PERFORMANCE OF EPOXY RESIN AS SELF-HEALING AGENT

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ABSTRACT

Formation of cracks due to the shrinkage effects during curing and mechanical loading can deteriorate the concrete performance especially in terms of durability aspect. Chemical and harsh solutions will easily penetrate into the concrete and cause damage to the concrete. In order to solve this problem, researchers have introduced a self-healing concrete; the mechanism of automatically repairing concrete cracks without external intervention. Nowadays, the self-healing concrete by using bacteria as a healing agent had gained interest among researchers. In contrast, this paper presents the study on performance of epoxy resin without hardener as a self-healing agent in concrete. Mortar specimens were prepared with mass ratio of 1:3 (cement: fine aggregates), water-cement ratio of 0.48 and 5 to 20% epoxy resin of cement content. All tested specimens were subjected to wet-dry curing; where compressive strength, apparent porosity and self-healing evaluation were measured. Result shows that, the compressive strength of mortar with addition of epoxy resin by 10% increased significantly compared to normal mortar. Epoxy resin as a healing agent was found to be functioned well as the compressive strength and ultrasonic pulse velocity regain the initial reading with prolonged curing time. These results together with microstructure test indicate that epoxy resin can be used as a self-healing agent.

Keywords: epoxy resin; compressive strength; self-healing; healing agent

1. INTRODUCTION

Concrete structures often suffer from cracking much earlier than designed service life. To prevent such deterioration, regular inspection of cracks in concrete structures and their repair are usually carried out by means of some kind of human intervention [1]. Cracks in concrete can occur at any stage of the service life due to volume instabilities such as shrinkage or drying shrinkage. The repairing works of concrete cracks take great cost and materials [2].

Related to this matter, an automatic cracks repairing concrete best known as self-healing concrete was introduced. Previous researcher had demonstrated that the microcracks in cementitious materials are able to heal under certain circumstances [3,4]. When cracks occurred, the water and oxygen gases penetrated into the concrete and reacted with bacteria to produce calcium carbonate that can heal the cracks [5]. Limited research regarding self-healing concrete by using epoxy resin as a healing agent has been carried out. In this paper, the performance of epoxy resin without hardener as a healing agent was evaluated and reported.

2. EXPERIMENTAL PROGRAM

2.1 Materials

Cement. The cement used in the study was ordinary Portland cement (OPC) conforming to ASTM C150 / C150M-12 [6] standard.

Fine Aggregates. Local river sand in which specific gravity of 2.62 and fineness modulus of 2.85 in saturated surface dry conditions was used.

Epoxy Resin. Diglycidyl Ether of Bisphenol A-type epoxy resin without hardener was used in the mix proportion and stored in room temperature to avoid damage. The amount of epoxy resin added in the mix was in the range of 10 % of the cement content. The viscosity of epoxy resin chosen was high as to create a bonding between epoxy resin and hydroxyl ion. Epoxy resin acted as a self-healing agent which when cracks occurs inside the specimens, epoxy resin reacts to heal the cracks automatically.

2.2 Preparation of Epoxy-modified Mortar

With reference to JIS A 1171-2000 [7], the epoxy-modified mortars without hardener were mixed with a mass ratio of cement to fine aggregates of 1:3; epoxy ratio of 5, 10, 15, and 20 % of cement; and a water-cement ratio of 0.48. The flow spread diameter was in the range of 170 ± 5 mm. Mortar cube specimens of 70 x 70 x 70 mm were cast for compressive strength test and 100 x 100 x 100 mm mortars for self-healing evaluation test. The mixing procedure was basically same as the ordinary cement mortar but with the addition of epoxy resin in the fresh mix. Table 1 shows the mix proportion of the epoxy-modified mortar. In order to have an optimum percentage of epoxy resin, the various epoxy resin was used in the initial experimental work. Normal mortar mix was prepared as control specimens.

Table 1 Mix proportion of epoxy-modified mortar

Sand (kg/m^3)	Cement (kg/m^3)	Water (kg/m^3)	Epoxy ratio (%)	Water /Cement ratio	Sand: Cement
1517	506	228	0 5 10 15 20	0.48	3:1

2.3 Curing Regime

For initial curing, wet-dry curing was applied to the specimens where the specimens were placed under wet burlap for two days followed by five days in water. After that, the specimens were taken out and placed at room temperature for 21 days [8]. After the specimens matured and crack was initiated, prolong dry-curing was applied until the day of testing. The normal mortar went through water curing.

2.4 Tests

Several tests were conducted to investigate the performance of epoxy resin without hardener in the mortar specimens. From the various percentage of epoxy resin added, the percentage that gives the highest compressive strength was chosen as the optimum mix proportion and was used in self-healing evaluation. The self-healing evaluation was tested up to one year of curing period.

2.4.1 Apparent Porosity

Determination of apparent porosity of mortars was done in accordance to ASTM C1403-13 [9]. Three cubes of mortars were oven-dried at 85°C for 24 hours and then immersed in water for 48 hours. The cubes were further suspended in water and weighted. The data were recorded and calculated for average. The percentage of apparent porosity was determined at the age of 28, 56, 90, 120, 180, 270, and 360 days.

2.4.2 Compressive Strength

The compressive strength test for epoxy-modified mortar was conducted using a compression test machine with a maximum load capacity of 2000 kN and the loading rate was 0.3 $N/mm^2/s$ after 28 days of curing. The test was conducted in accordance to BS EN 12390-3 [10]. An increasing compressive load was applied to the specimen until failure occurred to obtain the maximum compressive load.

The cube size used was 70 x 70 x 70 mm and the calculated compressive strength was based on the average of three values. For strength development test, the compressive strength test was conducted at the age of 28, 56, 90, 120, 180, 270, 360, 425 and 485 days.

2.4.3 Self-Healing Evaluation

The 100 x 100 x 100 mm cube specimens were used for self-healing evaluation tests. Several tests were conducted to check the performance of epoxy resin without hardener as a self-healing agent.

Pre-loading and dry curing. After the initial curing, the micro cracks were generated in the specimen using compressive machine at loading rate ratio of 0, 50 and 80% of maximum load of each specimen. Then the specimens were dry-cured at room temperature until the desired day of testing. The propagating crack was checked by using non-destructive test equipment, ultrasonic pulse velocity (UPV) machine in accordance to ASTM C597 [11].

Microstructure. Scanning Electron Microscopy (SEM) was performed by coating the samples with gold prior to analysis, and Energy-dispersive X-ray (EDX) rendered at an accelerating voltage of 15 KV. The specimens analysed by Field Emission Scanning Electron Microscope (FESEM) analysis were taken from the fractured pieces of the samples.

3. RESULTS AND DISCUSSIONS

3.1 Optimum Mix Proportion

The optimum mix proportion was selected based on compressive strength of the specimens. Figure 1 exhibits the different ratios of epoxy resin in mortar under wet-dry curing, which has been added to the mortar to study its effect on compressive strength. The compressive strength of normal mortar was 30 MPa at 28 days while the compressive strength of the 10 % epoxy resin was 36 MPa, which was the highest strength among all epoxy ratios and higher than the strength of normal mortar. This was achieved due to the presence of alkalis from the hydration process to react with epoxy resin [12].

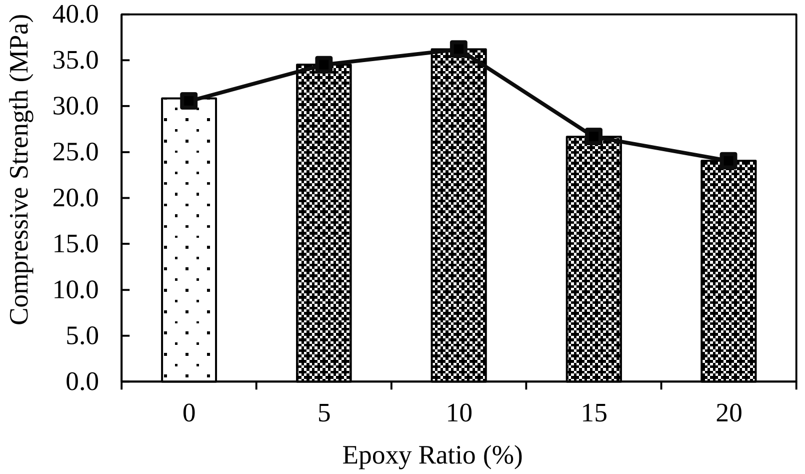


Figure 1: Relationship between compressive strength and epoxy ratio at 28 days in wet-dry curing condition.

The increase in epoxy ratio beyond 10 % has decreased the compressive strength probably due to the presence of epoxy resin that was not hardened and disturbs the bonding between hydroxyl ion and epoxy resin, which had been previously reported by Ohama *et al.* [12]. According to Ohama and Takahashi; Ohama *et al.*; Ohama [14-16], the reductions in the compressive strengths of the polymer-modified mortars using epoxy resin without the hardener at polymer-cement ratios of 10 % or more may be explained by the presence of considerable amount of epoxy resin which cannot harden in the polymer-modified mortars. The unhardened epoxy resin become excessive and lowers the compressive strength of the mortar. Therefore, 10 % epoxy ratio was taken as the optimum content as to be used without hardener which gives higher compressive strength and was applied to others tests.

Figure 2 shows the strength development of epoxy-modified mortar in the period of one and a half years of dry-curing.

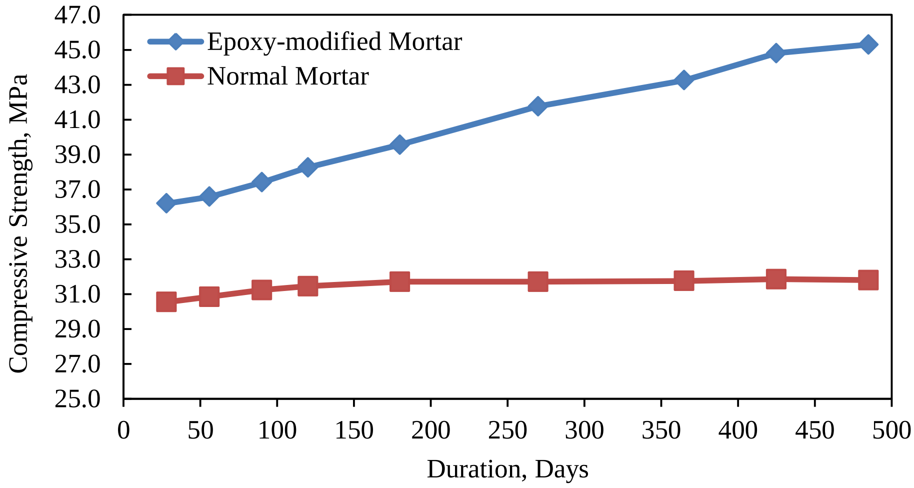


Figure 2: Strength development of epoxy-modified mortar with prolong dry-curing.

From the Figure, the strength development of epoxy-modified mortar keeps increasing after 485 days of curing. The initial compressive strength of epoxy-modified mortar was 36 MPa however, after 485 days; the compressive strength was 45 MPa. This trend shows that the increase of 25 % of compressive strength was recorded. On the other hand, for the normal mortar after 180 days curing, the compressive strength starts to be constant. No further increasing in compressive strength was recorded.

This phenomenon occurs to epoxy-modified mortars due to the further reaction between epoxy resin and hydroxyl ion. The reaction between unhardened epoxy resin and hydroxyl ions has produced a denser and stronger mortar [17].

3.2 Porosity

The porosity of the mortars highly influences the strength of mortar. The reduction of porosity in concrete will increase its strength as it make the concrete and mortar denser [18]. Figure 3 shows the results of strength development and apparent porosity of epoxy-modified mortar subjected to wet-dry curing condition. As shown in Figure 3, the apparent porosity of 6% was recorded at the age of 28 days.

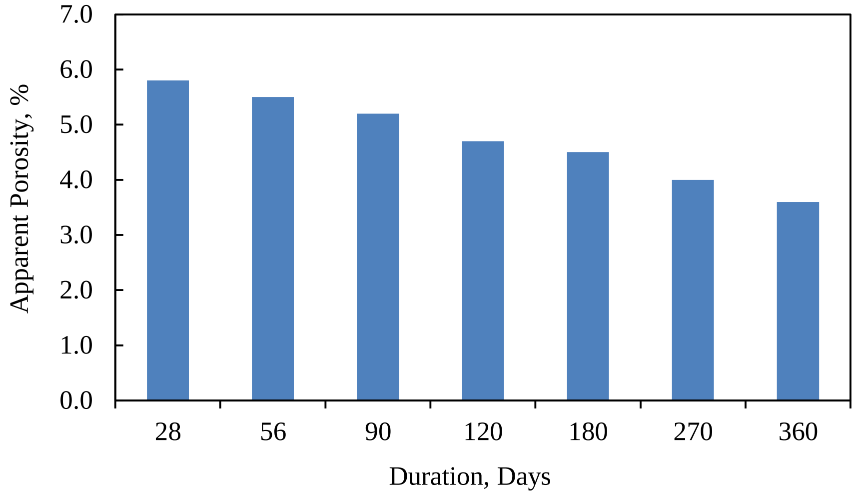


Figure 3: Relationship between porosity of epoxy-modified mortar with curing age.

Prolonged curing period has shown to lower the porosity which at 360 days curing, the porosity was 3.7 %. The decreasing porosity at 365 days was recorded almost 40 % of its initial porosity. The epoxy resin without hardener added into the mortar mixture was active even in dry condition that produced higher compressive strength and closed the pores within. The results of the apparent porosity test were reliable to be correlated with the strength development results.

3.3 Self-healing Evaluation

The self-healing performance of epoxy-modified mortar was investigated by non-destructive test and compressive strength test. The non-destructive test conducted was ultrasonic pulse velocity (UPV) test. Upon the maturity of the mortar specimen, pre-loading stress was applied and the UPV time travel was measured. Figures 4 and 5 show the results of self-healing performance after 12 months curing period. UPV technique was used to evaluate self-healing performance in which the method was also used by Zhong and Yao [19].

Figure 4 shows a time travel of epoxy-modified mortars (EM) and normal mortar (NM) after pre-loading. It can be seen from the Figure that the normal mortar did not show any changes in time travel even after 12 months. However, the epoxy-modified mortar shows a better performance in time travel after 12 months.

The lower time travel indicates that the mortar is dense and less porous. The Figure shows that the performance of epoxy resin as a healing agent is well functioned.

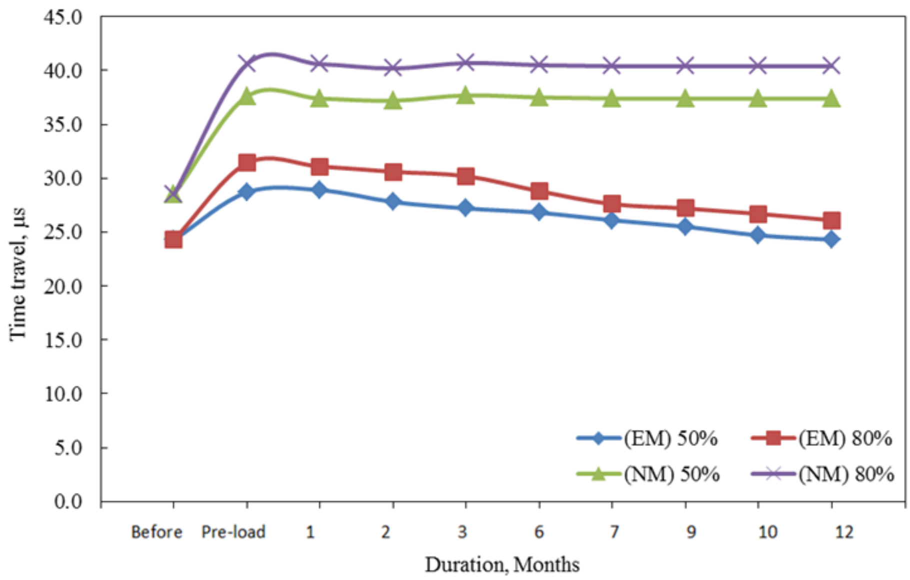


Figure 4: Relationship between time travel and duration of epoxy and normal mortar after pre-loading.

Figure 5 shows the compressive strength trend after pre-loading. From the Figure, after 12 months of curing, the compressive strength of epoxy-modified mortar starts to regain its initial reading. The initial compressive strength of epoxy-modified mortar was 36 MPa but after pre-loading, the compressive strength was 18 MPa for 50 % load while 27 MPa for 80 % load. After 12 months, the compressive strength was 29 MPa and 35 MPa for 50 % load and 80 % load, respectively. The result shows an indication that the epoxy resin has reacted well with hydroxyl ions in repairing and healing the cracks inside the specimens without external intervention.

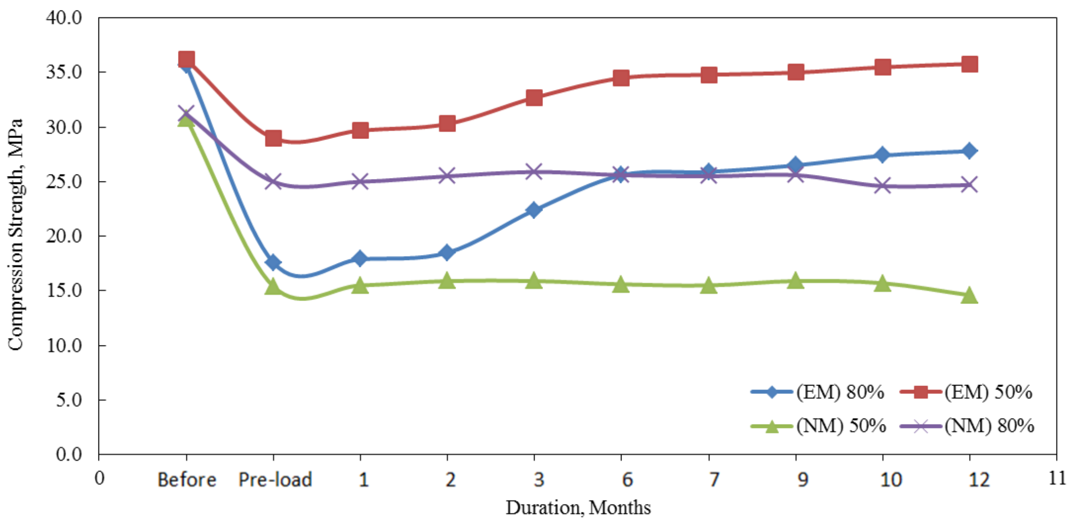
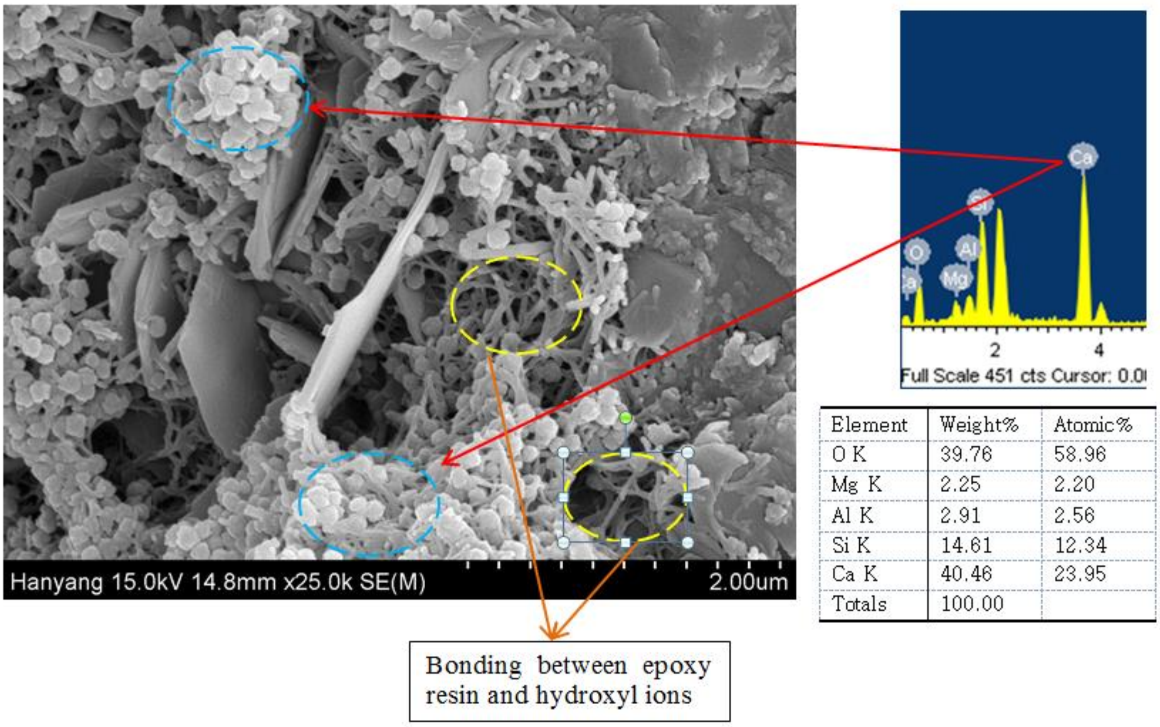


Figure 5: Relationship between compressive strength and duration of epoxy and normal mortar after pre-loading.

In contrast, the compressive strength of normal mortar did not have any changes after pre-loading. The compressive strength was reduced due to the penetration of water and aggressive ions that deteriorate the mortars.

3.4 Microstructure

The microstructure morphology was studied in order to see the components that activate the self-healing phenomenon. From the scanning electron microscopy test shown in Figure 6, it shows the bonding between epoxy resin and hydroxyl ions. The circle looks like ball was calcium based on EDX analysis. This component was the main components that contribute in self-healing function of epoxy resin.



Element	Weight%	Atomic%
O K	39.76	58.96
Mg K	2.25	2.20
Al K	2.91	2.56
Si K	14.61	12.34
Ca K	40.46	23.95
Totals	100.00	

Figure 6: SEM morphology of epoxy-modified mortar.

4. CONCLUSIONS

The conclusions that can be drawn from the study are as follows:

I. The optimum amount of epoxy content that produced the highest compressive strength was 10%.
II. The self-healing function of epoxy resin is functioning well based on the UPV and compressive strength tests which is after prolong the curing, the UPV and compressive strength start to regain its initial reading.
III. The microstructure study also indicated the self-healing process occurred in the samples tested.

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