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Abstract: Bond defects at rubber-cement matrix interface are detrimental to durability of the cement composite. Therefore, coating rubber aggregates with copolymer has been suggested to overcome this defect. This paper aims to investigate the effect of an improved rubber-cement matrix bond on frost resistance. Freeze-thaw temperature cycles were controlled by a thermal sensor embedded inside the core of a mortar specimen. Measurements of relevant quantities, such as mass loss, length change, mechanical properties (relative dynamic modulus of elasticity, compressive and flexural strengths), and durability factor,

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demonstrated that rubberized cement-based materials were more resistant under freeze-thaw environments than the control one. Especially, regardless of slight length gain of mortar incorporating coated rubber aggregates, copolymer coating still made the composite durable in frost conditions owing to its improved strain capacity and higher residual post-peak tensile strength.

Keywords: Rubber aggregates; rubber-cement matrix interface; rubber coating; freeze-thaw resistance; durability factor; strain capacity.

1 Introduction

Rubber aggregates (RA) addition into cementitious mixtures was reported to improve resistance of cement-based composites to freeze-thaw action [1–8]. Benazzouk et al. [1] studied the frost behaviour of cement-rubber composites in which RA contents were ranging from 0% to 40%. The authors reported a reduction in both mass loss and relative dynamic modulus loss of the materials containing 30% and 40% of RA by volume, demonstrating an improvement in frost resistance of the composites. According to Paine et al. [2], 6% volume of RA incorporation was suitable to improve frost durability in terms of resisting scaling phenomenon and of limiting a decrease in relative dynamic modulus. However, due to bond defects at rubber-cement matrix interface, performance of rubberized concrete under freeze-thaw conditions appeared less impressive than that of the one manufactured with a high content of air entrained [2]. Al-Akhras et al. [3] investigated relative dynamic modulus of elasticity during freeze-thaw actions and reported that, compared to the control mortar, the ones incorporating rubber ash (size 0.15 mm) as natural sand replacement at two distinct levels of 5% and 10% by weight exhibited higher durability factor, which was determined according to ASTM C666/C666M standard [9]. It should be noted that relative dynamic elastic modulus is defined as a variation in the dynamic modulus of the specimens.

The detrimental effect of RA addition on freeze-thaw resistance of rubberized cement-based composites was also reported. Karahan et al. [10] partially replaced natural sand in self-consolidating concretes with RA (size 4.75-0.15 mm) at different RA contents of 0%, 10%, 20% and 30%, by volume. Their experimental results showed some spalling on surface of rubberized concrete specimens, and a gradual loss in flexural strength and mass was observed with an increase of rubber content. Similarly, acording to investigations of Savas et al. [11], increasing rubber volume would decrease the freeze-thaw durability of rubberized concrete, which was measured according to ASTM C666/C666M standard [9] using procedure A. Among previous studies, Richardson et al. [7] found that washing RA before adding them to cementitious mixtures led to a composite with lower pulse velocity but reduced weight loss under freeze-thaw conditions. As reported by Si et al. [12], resistance of rubberized concrete to freeze-thaw environments appeared more significant in composites incorporating 15% by volume of sodium-treated RA replacing fine aggregates (rubber size 1.44 - 2.83 mm and 40-minute sodium treatment with the concentration of

4%), especially in terms of preventing both mass loss and relative dynamic elastic modulus reduction.

As briefly summarized above, there is still no consensus about the role of RA in cementitious mixtures against freeze-thaw conditions. Also, all previous studies have only evaluated durability of rubberized cement-based composites to frost actions thanks to mass loss and changes in relative dynamic modulus of elasticity. Moreover, no investigations have been assessed on bond effects between RA and cement matrix on freeze-thaw durability of the composites. This study therefore aims to characterize freeze-thaw resistance of two rubberized mortars, one of them incorporating polymer-coated RA, as demonstrated by Pham et al. [13, 14], to obtain an enhanced RA-cement matrix interfacial transitional zone. The mass loss, changes in ultrasonic pulse velocity and relative dynamic modulus of elasticity, residual mechanical properties (compressive and flexural strengths), durability factor, and especially length change of these mortars under freeze-thaw actions were investigated and compared to the ones of control mortar.

2 Materials and methods

2.1 Materials and mix proportions

As mentioned earlier, control mortar and two rubberized ones were investigated. Materials used for making these mortars include cement CEM I (52.5 R), natural sand (0-4 mm), RA (similar size as sand), and water. It should be noted that, in these rubberized mortars, only 30% volume of sand was replaced by RA. Compared to higher specific gravity (2.62) and significant water absorption (1.9%) of sand, RA have a lower density of 1.2 and are hydrophobic materials. These characteristics can explain a reduction in workability and segregation phenomena of rubberized cement-based mixtures. Hence, superplasticizer and viscosity agent were used to maintain workability and to make sure homogeneity of the composite, respectively. It is worth recalling that hydrophobic nature of RA is a main reason of higher porosity in rubberized cement-based composites due to air-entrapment effect when RA are in contact with mixing water. Obviously, bond defects at untreated rubber-cement matrix interface also contribute to such property. The difference in size distribution between RA and sand used in this work is described in Fig. 1.

Three mortars studied (control, untreated and coated rubberized ones) and their mix proportions are presented in Table 1. It should be noted that acronyms UR and CR denote Untreated Rubber and Coated RA, respectively; letter P stands for coPolymer, which is as bonding material to enhance the interfacial zone between RA and cement matrix. The rubber-cement matrix enhancement demonstrated in Fig. 2 was obtained using a coating method [14]. Firstly, RA were required to precoat with styrene-butadiene-type copolymer (2% mass of RA). The processed RA were then maintained in a conditioned room fixed at 20 °C temperature and at 50% relative humidity for 1 hour. This step is necessary for copolymer's condensation and stabilization on RA surface. Finally, pre-mixed cementitious mixture was prepared for a light and short mixing with coated RA.

Prismatic mortar specimens (40 mm x 40 mm x 160 mm) were prepared for freeze-thaw test. For monitoring length change, during mould preparation and casting process, two steel pinholes were embedded at the center of two head ends of mortar specimens. Then 24 hours after casting, the specimens were demoulded and placed in the curing room maintained under controlled atmosphere (20 °C temperature and 95% relative humidity) for 27 days before starting freeze-thaw cycles.

2.2 Freeze-thaw test programme

The freeze-thaw resistance test of control and rubberized mortars was carried out according to NF P18-424 standard [15] in combination of ASTM C666/C666M-15 standard - Procedure A [9]. An environmental chamber is used to simulate freeze-thaw cycles. It is able to induce the highest and lowest temperatures, namely 150 °C and -40 °C.

In order to induce frost actions, temperature inside the chamber can be controlled by either a chamber sensor or the one embedded in the core of a specimen (Fig. 5). In this work, freeze-thaw cycles were established in accordance with the latter case. Actual temperatures in the chamber and at the core of a mortar specimen were recorded during the test. The temperature-controlled specimens illustrated in Fig. 3 were made of untreated rubberized mortar (30UR). The authors' experience showed that during casting and hardening process or under freeze-thaw actions, the thermal sensor embedded at the center of the temperature-controlled specimen can fail, stop to work and need to be replaced by another specimen. Hence, to prevent these hazards, several temperature-controlled specimens were prepared and placed in the chamber as tested specimens. The curing process of such specimens was similar to the one of tested mortars. One should notice that the connecting systems including electronic wire lines and the sensor connectors must be protected under high moisture condition of curing.

Fig. 4 shows the temperature cycle set-up for the freeze-thaw test. The black, red, green colors indicate the target freeze-thaw temperature, and the ones of the chamber and at the core of the control specimen, respectively. The actual core temperature was dropped from 4 ± 2 °C to -18 ± 2 °C for around 3.0 hours, kept at -18 ± 2 °C for 0.5 hour, raised from -18 ± 2 °C to 4 ± 2 °C for other 2.0 hours, and kept at 4 ± 2 °C for 0.5 hour. The duration of a freeze-thaw cycle was 6 hours. It therefore allowed 4 cycles per day to be conducted. This core temperature cycle obtained was quite adapted to the requirement of standards [9,15]. Indeed, it was difficult to set up the core temperature of the controlled specimen close to the one inside the chamber due to requirement of thermal conductivity time into mortar specimens.

The arrangement of mortar specimens in the chamber is shown in Fig. 5. Before transferring the specimens into the chamber, the initial length, mass, and ultrasonic pulse velocity of prismatic mortar specimens were measured. Compressive and flexural tests were also carried out in order to determine initial strengths. It should be noted that the specimens were required to dry carefully using a sponge to remove only surface water before weight measurement. In general, the freeze-thaw test is continuing until the mortar specimens have been subjected to 300 cycles or terminated earlier if the relative length change overpasses 500 $\mu m/m$ [14] or 1000 $\mu m/m$ [9]; or the relative dynamic modulus of elasticity falls below 60%, as recommended in standards [9, 15]. In this study, the test was finalized when the expansion of mortar specimens exceeds 0.1% (1000 $\mu m/m$) of its original dimension.

After a given number of freeze-thaw cycles, length change, mass loss, ultrasonic pulse velocity were measured. Flexural and compressive tests on the prismatic mortar specimens were only performed at 130 cycles and at the test end. Details of necessary tests are described as below.

2.2.1 Mass loss and length changes

During the freeze-thaw test, the specimen mass was simply measured by weighing the surface-dried specimens using a scale with an accuracy of 0.01 g. The mass loss is determined according to Eq. (1), where m_o and m_i are weights before starting freeze-thaw test and after i cycles of freezing and thawing, respectively. A length sensor with a precision of 1 μm was used for determining the length of mortar specimens. Length gain (dimensional expansion) is then calculated according to Eq. (2), where L_1, L_i are the readings on the length sensor at the beginning of the test and at the i_{th} freeze-thaw cycle, respectively; and L_0 is the initial distance between two steel pinholes (specimen length of 160 mm). The average mass loss and length change from three specimens of each mortar were reported.

$$Mass\, loss\, (\%) = \frac{m_0 - m_i}{m_0}\, .\, 100 \tag{1}$$

$$Length\ gain\ (\mu m/m) = \frac{L_i - L_1}{L_0} \tag{2}$$

2.2.2 Ultrasonic pulse velocity test

The ultrasonic pulse velocity was determined according to NF EN 12504-4 standard [16]. The tester used mainly consists of a control unit (an electrical pulse generator, an amplifier, and an electronic timing device) and a pair of transducers. The devices with a frequency of 54 kHz are used to generate an ultrasonic pulse to travel on the path length of 160 mm from the transmitting transducer to the receiving one. The apparatus must be calibrated at every testing time using a calibration bar. The time duration for acoustic wave to propagate through the longitudinal direction of the specimen and the ultrasonic pulse velocity were recorded. Three specimens of each mortar type and at least five measurements for each specimen were taken to make sure that the variation between the measured transit time on single tested specimen should be within ± 1 % of the mean value of these three measurements. Note that the specimen surface contacted with transducers must be smooth enough by coating a quick-setting epoxy resin, especially when mortars are subjected to damage after a given number of freeze-thaw cycles.

From values of ultrasonic pulse velocity, a relative dynamic modulus of elasticity (P_c) is calculated as Eq. (3) [17], where v_c and v_o are ultrasonic pulse velocities at the c_{th} cycle of freezing and thawing and at time right before starting freeze-thaw test, respectively. The durability factor (DF) is finally determined at the end of freeze-thaw test using Eq.

(4), as recommended by ASTM C666/C666M-15 standard [9].

$$P_c\,(\%) = \frac{v_c^2}{v_0^2}\,.\,100 \tag{3}$$

$$DF = \frac{P.N}{M} \tag{4}$$

where P (%) is relative value of dynamic elastic modulus at N^{th} cycle; N is the number of freeze-thaw cycles at which P drops to the minimum value for terminating the frost test (60% as required by standards [9, 15]) or the selected number of freeze-thaw cycles when frost actions are to be ended (whichever is less); and M is the specific number of cycles at the end of freeze-thaw test.

2.2.3 Flexural and compressive tests

The flexural and compressive tests were carried out on prism mortar specimens according to NF EN 1015-11 standard [18]. Firstly, three point-bending tests were carried out on prismatic mortar specimens to get load-bearing capacity. The two rollers of the flexural tests were spaced at a distance of 100 mm. Two parts of specimens obtained after the bending tests were then compressed on an area of 40 mm x 40 mm to obtain the compressive strength. The loading rates of flexural and compressive tests were 0.05 kN/s and 0.5 kN/s, respectively.

3 Results and discussion

3.1 Mass loss

Fig. 6 shows mass loss versus number of freeze-thaw cycles for three types of mortars. The untreated rubberized mortar specimens were observed to increase slightly in mass for the first 50 cycles of freezing and thawing. It is due to the higher air-void density of this mortar, which still absorbs water to reach a critical degree of saturation [12]. The mass of the control mortar specimens started to decrease quickly since the 160^{th} cycles of freezing and thawing, and to become severely deteriorated at approximately 200 cycles compared to other rubberized mortar specimens (Fig. 7). A slight difference in mass loss was also observed between rubberized mortar using untreated RA and the one incorporating copolymer coated RA.

3.2 Length gain

The expansion of all mortar specimens under the freeze-thaw action is illustrated in Fig. 8. It can be clearly seen that the length gain of control mortar is much higher than the one of rubberized mortars. The smaller length change of rubberized mortar specimens exposed to freeze-thaw cycles can be explained as below:

(i) As demonstrated from Scanning Electron Microscope (SEM) observations and air permeability values from the same composites [13, 14], incorporation of RA in cementitious mixture induces high porosity not only at the poor rubber-cement matrix interface, but also especially high density of air pores in the core of cementitious matrix. It means that many escape spaces are formed in rubberized mortars. As explained by Mehta et al. [19], much lower temperature was required to freeze water in capillary pores than the one in gel pores. Higher energy state in gel pores forces water to transport to the capillary ones in order to balance the energy between these pores. Therefore, such escape spaces in rubberized cement-based composites play an important role to reduce energy gradient, leading to a decrease in expansion of rubberized mortars. Normally, spacing factor, a parameter associated with the distance from the periphery of an air void to adjacent ones in cementitious matrix measured on microscopic scanning pictures of air-void system according to ASTM C457/C457M [20] is used to estimate whether the cement-based composites are resistant to freeze-thaw actions [21]. However, due to unexpected problems related to polishing procedure of rubberized mortar specimens, spacing factor is therefore difficult to qualify accurately [11]. Additional voids would be generated due to low stiffness of RA and bond defects available at untreated rubber-cement matrix interface.

(ii) It is necessary to recall that low stiff RA should help in absorbing energy induced by a given damage process [22]. Hence, compared to the control mortar, high amount of energy from freeze-thaw actions in rubberized composites is assumed to be released.

(iii) Low water porosity and capillary absorption of rubberized cement-based composites compared to the control one can reduce the volume change due to ice formation. Moreover, high water absorption of natural aggregates (sand) is also detrimental to the durability under freeze-thaw performance than RA that do not absorb water.

(iv) According to Sahmaran et al. [23], tensile strain capacity and strain-hardening behaviour were also important to prevent damage from freeze-thaw actions. As demonstrated by previous authors [24–26], low stiffness property of RA is beneficial to improve strain capacity, the deformation at failure of rubberized cement-based composites, and to result in higher residual post-peak tensile strength. Hence, freeze-thaw resistance of rubberized mortars was consequently improved.

The difference in length change between rubberized mortar incorporating untreated RA and the one using coated RA was also observed. Rubberized mortars incorporating coated RA exhibited a slightly higher expansion. It can be explained by the fact that rubber coating resulted in an improved rubber-cement matrix bond and a partial reduction in gel and capillary pores in the composite. The frost resistance of mortar specimens can be evaluated through the total number of freeze-thaw cycles at which the relative length change exceeds 500 $\mu m/m$ [15] or 0.1% of the original length [9]. As illustrated in Fig. 8 and Table 2, it can be concluded that rubberized mortars are more resistant to freeze-thaw environments in term of length change due to a tolerance towards a greater number of freeze-thaw cycles.

3.3 Ultrasonic pulse velocity

The ultrasonic pulse velocity of mortar specimens over a freeze-thaw test period is presented in Fig. 9. The initial ultrasonic pulse velocities of rubberized mortars after 28 days in the curing room were lower than the one of control mortar. Higher density of pores in the cement matrix due to air entrapment phenomenon during casting process of rubberized cementitious mixtures, bond defects at untreated rubber-cement matrix interface, and low density of the composite are qualified explanations why the transmitting time of ultrasonic pulse waves in rubberized mortars is delayed, leading to low pulse velocity of rubberized mortars.

Under frost actions, the pulse velocity of all mortar specimens remained similarly as original values over the first 50 cycles of freezing and thawing. After this period, while pulse velocity of rubberized mortars seemed to be still constant, the one of control mortar decreased gradually and reached the value lower than the one of rubberized mortars after approximately 180 cycles of freezing and thawing. This is a consequence of damage induced by ice pressure inside the control mortar. On the contrary, presence of RA can absorb energy induced by volumetric expansion during phase change of water. Therefore, control mortar was less durable than the rubberized mortars, which highlighted the greater relevance of the pores induced by presence of RA in the composites. Coating RA with copolymer seemed to have no specific effect on a change of pulse velocity during a 200 freeze-thaw cycle testing period.

Based on ultrasonic pulse velocity, cement-based materials can be classified as excellent, good, questionable, poor and very poor qualities [27]. One found that initial pulse velocity values of all control and rubberized mortars were greater than 3660 m/s and less than 4575 m/s, thus classifying them as good mortars. At the end of freeze-thaw test, while rubberized mortars still remained their original classification, the control mortar dropped to a lower standard, a questionable one.

The number of freeze-thaw cycles where relative dynamic modulus of elasticity falls below 60% is also considered as a parameter to express the frost resistance of mortar/concrete [9]. Fig. 10 presents the relative dynamic modulus of elasticity over a period of 200 cycles of freezing and thawing. It can be clearly seen that none of control mortar specimens was freeze-thaw resistant after 200 cycles. On the contrary, rubberized mortar specimens exhibited a very good performance under frost action. Durability factor (DF) determined at the test end for three types of mortars is compared in Table 2. Based on durability factor values, Hansen [28] reported that freeze-thaw resistance of cement-based composites can be classified as follows: Nonresistance to freeze-thaw actions ($DF \leq 40\%$), doubtful frost resistance (40% $<$DF$\leq$60%), acceptable frost resistance (60% $<$DF$\leq$80%), and frost resistance ($DF > 80\%$). Therefore, it can be concluded that incorporating 30% volume of RA (0 - 4 mm) to cementitious mortars as a sand replacement produced "frost resistance" of rubberized mortars compared the "doubtful frost resistance" of the control one.

3.4 Loss of compressive and flexural strengths

The residual compressive and flexural strengths after 130 freeze-thaw cycles and at the test end are presented in Figs. 11 and 12, respectively. One should note that zero values of such mechanical properties of the control mortar at the test end because of a serve deterioration on the surface of the specimens. At the 130^{th} cycle of freezing and thawing, compressive and flexural strengths of the control mortar were decreased by 33% and 31%, respectively. Despite low expansion and insignificant change in pulse velocity of untreated rubberized mortar specimens, a slight reduction in compressive and flexural strengths of this type of mortar was still observed. It can be attributed to the additional water absorption of untreated rubberized mortar during the first period of freeze-thaw action. The enhanced bond between RA and cement matrix was observed to maintain mechanical properties (flexural and compressive strengths) with increasing the number of freeze-thaw cycles. It is explained by Sahmaran et al. [23] that, in addition to the air-void system, other parameters such as high tensile strain capacity and strain-hardening behaviour of cement-based composites are important for resisting cycles of freezing and thawing. As reported by Pham et al. [13, 14], coating RA with copolymer before mixing them with cementitious mixture was demonstrated to improve strain capacity and residual post-peak tensile strength compared to the untreated one. Therefore, the coated rubberized mortar was still durable under freeze-thaw cycles regardless of a slight length gain as reported above.

4 Conclusions

Freeze-thaw resistance of rubberized mortars were investigated and compared to the one of control mortar. From experimental results, the following conclusions can be drawn:

- Rubberized cement-based composites were more resistant to frost environments than the control one. It was especially validated based on the length change.

- Durability of rubberized mortars under freeze-thaw conditions can be attributed to high energy absorption and hydrophobic nature of RA and to lower water capillary absorption, high strain capacity and better residual post-peak performance of the composites.

- Rubber coating to enhance rubber-cement matrix interface led to a slight increase in length change of coated rubberized mortar under freeze-thaw actions, but the composite was still more durable than the control mortar under frost environment.

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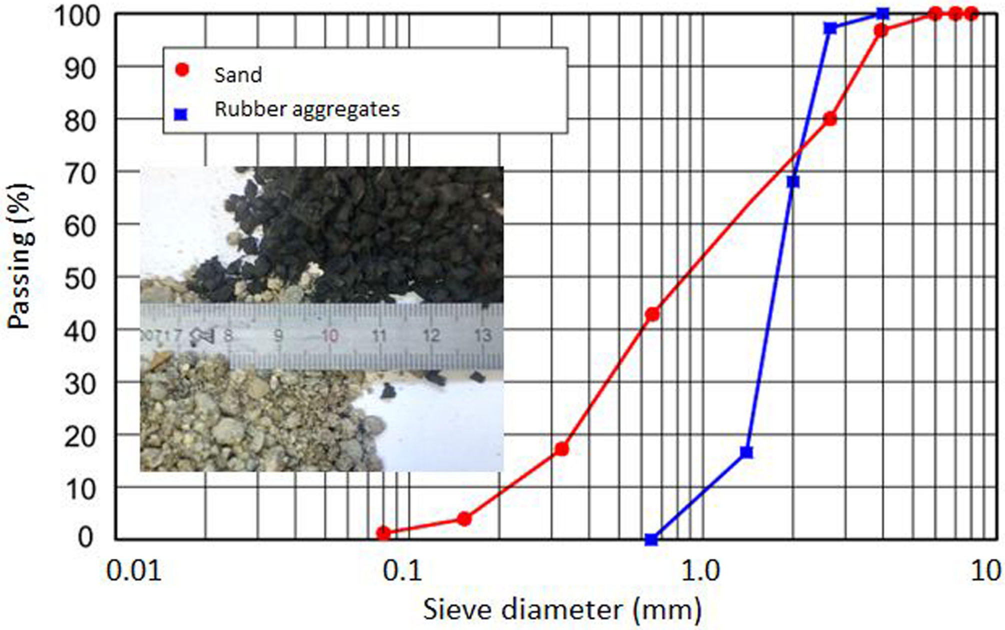

Figure 1: Difference in size distribution between sand and RA

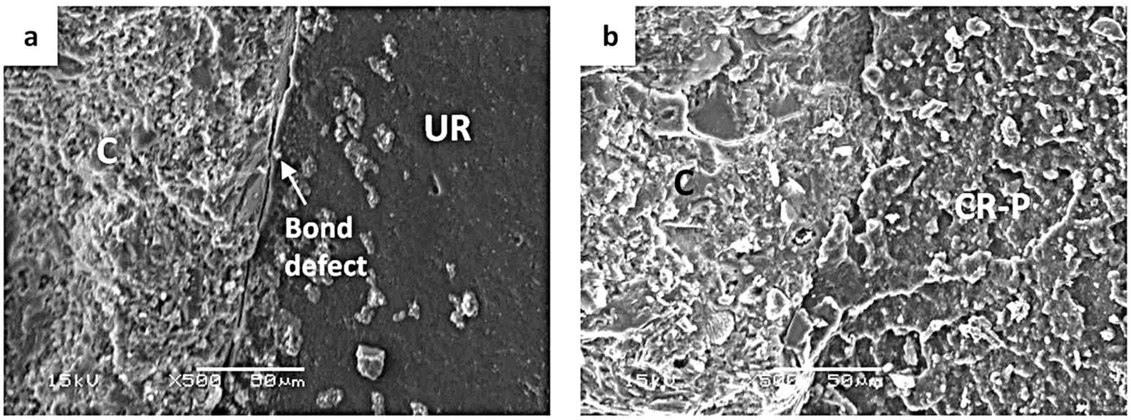

Figure 2: Effect of copolymer coating on rubber-cementitious matrix interface: (a) bond defects (UR-Untreated RA), and (b) bond enhancement between cementitious matrix (C) and coated RA (CR-P) [14]

Figure 3: Preparation of temperature-controlled specimens with thermal sensors

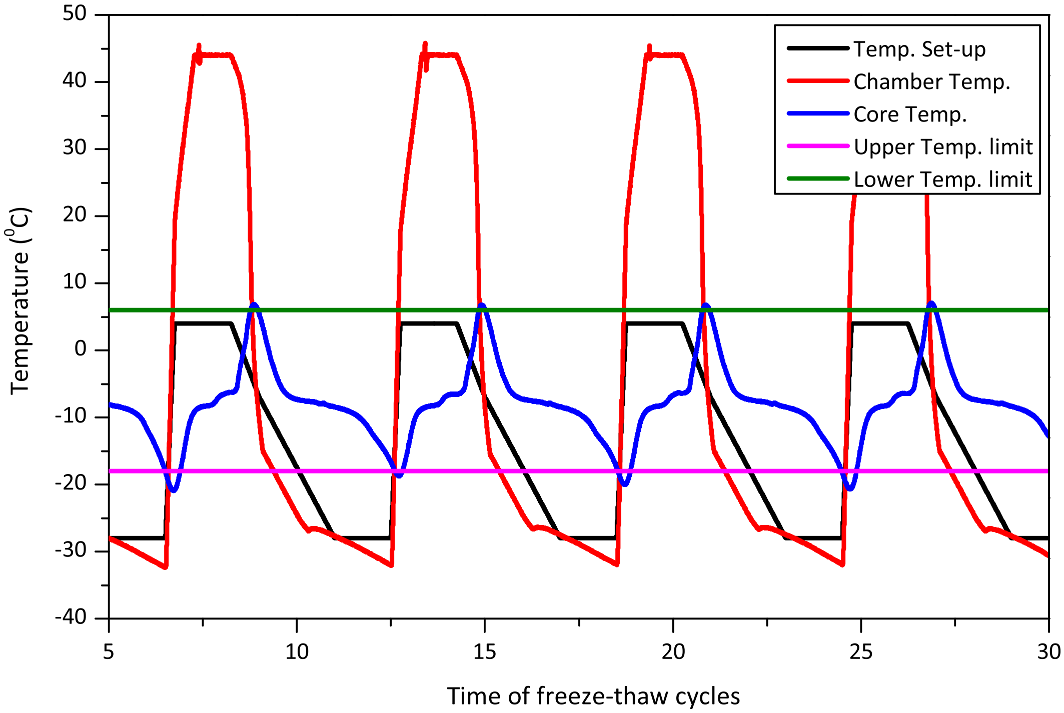

Figure 4: Freeze-thaw temperature cycle set-up

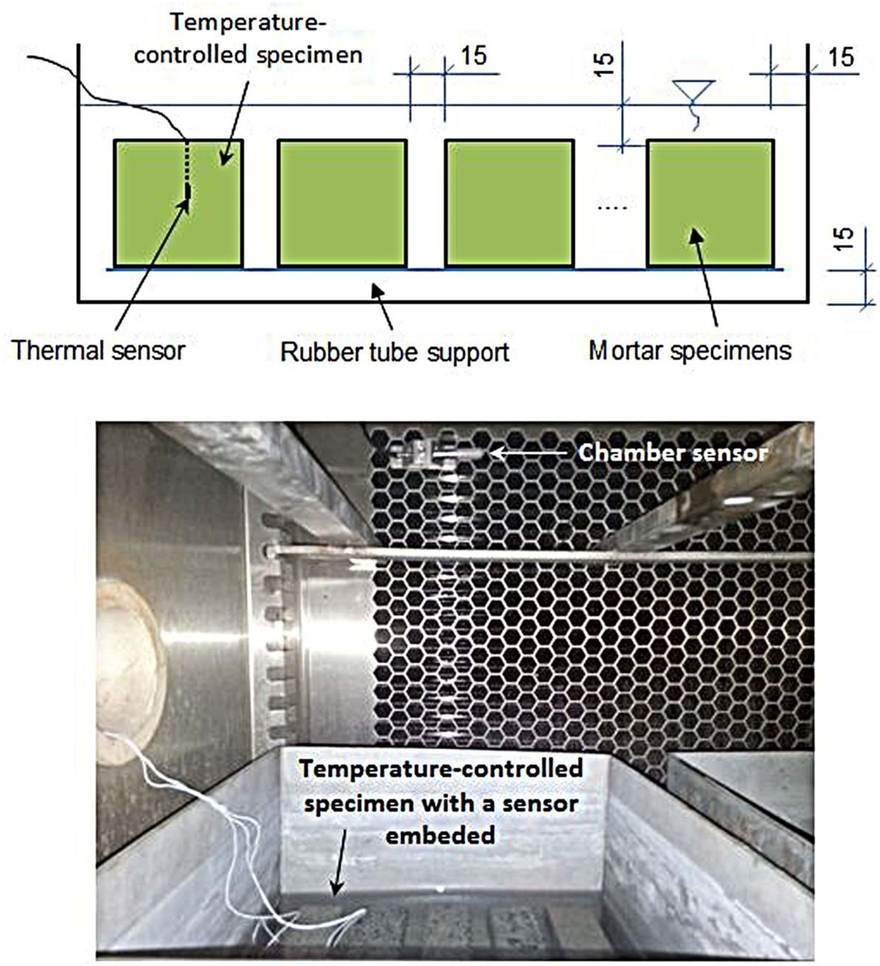

Figure 5: Specimen arrangement in freeze-thaw chamber (unit: mm)

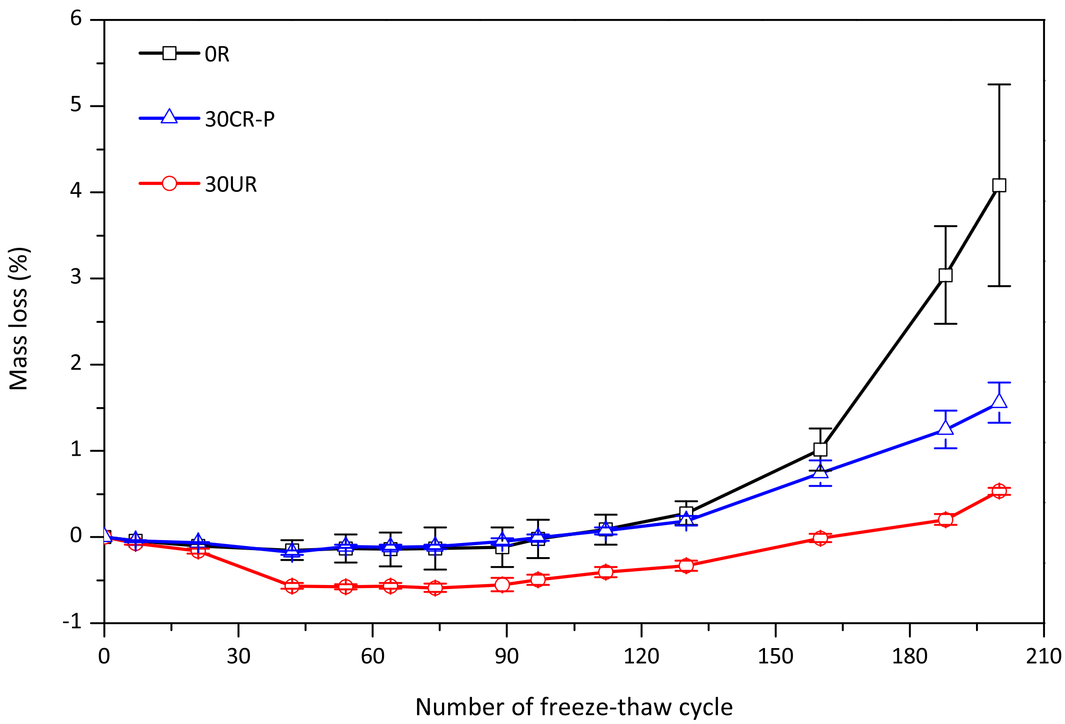

Figure 6: Comparison in mass loss between control and rubberized mortars

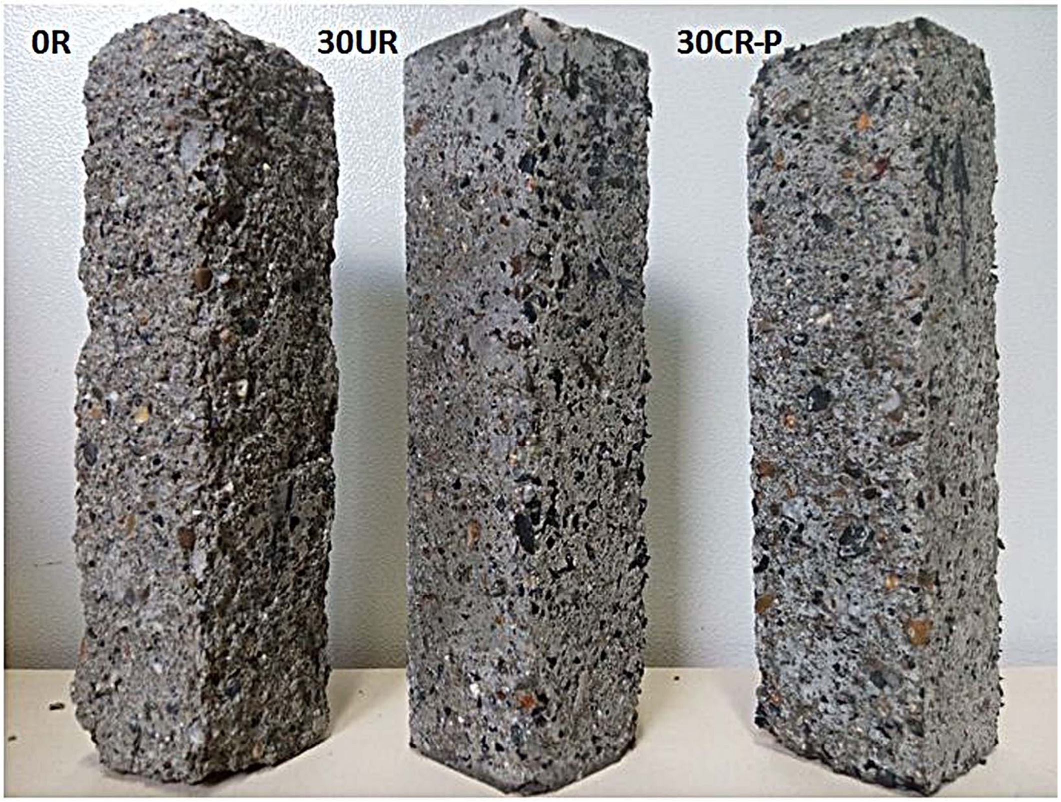

Figure 7: Degradation of mortar specimens at test end

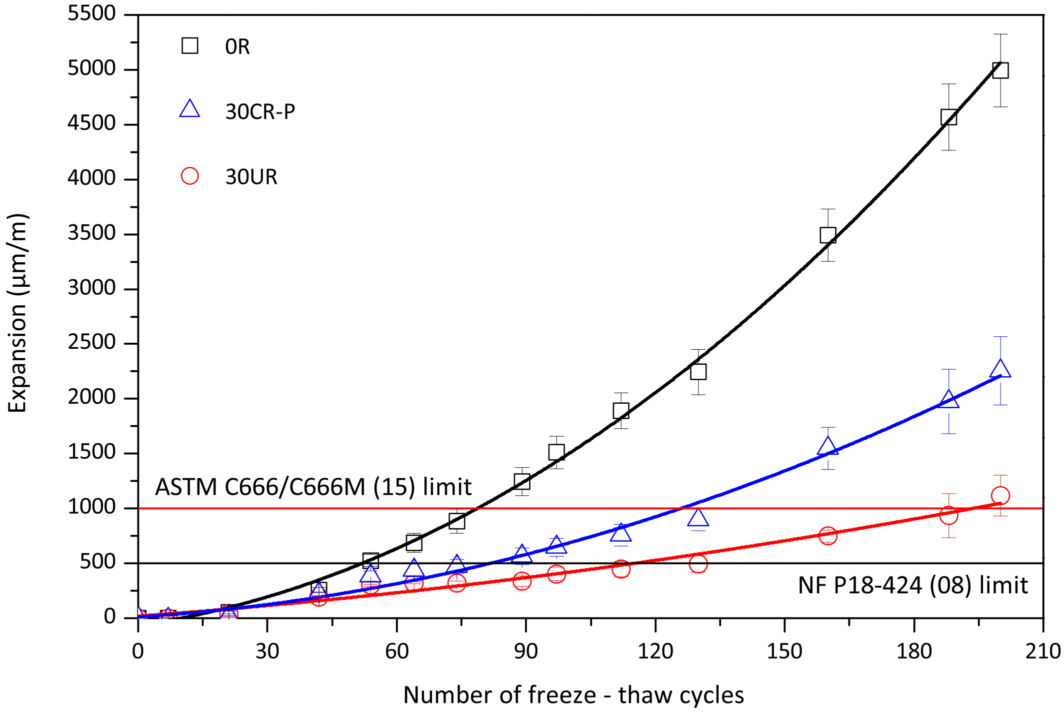

Figure 8: Length changes versus number of freeze-thaw cycles

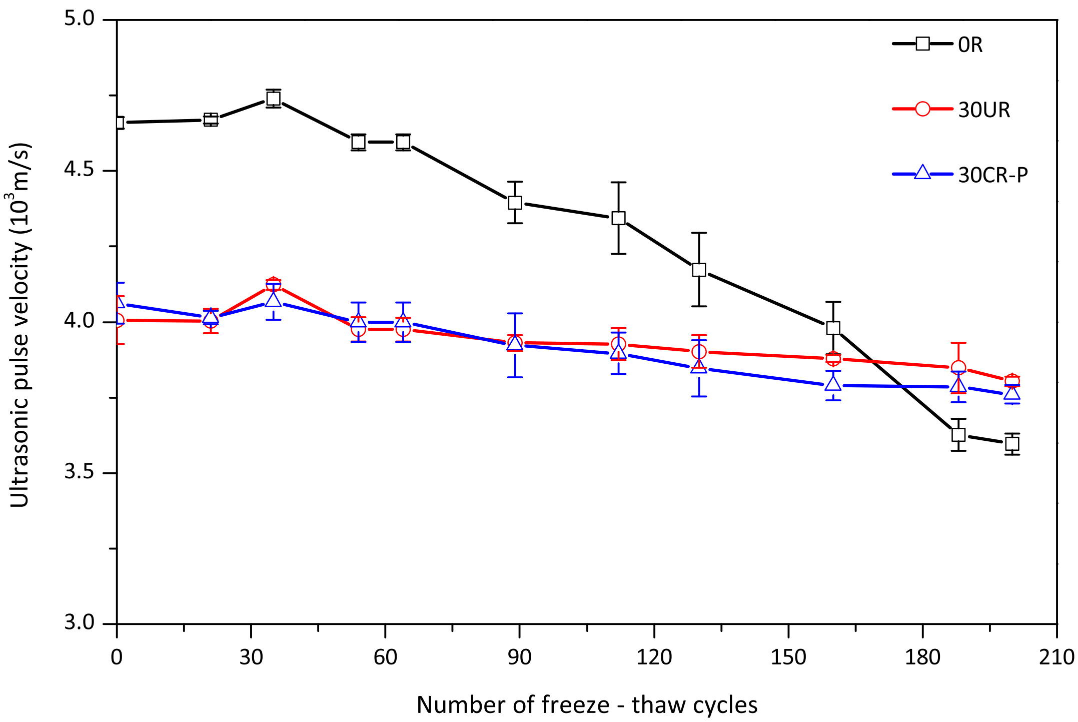

Figure 9: Changes of ultrasonic pulse velocity versus freeze-thaw cycles

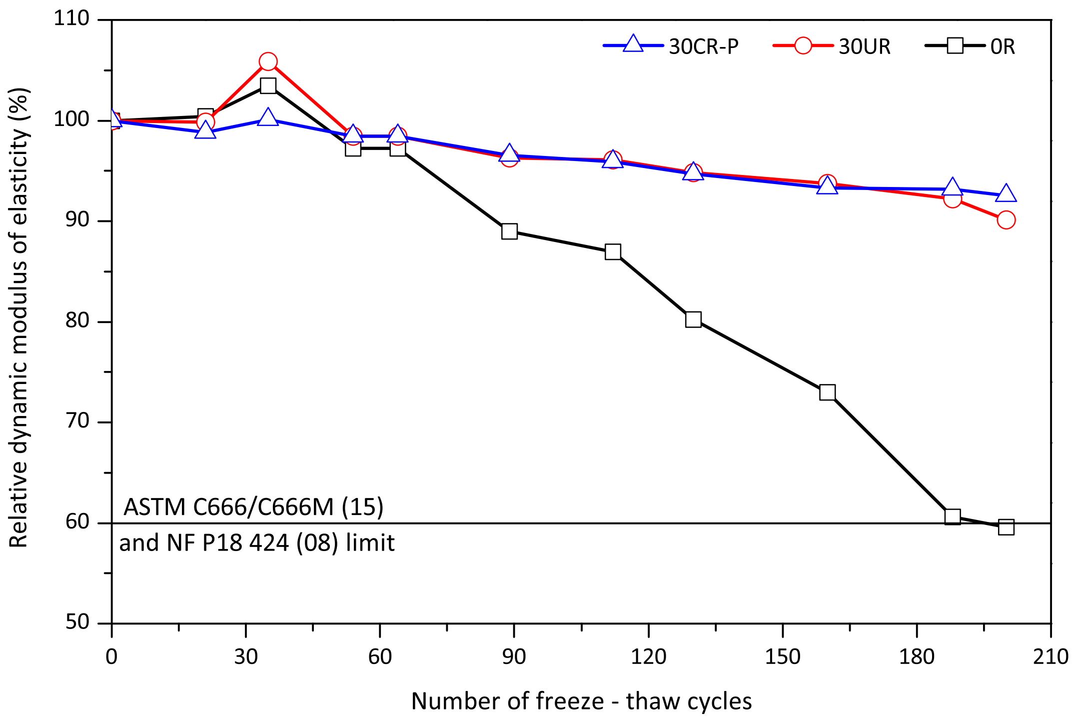

Figure 10: Relative dynamic modulus of elasticity versus freeze-thaw cycles

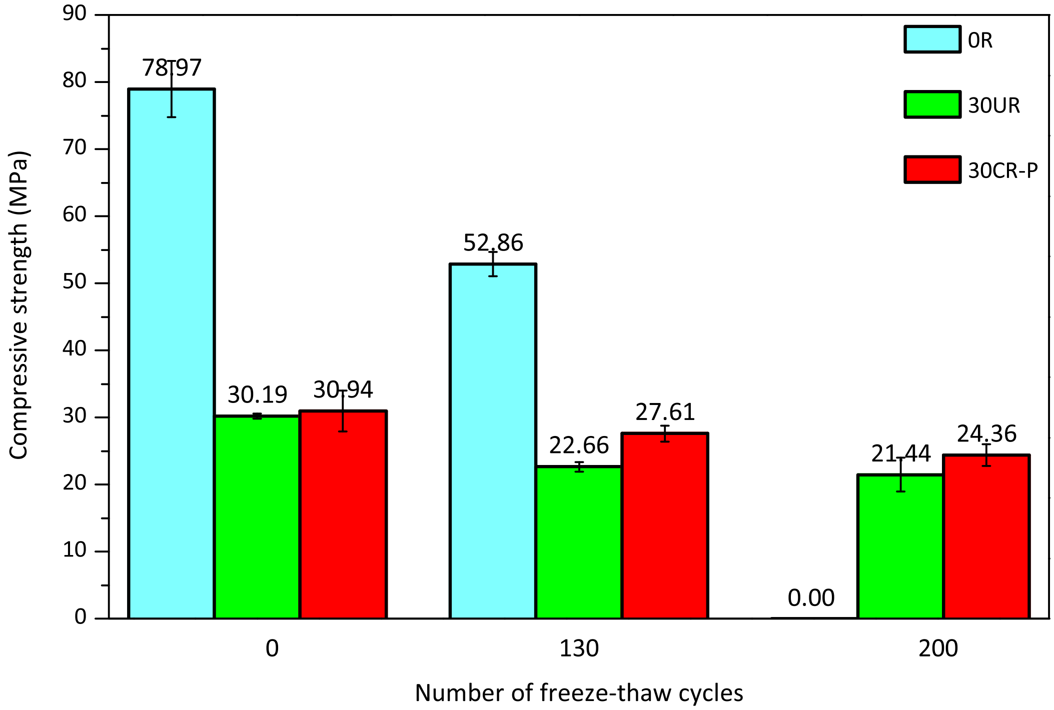

Figure 11: Compressive strength versus freeze-thaw cycles

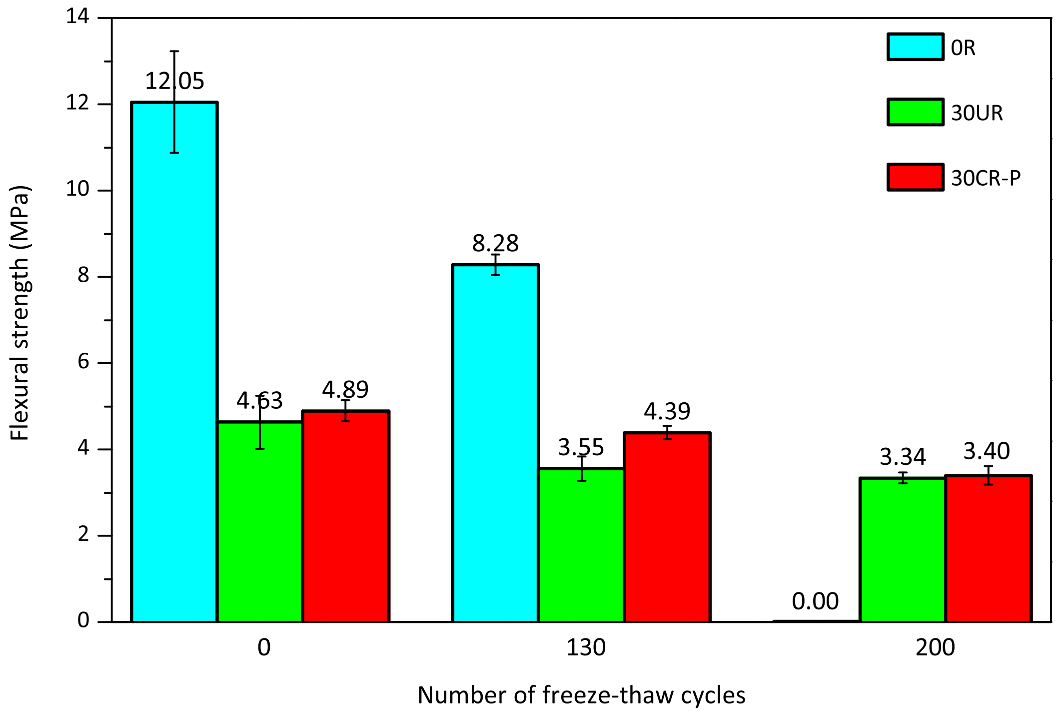

Figure 12: Flexural strength versus freeze-thaw cycles

Table 1: Mix design and proportions (values in kg/m^3)

Mix name	Cement	Sand	Water	RA	Superplasticiser	Viscosity agent
0R	500	1600	235	-	3.25	0.9
30UR	500	1120	235	220	3.25	0.9
30CR-P	500	1120	235	220	3.25	0.9

Table 2: Frost resistance of mortar specimens

Mix name	0R	30UR	30CR-P
Number of freeze-thaw cycles at limited length change (cycles)			
- Overpassing 500 $\mu m/m$ [15]	51.95	115.12	81.69
- Exceeding 0.1% of original length [9]	78.63	193.63	125.84
Relative dynamic modulus of elasticity at test end (%)	59.6	90.1	92.6
Durability factor (%)	59.6	90.1	92.6
Freeze-thaw durability [28]	Doubtful	Frost resistant	Frost resistant