Comparative study on sorption test of superabsorbent polymer (SAP) for use in cement-based materials

Anjali S K
Department of Civil Engineering
Government College of Engineering
Kannur, India

Aiswarya B Mohan
Department of Civil Engineering
Government College of Engineering
Kannur, India

Ajith Krishnan
Department of Civil Engineering
Government College of Engineering
Kannur, India

Devu K V
Department of Civil Engineering
Government College of Engineering
Kannur, India

Sharanya Raghu T P
Department of Civil Engineering
Government College of Engineering
Kannur, India

Dr. Ajith M S
Department of Civil Engineering
Government College of Engineering
Kannur, India

***Abstract*—The determination of absorption capacity of superabsorbent polymer is important for its application in cement-based materials. Currently, no standard methods are available for the measurement of absorption of superabsorbent polymer. The tea-bag method and filtration method are two methods that are generally used for this purpose. This study focuses on the determination of absorption capacity of superabsorbent polymer by the tea-bag method and filtration method. This work performs a comparative study between these two test methods and their application in cementitious environment is also studied.**

***Keywords*—superabsorbent polymer; tea-bag method; filtration method**

I. INTRODUCTION

Superabsorbent polymer (SAP) is a cross-linked chemical polymer that can absorb water many times of its own weight. SAP has been used in variety of fields due to its water absorption and other properties [1,2]. SAP is primarily used in concrete technology as an internal curing agent to prevent autogenous shrinkage [3, 4, 5]. Prior to adding SAP to cementitious materials, the absorption capacity (AC) of SAP must be known in order to estimate the appropriate amount of SAP for the mix. This study aims at identifying the absorption capacity of SAP for use in cement-based materials. Absorption capacity can be defined as the amount of liquid that can be absorbed by one gram of dry SAP, which may be calculated as the ratio of the mass of the absorbed liquid to the mass of dry SAP.

The test was carried out based on the recommendations of RILEM TC 260-RSC for testing sorption of superabsorbent polymer [6]. The RILEM TC recommends two methods for testing sorption of superabsorbent polymer; the tea-bag method and the filtration method. In the tea-bag approach, a dry tea-bag filled with SAP is immersed in a pre-described fluid for a set period of time, whereas in the filtration method, a measured amount of dry SAP is soaked in a pre-described fluid and subsequently filtered. The test fluid can be either de-ionized water or filtrate of cement slurry. The absorption capacity of SAP was determined in this test using both the tea-bag method and the filtration method. Since the goal of this experiment was to ascertain the capacity of SAP to absorb before being added to cementitious materials, the filtrate of cement slurry was used as the test fluid.

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II. MATERIALS

A. *Superabsorbent polymer*

The superabsorbent polymer used in this test was sodium polyacrylate. It is a sodium salt of polyacrylic acid and can absorb as much as 500 times its mass in water. The sodium polyacrylate used in this investigation was a white fine powder with particle sizes ranging from 230 to 100 mesh. Also, its free absorbency in distilled water was 350-500 g/g.

B. *Test liquid*

To replicate the conditions of high-performance concrete in which the superabsorbent polymer is usually used, the cement slurry was prepared using cement and other cementitious materials that will be used in the concrete. 53 grade ordinary Portland cement was used for the preparation of cement slurry. At 20% and 10% of the requisite total cementitious material content respectively, fly ash and silica fume were added. A water-cement ratio of 5 was used for the preparation of the cement slurry. 105 g cement, 30 g fly ash, 15 g silica fume and 750 ml water were taken in a 1 litre beaker and was stirred at 70 rpm for 30 minutes. The obtained slurry was then filtered and was used for both the tea-bag and filtration methods. Fig. 1 shows the filtration of the prepared slurry. The produced filtrate had a pale yellow colour.

III. TEST METHODS

A. *Tea-bag method*

A tea-bag measuring 8 cm×10 cm was used for the tea-bag procedure. The amount of dry SAP utilized in the test was

Fig. 1. Preparation of filtrate of cement slurry

0.2 g because the tea-bag's size nearly met the requirements in the RILEM recommendation. The test also made use of an electronic balance with a precision of 0.001 g.

Initially, the fluid absorbed by the tea-bag was measured by performing the test on ten individual tea-bags. The dry tea-bag was first weighed (m_1) and was then soaked in the test liquid. After one minute, the tea-bag was removed from the test liquid and carefully wiped with a dry cloth. The mass of the wet tea-bag was then measured (m_2). For the remaining nine tea-bags, the same procedure was followed. The average mass of test liquid absorbed by an empty tea-bag (m_0) was calculated using (1).

$$m_0 = \frac{1}{n}\sum_{i=1}^{n}\left(m_{2i} - m_{1i}\right) \tag{1}$$

For ensuring the reliability of the results, the remaining test was performed on three individual tea-bags (tea-bag 1, tea-bag 2 and tea-bag 3). The dry tea-bags were initially weighed (m_1) and were again weighed after inserting 0.2 g of SAP (m_3). The teabags were then hung in a beaker containing 200 ml of test liquid. A plastic bottle cap was used to tightly seal the beaker in order to prevent evaporation and carbonation. The tea-bags were removed from the beaker, gently rubbed with a dry cloth for about 30 seconds on its surface, and weighed (m_4) after a specified period of SAP/liquid contact time. The hydrogel-containing tea-bags were then returned to the test liquid in the beaker and carefully covered until the next measurement. The measurement intervals were 1, 5, 10, 30, 60 min, 3 and 24 h after the SAP-containing tea-bags were immersed in the test liquid for the first time. The absorption capacity at each time interval was then determined using (2).

$$AC = \frac{\left(m_4 - m_3 - m_0\right)}{\left(m_3 - m_1\right)} \tag{2}$$

B. Filtration method

For the filtration method, an excess amount of test liquid was required for the free swelling of SAP. Therefore, the amount of SAP required to absorb 50 ml of the test liquid was initially found. 50 ml of the test liquid was taken in a beaker and measured quantities of SAP was added into it until the whole liquid gets absorbed by SAP particles. Then the total amount of SAP taken was calculated (m_1). The same 0.001 g precision electronic balance that was used for the tea-bag method was also used for the filtration method.

For the actual test, m_1 g of dry SAP was placed in seven beakers and 100 g (m_2) of the test liquid was added to all the beakers. The seven beakers were kept for an SAP/liquid contact time of 1, 5, 10, 30, 60 min, 3 and 24 h respectively. All the beakers were tightly covered using a plastic bottle cap during the prescribed contact time. After the corresponding SAP/liquid contact time, the hydrogel-containing test liquid in each beaker was filtered using filter paper and a funnel. The filter paper was saturated in the test liquid before filtering to minimise the impact of liquid absorbed by the filter paper. Using the mass of the filtered fluid (m_3), the absorption capacity for each time interval was then determined (3).

$$AC = \frac{m_2 - m_3}{m_1} \tag{3}$$

IV. RESULTS AND DISCUSSIONS

SAP absorption capacity was determined using both the tea-bag method and the filtration method, as recommended by RILEM. According to the tea-bag method, the average quantity of test liquid absorbed by an empty tea-bag (m_0) was 1.714 g. For tea-bags 1, 2, and 3 respectively, the mass of dry tea-bags (m_1) were determined as 0.887 g, 0.0892 g and 0.891 g. The mass of those tea-bags with dry SAP (m_3) were obtained as 1.087 g, 1.092 g and 1.101 g respectively. For the filtration method, the amount of dry SAP taken (m_1) was 0.854 g and the mass of test fluid used in each beaker (m_2) was 100 g. Table 1 displays the mass of tea-bag containing hydrogel (m_4) and mass of filtered fluid (m_3) for the tea-bag method and filtration method respectively, at various SAP/test liquid contact periods as well as the associated absorption capacity estimated using (2) and (3).

Fig. 2 shows a comparison of the results obtained from both methods. The filtration procedure resulted in an increase in absorption capacity throughout the test. The rate of improvement in absorption capacity was slower up to 3 h of SAP/liquid contact time. Only a 2.94% difference in absorption capacity was discovered at 3 h against 5 min, and it was 3% at 5 min against 1 min contact duration. But the percentage increase in absorption capacity at 24 h was obtained as 31.43% from that at 3 h. In contrast to the findings from the filtration method, the tea-bag method showed a decline in the absorption capacity of SAP over the course of the test but the rate of decrease was found to be lower at 24 h contact period. The tea-bag method showed a 11.77% decrease in absorption capacity at 5 min contact time from that at 1 min. As the contact time increased, the rate of decrease was found to be higher until

TABLE I. Observed mass and absorption capacity obtained from tea-bag method and filtration method

SAP/test liquid contact time	Tea-bag method							Filtration method	
	Tea-bag 1		*Tea-bag 2*		*Tea-bag 3*		*Average AC (g/g)*	*Mass m_3 (g)*	*AC (g/g)*
	Mass m_4 (g)	*AC (g/g)*	*Mass m_4 (g)*	*AC (g/g)*	*Mass m_4 (g)*	*AC (g/g)*			
1 min	10.697	39.480	10.185	36.895	10.038	34.395	36.923	67	38.642
5 min	9.400	32.995	10.058	36.260	8.796	28.481	32.579	66	39.813
10 min	8.483	28.410	8.645	29.195	7.545	22.524	26.710	66	39.813
30 min	7.499	23.490	7.717	24.555	6.912	19.509	22.518	66	39.813
1 h	6.696	19.475	6.335	17.645	6.016	15.243	17.454	65	40.984
3 h	5.455	13.270	4.944	10.690	4.505	8.048	10.669	65	40.984
24 h	4.660	9.295	4.888	10.410	4.655	8.762	9.489	54	53.864

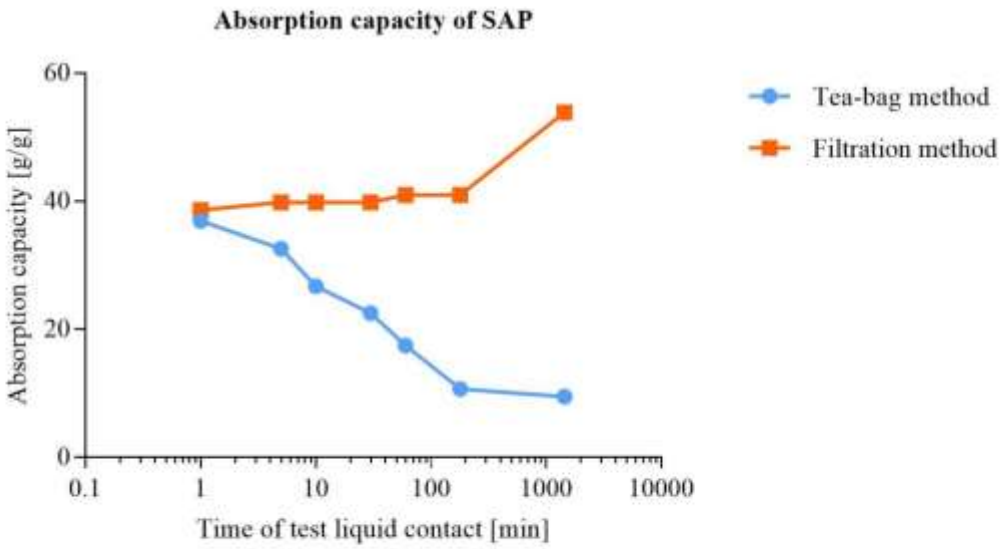

Fig. 2. Absorption capacity of superabsorbent polymer in filtrate of cement slurry at different SAP/liquid contact period using tea-bag method and filtration method

3 h of contact time but a change in the trend was observed at 30 min at which the rate of decrease was 15.69% which was lower than its preceding value of 18.01%. At 3 h, the rate of decrease in absorption capacity was found to be 38.87%, but at 24 h of contact, it was only 11.06%.

The trend observed in both tests were similar to that obtained from the RILEM round-robin test [7]. The difference in the value of absorption capacity in comparison to the RILEM round-robin test indicates that the type of superabsorbent polymer is a critical factor that affects the absorption capacity. Since a decline in the absorption capacity was seen throughout the test, the tea-bag method was the main focus of the investigation. While performing the tea-bag method of absorption test, a white precipitate was found in the test liquid which was not evident in the filtration method. Also, at the end of 24 h of SAP/liquid contact period, a white-coloured substance was found inside the tea-bag. The material found inside the tea-bag not even had the ability to absorb any liquid. In contrast to this, a transparent gel was found at 24 h of contact period in the filtration method. All these observations point out to a change in the properties of the superabsorbent polymer when it was used in the tea-bag method of absorption test.

Several investigations have found that the presence of ions in the test liquid may cause the chemical bonds in SAP to cleave when it is immersed in cement slurry filtrate [7, 8, 9]. The observations from the test also point towards such a reaction. The acrylate group present in SAP might have undergone a complex formation with the Ca^{2+} ions present in the test liquid. The rate of reduction in absorption capacity was discovered to be faster at younger ages than at older ages. This indicates that the ionic exchange between SAP and the test liquid was higher at earlier ages. The slower rate of decline in absorption capacity at 24 h may be due to the fact that the majority of ions in SAP have been exchanged with the test liquid. SAP was immersed in the test liquid in both techniques while comparing the tea-bag approach to the filtration method. The sole difference between the two methods was that the tea-bag procedure involved a change in the surrounding environment. In the tea-bag approach, the tea-bag containing hydrogel was removed from the beaker at the end of each contact period. In the filtration method, however, separate beakers were used for each measurement. That is, a disruption occurred to the sample during the tea-bag procedure. This shift in environment could have caused the ionic exchange between SAP and the test liquid.

The main application of superabsorbent polymer is as an internal curing agent in cement paste, mortar and concrete. Cement/aggregates are present in cement paste, mortar and concrete. However, neither the tea-bag approach nor the filtration method completely duplicates the conditions encountered in cement paste, mortar or concrete. As a result, a more accurate technique for detecting SAP absorption in cementitious environments is required. Although the tea-bag approach demonstrates desorption, which is a necessary step in internal curing, this desorption is not caused by the breaking of chemical bonds in SAP. Rather, it occurs as a result of the humidity difference that develops as a result of cement hydration [10]. As a result, it is not possible to say that the trend seen in the tea-bag approach is comparable to what occurs in a cementitious environment.

As stated earlier, the samples for measurement in the filtration method were kept in the same environment without any disturbance until the time of measurement. As a result, when compared to the tea-bag approach, the filtration method's results seem more appropriate. Hence, for the purpose of incorporating SAP in cement-based materials for internal curing, the absorption capacity obtained from the filtration method can be used. This test shows the absorption capacity at different SAP/liquid contact time. Among these, the required absorption capacity can be selected based on the initial setting time of the mixture in which SAP is to be incorporated.

V. CONCLUSIONS

In this paper, the absorption capacity of superabsorbent polymer was investigated. The test was carried out using two methods: the tea-bag method and the filtration method, in accordance with the recommendations of RILEM TC-260 RSC. The superabsorbent polymer used in this test was sodium polyacrylate. From the test results, the following conclusions can be made:

- When tested on the same type of SAP with the same test liquid, the tea-bag method and filtration method produce conflicting findings. In the case of the tea-bag approach, the absorption capacity decreased with the SAP/liquid contact period and vice versa for the filtration method.
- In the case of the tea-bag method, the decrease in absorption capacity can be attributed to the breaking of chemical bonds in SAP caused by ionic exchange between SAP and test liquid.
- The disturbance occurring to the sample in tea-bag method has caused the ionic exchange between SAP and test liquid.
- Both the tea-bag approach and the filtration method fall short of accurately describing the conditions present in cement paste, mortar or concrete.
- The absorption capacity produced from the filtration method is more appropriate for use in cement-based materials when compared to the tea-bag approach.
- Further studies are required for the measurement of the absorption capacity of superabsorbent polymer in exact cementitious environments.

VI. REFERENCES

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