Validation of Water Demand Studies as an Incompatibility Test for Binders and Superplasticizers

Swathy Krishna A K[1*], Sreeranjini T K[2]&Vijayakumari P L[3]

[1,2]*B Tech student, Jawaharlal College of Engineering and Technology (JCET), APJ Abdul Kalam Technological University, Kerala, India.*
[3]*Professor and Head, Department of Civil Engineering, JCET, Kerala, India.*

---***---

Biography:ACI member **Swathy Krishna A K** is a civil engineering graduate who received her B Tech from Jawaharlal College of Engineering and Technology, Kerala Technological University in 2019. Her research interest includesconcrete materials and construction technologies. She worked as a civil engineer in A.K Brothers builders and Engineering consultants, Palakkad. She is currently doing her M tech in construction engineering and management. Attended and presented papers in different conferences on civil engineering topics.

Sreeranjini T K is a B Tech civil engineering graduate from Jawaharlal College of Engineering and Technology, Kerala Technological University in 2019. Concrete technologies and construction methods are her area of research interest. Attended and presented papers in different conferences on civil engineering topics.

VijayalumariPillaiis currently working as professor and PG Dean in department of Civil Engineering, Marian Engineering College, Trivandram.She also worked as Professor in N.S.S College of Engineering and Jawaharlal College of Engineering and Technology. She received her B Tech in Civil Engineering from T.K.M College of Engineering, University of Kerala and Bachelor of Science (Chemistry Main) from Assumption College, University of Kerala. She received her M Tech from NIT Surathakal and Ph D from Indian Institute of Technology Madras in 2006.

Abstract- *Compatibility of cement and superplasticizer is a major factor we should consider to ensuredurability of concrete.Although we are not giving much attention to this fact due to lack of awareness andare using cement, chemical and mineral admixtures in predetermined amount and combinations. Compatibility depends on various factors but we are mostly using only marsh cone test to study compatibility and neglecting possible other ways of checking the compatibility. The study was conducted to introduce a new method to test compatibility and to analyze the scope of vicat's apparatus consistency or water demand test as a compatibility test. The addition of water relates to the workability and strength of concrete as well as issues like bleeding, setting time variation etc. So the variation in requirement of water which is also a major consistuent in the cement mix can be considered as a compatibility factor. The water demand characteristics of different type of cement mix with varying combination of two frequently used superplasticizersviz; Sulphonated Naphthalene Formaldehyde (SNF), Polycarboxylate Ether (PCE), fly ash, and coconut shell ash (CSA) were studied. Water demand variation is studied comparing hand mixed and machine mixed paste. The finding were proved correct when compatible and incompatible mix in vicat's apparatus water demand test was found to show same result in the standard test, marsh cone as well.*

***Keywords* : Superplasticizer, SNF, PCE, chemical admixtures, Compatibility, Water demand.**

1.INTRODUCTION

Studies and investigations are progressing on improving concrete to increase the durability of structure, reduce failures and environmental impacts; and as a process we use different high performace cement and additional materials as per required for the particular purpose of concrete. Here is where chemical and mineral admixtures play a crucial role in concrete as they can improve the properties without affecting the strength.Superplasticizers (SP) is one of the chemical admixture widely used in construction to reduce water demand by ensuring maximum workability and has additional advantages of reducing heat of hydration, segregation and bleeding. But use of SP in a predicted dosage might not give desired outcome as SP belong to different families varies in their chemical and physical properties and not necessarily be having good compatibility with the cement used. On the other hand mineral admixtures differ from cement in their particle size.Hence we cannot generalize the impact of admixtures on cement paste and their compatibility with cement paste should be analyzed separately. Marsh cone and mini slump test are the standard tests used for compatibility study and they analyze the incompatibility with respect to flow time. The aim was to analyze the water demand characteristics of different cement- superplasticizer paste, clarify whether incompatibility can be ascertained with respect to water demand studies and to introduce a new method to analyze the compatibility issues. So the possibility of using Vicat's apparatus consistency test (water demand test)to analyze the compatibility issue is studied. The compatibility of a popular brand of PPC and two major families of superplasticizers, viz., Sulphonated Naphthalene Formaldehyde (SNF) and Polycarboxylate Ether (PCE) are conducted at different conditions using the Vicat's apparatus water demand test. After checking the compatibility with SP alone, the same experiment is continued with addition of pozzolanic materials aswell to the mix.The effect of addition of more pozzolanic materials to fly ash based PPC, for increasing the sustainability potential was analyzed by replacing PPC with 10% of coconut shell ash (CSA). The experiment was repeated with 10% fly ash instead of CSA to analyze how the addition of a different pozzolanic material affects the compatibility of paste.The validation of these results has been done with Marsh cone which is a standard compatibility test. The study leads to the conclusion that the water demand studies could be considered an indicative to identify compatibility issues and SP over dosage..In water demand studies, the pastes are prepared for the required consistency unlike marsh cone and mini slump tests were the pastes are prepared with predetermined water content. So it is also possible to observe the pastes at optimum water content for its properties like workability, segregation, bleedingetc and even to predict optimum SP dosage by considering all these factors.

2.RESEARCH SIGNIFICANCE

The main objective of the study was to introduce a new method for compatibility study. There have been no much study undertaken in this topic so far, but being major elements of construction the cement and admixtures should be studied for compatibility based on various factors and circumstance. The possibility of using Vicat's apparatus consistency test as a new test for incompatibility problem in the cement- SP paste based on water demand is studied and the findings are ascertain through a standard test called marsh cone. The water demand characteristics of cement mix with addition of superplasticizerviz; SNF, PCE is studied. Understand the change in water demand characteristics if coconut shell ash (CSA) and fly ash are added to these mix were also a main concern of the study as chemical admixtures are widely used in construction. The study also checked whether there is huge difference in water demand and characteristics of cement- superplasticizer paste if hand mixing is replaced with machine mixing.

3.EXPERIMENTALINVESTIGATION

3.1 METHODOLOGY

The compatibility of SPs with cement was analyzed by conductingvicat's apparatus consistency test to determine water demand of SP at different dosages, keeping all other parameters like weight of cement, gauging time constant. The water demand- SP dosage graph plotted was analyzed for understanding the behavior of SP with cement at different dosage and incompatibility problem if any. While performing consistency test, SP dosage was taken in terms of solid content percentage. For that solid content of SP was determined and water correction was done. For the confirmation of results regarding the compatibility issues obtained from the consistency test, marsh cone test which is used as a standard test for incompatibility study has been used.

3.2 MATERIAL STUDY

Material study was conducted for the following materials.

- Portland Pozzolana Cement (PPC). The physical properties of PPC are listed in **Table 1**.

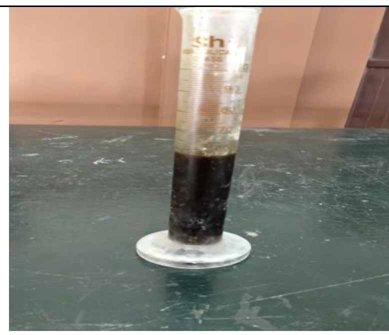
Table 1- Properties of Portland Pozzolana Cement.

Type of Cement	PPC
Fineness	9 %
Standard consistancy	33.3%(hand mix)
	32.67%(machine mix)
Initial setting time	1 hours 45 minutes (6300 seconds)
Final setting time	5 hours 20 minutes (19200

	seconds)
Specific gravity	3.35
Soundness	1mm (0.039 in.)

Superplasticizer: Sulphonated Naphthalene Formaldehyde (SNF), Polycarboxylate Ether (PCE). The properties of these superplasticiers are shown in **Table**

Table 2- Properties of Superplasticizers.

SP	SNF	PCE
Sample		
Solid content	27.14%	30.78%
Viscosity(visual observation)	Less	High

As specified in "ASTM C494/C494M-99ae1-Standard specification for chemical admixtures for concrete" the solid content determination of SNF and PCE are conducted. Solid content is determined as $m_5 \times 100/m_4$,where $m_4=m_2-m_1$ and $m_5=m_3-m_1$. Where;

m_1 isweight of 30g (1.058 oz) of sand taken in a jar and dried in oven for 17 hours (61200 second) at 105 degree Celsius (221 degree Fahrenheit).

m_2 is weight of this sample after adding 4ml (0.004 US liquid quarts) of SP over it.

m_3 is weight of this sample after drying for 17 hours at 105 degree Celsius.

The experimental results from the testing described above are shown in **Table 3.**

Table 3- Observations of solid content determination of SP.

Notation	m_1	m_2	m_3	m_4	m_5
Sample 1 (SNF)	49.165 g (1.73 oz.)	53.505 g (1.89 oz.)	50.343 g (1.78 oz.)	4.34 g (0.153 oz.)	1.178 g (0.041 oz.)
Sample 2 (PCE)	57.15 g (2.016 oz.)	61.47 g (2.17 oz.)	58.47 g (2.06 oz.)	4.31 g (0.152 oz.)	1.32 g (0.046 oz.)

Solid content for SNF= $m_5 \times 100/m_4 =$ 1.178×100/4.34=27.14%

Solid content for PCE= $m_5 \times 100/m_4 =$ 1.32×100/4.31=30.78%

- Pozzolanic materials: coconut shell ash (CSA), fly ash. **Table4**shows the importantproperties of thesePozzolanic materials.

Table 4- Properties of Pozzolanic materials.

Pozzolanic material	**Fly ash**	**CSA**
Sample		
Fineness as per IS:4031-part1-1996 (90 micron)	28%	70%
Fineness modulus As per IS :1727 (300 and 150 micron)	0.81	0.89

3.3 WATER DEMAND TEST

Superplasticizers are expected to decrease the water demand of the cement paste.Showing a difference in this property can be an incompatibility issue. Vicat's apparatus consistency test was conductedas specified in IS: 4031-Part4-1988 to understand trend in water demand variation with increasein SP dosage. SP dosage of 0.05%, 0.1%, 0.2%, 0.3%, 0.4%, 0.5%, 0.7%, 1%, 1.3%, 1.5% were taken for experiment.SP dosage was calculated as percentage of solid content for correcting water demand considering the liquid content of SP. The amount of SP is calculated as follows:

Weight of solid content of SP, W = weight of cement × percentage of SP used.

Weight of SP, Wsp=W/percentage solid content.

Consistency was determined for each SP dosage of each mix.

Table 5- Weight of SNF and PCE used with respect to percentage solid content in water demand test.

Weight of percentage by solid content/cement content	Weight of solid content of superplasticizer, W	Weight of SNF used	Weight of PCE used
0	0 g (0 oz.)	0 g (0 oz.)	0 g (0 oz.)
0.05	0.15 g (0.005 oz.)	0.552689757 g (0.019 oz.)	0.487329435 g (0.017 oz.)
0.2	0.6 g (0.02 oz.)	2.210759027 g (0.078 oz.)	1.949317739 g (0.069 oz.)
0.3	0.9 g (0.03 oz.)	3.316138541 g (0.11 oz.)	2.923976608 g (0.103 oz.)
0.4	1.2 g (0.04 oz.)	4.421518055 g (0.156 oz.)	3.898635478 g (0.138 oz.)
0.5	1.5 g (0.05 oz.)	5.526897568 g (0.19 oz.)	4.873294347 g (0.17 oz.)
0.7	2.1 g (0.074 oz.)	7.737656595 g (0.27 oz.)	6.822612086 g (0.24 oz.)
1	3 g (0.1 oz.)	11.05379514 g (0.39 oz.)	9.746588694 g (0.34 oz.)
1.3	3.9 g (0.13 oz.)	14.36993368 g (0.5 oz.)	12.6705653 g (0.44 oz.)
1.5	4.5 g (0.16 oz.)	16.5806927 g (0.58 oz.)	14.61988304 g (0.51 oz.)

Table 5 shows theweight of SNF and PCE used as described above.Paste prepared with 300g (10.58 oz) of cement and each SP dosage were filled in vicat'smould and plunger was gently lowered on the surface of the paste and reading was noted for different percentage of water. The percentage of water at which plunger penetrates to a depth of 5 to 7 mm (0.197 to 0.276 in.) from the bottom was determined by trial and error method which denotes standard consistency value [**Fig. 1**].

Fig. 1- Water demand test with SP using vicat's apparatus.

The experiment was repeated with addition of 10% CSA and fly ashas well.Mix 7 was prepared by mixing PPC with PCE using a cake mixer with a gauging time of 5 minutes (300 seconds) [**Fig. 2**].

Fig. 2- Mixing of paste using cake mixer.

The details of composition of all the 7 mixes used are listed in **Table 6.**

Table 6- Specification of mix

Type of mix	Composition of mix
Mix 1	PPC, SNF, hand mix
Mix 2	PPC, PCE, hand mix

Mix 3	PPC, SNF, 10% CSA, hand mix
Mix 4	PPC, PCE, 10% CSA, hand mix
Mix 5	PPC, SNF, 10% fly ash, hand mix
Mix 6	PPC, PCE, 10% fly ash, hand mix
Mix 7	PPC, PCE, machine mix using cake mixer

Graphs were plotted showing the variation of cement-super plasticizer interaction from the results of all the tests, where X axis indicate the water demand and Y axis indicate SP dosage.

3.4 MARSH CONE TEST

Marsh cone test was conducted with SP dosage as varying parameter. Each sample was made of 2kg (4.4 lbs.) of cement with water content 0.55. Tests were done at SP dosage from 0.2% onwards up to 0.6% by increasing dosage in rate of 0.1%. Additional points 0.9%, 1.2%, 1.5% are also taken to analyze the behavior at even high dosage of SP. The time taken for the prepared slurry of cement, SP and water to pass through marsh cone after 60 minute (3600 seconds) of retention was noted.

Table 7- Marsh cone flow time for PCE and SNF at 60 minute (3600 seconds) retention.

Cement	W/C ratio	Water	SP dosage (%)	Amount of PCE(Considering solid content of SP)	Marsh Cone Time for PCE at 60 min (3600 seconds) Retention	Amount of SNF (Considering solid content of SP)	Marsh Cone Time for SNF at 60 min (3600 seconds) Retention
2 kg (4.4 lbs.)	0.55	1100 ml (0.29 gallon)	0.2	13 ml (0.0034 gallon)	1 min 32 sec 94 ms	14.7 ml (0.0039 gallon)	1 min 21 sec 87 ms
2 kg (4.4 lbs.)	0.55	1100 ml (0.29 gallon)	0.3	19.5 ml (0.0051 gallon)	1 min 11 sec 53 ms	22.1 ml (0.0058 gallon)	1 min 2 sec 81 ms
2 kg (4.4 lbs.)	0.55	1100 ml (0.29 gallon)	0.4	30 ml (0.0079 gallon)	1 min 0.6 sec 66 ms	29.5 ml (0.0078 gallon)	49 sec 44 ms
2 kg (4.4 lbs.)	0.55	1100 ml (0.29 gallon)	0.5	32.5 ml (0.0086 gallon)	1 min 0.3 sec 56 ms	36.8 ml (0.0097 gallon)	53 sec 0.9 ms
2 kg (4.4 lbs.)	0.55	1100 ml (0.29 gallon)	0.6	40 ml (0.01 gallon)	56 sec 00 ms	44.2 ml (0.011 gallon)	59 sec 0.8 sec
2 kg (4.4 lbs.)	0.55	1100 ml (0.29 gallon)	0.9	58.5 ml (0.0154 gallon)	1 min 4 sec 22 ms	66.3 ml (0.018 gallon)	1 min 5 sec 84 ms
2 kg (4.4 lbs.)	0.55	1100 ml (0.29 gallon)	1.2	80 ml (0.021 gallon)	1 min 04 sec 56 ms	88.4 ml (0.02 gallon)	59 sec 82 ms
2 kg (4.4 lbs.)	0.55	1100 ml (0.29 gallon)	1.5	97.5 ml (0.026 gallon)		110.5 ml (0.029 gallon)	1 min 0 sec 75 ms

Fig. 3- Performing marsh cone test.

The experimental values are shown in **Table 7** and the test setup is shown in **Fig. 3.**Flow time- SP dosage graph is plotted for predicting incompatibility.

4. EXPERIMENTAL RESULTS AND DISCUSSION

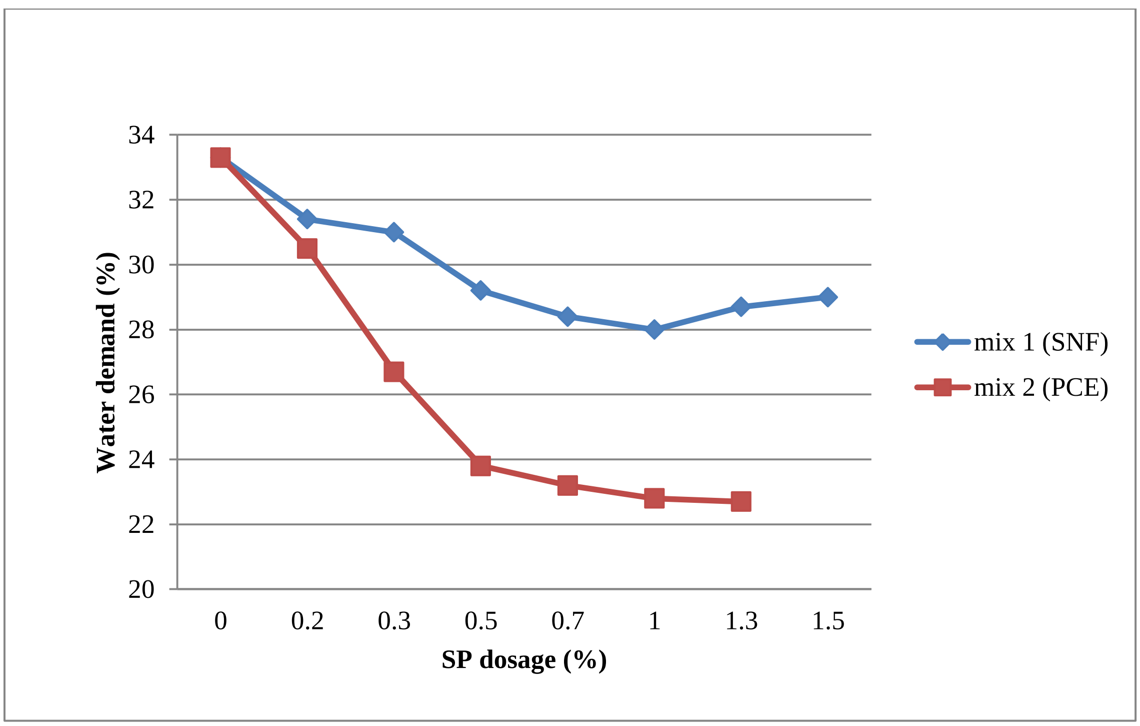


Fig.4- Comparison of water demand graph of PPC at gradually increasing dosage of SNF and PCE.

Fig.4 compare the water demand of same cement with different superplasticizerviz SNF (blue) and PCE (red) with gradually increasing dosage. Water demand is decreasing in case of PCE with increasing SP dosage. While in case of SNF water demand decreases but starts to increase after a particular dosage. Hence the addition of SNF should be done carefully as it shows a reverse trend in water demand. Also PCE is showing much decrease in water demand compared to SNF as the dosage starts to increase. In case of over dosage of SP (after 0.7% in **Fig.4)** PCE is demanding constant water while SNF starts to demand more. So here we can say PCE is more compatible to the particular brand of cement used than that of SNF [1].

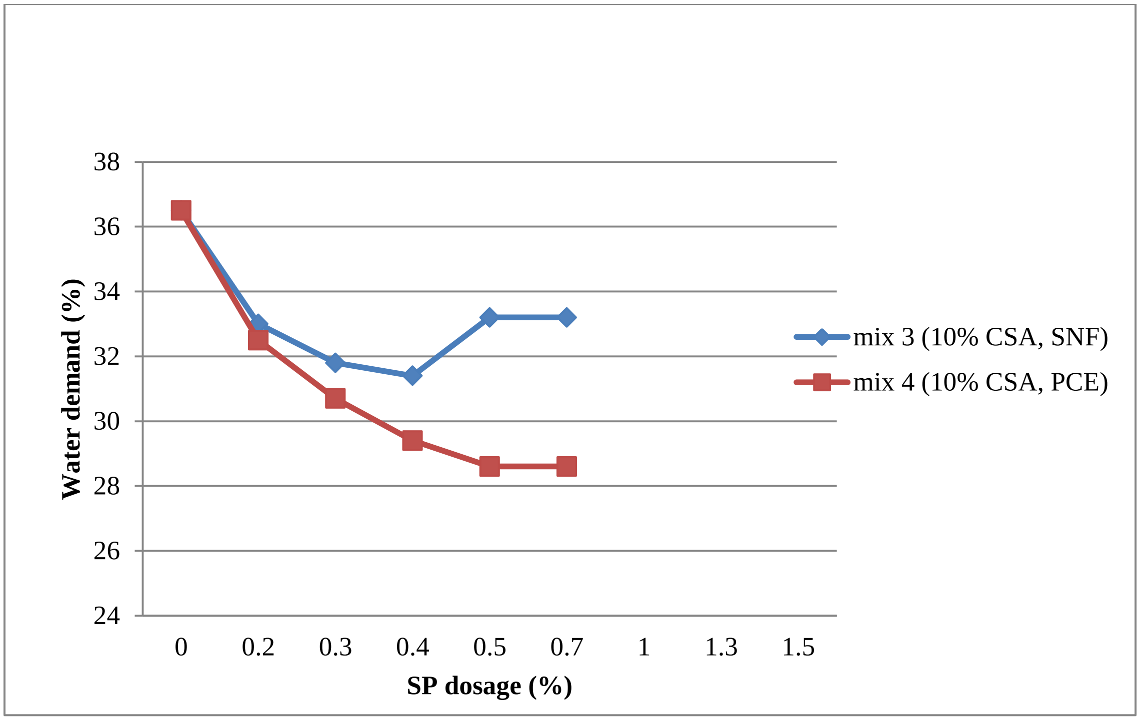


Fig. 5- Comparison of water demand graph of PPC at gradually increasing dosage of SNF and PCE with addition of 10% coconut shell ash.

Fig.5is a comparison of water demand for mix 3 prepared with 10% of CSA added to increasing dosage of SNF and mix 4 with 10% CSA and increasing dosage of PCE. The graph of **Fig.5** and **Fig.4** are ofalmost similar shapes and this shows mix 4 with PCE shows decreasing water demand while mix 3 with SNF shows a reversing trend of water demand.

While considering the water demand of fig.4and fig.5, the values are more for **Fig.5**. That shows addition of mineral admixtures like CSA increased the water demand. Over dosage is also reached at comparatively earlier stage when CSA is added, because we can see the change in behavior is shown in **Fig.5**at 0.5% dosage while that in **Fig.4** it is shown after 0.7% only. This shows the compatibility is decreasing when CSA is added when we consider water demand as a factor.

We cannot conclude these results are true for all mineral admixtures as admixtures vary in size, shape and other properties. Hence we are analyzing the case with addition of a different admixture called flyash which is shown in **Fig.6**.

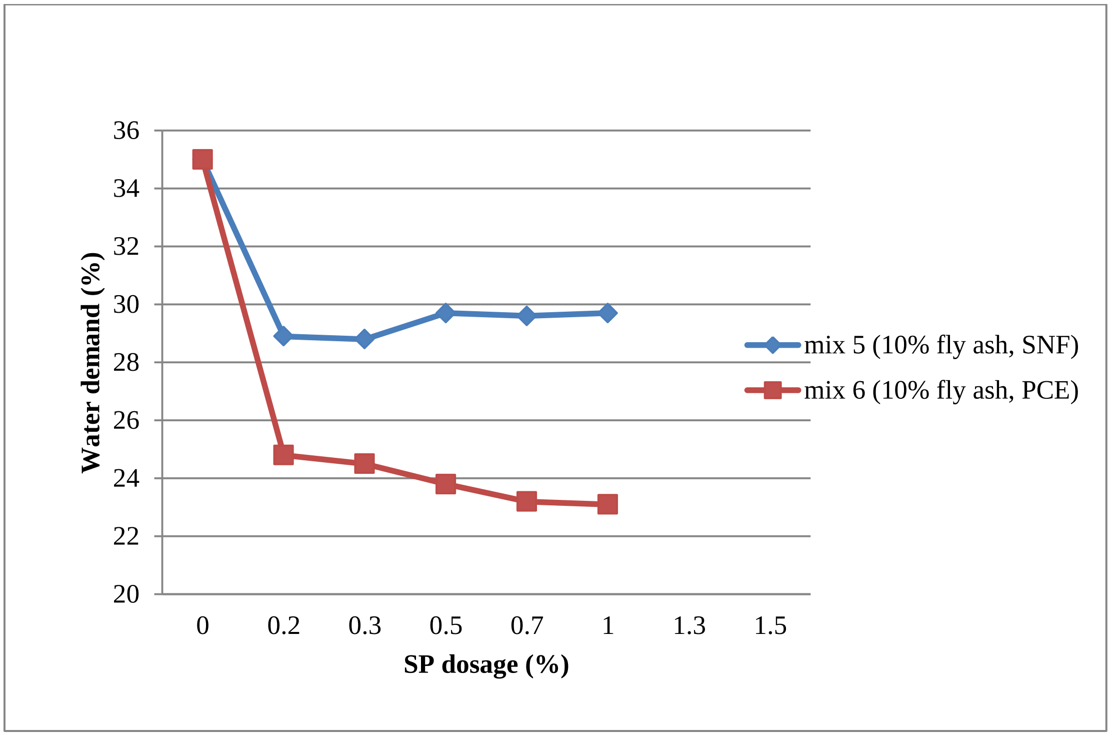


Fig. 6- Comparison of water demand graph of PPC at gradually increasing dosage of SNF and PCE with addition of 10% fly ash.

The graphs are much steeper in **Fig.6** than that of the previous ones. Just the addition of 10% fly ash largely decrease the water demand at 0.2 SP dosage, then gradual decrease is shown as that of the other cases. The reason for immediate depression of water demand can be the smooth spherical surface of fly ash [2]. The water requirement is very less when fly ash is added compared to that of CSA. It is noticeable that water demand even reduces to nearly same values of that of mix without any chemical admixtures (mix 1 and 2) when over dosage is reached and constant value is attained. The mix with fly ash and SP can be considered more compatible than rest of themixes because it requires very less water to form a consistent mix.

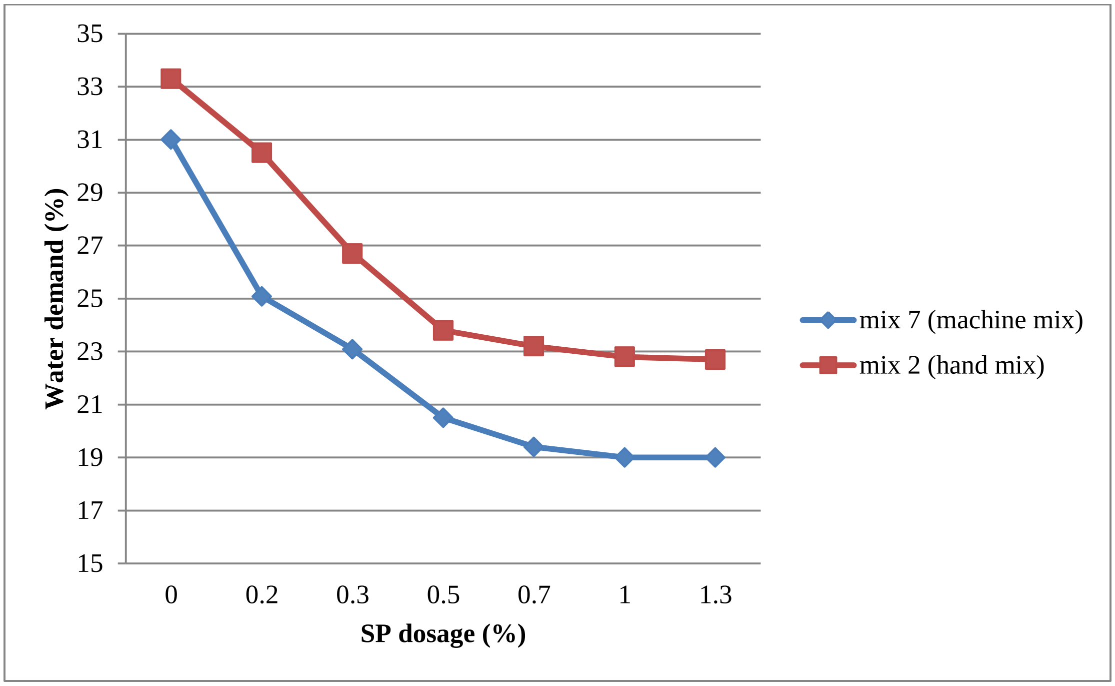


Fig. 7- Comparison of water demand graph of PPC at gradually increasing dosage of PCE with hand mix and machine mix.

While comparing the water demand characteristicsof mix 2with mix 7 in **Fig. 7**, where only difference is the type of mixing, we can see a difference in water demand. The shape of the graph is similar in both cases. The graphs are almost parallel to each other. But water required by machine mixing is less compared to that of hand mixing. It is found that over dosage of SP is reached at same point (0.7%). Most of the cement mixing works are done using machine mixing these days due to reduce time and labor. But there are cases we only require hand mixing in the same site. By studying the **Fig. 7**, we can say that compatibility of cement and SP does not change much with difference in mixing as both cases shows similar trend in graph and over dosage point.But we have to consider the fact that the water demand is less for machine mixing and therefore consistency test should be done separately for these cases. Also machine mixing should be preferred over hand mixing as they are slightly more compatible and better mixing.

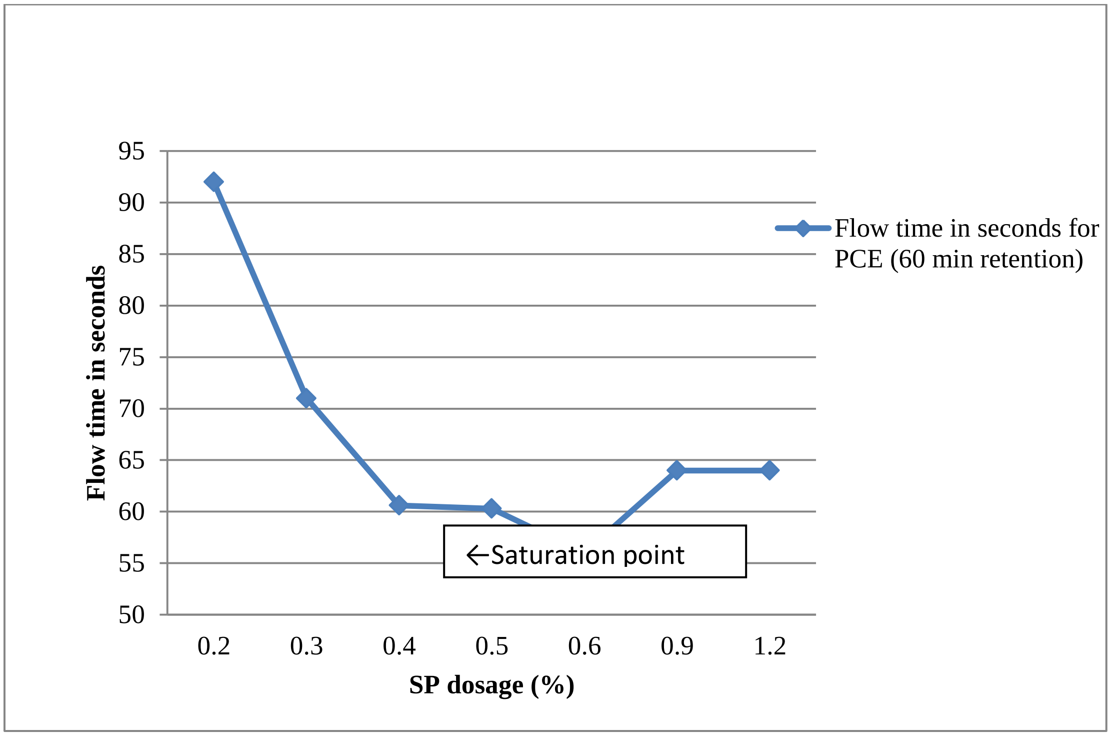


Fig. 8- Flow time in seconds for PCE (60min (3600 seconds) retention) using marsh cone.

The graph shown in **Fig. 8** illustrates the flow time variation of PCE at 60 min (3600 seconds) retention time with respect to increasing SP dosage. The flow time decreases with increasing PCE till 0.6 SP dosages. Then it slightly increases and remains constant regardless of the increasing dosage. The minimum flow time is observed at 0.6 and thereafter constant value is obtained, so it is taken as the optimum or saturation point of SP. Since we are able to identify the saturation point PCE – cement mix is compatible [3].

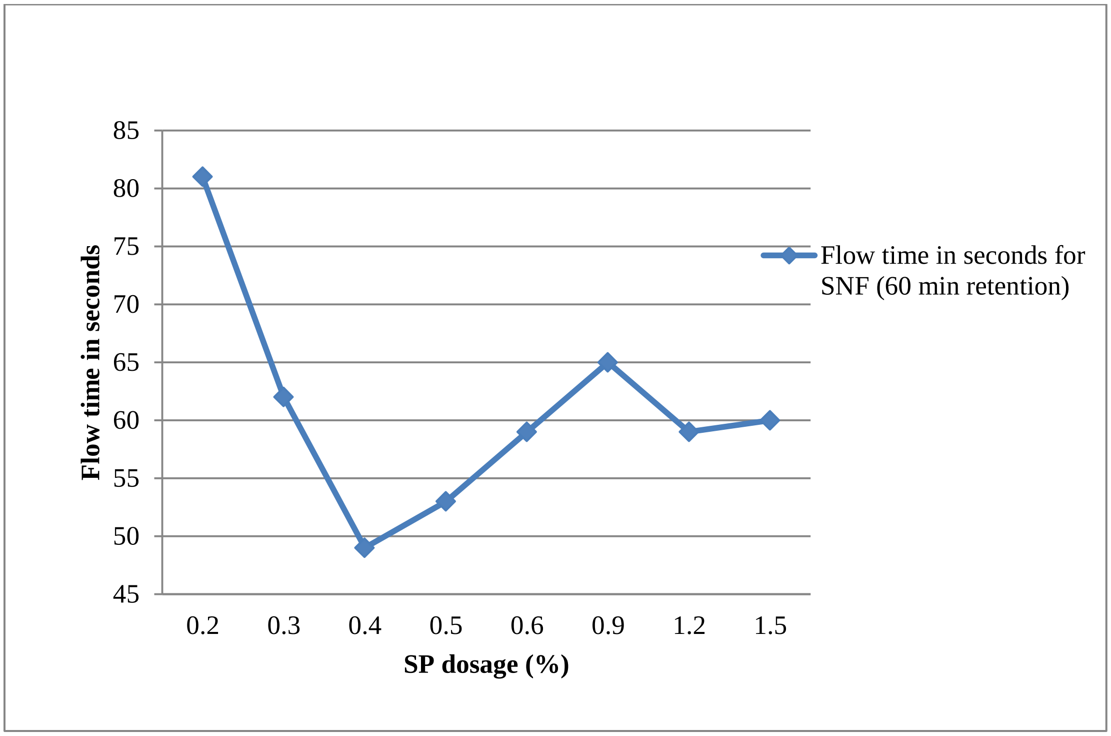


Fig. 9- Flow time in seconds for SNF (60 min (3600 seconds) retention) using marsh cone.

Fig. 9 shows the marsh cone flow time graph of SNF at 60 min (3600 seconds) retention. There is no desired saturation point for the graph. Flow time initially decreases then starts to show irregular variation. It is the indication of incompatible condition as there is difficulty to determine saturation point and flow time does not turn to be constant.

5. SUMMARY AND CONCLUSIONS

The possibility of vicat's apparatus consistency test as a compatibility test is validated in comparison with marsh cone test.However the optimum dosage of marsh cone test cannot be compared with that of water demand test as the mixes are prepared differently and both are two different experiments in which compatibility is considered in terms of flow time in marsh cone while consistency in vicat's apparatus. According to water demand studies, PCE is much more compatible to the particular brand of PPC used than that of SNF. Cement paste which shows increase in water demand or reversing trend after decreasing to certain point as the SP dosage increase is considered to be incompatible. Compatibility issue of cement and SNF remained even when fly ash and CSA were added. Use of fly ash and SP together decreased the water demand while CSA and SP mix demanded more water than mix without any chemical admixtures. There is faster reaction and quick over dosing in case of mix with chemical admixtures and SP.Change in mixing method should also be considered for compatibility since machine mixing method reduce the water requirement. But the optimum dosage of SP is not varying with change of mixing method unlike that of other cases.

6. ACKNOWLEDGEMENTS

The authors acknowledge the support of Indian Institute of Technology, Palakkad and Jawaharlal College and Technology, Lakkidi for the lab facilities. The authors also acknowledge A.K Brothers builders and Engineering consultants, Palakkad for disclosing the type of cement and SP used in construction and having provided some materials used in the study. The help of our friend Mr.Sreenath S in performing experimental works is also acknowledged.

NOTATION

SP: Superplasticizer

SNF: Sulphonated Naphthalene Formaldehyde

PCE: Polycarboxylate Ether

CSA: Coconut shell ash

REFERENCES

[1]. P.R.KannanRajkumar, P.T.Ravichandran, J.K.Ravi and L.Krishnaraj (2016) Investigation on the compatibility of cement paste with SNF and PCE based superplasticizers, Indian Journal of Science and Technology, Vol 9(34).

[2].VanissornVimonsatit, PrinyaChindaprasirt, SeksunRuangsiriyakul and VanchaiSata (2015) Influence of fly ash fineness on water requirement and shrinkage of blended cement mortars, KKU Engineering Journal.

[3]. Tiji K James and Liji Anna Mathew (2016) Compatibility study of an admixture with different cement brands of varying chemical composition for SCC, International Journal of Engineering Research & Technology, Vol.5 Issue 09.