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INFLUENCES OF SHRINKAGE REDUCING ADMIXTURE ON THE MECHANICAL PROPERTIES, DRYING SHRINKAGE, WATER ABSORPTION AND POROSITY OF PORTLAND CEMENT MORTAR

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Abstract

Drying shrinkage is the main cause of early age cracking of concrete and mortar. A wide range of research has been conducted to reduce the drying shrinkage, including using fibres or chemical admixtures. This paper investigated the effect of shrinkage reducing admixture on the flexural strength, compressive strength, drying shrinkage, water absorption and porosity of mortar. The mix compositions were ordinary Portland cement (OPC) : sand : liquid = 1 : 1 : 0.38 in which liquid consisted of water and shrinkage reducing admixture (SRA). SRA was used at the proportions of 2%, 4%, and 7% by weight of cement. The test results show that SRA reduces the flexural and compressive strengths of mortar. The reduction in flexural strength and compressive strength at 28 days is 14% and 25%, respectively at 7% SRA dosage. In addition, SRA significantly reduces the drying shrinkage and water absorption of mortar. At 7% SRA dosage, the drying shrinkage at 53 days is reduced by 60% while the water absorption rate at 24 hours is reduced by 54%. However, SRA has a minor effect on the pore size distribution, effective porosity, and cumulative intrusion volume of mortar.

Keywords: mortar; shrinkage reducing admixture; strength; drying shrinkage; water absorption; porosity.

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1. Introduction

Drying shrinkage is one of the primary reasons causing early age cracking of mortar and concrete. This phenomenon is caused by the loss of capillary water during the hardening of cement paste [1]. Drying shrinkage depends on many factors including mix composition, moisture, curing environment, etc. . . [2]. It is reported that drying shrinkage is also affected directly by the C-S-H characteristics and pore size distribution of cement paste [3, 4]. There are various solutions for reducing drying shrinkage including using polypropylene fibre [5], steel fibre [6] and shrinkage reducing admixtures [7]. A wide range of research has been conducted to investigate the effect of SRA on the shrinkage of mortar and concrete [8–11]. It is reported that when SRA was added to the mixes, the surface tension of the pore water decreased. As a result, the capillary tension within the pore structures reduced leading to the water evaporation [8, 12, 13]. Although SRA has its typical chemical composition depending on the suppliers, it can reduce the surface tension of the pore solution by more than 50% [14]. SRA decreases

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the shrinkage of concrete as it reduces the relative humidity in pore solution, which plays the most important part in shrinkage deformations [1].

Shrinkage reducing admixture was initially introduced in Japan in the 1980s [15] and used in the US by the late 1990s [16, 17]. It is a liquid organic compound consisting of a blend of propylene glycol derivatives. During the hydration of Portland cement, water evaporation leads to forming a layer at the air solution interface of the capillary pore when the internal humidity is decreased. The pore walls are pulled inward by the surface tension leading to shrinkage [18]. However, various surfactants in SRA adsorbed in the water -air interface in the pore structure leads to the reduction in the surface tension stress and reducing shrinkage [19]. In addition, SRA remains in the pore water after the cement paste hardening contributing to the continuous reduction in surface tension stress and drying shrinkage [18]. SRA is recognised as one of the most effective methods to reduce the shrinkage of concrete or mortar [20, 21].

A wide range of research has proved that SRA improves the performance of mortar and concrete. For example, SRA enhances durability due to reduced sorptivity [22] and reduces the diffusion coefficient of ions due to the increase in viscosity in pore water [14]. In contrast, SRA shows some adverse effects, including the delayed setting, decreased cement hydration rate and delayed strength development [23]. Although the effects of SRA on the strengths and drying shrinkage of concrete or mortar have been studied by many researchers the influences of SRA on the water absorption, microstructure, porosity and pore size distribution of mortar have not been fully investigated. The paper is aimed to study the influences of three proportions of SRA (2%, 4%, and 7% by weight of cement) on drying shrinkage, strengths, water absorption, microstructure, and porosity of Portland cement mortar.

2. Experimental programme

2.1. Materials and mixture compositions

A strength class of 52.5R Ordinary Portland CEM I from the supplier in Sheffield, UK was used in this test. The physical and chemical properties of ordinary Portland cement (OPC) are shown in Table 1. Fine aggregate was CEN standard supplied by the manufacturer in Cambridge, UK meeting the requirements of BS EN 196-1 [24]. The density of sand was 2.65 g/cm^3. Before conducting the test, sand was oven dried at temperature of 100°C for 24 hours to eliminate totally the moisture. Sand was then left in the laboratory air (20°C, 65% relative humidity) for additional 24 hours to reduce its temperature to about 20°C. Details of the mixture compositions are showed in Table 2. As can be seen that cement to sand to liquid ratio was remained constantly as 1:1:0.38 for all mixes in which liquid was the total of tap water and SRA. SRA was supplied by the company in Bradford, UK. It was a liquid- based admixture with the specific gravity at 20°C of 1.012 and the pH of about 7. SRA was used at the proportions of 2%, 4% and 7% by weight of cement, except for the control mix without SRA.

2.2. Casting and curing

Firstly, the liquid was prepared by mixing SRA and tap water. OPC and sand were then mixed in the 12 litres three speeds Hobart mixer for 1 minute. After that, the prepared liquid was added gradually to the mix while the mixer was running for about 2 minutes. Test samples were cast and cured as described in details in section 2.3.

Table 1. Physical and chemical properties of ordinary Portland cement

Physical properties	
Fineness by Blain method (cm^2/g)	4200
Specific gravity	3.15
Chemical compositions	
MgO (%)	0.98
Al_2O_3 (%)	5.51
SiO_2 (%)	18.31
P_2O_5 (%)	0.06
SO_3 (%)	2.54
K_2O (%)	1.01
CaO (%)	68.62
TiO_2 (%)	0.11
Fe_2O_3 (%)	2.61
ZnO (%)	0.08
SrO (%)	0.04
Loss on ignition (%)	2.20

Table 2. Mix composition of cement mortar

Sample Identification	OPC	Sand	*SRA(% by weight of OPC)	Water/OPC	**Liquid/OPC
M0 (0%SRA)	1	1	0	0.38	0.38
M1 (2%SRA)	1	1	2	0.36	0.38
M2 (4%SRA)	1	1	4	0.34	0.38
M3 (7%SRA)	1	1	7	0.31	0.38

*SRA = shrinkage reducing admixture; **Liquid = Water + SRA.

2.3. *Test procedure*

a. Mechanical properties

The flexural and compressive strengths of mortars were tested on the prisms of dimensions of 40 × 40 × 160 mm meeting the requirements of BS EN 196-1: 2005 [24]. Six samples were cast and demoulded after 24 hours of curing in the laboratory air (20°C, 65% relative humidity). After demoulding, specimens were cured in water to determine the strengths at 2 days and 28 days. The flexural strength was determined by three points bending test with the loading rate of 50N/s, and each value of flexural strength was the mean value of three specimens. The compressive strength of mortar was determined from the six broken halves obtained from the three prisms used to determine the flexural strength. The loading rate for compressive strength testing was 2 kN/s.

In order to study the microstructure of mortar, Scanning Electron Microscope (SEM) was conducted by using the SEM QUANTA 650 machine. Specimens used for SEM were derived from the inner core of prisms used for flexural strength tests at 28 days. The inner core of prism was used as it had better quality than the cast face of prism did for SEM. They were then oven dired at 45°C for 4 hours to eliminate the moisture content before gold coating. SEM images were obtained with an ETD detector, a working distance of about 10 mm, an accelerating voltage of 5 kV and a spot size of 4 nm.

b. Drying shrinkage

ASTM C596-18 [25] was applied to measure the drying shrinkages of mortars. Three prisms with dimensions of $40 \times 40 \times 160$ mm were cast and demoulded after 24 hours. After demoulding, each face of the prism was fixed with two demecs at a gauge length of 100 mm. The specimens were then continuously cured in water at 20°C, 45%RH. At 7 days, samples were removed from the water, dried with a cloth and the first (datum) strain reading was obtained with a demec extensometer. The distance between two demecs measured with an extensometer is strain (see Fig. 1). The samples were then cured in the humidity room at 20°C, 45%RH. Subsequent shrinkage readings were obtained at regular intervals up to 53 days.

Figure 1. Shrinkage measurement of mortar samples

c. Water absorption test

Water absorption rates of all mortars with and without SRA were determined in accordance with ASTM C1403-15 [26]. Samples used in this test were cubes of dimensions of $50 \times 50 \times 50$ mm. They were cast in steel moulds, demouled after 24 hours of curing in the laboratory air. They were then stored in airtight plastic bags and cured in a desiccator for 28 days before conducting the water absorption test. Each value of the water absorption rate was the mean value of three samples. The test cube surface (as cast) area was calculated by using callipers at three locations along with its height and recorded the average length (L_1) and width (L_2) in millimetres. The initial weight of each specimen was recorded immediately prior to testing (W_o). The test was conducted in the uptake container placing on a flat level surface. All specimens in the uptake container with their top faces (as cast) in contact with the specimen supports (see Fig. 2). Water was added to the uptake container ensuring that the cube samples were partially immersed in 3.0 ± 0.5mm of water. Then the uptake container was covered by the lid. During the test, the water level needs to be checked and ensured adequately at the designed level. The weight of cube samples was measured at interval times of 15 minutes, 1 hour, 4 hours, 24 hours, and 120 hours and recored as W_T. The water absorption at each time was calculated in Eq. (1).

$$A_T = \frac{10000 \times (W_T - W_o)}{L_1 \times L_2} \tag{1}$$

where W_T is the weight of the specimen at time T (g); W_o is the initial weight of the specimen (g); L_1 is average length of the test surface of the cube samples (mm); L_2 is average width of the test surface of the cube samples (mm).

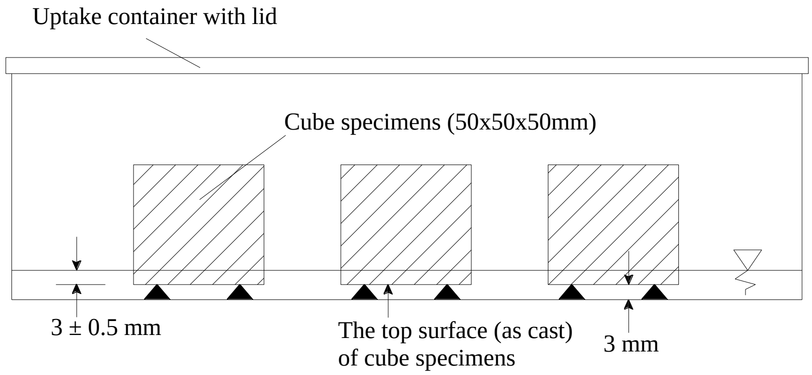


Figure 2. Schematic of water absorption test

d. Porosity

The porosities of the mortars with and without SRA were measured by Mercury Intrusion Porosimetry (MIP) PASCAL 140/240. After the flexural and compressive strength test, the inner core between 1 - 2g (0.002 - 0.004 lb) with an average length of 1 cm (0.39 in) of broken samples were derived and served as the porosimeter samples. The inner core of prism was used as it had better quality than the cast face of prism did for porosity test. They were then oven dried at 50°C for three days to eliminate the moisture content in the pore structures of cement paste. The next step was to treat the oven dried specimens in acetone for four hours and store them in a desiccator for 24 hours for preventing moisture migration from the air environment. It has been noted that silica gel was applied at the bottoms of the desiccator to isolate moisture migration from the air environment.

MIP samples were run in a PASCAL 140/240 machine consisting of two parts. The pressures were applied of up to 100 MPa and 200MPa for Pascal 140 part and Pascal 240 part, respectively. The range of pore sizes of 0.007 to 100 µm was applied in the test. The radius of pores was computed by using the Washburn equation (2). Data of effective porosity, pore size distribution was calculated and exported from the software connected to the PASCAL 140/240 machine.

$$r = 2\gamma \cos\theta / P \tag{2}$$

where r is the radius of pores (nm); γ is the surface tension of mercury (N/m) (assumed as 0.48 N/m); θ is the contact angle between mercury and concrete (assumed as 140°).

3. Results and discussion

3.1. Flexural and compressive strengths

Fig. 3 shows the flexural strengths of the control and SRA mortars. SRA reduced the flexural strengths at both 2 days and 28 days. The higher proportion of SRA used in the mix the more decrease in flexural strength. At 2 days, the flexural strengths of the M1(2% SRA), M2 (4% SRA) and M3 (7% SRA) were 3.81 MPa, 3.30 MPa and 2.64 MPa respectively while it was 4.27 MPa for the control mortar M0 (0% SRA). In comparison to the control mortar, the flexural strengths of SRA specimens were reduced by about 11%, 23% and 38% for M1 (2% SRA), M2 (4% SRA), M3 (7% SRA), respectively. At 28 days, the flexural strength of the M1 (2% SRA), M2 (4% SRA) and M3 (7% SRA) were 7.27 MPa, 6.72 MPa and 6.63 MPa respectively while it was 7.67 MPa for the control mortar M0 (0% SRA). It means that in comparison to the control specimen the reduction of about 5%, 13% and 14% for M1(2% SRA), M2 (4% SRA), M3 (7% SRA), respectively, were observed.

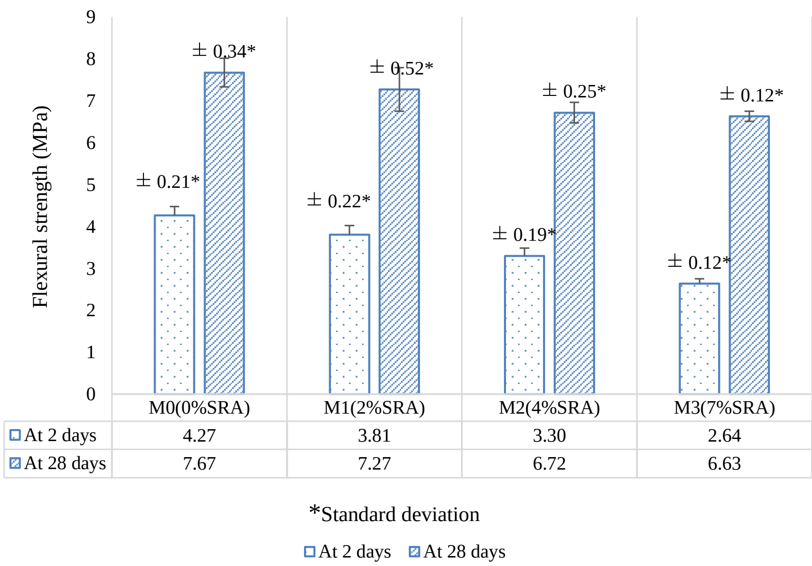
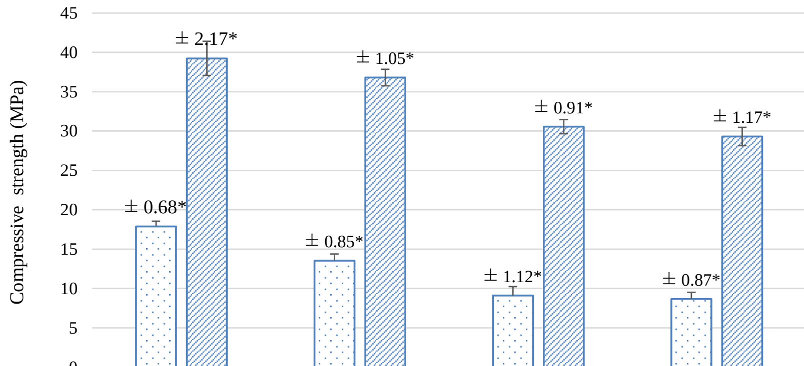


Figure 3. Effect of SRA on the flexural strength of mortars

Fig. 3 also shows that at the same SRA proportion, the reduction in flexural strength of SRA mortar in compared with the corresponding control sample at 2 days was higher than that of 28 days. For example, when 2% of SRA was added to the mixes, the reductions in flexural strengths in compared with the corresponding control samples were 11% and 5% at 2 days and 28 days, respectively. When 4% of SRA was added to the mixes, the reductions in flexural strengths in compared with the corresponding control samples were 23% and 13% at 2 days and 28 days, respectively. When 7% of SRA was added to the mixes, the reductions in flexural strengths in compared with the corresponding control samples were 38% and 14% at 2 days and 28 days, respectively. It is suggested that the effect of SRA on the delayed hydration process at later age reduced due to the water evaporation leading to the lower reduction in flexural strength at longer age.

Fig. 4 presents the effects of SRA on the compressive strengths of mortars. It is observed that SRA reduced the compressive strengths of mortars at both 2 days and 28 days. The higher proportion of SRA used in the mortar, the more decrease in compressive strength. For example, at 2 days the compressive strengths of the M1 (2% SRA), M2 (4% SRA) and M3 (7% SRA) were 13.54 MPa, 9.11 MPa and 8.66 MPa, respectively, while it was 17.87 MPa for the control mortar M0 (0% SRA). It means that in comparison to the control specimen, SRA reduced about 24%, 49% and 52% for M1 (2% SRA), M2 (4% SRA), M3 (7% SRA), respectively. At 28 days the compressive strengths of the M1 (2% SRA), M2 (4% SRA) and M3 (7% SRA) were 36.81 MPa, 30.55 MPa and 29.30 MPa respectively while it was 39.23 MPa for the control mortar M0(0% SRA). It means reducing about 6%, 22% and 25% for M1 (2% SRA), M2 (4% SRA), M3 (7% SRA), respectively, in comparison to the control mortar.

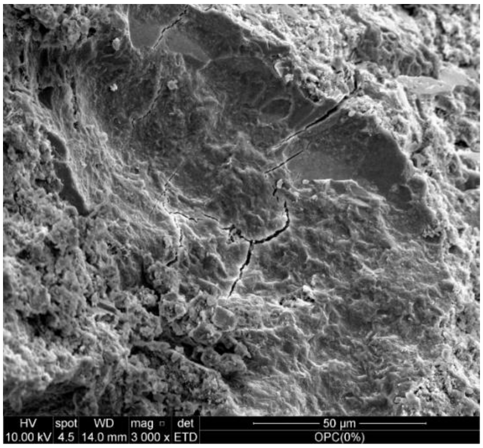


	M0(0%SRA)	M1(2%SRA)	M2(4%SRA)	M3(7%SRA)
At 2 days	17.87	13.54	9.11	8.66
At 28 days	39.23	36.81	30.55	29.30

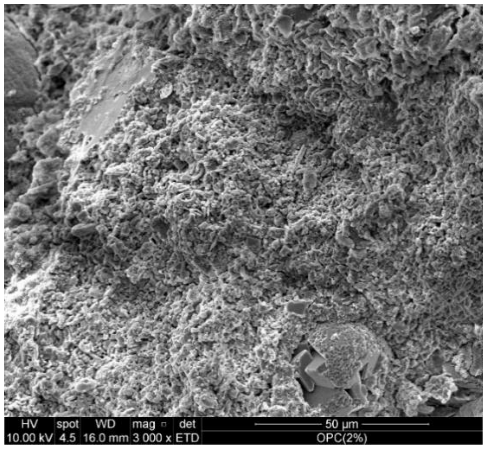
Figure 4. Effect of SRA on the compressive strength of mortars

Similar to flexural strength, at the same SRA proportion, the reduction in compressive strength of SRA mortar in compared with the corresponding control sample at 2 days was higher than that at 28 days. For example, when 2% of SRA was added to the mix, the reductions in flexural strengths in compared with the corresponding control samples were 24% and 6% at 2 days and 28 days, respectively. When 4% of SRA was added to the mixes, the reductions in flexural strengths in compared with the corresponding control samples were 49% and 22% at 2 days and 28 days, respectively. When 7% of SRA was added to the mix, the reductions in flexural strengths in compared with the corresponding control samples were 52% and 25% at 2 days and 28 days, respectively. It is suggested that the effect of SRA on the delayed hydration process at later age reduced due to the water evaporation leading to the lower reduction in compressive strength at longer age.

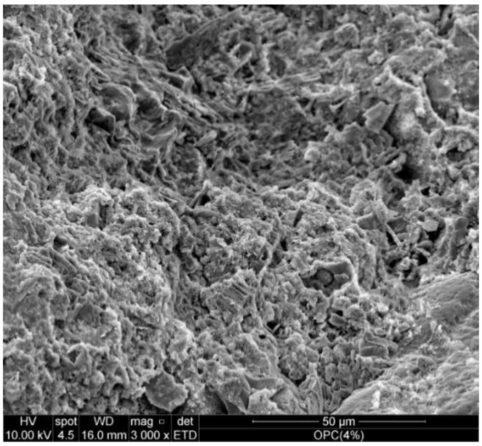
The reduction in flexural and compressive strengths due to the addition of SRA can be explained by the delayed hydration process of Portland cement [23, 27, 28]. In addition, SEM images at 28 days also show that SRA mortar samples have a less dense matrix than that of the control sample M0 (0% SRA), resulting in the reduction in strengths in comparison to the control specimen (Fig. 5). The more addition of SRA, the less dense in cement matrix. The reduction in strength of concrete and mortar due to addition of SRA is also confirmed by previous research [1, 29–32]. It is reported that SRA reduces the hydration kinetics resulting the decrease of portlandite after 24 hours of cement hydration process. Moreover, SRA also contributes to the increase in initial and final setting time of mortar [33, 34].



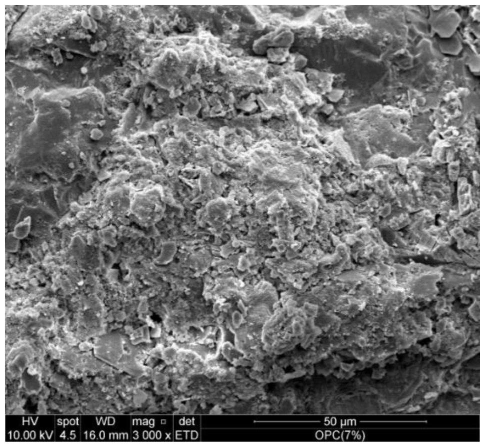
(a) M0 (0% SRA)



(b) M1 (2% SRA)



(c) M2 (4% SRA)



(d) M3 (7% SRA)

Figure 5. SEM micrographics at 28 days

3.2. Drying shrinkage

Fig. 6 shows the influences of SRA on the drying shrinkage of Portland cement mortars up to 53 days. The mean value of 6 readings obtained from three specimens was used for each data in Fig. 6. It can be seen clearly that SRA decreased the drying shrinkage of mortar and the more dosage used the more reduction in drying shrinkage. At 28 days, the drying shrinkage of all samples is around 820, 619, 442 and 241 microstrain for M0 (0% SRA), M1 (2% SRA), M2 (4% SRA), M3 (7% SRA), respectively. The decrease in drying shrinkage of SRA mortars compared with the M0 (0% SRA) sample is 25%, 46%, 70% for M1 (2% SRA), M2 (4% SRA), M3 (7% SRA), respectively. Therefore, although the strength of SRA mortar reduced the total drying shrinkage also reduced leading to the reduction in early age cracking of mortar. At 53 days, the drying shrinkage of all samples is 1013.04, 884.2, 6651,2 and 402 microstrain for M0 (0% SRA), M1 (2% SRA), M2 (4% SRA) and M3 (7% SRA), respectively. The drying shrinkages of SRA samples reduced by 17%, 36% and 60% compared with the control sample when SRA was added at 2%, 4% and 7% by weight of OPC, respectively. This agrees well with previous research where SRA decreases the drying shrinkage of mortar and concrete [1, 19, 35, 36].

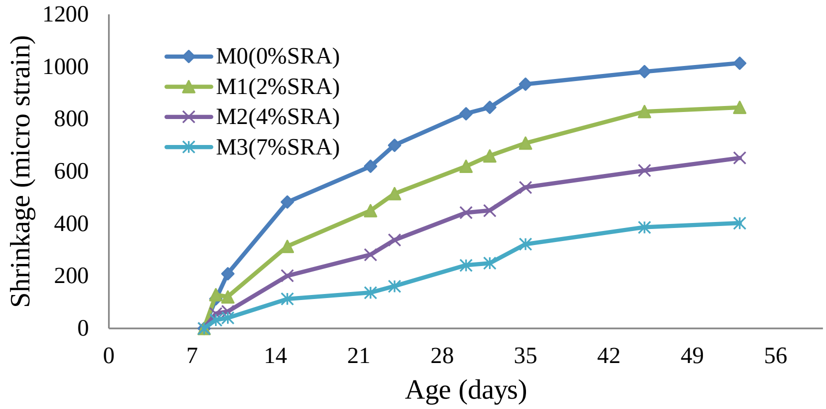


Figure 6. Drying shrinkage of mortars with and without SRA

The mechanism of SRA to reduce the shrinkage has been studied by many researchers. SRA causes the decrease of surface tension of pore water [8, 11, 37]. As a result, shrinkage deformation caused by drying is reduced. Other research has confirmed that the internal relative humidity of mortar has been changed due to the addition of the SRA [11]. This is explained by the appearance of a protective outer layer of mortar with high SRA concentration in the initial drying stage. This outer layer prevents the drying process in the inner layer due to its lower surface tension [1].

3.3. Water absorption test

Fig. 7 presents the water absorption rates of the control and SRA mortar specimens. It can be seen clearly that SRA decreased the water absorption of mortar, and the more proportion of SRA, the less water absorption. At 24 hours the water absorption rates of M1 (2% SRA), M2 (4% SRA) and M3 (7% SRA) are 79.2, 68.6 and 51.6 (g/100cm^2) while it was 113.3 (g/100cm^2) for the control sample M0 (0% SRA). It means that the water absorption rate is reduced by 30%, 39%, and 54% at 2%, 4% and 7% SRA dosage, respectively. This reduction can be explained by the presence of SRA in the pore structures as it may not be consumed during the hydration of cement paste [1]. SRA reduces the surface tension stress of water with the wall of capillary pores. As a result, capillary suction of water

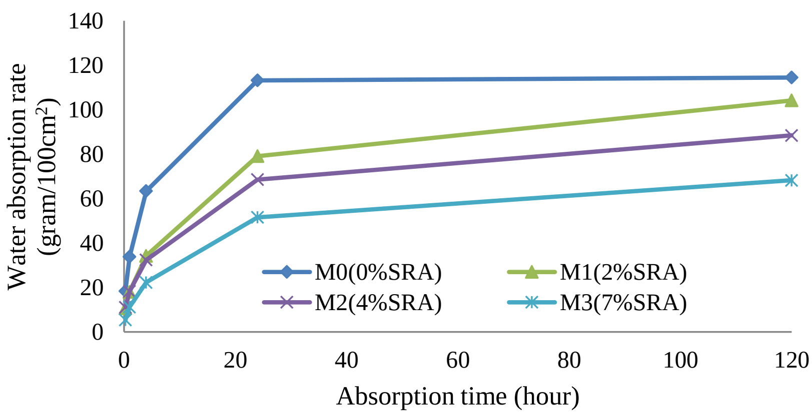


Figure 7. Water absorption of all mortar samples

into cavities reduces leading to the decrease in the water absorption [38–41]. The test results agree well with the previous research in both experimental work and theoretical modelling. SRA contributes to the reduction in the sorptivity and moisture diffusity and improves the durability of cement paste as it reduces the chloride and deleterious ion absorption and migration [42].

3.4. Porosity

Fig. 8 shows the pore size distribution of the mortars with and without SRA. The significant levels of differential pore volume are observed in the range of pore diameter of 0.01 µm to 1 µm while the insignificant differential pore volume (zero or near zero) are observed in the range of pore diameter of 1 µm to 100 µm. The porosity is observed at the zone with significant differential pore volume, while the zero differential pore volume represents a non-porous zone. It is observed from Fig. 8 that all mortar samples had a unimodal pore distribution as a single range of pore volume was observed within the differential pore volume curves [43]. It can been seen that SRA has a minor influence on the pore size distribution of mortar. Moreover, Table 3 also shows the insignificant influences of SRA on the cumulative intrusion volume and effective porosity of mortars, and this is also confirmed by previous research [10, 44]. The effective porosities of M1 (2% SRA), M2 (4% SRA) and M3 (7% SRA) are 15.868%, 16.467% and 16.773%, respectively while it is 16.808% for the control mortar M0 (0% SRA).

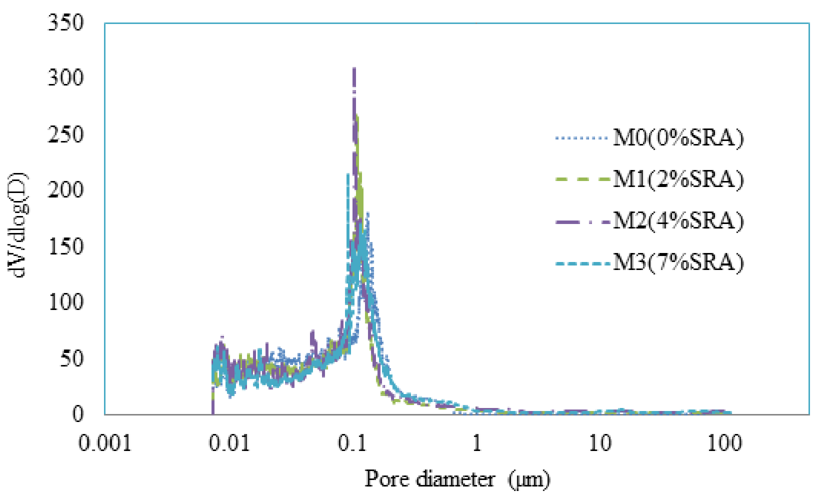


Figure 8. Differential volume of intruded mercury versus pore diameter of mortar at 28 days

Table 3. Effective porosities and cumulative intrusion volumes of all mortars

Sample ID	Effective porosity (%)	Cumulative intrusion volume (mm^3/g)
M0 (0%SRA)	16.808	89.38
M1 (2%SRA)	15.868	86.94
M2 (4%SRA)	16.467	91.14
M3 (7%SRA)	16.773	89.63

4. Conclusions

The paper has investigated the effects of three proportions of SRA on strengths, drying shrinkage, water absorption rate, SEM and porosity of mortars. The test results re-confirm that SRA reduces the strengths and drying shrinkage of mortar. In addition, the paper also studied the influences of SRA on the water absorption and porosity, pore size distribution which were not fully investigated in the previous work. The following conclusions can be drawn from the test results:

- SRA reduces the flexural and compressive strengths of mortars, the higher proportion of SRA the more decrease in strengths. The reductions in flexural strength and compressive strength at 28 days are 14% and 25%, respectively at 7% SRA dosage. The reduction in strength of SRA mortar at later age is less than that at early age.

- SRA reduces significantly the drying shrinkage of mortar and the higher proportion of SRA the more reduction in drying shrinkage. At 53 days, the drying shrinkage is reduced by 17%, 36%, and 60% at 2%, 4% and 7% SRA dosage, respectively.

- SRA reduces the water absorption rate of mortar and the more SRA added the less water absorption rate. This improvement can be explained by the presence of SRA in the pore structures as it may not be consumed during the hydration of cement paste. At 24 hours, the water absorption rate is reduced by 30%, 39%, and 54% at 2%, 4% and 7% SRA dosage, respectively.

- SRA has a minor effect on the pore size distribution, effective porosity, and cumulative intruded volume of mortar.

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