



Study of Variability in the Results of the Marsh Cone and Mini-slump tests

Ashwini Mahisbadwe^{1*}, Ravindra Gettu², C. Jayasree³

¹M. Tech Graduate, Indian Institute of Technology Madras, Chennai, Tamil Nadu, India.

²V.S. Raju Chair Professor, Civil Engineering Department, Indian Institute of Technology Madras, Chennai, Tamil Nadu, India, Email: gettu@iitm.ac.in

³Former Research Scientist, Kuwait Institute for Scientific Research, Safat, Kuwait. Email: jchakkolath@gmail.com

*Corresponding author

ABSTRACT

The flow behavior of cement paste depends significantly on the type and dosage of the superplasticizer used. The Marsh cone and mini-slump tests of the cement paste are simple and easy tests for assessing the flow properties of cement paste. In this paper, the ability of these tests to replicate the results under constant execution conditions has been studied. The variability introduced by inherent random errors during the testing process has been quantified in terms of repeatability values for these tests. The test results confirmed good repeatability, indicating that the test methods are robust.

Keywords : *Marsh cone, Mini-slump, Optimum superplasticizer dosage, Repeatability values*

1. Introduction

The incorporation of mineral and chemical admixtures in the cement paste composition results in a balance between the cohesion and fluidity of cement-based materials such as cement grouts, underwater concrete, and self-compacting concrete (SCC) (Aïtcin, 1994, 2019; Lachemi et al., 2004, 2007). SCC is characterized by its ability to fill the formwork and consolidate under its own weight. Also, SCC possesses the ability to pass through congested reinforcement. In spite of high flowability, SCC demonstrates excellent segregation resistance. These fresh state properties of SCC therefore largely depend on the properties of the cement paste. Hence, the fluidity studies of cement paste are crucial in order to achieve the performance criteria. The fluidity tests on cement paste have been conducted using viscometers over a period of time. But these viscometers are expensive and require skill for operation and their application is limited to sophisticated laboratories. This necessitates the need for simpler tests, such as Marsh cone and mini-slump tests, easy to perform even at the job site (de Larrard et al., 1998; Agullo et al., 1999; Giaccio, 2002; Roussel et al., 2004, 2005; Jayasree and Gettu, 2008, 2010; John and Gettu, 2014; Chakkamalayath et al., 2022).

However, during any testing operation, certain inherent unavoidable random errors are introduced. As a result, no two results of the test conducted on similar materials under almost identical testing conditions are found to be the same. The present study focuses on the aspect of variability introduced during the testing of cement paste using the Marsh cone and mini-slump tests. The variability is expressed in terms of measures of precision like the repeatability value (r). Certain factors like the operator, equipment, calibration of the equipment, the environmental conditions and the time lapsed

between the measurements introduce errors. Although these factors are maintained constantly, there still exist some variability in the results. Variability obtained under these conditions is quantified and is termed as repeatability by ISO-5725 (Part 1-6): 2023 and IS 15393 (Parts 1-6): 2003 which is the standard deviation obtained under conditions of repeatability.

2. Experimental details

The Marsh cone and mini slump tests used in this study are simple empirical methods to characterize the flow behaviour of superplasticized cement paste.

2.1 Materials

53 grade Ordinary Portland Cement (OPC) was used throughout the study. SP1, a new generation polycarboxylic ether based superplasticizer was used for the experiments; the solid content of which is 33 %; confirmed in the laboratory following the guidelines prescribed in IS 9103:1999. The specific gravity of SP1 is 1.03 kg / litre and the recommended dosage is 0.5 to 1.6 litres per 100 kg of cement. SP2, a synthetic polymer-based superplasticizer, with a solid content of 44 % was also used in the study. Three combinations, water to cement ratio (w/c) = 0.35 and SP1; w/c = 0.4 and SP1; w/c = 0.35 and SP2 were studied. Figure 1 shows the structure of the experiments.

2.2 Marsh cone test of cement paste

The Marsh cone is an inverted hollow metal cone with fabricated using non-corrosive material, open at the top and has an aperture at the bottom. The dimensions of the cone used for the study are shown in Figure 2.

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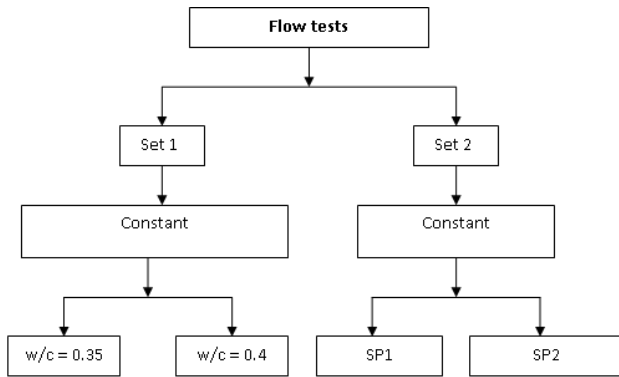


Figure 1: Experimental structure for the Marsh cone and mini-slump tests

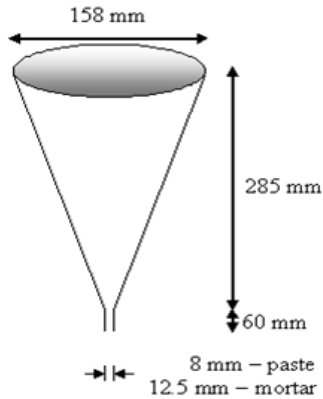


Figure 2: Geometry and dimensions of Marsh cone.

The Marsh cone has been used to study the fluidity of cement pastes and mortar, to determine the optimum superplasticizer (SP) dosage, and to study the loss of fluidity with time. Certain volume (V) of cement paste is poured into the cone and the time required for a fixed volume to flow through the aperture is measured. Thus, it acts as an inverse measure of fluidity. Higher the fluidity, lower is the flow time and vice versa. In the present study, the Marsh cone has been used for the determination of the saturation dosage of the superplasticizer (Roncero et al., 2000; Jayasree and Gettu, 2008). Addition of superplasticizers improves the fluidity. But beyond a certain superplasticizer dosage no significant improvement in fluidity can be seen. This dosage is termed as the saturation dosage. The Marsh cone test has been used for the objective determination of the optimum dosage of superplasticizer.

2.2.1 Test Procedures

Conditions like the operator, equipment and environmental conditions were maintained constant to carry out the variability studies. All the material was placed in the environmental chamber for 24 hours at 27°C and at a relative humidity of 65 %. No mineral admixture was used in this study. The superplasticizer dosage is expressed as the ratio of solid content of the superplasticizer to the cement content by weight and is denoted as sp/c (%). Hobart mixer was used for mixing the cement paste. The cement paste was mixed at a speed of 60 rpm. Distilled water was used for mixing the paste.

Entire cement was added to the mixing bowl and mixed with 70 % of the total water requirement for 2 minutes in the Hobart mixer at a speed of 60 rpm. The mixing bowl and the blade were scraped, and the remaining water and superplasticizer were added to the partially mixed cement paste. It was mixed for another 3 minutes at the same speed. 1000 ml of the paste was measured in a measuring cylinder. The nozzle at the bottom of the cone was closed with finger and the paste was poured into the cone. The nozzle was opened, and simultaneously a stop watch was started and the time taken for 500 ml to flow through the cone was measured. The superplasticizer dosage was increased and the above-mentioned procedure was repeated.

A draft proposal for the methodology to be followed to conduct the Marsh cone test has been sent to the cement and concrete sectional committee CED 02 of Bureau of Indian Standards to develop it as an Indian standard to standardize the particular test method.

The method proposed by Gomes et al. (2002) was followed for the objective determination of the saturation dosage. The superplasticizer dosage as expressed by Gomes et al. is the ratio of the solid content of the superplasticizer to the mass of cement. A plot of the sp/c (%) as the abscissa and the log (Marsh cone flow time) as the ordinate is shown in Figure 3. The internal angle was calculated at every data point. The superplasticizer dosage corresponding to the data point with an internal angle of $140^\circ \pm 10^\circ$ was considered as the optimum dosage as shown in Figure 3.

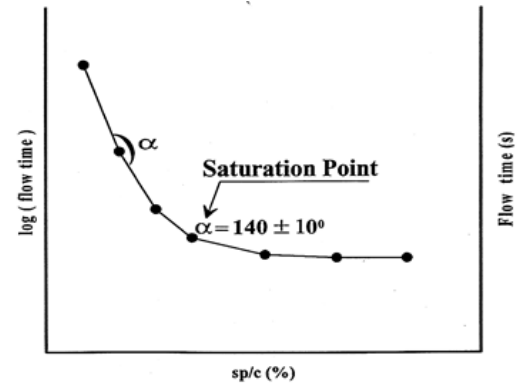


Figure 3: Approach for the determination of saturation dosage, Gomes et al. (2002)

2.3 Mini-slump test of Cement Paste

The mini-slump test developed by Kantro (1980) has also been used to study the fluidity of superplasticized cement paste (Gomes, 2002; Jayasree and Gettu, 2008). The mini-slump cone is a smaller version of the Abrams cone, but the proportions of the cone are maintained. Figure 4 shows the mini-slump cone used in the present study.

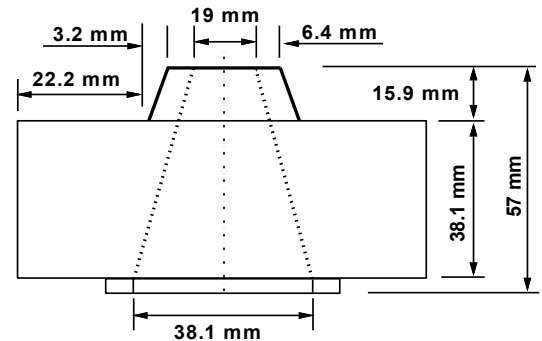


Figure 4: Mini-slump cone

The cement paste is poured into the cone, and the cone is lifted in the vertically upward direction. The paste is allowed to spread, and the spread diameter is measured in two perpendicular directions. The average of these two values is recorded as the average spread diameter. Thus, the fluidity of the cement paste can be studied.

2.3.1 Test Procedure

The cement paste was prepared in the same manner as described in the case of Marsh cone test. The clean mini-slump cone was placed on a clean glass plate. Cement paste was poured into the cone, and the cone was lifted in a vertically upward direction. The cement paste was allowed to flow over the glass plate. The spread diameter was measured in two perpendicular directions, and the average value was recorded as the average spread diameter. The superplasticizer dosage increased, and the same procedure was repeated. A plot of the sp/c (%) is plotted against the

average spread diameter (mm). The superplasticizer dosage beyond which a significant increase in the spread diameter is not observed is considered as the saturation dosage. A draft proposal for the methodology to be followed to conduct the mini-slump test has been sent to the cement and concrete sectional committee CED 02 of Bureau of Indian Standards to develop it as an Indian standard to standardize the particular test method.

3. Results and discussion

3.1 Marsh cone test

The tests were repeated fifteen times for each combination mentioned in Figure 1. Graphs were plotted both with respect to $\log_{10}$ (flow time). Figures 5–7 show the plots for the fifteen tests performed for each combination.

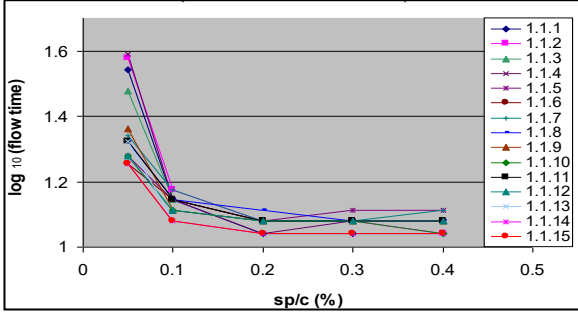


Figure 5: Flow time curves for SP1 and w/c = 0.35 calculated with respect to $\log_{10}$

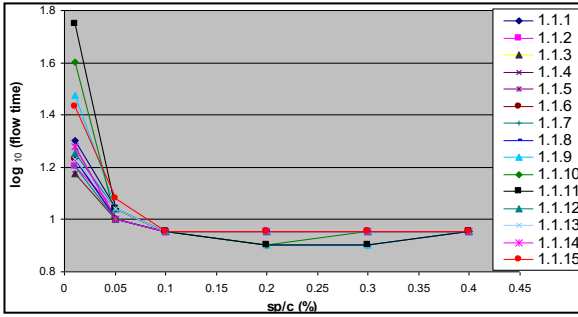


Figure 6: Flow time curves for SP1 and w/c = 0.4 calculated with respect to $\log_{10}$

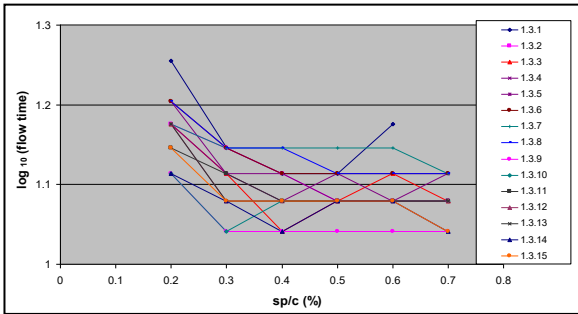


Figure 7: Flow time curves for SP2 and w/c = 0.35 calculated with respect to $\log_{10}$

The above graphs show greater variability in the flow time values in the pre saturation regime than the post saturation regime in case of w/c = 0.35 and 0.4 used along with SP1. In the post saturation regime, SP1 used with w/c = 0.4 shows less variability than the other two combinations. Maximum variability was seen in the case of SP2.

The optimum superplasticizer dosages obtained from all the tests have been tabulated in Table 1. Note that the values given in bold and italics are outliers. The statistical measures of central tendency like the mean and the median and the measures of the dispersion of the data like the quartiles and the standard deviation were determined for the three combinations. These values are tabulated in Table 2.

Table 1: Optimum superplasticizer dosage values from the Marsh cone test

Number of replicates (n)	SP1, w/c = 0.35 (Saturation Dosage (%))	SP1, w/c = 0.4 (Saturation Dosage (%))	SP2, w/c = 0.35 (Saturation Dosage (%))
1	0.136	<i>0.10</i>	0.22
2	0.122	0.077	0.245
3	0.090	0.084	0.229
4	0.109	0.084	0.24
5	0.125	0.062	0.30
6	<i>0.20</i>	0.079	0.242
7	0.172	0.074	0.259
8	0.080	0.074	0.267
9	0.080	0.073	0.278
10	0.081	<i>0.046</i>	0.322
11	0.099	<i>0.10</i>	0.244
12	0.081	0.075	0.30
13	0.080	0.076	0.30
14	0.082	0.062	0.242
15	0.082	0.073	0.272

Table 2: Statistic measures of the data obtained from the Marsh cone test

Statistic	SP1 w/c = 0.35	SP1 w/c = 0.4	SP2 w/c = 0.35
Minimum	0.08	0.046	0.22
Maximum	0.20	0.10	0.322
Range	0.12	0.054	0.102
Mean	0.108	0.076	0.264
Median	0.090	0.075	0.259
Lower quartile (Q_1)	0.081	0.073	0.242
Upper quartile (Q_3)	0.123	0.081	0.289
Inter quartile range (IQR)	0.042	0.008	0.047
Standard deviation (σ) (%)	0.037	0.014	0.030
Coefficient of variation	0.342	0.184	0.11
Outliers	0.2	0.046, 0.1	No outliers

The outliers present in the data collected through experimentation within the laboratory were identified and were discarded to arrive at the final values of standard deviation under conditions of repeatability. A range was specified to identify the outliers. The lower limit of the range is given by $Q_1 - 1.5 (IQR)$ and the upper limit of the range is $Q_3 + 1.5 (IQR)$. The variability is expressed in terms of repeatability value. This value is the standard deviation of the data obtained from tests conducted under conditions of repeatability (factors like operator, equipment, temperature etc. are maintained constant) after discarding the outliers. The average of the standard deviation values for the three combinations was concluded to be the repeatability value. The repeatability value for the Marsh cone test was 0.022% when calculated with respect to $\log_{10}$ (flow time).

3.2 Mini-slump test

Figures 8–10 show the collective plots of all the fifteen tests conducted for each combination.

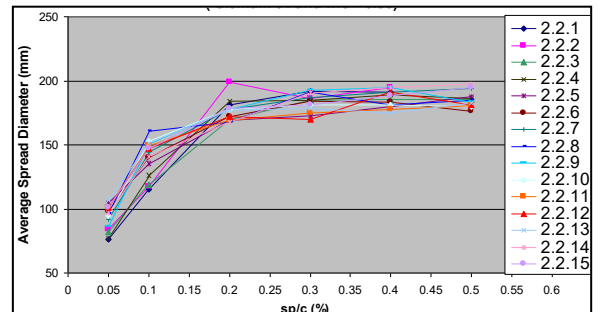


Figure 8: Mini-slump test results for SP1 and w/c = 0.35

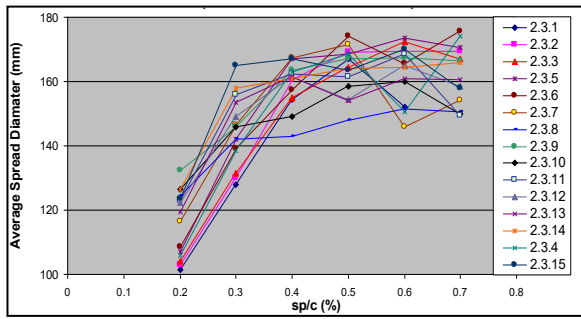


Figure 9: Mini-slump test results for SP1 and w/c = 0.4

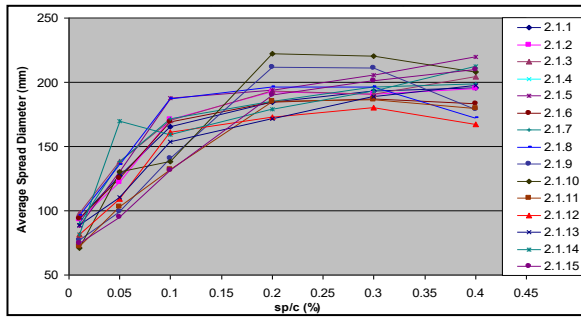


Figure 10: Mini-slump test results for SP2 and w/c = 0.35

Table 3: Optimum superplasticizer dosage values from the mini-slump test

Number of replicates (n)	SP1, w/c = 0.35 (Saturation dosage (%))	SP1, w/c = 0.4 (Saturation dosage (%))	SP2, w/c = 0.35 (Saturation dosage (%))
1	0.2	0.1	0.4
2	0.2	0.1	0.4
3	0.2	0.1	0.4
4	0.2	0.1	0.4
5	0.2	0.1	0.4
6	0.2	0.1	0.5
7	0.1	0.1	0.4
8	0.1	0.1	0.3
9	0.1	0.2	0.4
10	0.1	0.2	0.3
11	0.1	0.2	0.3
12	0.1	0.1	0.4
13	0.1	0.1	0.3
14	0.1	0.05	0.3
15	0.2	0.2	0.3

Table 4: Statistic measures of the data obtained from the mini-slump test

Statistic	SP1 w/c = 0.35	SP1 w/c = 0.4	SP2 w/c = 0.35
Minimum	0.1	0.05	0.3
Maximum	0.2	0.2	0.5
Range	0.1	0.15	0.2
Mean	0.147	0.123	0.367
Median	0.1	0.1	0.4
Lower quartile (Q ₁)	0.1	0.1	0.3
Upper quartile (Q ₃)	0.2	0.15	0.4
Inter quartile range (IQR)	0.1	0.05	0.1
Standard deviation (s) (%)	0.052	0.049	0.062
Coefficient of variation	0.35	0.39	0.168
Outliers	No outliers	No outliers	No outliers

From the above graphs, least variability was seen for the combination of SP1 and w/c = 0.35, and maximum variability in the results was seen in case of SP2 and w/c = 0.35.

The analysis of the data was carried out in a similar manner as discussed previously. The optimum superplasticizer dosage obtained from all the tests is tabulated in Table 3.

The statistical measures which describe the central measures of the data and those which describe the dispersion of the data like the quartiles and the standard deviation were calculated. These values are tabulated in Table 4. The outliers were determined in a similar manner as discussed previously. The standard deviations obtained were averaged to arrive at the repeatability value for the mini-slump test. This value was found to be 0.054% of the superplasticizer dosage.

3. Conclusions

The repeatability values are the allowable variations possible in the test results when the tests are conducted under controlled conditions. The repeatability value of the Marsh cone test was obtained to be 0.022% of the superplasticizer dosage, and the repeatability value for the mini-slump test was obtained as 0.054% of the superplasticizer dosage. These values were obtained after performing forty-five tests following the given test procedures. The repeatability values obtained show that the proposed test procedures can replicate the results for both the Marsh cone test and the mini-slump test, and that small variations are introduced during the application of these test methods. Therefore, these tests are said to be robust. From these values, it can be concluded that the allowable variation when the same operator performs the test using the same equipment within a short interval of time is higher in the case of the mini-slump test than in the case of the Marsh cone test.

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