRA demand. In contrast, class C fly ash contains irregular grain shapes reducing some of these benefits. Malhotra and Mehta [13] stated that using cementitious material it was possible to have a favorable influence on many properties of cement paste with presence of very fine particles or physio-chemical effects associated with pozzolanic and cementitious reaction which result in pore-size reduction and grain–size reduction phenomena. The use of FA in OPC reduces the water requirement to obtain the given consistency. Water reduction caused by FA was attributed to very fine particles adsorbed on oppositely charged surface of cement particle and prevent them from flocculation. Therefore, cement particles are effectively dispersed. Lomboy [14] reported that in cementitious pastes, replacement with class C fly ash decreases the values of the rheological parameter while replacement with GGBS and MS increases these values.

Z. X. Zhang and J. Han [15] after testing very fine additives (granulated blast furnace slag, micro silica, ashes) effect on cement slurry rheological properties have found, that while increasing quantity of such additives, cement slurry yield stress most often are decreased. But slurry viscosity depends on the additives type and quantity. Very finely ground slag reduces slurry viscosity efficiently, when the additive is added by more than 15 % of cement mass. Slag grinding technology has great influence on GGBS cement slurry rheological properties.

H. Wan, Z. Shui, Z. Lin [16] investigated granulated blast furnace slag granulometry and particles shape influence on cement properties. Out of the test results we can see, that in the slag ground in air stream mill particle diameter is dispersed in a narrow interval, and when granulated slag was taken, after grinding it in a ball mill, the dispersion was in a large interval. Most of the particles gained of slag ground in vibrating mill have got a spherical shape and they are characterized by a smooth and plane surface. At the same time while grinding in ball and air stream mills, the particles are angulated and sharp edged. Slump of the cement pastes that were prepared using granulated blast furnace slag depended on the slag particles size dispersion, i.e. if particle size of granulated slag was dispersed in a narrow interval, the cement paste were characterized by greater slump.

Diab et al. [17] reported that Micro Silica has been recognized as a pozzolanic and cementitious admixture which is effective in enhancing the mechanical properties to a great extent. The pozzolanic reaction results in a reduction of the amount of calcium hydroxide in cement paste, and Micro Silica reduces porosity and improves durability. It accelerates the dissolution of C-S and formation of C-S-H with its activity being inversely proportional to the size, and also provides nucleation sites for C-S-H. It is responsible for an additional increase in strength and chemical resistance and decrease in water absorption. Malhotra and Mehta [13] reported particle packing effect responsible for water reduction. The physical effect of particle packing by MS would reduce void space and correspondingly water requirement for plasticizing system. Very fine cement particle should also behave in the same way, but they tend to dissolve rapidly in water so ineffective as space filler. The MS particles were relatively inert and should be effective. Haist and Muller [18] investigated that quality of cement paste was significantly influenced by properties at fresh state which could be adjusted by using organic or inorganic additivies, not only systematic approach in research or development but also in quality control. Empirical test method do not provide relevant information. Modern rheometry allowed in-depth characterization of elastic and viscous properties of freshly prepared cement paste suspension.

III. AIMS AND OBJECTIVES OF INVESTIGATION

On the backdrop of the afore-mentioned review of literature, the present investigation investigate the study of the rheological behavior of OPC in conjunction with different cementitious materials like FA,GGBS and MS by differential weight% for water/binder ratio:-0.45.To study Non-newtonian behavior of cement paste samples, instead of using shear stress and shear rate the torque and rotational speed are used as relative parameters for plotting flow curve using viscometer [18,19,20].The investigation is extended to study the time behavior effect on these samples for the rheological properties. The investigation does not entail the use of chemical admixtures. In a nutshell, the investigation aims at quantifying the effect of the varying amounts of cementitious material on the rheological behavior of cement paste.

IV. MATERIALS AND PROPORTIONS OF MIXES

In the present work, cement used was ordinary Portland cement (53 grade) confirming to IS 12269 whose chemical composition is described in Table I. This cement was used for

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all the rheological test of cement paste. Chemical compositions of the cementitious materials used in this investigation are also shown in Table I. The cement paste composition was varied to explore the influence of supplementary cementitious materials dosage and type, on the rheological properties. The performance differences in the pastes due to the replacement of SCM by weight%, were determined by measuring their rheological properties at w/b ratio-0.45, keeping volume of the mix paste constant while testing samples. All the measurements were taken at ambient temperature (temperature of cement paste varying between 22^{o} to 30^{o} C). The additional details on the mix proportions are provided in Table II.

Table II shows mix proportion used in present investigation. Composition I (control mix) of cement paste is designed only with Ordinary Portland Cement. Composition II is designed mixing 80% of OPC and 20% of Fly ash by wt% of paste volume. Composition III is designed mixing 60% of OPC and 40% of Fly ash. The content of GGBS is 40% in composition IV while 70% in composition V by wt% of paste volume.5% of MS is replaced in composition VI with OPC while 7.5% in composition VII with wt% of paste volume. Other than supplementary cementitious materials, no chemical admixtures are not used in present study.

Some of the most common tests used to evaluate the characteristics of cement paste are: the mini slump test and viscometer or rheometer test. Out of these, the most convenient and simple test is the mini slump test which is an indirect test used to estimate the yield shear stress and plastic viscosity of flowable paste. However, the geometry of the cone largely affects the test results [19]. The other common apparatus used is the rotational viscometer, which is a direct method of measurement. However, the results depend highly on the accuracy of measurement and the geometry of the cylinder.

TABLE 1. CHEMICAL COMPOSITIONS OF THE CEMENTITIOUS MATERIALS USED (WT. %)

COMPOUND	OPC	FA	GGBS	MS
SIO	21.4	68.1	33.79	88.9
$A1_2O_3$	4.3	25.8	20.73	0.4
FE_2O_3	2.4	6.9	1.12	0.4
CAO	64.4	8.7	31.06	1.6
MGO	2.1	1.39	11.23	-
SO_3	2.3	0.26	0.10	0.4
NA_2O	0.60	0.18	0.30	0.5

TABLE 2. MIX PROPORTIONS FOR LABORATORY TRIALS

Composition	OPC (%)	FA (%)	GGBS (%)	MS (%)	w/b ratio(%)
I.	100	-	-	-	45
II.	80	20	-	-	45
III.	60	40	-	-	45
IV.	60	-	40	-	45
V.	30	-	70	-	45
VI.	95	-	-	5	45
VII.	92.5	-	-	7.5	45

There, are several equations obtained to relate the Bingham constants with the viscometer readings (torque and rotational velocity). There have also been attempts to relate the mini slump test results with the viscometer test results [19].

V. MIXING PROCEDURES AND APPARATUS

The cement paste preparation is very important because the shear history of a mixture will influence its rheological behavior. In the presence of only cement, water and supplementary cementitious materials, the approach to mixing is different than the concrete, due to the absence of aggregates. The ASTM C1738/C1738M-14, provides the guidelines to carry out the tests. As stated in the standard, the method has been useful in testing the rheology of the paste because it gives similar results to those obtained in a concrete mix where the aggregates have been removed. Mixing of paste does not falls under ASTM C305, since it should not be thoroughly mixed due to the absence of sand. This practice is known as high shear mixing, because, it imparts a significantly higher amount of shear than in ASTM C305.

A. Brook-field Viscometer

The Bingham constants were evaluated using a Brook-field rotational type viscometer RV DV 2+PRO model. This is a direct test method of obtaining yield shear stress and plastic viscosity. The test was performed according to the guidelines given in ASTM C1749- 12: "Standard Guide for the measurement of rheological properties of a hydraulic cementitious paste using a rotational viscometer [20]. The Brook-field rotational viscometer was used to determine the yield stress and plastic viscosity as defined by bingham model. The spindle of viscometer should be selected based on the type of fluid based on trial and error method. For present investigation number 04 spindle is selected. All samples were pre-sheared for 5 minute at 100 rpm. This reduced substantially the torque; close enough to plateau value to consider the sample to have been well preconditioned in which a steady state had been reached. The rotation frequency was then reduced at spaced steps (100, 50, 20, 10, 5 &1 minute), each step lasting for 5 minute. The expected variation of torque with the rotational velocity is indicated in the Figure 2.

It is generally agreed that during a rheology test, the attractions and agglomerations of the particles in the tested sample are broken down during the period of the increased shear rate (up curve of the test result).

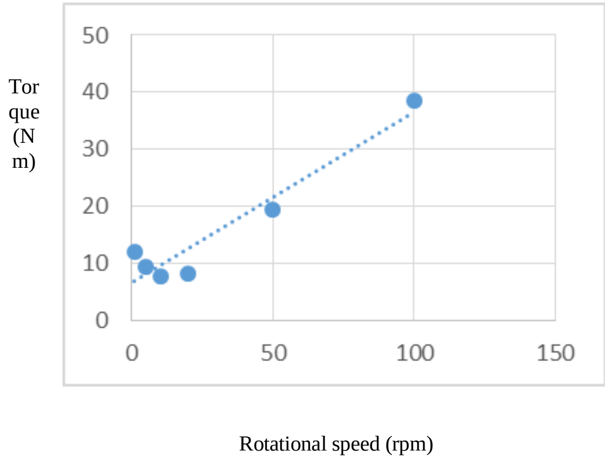

Figure 2. Viscometer observation and trend line

After the attractions and agglomerations of the particles in the tested sample have fully broken down, the rheological behavior of the tested material generally agrees with the Bingham model, as shown by the down curve of the test result. Due to the complicated flow pattern applied to the tested cement paste by the spindle, it is difficult to obtain the exact value of shear stress and shear rate of the tested cement paste. Therefore, the torque and the speed of the spindle were reported during the cement paste rheology tests and their relationship was plotted. The following equation was used to fit the down curve from a Viscometer rheology test based on Bingham model:

$$T = G + H \times N \quad (2)$$

Where T was the torque acting on the spindle and N was the rotation speed of the spindle. G was the interception of the linear portion of a Viscometer down curve and the y-axis. H was the slope of the linear portion of the Viscometer down curve. Direct calculation of yield stress and plastic viscosity in fundamental units is not possible due to the complicated flow pattern, the down curves of the torque-rotation speed were found to fit well with Bingham model, The slope (H) and interception (G) obtained from Viscometer measurements were considered to be directly proportional to yield stress and viscosity of cement paste respectively. In the present paper, G is therefore used to describe the yield stress and H is used to describe the plastic viscosity of the tested cement paste [18, 19, 21].

VI. RESULTS AND DISCUSSION

The parameters obtained in view of the rheology of fresh cement paste using supplementary cementitious materials such as fly ash (FA), ground granulated blast furnace slag (GGBS) and micro-silica (MS) are discussed below. Composition I is prepared from Ordinary Portland Cement which is control sample(No supplementary cementitious materials added) to compared with other composition to study rheological as well as flow property. It is observed that control sample shows linear increasing in variation for yield stress as well as for plastic viscosity as test time elapsed.

A. *Effect of fly ash on rheology*

Rheological parameters for increasing weight percentage replacement of fly ash are study in this investigation. Composition II & III are prepared with 20% and 40% replacement level with OPC. Figure 3.shows yield stress variation with time for increasing weight percentage level of fly ash. After mixing of samples, initially composition II result in 43.40% increase in yield stress while composition III show 3.40% decrease in yield stress as compared to control sample. After 45 minute time interval, 8.46 and 2.86% of increase in yield stress can be calculated for composition II and III respectively. At 90 minute interval, yield stress of composition II and III are decreases to 23.69% and 18.86% respectively. It is observed that composition II shows approximate constant value for yield stress as test time elapsed.

Figure 4.shows plastic viscosity variation with time for increasing weight percentage level of fly ash. After mixing of samples, initially composition II and III result in decrease in plastic viscosity value by 20.86% and 3.60% respectively. After 45 minute of time interval, 5.13% and 20.82% decrease in plastic viscosity is observe in composition II and III respectively.

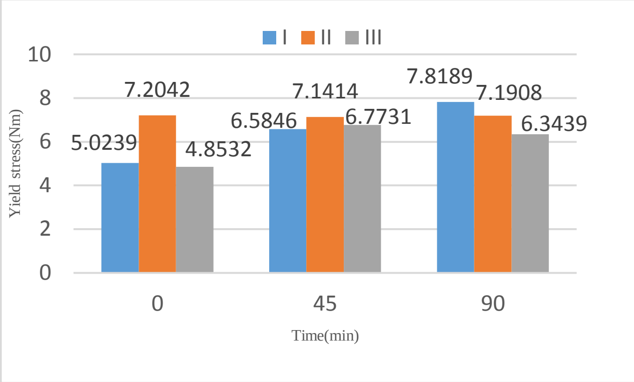

Figure 3. Yield stress variation with test time elapsed

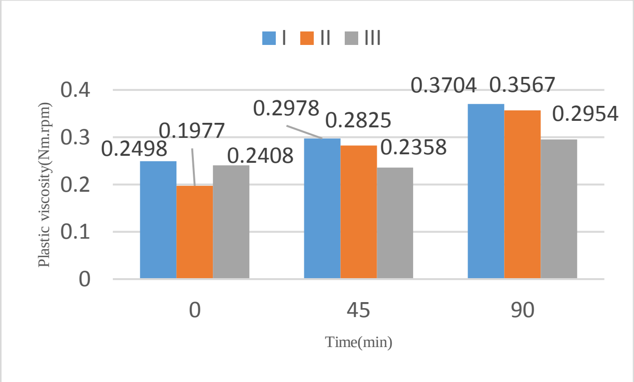

Figure 4. Plastic viscosity variation with test time elapsed

Further at 90 minute interval, decreasing variation in plastic viscosity is observe with values 3.70% and 20.25% for Composition II and III respectively. It is observed that plastic viscosity variation is linearly increasing for composition II as test time elapsed.

B. *Effect of ground granulated blast furnace slagon rheology*

Rheological parameters for increasing weight percentage replacement of ground granulated blast furnace slag are study in this investigation. Composition IV & V are prepared with 40% and 70% replacement level with OPC. Figure 5.shows yield stress variation with time for increasing weight percentage level of ground granulated blast furnace slag .After mixing, initially composition IV & V result in 14.49% and 143.32% increase in yield stress compared to control sample. After 45 minute time interval, composition V show 55.40% increase in yield stress while composition IV show 1.94% decrease in yield stress. At 90 minute interval, composition IV show 23.69% increase in yield stress while composition V show 24.57% decrease in yield stress. It is observed that yield stress variation is linearly increasing for composition IV while linearly decreasing for composition V as test time elapsed.

Figure 6.shows plastic viscosity variation with time for increasing weight percentage level of ground granulated blast furnace slag. After mixing of samples, initially composition IV result in increase in plastic viscosity value by 13.61% and decrease in plastic viscosity value by 19.62% for composition V. After 45 minute of time interval, 27.73% and 44.80% increase in plastic viscosity is observe in composition IV and V respectively. Further at 90 minute interval, increasing variation

in plastic viscosity is observe with values 17.09% and 30.21% for

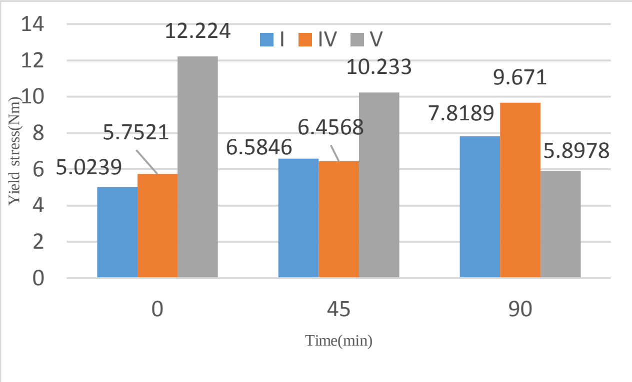

Figure 7. Yield stress variation with test time elapsed

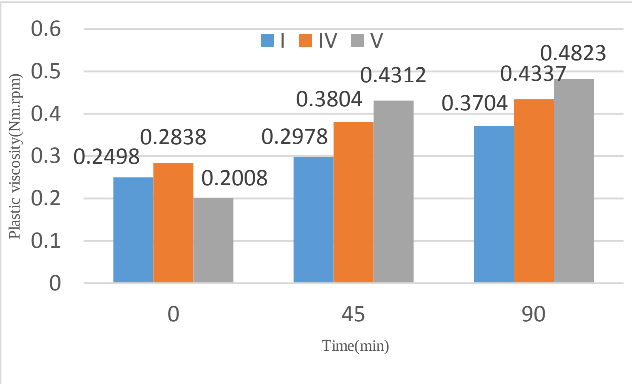

Figure 8. Plastic viscosity variation with test time elapsed

Composition IV and V respectively. It is observed that plastic viscosity variation is linearly increasing for both composition IV and V as test time elapsed.

C. Effect of micro silica on rheology

Rheological parameters for increasing weight percentage replacement of micro silica are study in this investigation. Composition VI & VII are prepared with 5% and 5.7% replacement level with OPC. Figure 7.shows yield stress variation with time for increasing weight percentage level of micro silica. After mixing, initially composition VI & VII result in 464.10% and 258.39% increase in yield stress compared to control sample. After 45 minute time interval, composition VI show 37.94% increase in yield stress while composition VII show 55.61% decrease in yield stress. At 90 minute interval, composition VI show 75.77% increase in yield stress while composition VII show 32.88% decrease in yield stress. It is observed that composition VI and VII, yield stress is decreases at 45 minute time interval which again increases at 90 minute time interval though not to original level as test time elapsed.

Figure 8.shows plastic viscosity variation with time for increasing weight percentage level of micro silica. After mixing of samples, initially composition VII result in decrease in plastic viscosity value by 26.66% while control sample and composition VI have same plastic viscosity. After 45 minute of time interval, 96.07% and 47.08% increase in plastic viscosity is observe in composition VI and VII respectively. Further at 90 minute interval, increasing variation in plastic viscosity is observe with values 79.02% and 38.34% for Composition VI and VII respectively. It is observed that plastic viscosity variation is

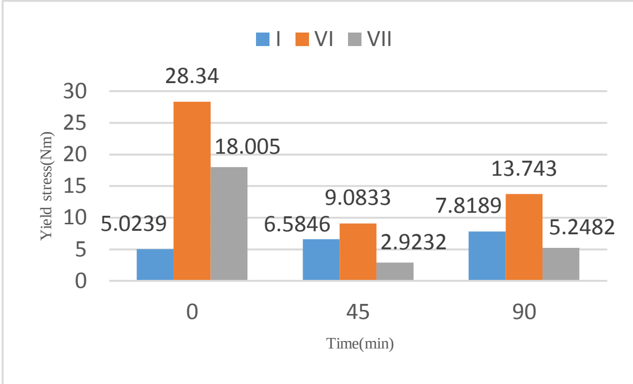

Figure 7. Yield stress variation with test time elapsed

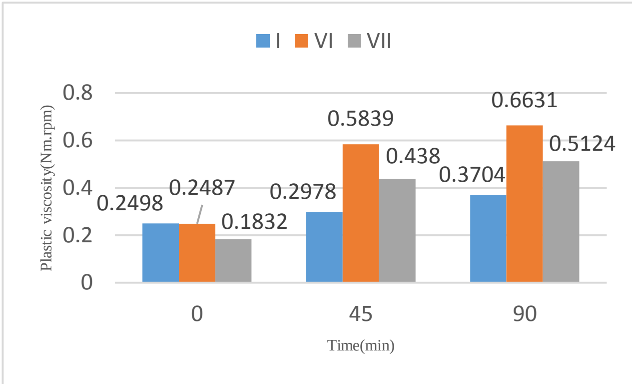

Figure 8. Plastic viscosity variation with test time elapsed

linearly increasing for both composition VI and VII as test time elapsed.

D. Relation between mini slump flow and Viscometer yield stress

Fluid viscometer for cement paste is not widely available in the construction industry because of two main reasons is: (I) the instrument is relatively expensive and (2) the knowledge of using such a device for cement paste is limited. Therefore, it is advantageous to use simple test such as the mini slump test. The plot of yield stress vs. mini slump spread diameter(Fig. 9) shows

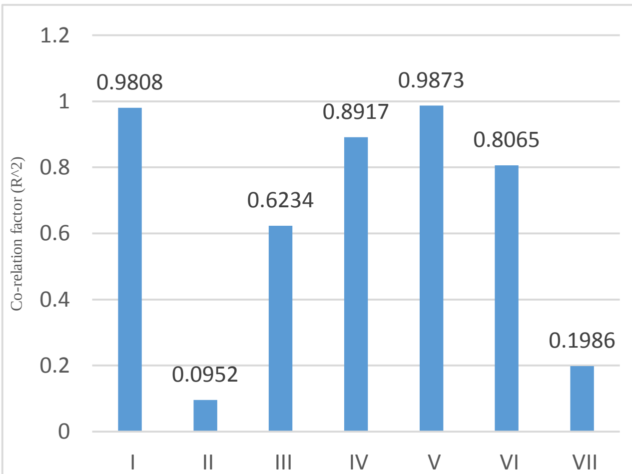

Figure 9. Co-relation factor for yield stress and spreaddiameter of mini slump flow

a good correlation for all compositions except composition II (20% replacement by fly ash) and VII (7.5% replacement level by micro silica: higher yield stress corresponds to a lower spread in the mini slump test. This result shows that the cement paste in a mini slump test will only flow if the stress due to the weight of the cement paste contained in the cone is high enough i.e., higher than the yield stress of the cement paste.

VII. SUMMARY AND CONCLUSIONS

An experimental study was carried out to study the effect of various cementitious materials on the rheological behavior of cement paste. The cementitious materials such as fly ash, ground granulated blast furnace slag and micro-silica in conjunction with OPC were used in different percentages. Cement paste is binding matrix in concrete, which affects the pumpability of resulting concrete provided other parameters remains same it was found that FA highly improve rheology followed by GGBS and MS compared to control sample. It was observed that the replacement of cement by MS results in an increase in the water demand and requires high range water reducer dosage to maintain same the rheological properties. The possible reason for increasing in the rheological parameter is its extreme fineness and very high amorphous silicon dioxide content, which make MS highly reactive pozzolanic material while FA having lower silicon dioxide content makes FA less reactive. In contrast the replacement of cement by FA resulted in a reduction of the water demand hence lower HRWR dosage to maintain the same rheological properties. GGBS showed intermediate properties in between FA and MS.

The instrument RV DV 2+PRO Rotational Viscometer employed in the present study for evaluating the rheological behavior of cement paste to detect systematic changes in flowability, i.e., flow curve to determine yield stress and plastic viscosity behavior with time. The cement paste containing OPC and cementitious materials like FA, GGBS, MS demonstrates different rheological behavior which will guide the concrete engineers in arriving upon a decision for design of proper pumpable and economical concrete design.

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