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Experimental investigation of self-healing concrete after crack using nano-capsules including polymeric shell and nanoparticles core

Mojtaba Naseri Taheri [1], Seyyed Ali Sabet [*1] and Reza Kolahchi [2]

[1] *Department of Civil Engineering, Kashan Branch, Islamic Azad University, Kashan, Iran*
[2] *Institute of Research and Development, Duy Tan University, Da Nang 550000, Vietnam*

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Abstract. In this paper, we focused on the self-healing concrete using new nano-capsules. Three types of nano-capsules with respect to availability, high strength and temperature tolerance are used; type 1 is URF and polyethylene (PE) as shell and nano titanium oxide (TiO_2) as core, type 2 is URF and PE as shell and nano silica oxide (SiO_2) as core, type 3 is PE as shell and nano silica oxide (SiO_2) as core. The concrete samples mixed by nano-capsules with three percents of 0.5, 1 and 1.5. Based on experimental tests and the compressive strength of samples, the URF-PE-SiO_2 is selected for additional tests of compressive strength before and after recovery, ultrasonic test, ion chlorine and water penetration depths. After careful investigation, it is concluded that the optimum value of URF-PE-SiO_2 nano-capsules is 0.5% since leads to higher compressive strength, ultrasonic test, ion chlorine and water penetration depths.

Keywords: self-healing concrete; nano-capsules; compressive strength; ultrasonic test; ion chlorine and water penetration depths

1. Introduction

Due to the different static and dynamic loads in concrete structures, the crack can be created and extended. On the other hand, the tension strength of concrete with respect to compressive strength is low which leads to adding steel rebar as reinforcement. This makes crack in the concrete and increases the water penetration depths and consequently corrosion. Recently, with the help of nanoparticles, the concretes with higher stiffness have been built. In order to produce the self-healing concrete, the nano-capsules can be used with a polymer shell and nanoparticle core.

For the self-healing concrete, Joseph *et al.* (2010) studied the repair or healing of cracks and filling of voids in cementitious matrices by the internal release of repair chemicals from inside fibers into the hardened matrix. Van Tittelboom and De Belie (2013) presented the types of healing agents and capsules used. In addition, the various methodologies were evaluated based on the trigger mechanism used and attention was paid to the properties regained due to self-healing. Joseph *et al.* (2010) presented the results of a series of self-healing experiments conducted on reinforced mortar beams containing adhesive-filled glass reservoirs. As synthetic polymers, currently used for concrete repair, may be harmful to the environment, the use of a biological repair technique was investigated by Van Tittelboom *et al.* (2010). Qian *et al.* (2010) investigated the self-healing behavior of Engineered Cementitious Composites (ECC) with focus on the influence of curing condition and precracking time. Nano-materials for corrosion control in reinforced concrete was studied by Koleva *et al.* (2011). Kanellopoulos *et al.* (2015) presented the encapsulation of mineral compounds as healing materials for cement-based composites. Three liquid (sodium silicate, colloidal silica and tetraethyl orthosilicate) and one powdered (magnesium oxide) minerals were encapsulated in thin walled soda glass capsules. Sealing of cracks in cement using micron capsulated sodium silicate was presented by Giannaros *et al.* (2016). Numerical analysis of large amplitude free vibration behaviour of laminated composite spherical shell panel embedded with the piezoelectric layer was presented by Singh and Panda (2015a). The nonlinear free vibration behaviour of laminated composite single/doubly curved shell panel embedded with the piezoelectric layer was investigated numerically by Singh and Panda (2015a). Singh and Panda (2016) investigated the geometrical nonlinear free vibration characteristic of cylindrical composite shell panel embedded with piezoelectric layers. The geometrically nonlinear transient response of the smart laminated composite plate was investigated by Singh *et al.* (2016a) under the coupled electromechanical load. Singh *et al.* (2016b) studied geometrical nonlinear flexural behaviour of laminated composite shell panels integrated with the piezoelectric fibre reinforced composite (PFRC) layer. New self-healing material for concrete repair was fabricated by Tan *et al.* (2017) through microencapsulation of silica sol via interfacial polymerization of poly (urea-urethane). Mohajeriand and Goher (2017) investigated the nanocapsule with polymer core and silica coating as cement additive for concrete with self healing property. Self-healing

*Corresponding author, Ph.D., Professor,
E-mail: sabet@iaukashan.ac.ir

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microcapsules were synthesized by Li *et al*. (2016) in situ polymerization with a melamine urea-formaldehyde resin shell and an epoxy resin adhesive. Ahn *et al*. (2017) examined the applicability and limitation of various ultrasonic test methods in assessing the self-healing performance. Dong *et al*. (2017) presented the development of self-healing materials that hold promise for permeability healing of concrete or other cementitious composites. Microcapsules, with sodium silicate solution as core, were produced by Kanellopoulos *et al*. (2017) using complex coacervation in double, oil-in-water-in oil, emulsion system. UHPC were developed by Calvo *et al*. (2017) incorporating an innovative self-healing system based on two micro/nano-additions: silica microcapsules containing epoxy sealing compound (CAP) and amine functionalized silica nanoparticles. Vijay *et al*. (2017) reviewed the types of bacteria used in concrete and the ways it can be applied as healing agents. The effects on transmission of chloride were test by Ling and Qian (2017) through multiple methods. The feasibility of expanded perlite (EP) as a novel bacteria carrier on quantifying cracks-healing in concrete via immobilization of Bacillus cohnii was demonstrated by Zhang *et al*. (2017). Palin *et al*. (2017) presented a bacteria-based self-healing cementitious composite for application in low-temperature marine environments. Self healing concrete by bacterial and chemical admixtures was investigated by Shaikh and John (2017). The flexural behaviour of the laminated composite plate embedded with two different smart materials (piezoelectric and magnetostrictive) and subsequent deflection suppression were investigated by Dutta *et al*. (2017). Static bending and strength behaviour of the laminated composite plate embedded with magnetostrictive (MS) material was computed numerically by Suman *et al*. (2017). Evaluation of self-healing concrete attributes as sustainable and smart construction material was studied by Huseien *et al*. (2019). The eigenfrequency responses of a nanoplate structure were evaluated numerically by Mehar *et al*. (2018) via a novel higher-order mathematical model and finite-element method including nonlocal elasticity theory. Thermal buckling temperature values of the graded carbon nanotube reinforced composite shell structure was explored by Mehar and Panda (2019).

To the best of author's knowledge, no report has been found in the literature for study of self-healing concrete with polymeric nano-capsules. Three types of nano-capsules including URF-PE-SiO_2, URF-PE-TiO_2 and PE-SiO_2 are tested in the concrete samples. The objective of this work is investigation of compressive strength before and after recovery, ultrasonic test, ion chlorine and water penetration depths.

2. Construction of samples

In order to build the samples, a mix design as shown in Table 1 is used. Since the nano-capsules are not solved in water without any specific process, before producing concrete samples, nano-capsules with 0.5%, 1% and 1.5% are dispersed by using shaker, magnetic stirrer, ultrasonic devices and finally mechanical mixer (This procedure is shown in Fig. 1) (Ying *et al*. 2017, Jarali *et al*. 2017).

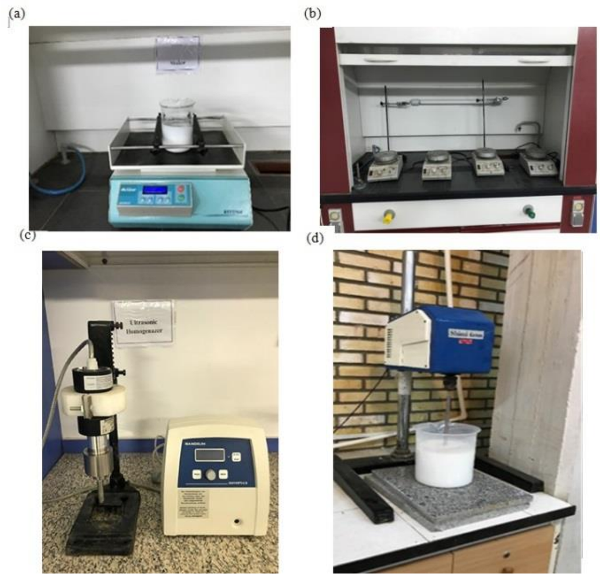

Fig. 1 The procedure of dispersing nano-capsules in the water (a) shaker; (b) magnetic stirrer; (c) ultrasonic devices (d) mechanical mixer

Table 1 The proposed mix design for construction of concrete samples

Weight of nano capsules (gr)	Sand	Fine gravel	Coarse gravel	Cement	water	Slump	W/C	Nano-capsule percentage	Sample types
0.00	696	471	650	376	163	7-9	0.43	0.00	without nano-capsules
1.88				374.12				0.5	PE&TiO2&URF
3.76				372.24				1	PE&TiO2&URF
5.64				370.36				1.5	PE&TiO2&URF
1.88				374.12				0.5	PE&SiO2&URF
3.76				372.24				1	PE&SiO2&URF
5.64				370.36				1.5	PE&SiO2&URF
1.88				374.12				0.5	PE&SiO2
3.76				372.24				1	PE&SiO2
5.64				370.36				1.5	PE&SiO2

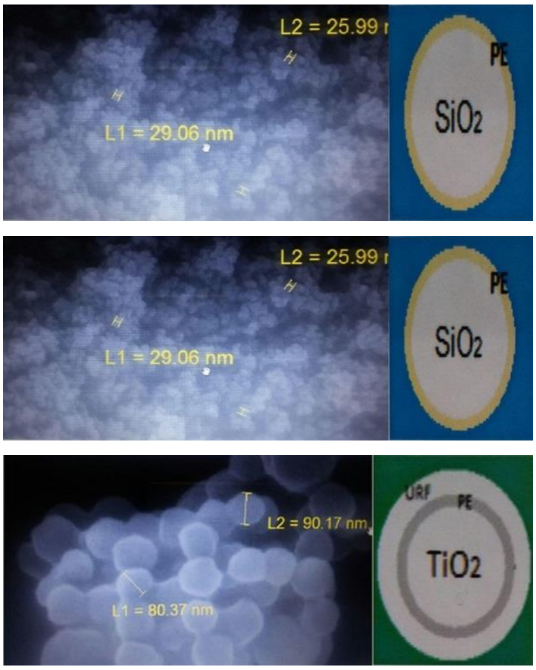

Fig. 2 The SEM image of (a) PE-SiO_2; (b) PE-URF-SiO_2; (c) PE-URF-TiO_2

Fig. 3 Compressive strength test for (1) 0.5% URF-PE-TiO_2; (2) 1% URF-PE-TiO_2; (3) 1.5% URF-PE-TiO_2; (4) 0.5% URF-PE-SiO_2; (5) 1% URF-PE-SiO_2; (6) 1.5% URF-PE-SiO_2; (7) 0.5% PE-SiO_2; (8) 1% PE-SiO_2; (9) 1.5% PE-SiO_2

The 15×15×15 cm cubic concrete samples for determining the compressive strength, ultrasonic and water penetration depth and 10×5 cm (diameter and height) cylinder concrete samples for calculation the ion chlorine penetration depth are built.

Three types of nano-capsules with respect to availability, high strength and temperature tolerance are used; type 1 is URF and polyethylene (PE) as shell and nano titanium oxide (TiO_2) as core, type 2 is URF and PE as shell and nano silica oxide (SiO_2) as core, type 3 is PE as shell and nanosilica oxide (SiO_2) as core. The scanning electron microscope (SEM) images of these materials are shown in Fig. 2 and their technical material properties are presented in Table 2.

3. Experiment tests

This section is divided to 2 sections. In the first section the compressive strength for the cubic concrete samples with three types of nano-capsules (URF-PE-SiO_2, PE-SiO_2, URF-PE-TiO_2) is measured. After selecting the best nano-capsules, in the second section, the ultrasonic test, ion chlorine and water penetration depths are tested. For measuring the compressive strength of the concrete samples after recovery, the samples are subjected to 80% of the final defect load (Dry 1994).

3.1 Compressive strength

As shown in Fig. 3, the cubic concrete samples with 0.5,1 and 1.5 percent of URF-PE-SiO_2, PE-SiO_2 and URF-

Table 2 The technical material properties of nano-capsules

Nano capsule type	Color	Property	Density	Temperature tolerance (°C)	Acidity
PE&TiO_2 & URF	Milky white	Hydrophobic, resistant to moisture	0.5	180	13-4
PE&SiO_2 & URF	Milky white	Hydrophobic, resistant to moisture	0.4	180	13-4
PE & SiO_2	White	Hydrophobic, resistant to moisture	0.5	100	13-4

Table 3 The compressive strength of samples with URF-PE-TiO_2 nano-capsules

Strength increase percentage	The length of recovery period	Resistance after restoration	Resistance before restoration	Sample age (day)	Nano capsule type
41	14	405	288	7	PE & TiO_2 & URF (0.5%)
18	14	386	328	28	PE & TiO_2 & URF (0.5%)
31	14	347	264	7	PE & TiO_2 & URF (1%)
15	14	360	313	28	PE & TiO_2 & URF (1%)
30	14	331	255	7	PE & TiO_2 & URF (1.5%)
--------	14	266	304	28	PE & TiO_2 & URF (1.5%)

Table 4 The compressive strength of samples with URF-PE-SiO_2 nano-capsules

Strength increase percentage	The length of recovery period	Resistance after restoration	Resistance before restoration	Sample age (day)	Nano capsule type
31	14	337	258	7	PE & TiO_2 & URF (0.5%)
25	14	371	323	28	PE & TiO_2 & URF (0.5%)
35	14	324	240	7	PE & TiO_2 & URF (1%)
30	14	380	292	28	PE & TiO_2 & URF (1%)
38	14	313	227	7	PE & TiO_2 & URF (1.5%)
31	14	351	268	28	PE & TiO_2 & URF (1.5%)

Table 5 The compressive strength of samples with PE-SiO_2 nano-capsules

Strength increase percentage	The length of recovery period	Resistance after restoration	Resistance before restoration	Sample age (day)	Nano capsule type
14	14	266	233	7	PE & SiO_2 (0.5%)
22	14	341	280	28	PE & SiO_2 (0.5%)
26	14	244	193	7	PE & SiO_2 (1%)
14	14	268	235	28	PE & SiO_2 (1%)
29	14	224	173	7	PE & SiO_2 (1.5%)
15	14	272	236	28	PE & SiO_2 (1.5%)

PE-TiO_2 nano-capsules are tested by ELE ADR 2000 machine. The obtained results for the compressive strength before and after recovery are presented in Tables 3-5, respectively for URF-PE-TiO_2, URF-PE-SiO_2 and PE-SiO_2.

The compressive strength of samples is measured before and after 14 days restoration. In the concrete samples with the age of 7 days, it is found that the compressive strength increases percentages are 41, 31 and 30 for nano-capsules of URF-PE-TiO_2, 31, 35 and 38 for nano-capsules ofURF-PE-SiO_2 and 14, 26 and 29 for nano-capsules of PE-SiO_2. In addition, for the 28 days, the compressive strength after recovery is increased 18, 15 and 12 for nano-capsules of URF-PE-TiO_2, 15, 30 and 31 nano-capsules of URF-PE-SiO_2 and 22, 14 and 15 for nano-capsules of PE-SiO_2.

However, it can be concluded that the compressive strength increases percentages after restoration is better for the nano-capsules of URF-PE-SiO_2 with the volume percent of 0.5%.

3.2 Compressive strength

For additional experiment, the concrete samples with URF-PE-SiO_2 nano-capsules are selected. However, the cubic samples for ultrasonic test and water penetration depths and cylinder samples for ion chlorine and water penetration depths are built.

3.2.1 Ultrasonic test

In this test, two probes smeared to Glycerin of ultrasonic devise are connected to the concrete sample and the output axial compressive wave is displayed in oscilloscope (Fig. 4).

The output results from this test for the control sample (without nano-capsules) and concrete samples with 0.5, 1 and 1.5 percent of URF-PE-SiO_2 nano-capsules are

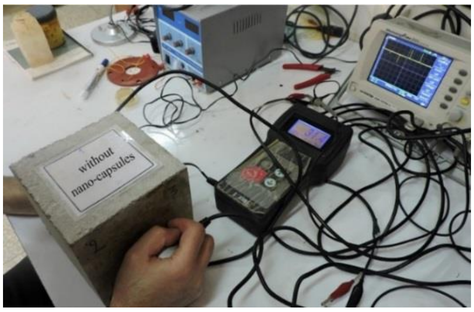

Fig. 4 The ultrasonic test for the cubic concrete samples

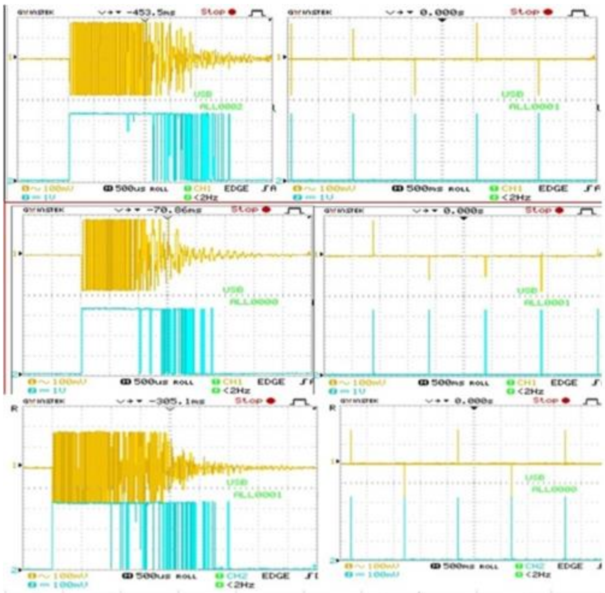

Fig. 5 The axial compressive wave in the control sample (without nano-capsules)

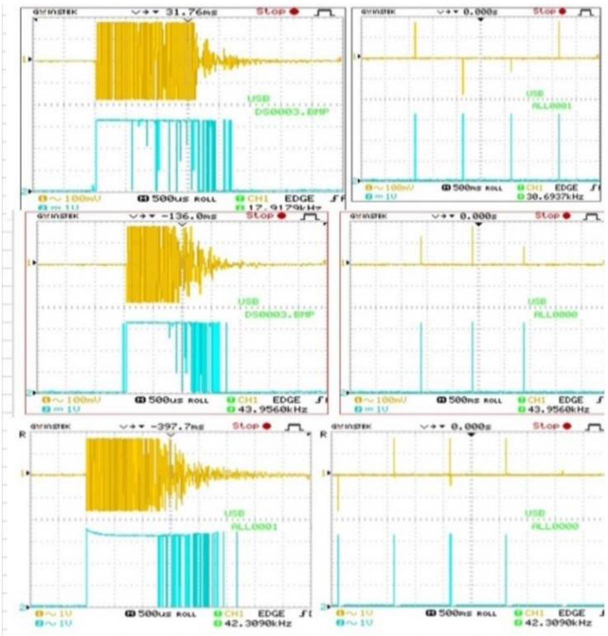

Fig. 6 The axial compressive wave in the concrete sample with 0.5% URF-PE-SiO_2

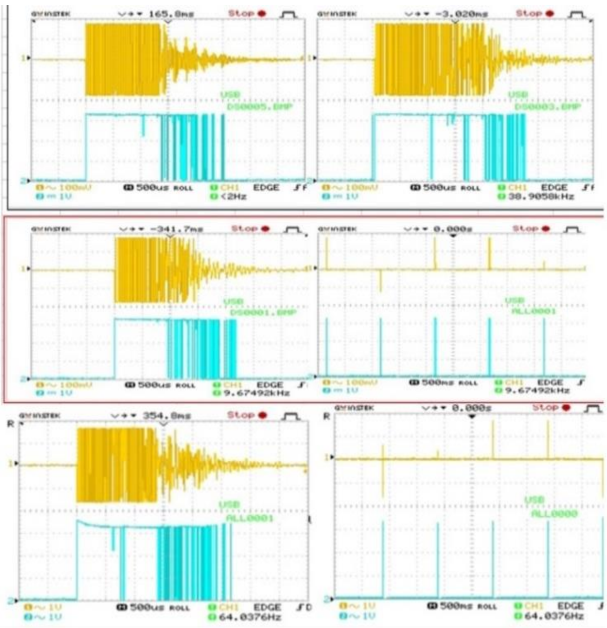

Fig. 7 The axial compressive wave in the concrete sample with 1% URF-PE-SiO_2

illustrated in Figs. 5-8, respectively. Form the figures, it is found that the time of wave transfer in the concrete sample is lower for the sample with 0.5% URF-PE-SiO_2 compared to other samples. Hence, it can be resulted that the self-healing of the concrete with 0.5% URF-PE-SiO_2 nano-capsules is a good choice with respect to other cases.

3.2.2 Water penetration depth

In this test, the concrete samples are subjected to water pressure of KPa and wind pressure of 5 bar for 72 hours

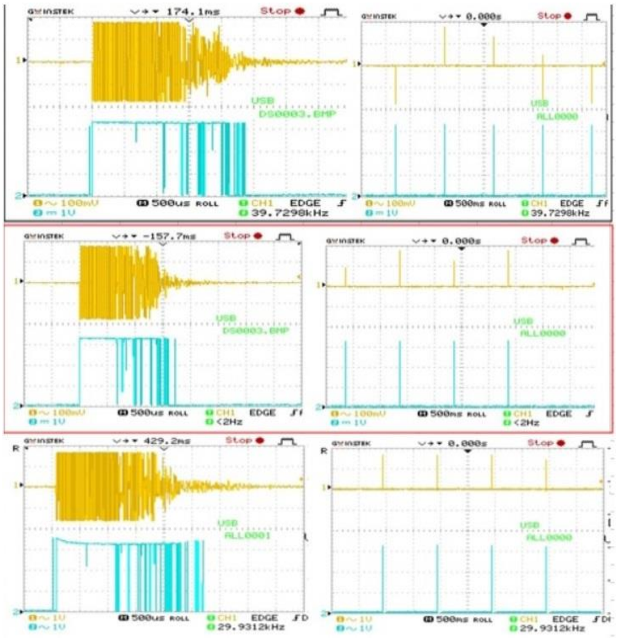

Fig. 8 The axial compressive wave in the concrete sample with 1.5% URF-PE-SiO_2

Fig. 9 The water penetration depth device

(Fig. 9). After the test, the concrete samples are broken by automatic jack and the water penetration depth is measured.

The images of sample after the test are demonstrated in Fig. 10. As can be seen, the water penetration depth for the concrete samples with 0, 0.5, 1 and 1.5 percent URF-PE-SiO_2 are respectively 60, 33, 112.5 and 71 mm. With respect to standard of 50 mm, it is found the concrete sample with 0.5% URF-PE-SiO_2 is the better choice.

3.2.3 Ion chlorine penetration depth

For this test, the cylinder concrete samples with diameter of 100 mm and height of 50 mm are built. The samples are based on the standard of AASHTO TP64 after saturation with water is located in isolate rubber sheath with 3% soda density. This complex is putted in NaCl with 10% density so that the below surface of concrete sample be in contact with NaCl (Fig. 11).

After the test, the results of Ion chlorine penetration depth for the 0, 0.5, 1 and 1.5 percent URF-PE-SiO_2 nano-capsules in the concrete samples are shown in Fig. 12.

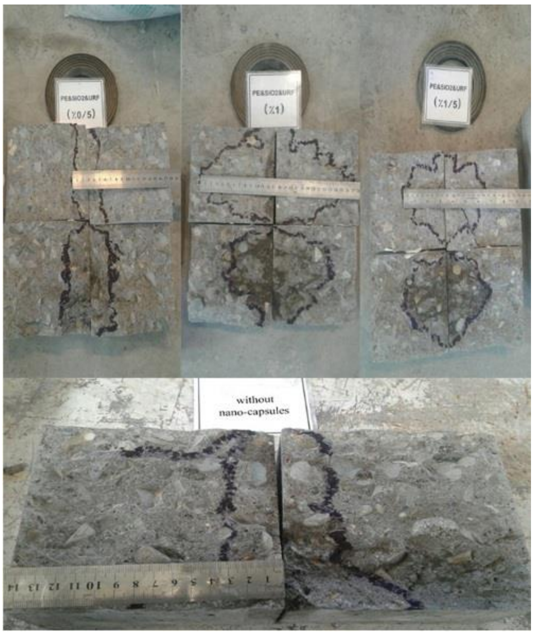

Fig. 10 The water penetration depth for the concrete samples

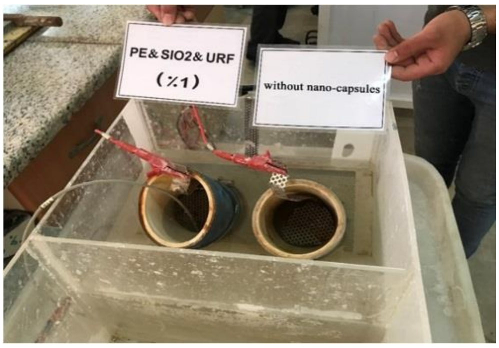

Fig. 11 The schematic of Ion chlorine penetration depth device

Fig. 12 The output of Ion chlorine penetration depth for (1) 0;(2) 0.5; (3) 1; (4) 1.5 percent URF-PE-SiO_2 nano-capsules in the concrete samples

can be observed that the Ion chlorine penetration depth for the samples with 0, 0.5, 1 and 1.5 percent URF-PE-SiO_2 nano-capsules are respectively, 40, 34.5, 46.7 and 54 mm. These results show that the Ion chlorine penetration depth in the concrete sample with 0.5 percentURF-PE-SiO_2 nano-capsules leads to better results.

4. Conclusions

Investigation of self-healing concrete based on nanotechnology was the main contributing of this work. Three special nano-capsules namely as URF-PE-SiO_2, URF-PE-TiO_2 and PE-SiO_2 were tested. In the first phase of this project, the nano-capsule of URF-PE-SiO_2 was selected due to high compressive strength after the recovery with respect to two other materials. In the second phase, the additional experiments of ultrasonic test, ion chlorine and water penetration depths were done for the concrete samples with URF-PE-SiO_2 nano-capsules. It can be concluded that the compressive strength increases percentages after restoration was better for the nano-capsules ofURF-PE-SiO_2 with the volume percent of 0.5%. It was found that the time of wave transfer in the concrete sample is lower for the sample with 0.5% URF-PE-SiO_2 compared to other samples. The water penetration depth for the concrete samples with 0, 0.5, 1 and 1.5 percent URF-PE-SiO_2 were respectively 60, 33, 112.5 and 71 mm. With respect to standard of 50 mm, it was found the concrete sample with 0.5% URF-PE-SiO_2 was the better choice. In addition, the Ion chlorine penetration depth for the samples with 0, 0.5, 1 and 1.5 percent URF-PE-SiO_2 nano-capsules were respectively, 40, 34.5, 46.7 and 54 mm which show the better results for 0.5 percent URF-PE-SiO_2 nano-capsules.

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