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SYNTHESIS AND CHARACTERIZATION OF SELF-HEALING MORTAR WITH MODIFIED STRENGTH

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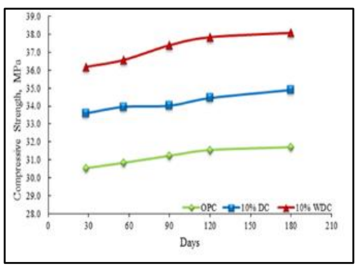
Graphical abstract



Abstract

Cementitious materials being the most prospective building blocks achieving their absolute strength to avoid the deterioration in the early stage of service life is ever-demanding. Minimizing the labor and capital-intensive maintenance and repair cost is a critical challenge. Thus, self-healing mortars with modified strength are proposed. Lately, self-healing of micro-cracks by introducing bacteria during the formation of mortar or concrete became attractive. Self-healing with polymeric admixtures is considered to be relatively more durable and faster process. Certainly, the self-healing of synthetic polymeric materials is inspired by biological systems, where the damage triggers an autonomic healing response. This emerging and fascinating research initiative may significantly improve the durability and the safety limit of the polymeric components potential for assorted applications. In this work, using epoxy resin (diglycidyl ether of bisphenol A) without any hardener as admixture polymeric-cementitious materials is prepared. These epoxy-modified mortars are synthesized with various polymer-cement ratios subjected to initial wet/dry curing (WDC) together with long term dry curing (DC). Their self-healing function and hardening effects are evaluated via preloading and drying of the specimens, chemical analysis, and ultrasonic pulse velocity testing. It is demonstrated that 10% of polymer is the best proportion for polymer-cement ratio. Furthermore, the wet/dry curing is established to be superior process for healing hairline cracks present in the mortar. The excellent features of the results suggest that our novel method may constitute a basis for improving the compressive strength and self-healing features of mortars.

Keywords: Self -healing, the ultrasonic pulse velocity (UPV) and compressive test

Abstrak

Over recent years, Bahan bahan bersimen merupakan bahan binaan yang paling prospektif. Tuntutan bagi penambahbaikan kekuatan bahan bersimen yang juga disertakan dengan tuntutan bagi mendapatkan bahan binaan bersimen yang tidak mudah merosot prestasinya di peringkat awal adalah sangat tinggi dan sentiasa mendapat perhatian. Hal yang tersebut dapat mengurangkan kos buruh serta kos penyelenggaraan yang juga memerlukan modal yang amat besar untuk diselenggarakan. Justeru, rawatan penyembuhan mortar yang berlaku secara sendiri dicadangkan di dalam penyelidikan ini. Akhir-akhir ini, kaedah penyembuhan retakan mikro secara sendiri dengan menggunakan bakteria sewaktu terbentuknya motar semakin mendapat perhatian. Kaedah penyembuhan secara sendiri dengan menggunapakai bahan tambah sintetik polimer adalah dijangka lebih tahan lama dengan tempoh pemprosesan yang lebih cepat. Pastinya, kaedah penyembuhan secara secara diri bahan sintetik polimer ini dicetuskan oleh sistem biologi semulajadi, apabila proses penyembuhan secara otomatik akan berlaku pada tempat yang mengalami kerosakan. Inisiatif daripada penyelidikan baru ini memberikan satu inisiatif dan prospek yang menarik dalam meningkatkan tahap ketahanan dan tahap keselamatan komponen polimetrik yang ketara bagi pelbagai aplikasi. Dalam penyelidikan ini, penggunaan epoksi resin (diglycidyl eter daripada Bisphenol A) tanpa bahan pengeras sebagai bahan tambah kepada bahan polimer bersimen dicadangkan. Motar yang telah dimodifikasikan dengan epoksi ini telah disentisiskan dengan pelbagai nisbah simen polimer yang kemudiannya dibandingkan prestasinya setelah keras pada peringkat awal (WDC) dan juga prestasinya setelah keras untuk jangka masa panjang (DC). Kaedah penyembuhan motar yang berlaku secara sendiri ini serta tahap kekerasan motar tersebut kemudiannya diukur dan dinilai dengan memasukkan dan mengeringkan spesimen, analysis kimia dan ujian denyutan ultrasonik halaju. Hasil kajian mendapati bahawa nisbah 10% polimer simen merupakan jumlah nisbah yang terbaik. Kajian juga mendapati bahawa pengerasan sewaktu basah/kering adalah lebih ketara bagi penyembuhan retakan garis halus. rambut yang terdapat dalam motar. Hasil dapatan memberansangkan dari kajian yang perintis ini justeru dapat dijadikan sebagai asas terhadap penambahbaikan kemampatan dan kekuatan motar serta fungsi penyembuhan motar secara sendiri.

Kata kunci: Penyembuhan dirisendiri, halatujn denyutan ultrasonik and ujian kemampatan

1.0 INTRODUCTION

Undoubtedly, the exposure of concrete to external environmental conditioning including extreme heat, cold and stress during service life is inevitable. Concrete is known to shrink and expand with the variations of moisture contents, pressure, and temperature. Consequently, the cracks that emerge to accommodate these changes are not implemented in the design and development of cementitious materials. [1] The other factors that often affect the concrete durability are the shrinkage, design flaws, or poor quality of construction materials. [2] Subsequently, a sequence of critical events originates within these structures due to the formation of cracks. [3], [5] This not only affects the functionality of the structure but also their durability and strength. To enhance the concrete resistance against these defects and degradations, the innovative concept called self-healing is coined. [7]

By definition, self-healing is the material's ability to heal/ recover and repair damages automatically and autonomously in the absence of any external involvement. [9] Several terms including self-repairing, autonomic-healing, and autonomic-repairing are invariably used to signify such material property. Often, incorporation of self-healing properties in manmade materials does not effectively manifest without an external trigger. [4]

It is established that concrete can withstand compressive forces very well but not tensile forces. Truly, it generates cracks when subjected to tension. Thus, concretes are reinforced with steel to resist the tensile forces. Structures such as underground basements and marine constructions those built in great aqueous environment are particularly vulnerable to corrosion of steel reinforcement. [8] Motorway bridges are also susceptible because the salts used to de-ice the roads gets penetrated into the cracks and thereby accelerates the corrosion. In most civil engineering constructions, tensile forces mediated cracks appear soon after the structure is built. Usually, the conventional concrete structures are repaired by applying a concrete mortar bonded to the damaged surface. Occasionally, mortars are tied with metal pins to ensure the permanent attachments. Indeed, these repairs are not only time consuming but expensive too due to the inherent difficulty in accessing these structures, especially for deep underground or highly elevated one. [3]

In 1996, Dry first developed polymer matrix composites and demonstrated its feasibility to self-repair internal cracks created via mechanical loading. The study focused on the cracking of hollow repair fibers dispersed in a matrix and the subsequent timed release of repair chemicals which resulted the sealing of cracks, the restoration of strength in damaged areas and the ability to retard crack propagation. [4] We characterize epoxy resin modified polymeric-cementitious materials (devoid of any hardener) prepared with different proportions of polymer and cement using WDC accompanied by long term DC method. Self-healing properties and strength of these mortars are determined via various techniques.

2.0 EXPERIMENTAL

2.1 Self-Healing Concrete

As aforementioned, concrete displays shrinkage and expansion under the exposure of extreme heat, cold, and external stress during usage. [1] Design flaws and poor quality of construction materials are the other factors that severely affect the concrete lifecycle. Furthermore, various loadings from heavy vehicles, earthquakes, and strong winds result cracks in concrete. [5] Generally, cracks are initiated inside the concrete structures through a series of vital events. To boost the concrete structures functionality, durability, and strength against defects and degradations, self-healing concrete revolution begun. [1] Self-healing agents are automatically healed concrete structures subjected to cracks. They are conveyed through strong core microcapsules, hollow reinforced fibers, and forms of organic matter. Currently, their testing and analysis regarding durability and longevity are in progress. Meanwhile, several self-healing chemicals that existed in the micro-encapsulation process are identified. [6]

2.2 Materials and Methods

The recurring theme of this work is to develop a self-healing polymeric-cementitious material using epoxy resin (Diglycidyl Ether of Bisphenol A Type) and determine their strength and hardness. The engineering properties of epoxy-modified mortars as self-healing materials and the mechanism responsible for hardening and self-healing function of epoxy resin are inspected. The performance effectiveness of epoxy-modified materials is evaluated using UPV techniques. Ordinary Portland cement (OPC), fine aggregate, and epoxy resin as illustrated in Figure 1 are used as raw materials.

Figure 1 Raw material of OPC and fine aggregate

2.3 Proportion of Mixtures

As displayed in Figure 2, three series of control mortar mixtures are prepared in the Laboratory of Structures and Materials, Universiti Teknologi Malaysia. The control mixtures are made of OPC, fine aggregates and water. The water to binder ratio for all mixtures is set at 0.48 and the polymer to binder ratios is varied from 5 - 20%.

Figure 2 Synthesis of control mortar mixtures for UPV test

The mixing process of epoxy resin self-healing mortar is same as that of OPC. A strict quality control is followed during materials preparation and mixing to achieve the specific design strength. All specimens are made with fine aggregate to cement ratio by mass of 3:1. Fine aggregates are maintained with saturated surfaces under dry condition. The polymer to cement ratios is varied to 0, 5, 10, 15, and 20% to acquire optimized composition as summarized in Table 1.

Table 1 Proportions of mixtures in self-healing mortars

kg/m^3			%		
Cement	**Water**	**Sand**	**P/C**	**W/C**	**Sand: Cement ratio**
506	228	1517	0	0.48	3:1
			5		
			10		
			15		
			20		

2.4 Curing Conditions

Two curing conditions including WDC and DC are applied for epoxy resin mortars. For WDC, the specimens are put under wet gunny for 2 days followed by 5 days in water. Then, the specimens are taken out and kept at room temperature for 21 days. Conversely, DC of the specimens is performed for 2 days under wet gunny condition followed by room temperature exposure for 26 days

2.5 Tests

The tests for self-healing evaluation are conducted after the mortars are matured. Test time duration of 28, 56, 90, and 180 days are chosen to identify the reaction of self-healing agent with the crack inside the mortars. Microstructures self-healing evaluation, tests for strength development and pre-loading stress impacts are carried out. After the initial curing, the micro cracks are allowed to propagate by subjecting compressive loading at the rate of 0, 50, and 80% of maximum load for each type of specimen. Next, the specimens are dry-cured at room temperature for 0, 1, 3, and 6 months. Non-destructive test (Figure 3) is performed using UPV machine to confirm the preloading.

Figure 3 Confirming the preloading via UPV machine

3.0 RESULTS AND DISCUSSION

3.1 Analysis of Compressive Strength

Compressive strength is determined using cubic shaped specimens each of size 100 mm x 100 mm x 100 mm. Five types of mixtures are prepared by blending different ratio of polymer to cement (p/c), followed by their initial screening. Sample with 10% p/c is observed to be the strongest as illustrated in Figure 4.

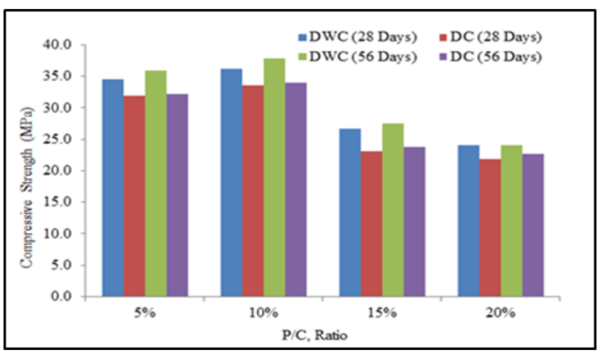


Figure 4 Polymer to cement ratio dependent compressive strength

This sample is further subjected to the mechanical testing at 28 and 56 days followed by DC and WDC. The increasing compressive load is applied until the failure and the maximum load for failure is recorded. Figure 4 shows the p/c ratio dependent compressive test results for two different duration and types of curing. The compressive strength of the control samples (OPC) at 28 and 56 days are determined to be 30.84 and 30.93 N/mm^2, respectively. For p/c ratio of 5, 10, 15, and 20 the strength at the 28 days DC test are observed to be 31.90, 33.6, 23.14, and 21.81 N/mm^2, respectively. Conversely, tor the same ratios the compressive strength at 28 days WDC test is measured to be 34.5, 36.19, 26.68, and 24.04 N/mm^2, respectively. In addition, the compressive strength for the same set of samples for 56 days of DC test is discerned to be 32.20, 34.03, 23.79, and 22.70 N/mm^2, respectively. The compressive strength for 56 days of WDC test is 35.90, 37.90, 27.5, and 24.11 N/mm^2, respectively. These results clearly exhibit that p/c ratio of 10% WDC is the optimum composition and the value to compressive strength is decreased thereafter.

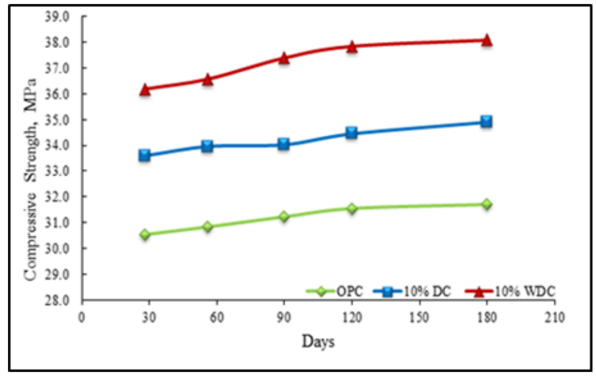


Figure 5 Time dependent compressive strength development

Figure 5 compares time dependent improvement of compressive strength for OPC, 10% of p/c under WDC and DC. The WDC process is evident to be optimum with the emergence of hardening over time.

3.2 Self-Healing

The self-healing function is determined by pre-loading the mortars to 50 and 80% of maximum load followed by non-destructive testing. Tables 2 and 3 enlist the self-healing progression of specimens after 6 months of WDC.

Table 2 UPV data after 6 months of WDC with 80% pre-loading

	Before		After								
Type	Pre-loading		Pre-loading 80%	1 month		2 months		3 months		6 months	
	UPV, km/s	MPa	UPV, km/s	UPV, km/s	MPa	UPV, km/s	MPa	UPV, km/s	MPa	UPV, km/s	MPa
10% WDC	24.3	36.2	31.4	31.0	28.7	28.3	31.3	27.7	33.7	26.1	34.2
OPC	28.5	31.2	35.6	35.6	24.5	34.2	25.0	33.7	25.9	33.5	27.6

Table 3 UPV data after 6 months of WDC with 50% pre-loading

	Before		After								
Type	Pre-loading		Pre-loading 50%	1 month		2 months		3 months		6 months	
	UPV, km/s	MPa	UPV, km/s	UPV, km/s	MPa	UPV, km/s	MPa	UPV, km/s	MPa	UPV, km/s	MPa
10% WDC	24.3	35.7	28.7	27.6	18.9	26.7	23.5	26.1	25.4	25.6	27.6
OPC	28.5	30.8	35.6	35.6	15.0	34.2	15.5	33.7	16.9	33.5	17.6

Pre-loading with 50 and 80% of the load failure resulted internal cracks inside the sample. Afterwards, the specimens are subjected to DC for 1, 2, 3 and 6 months and subsequent UPV tests are performed as depicted in Figure 6.

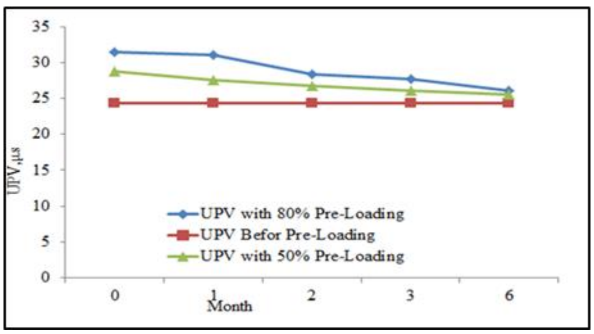


Figure 6 UPV (km/s) readings after 6 months dry curing (10% p/c)

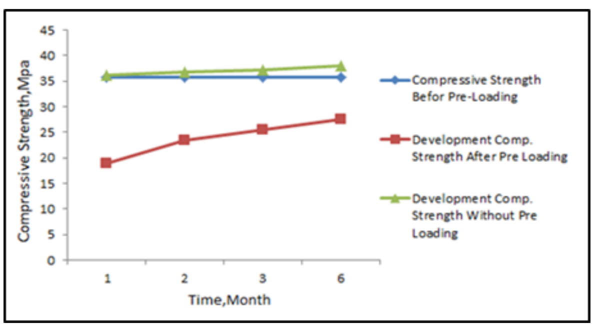


Figure 7 Development of compressive strength after 6 months of DC for specimen with 10% p/c at 50% pre-loading

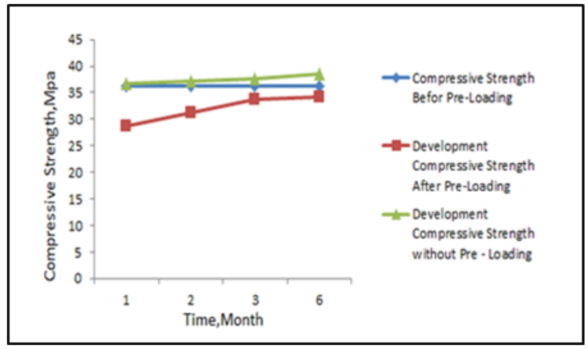


Figure 8 Development of compressive strength after 6 months of DC for specimen with 10% p/c at 80% pre-loading.

Figure 7 and 8 shows the time dependent variation in the compressive strength after pre-loading of 50 and 80% of the maximum load. The specimens are subjected to DC after pre-loading and the data are recorded. From Table 2 it is evident that 10% of epoxy has modified the UPV reading, meaning the cracks are produced under pre-loading and simultaneous repair occurred. After 6 months of curing, the compressive strength and UPV reading is further improved. This development in the compressive strength over time indicates that the polymer is capable of closing the internal crack automatically. The achieved modification in the compressive strength with 50% pre-loading (with smaller crack) is found to be better that that of 80% pre loading (with greater cracks).

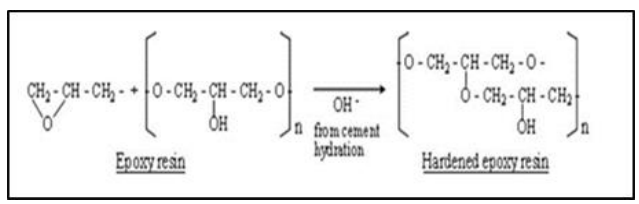


Figure 9 Chemical reactions between epoxy resins and cement hydrates

The mechanism of self-healing is understood in terms of the chemical reactions between epoxy resins and cement hydrates as illustrated in Figure 9. The self-healing is initiated from the reaction of epoxy resin with the OH ion (hydroxide ion) produced from cement hydration process. The occurrence of 10% epoxy resin in mortar is found to be superior most for the self-healing and functioning.

4.0 CONCLUSION

We report the preparation and subsequent characterization of self-healing mortars. Various test under loading confirmed the achievement of modified strength for the optimized compositions. It is established that WDC is the best and suitable curing for epoxy-modified mortar because it provided an efficient hydration and polymerization process to occur. Based on the compressive strength after 28 days, the optimum polymer to cement ratio is determined to be 10%. The hydroxide ion from cement hydration process that reacted with epoxy resin is observed to be responsible for self-healing. Our systematic methodology and the admirable features of the results may contribute towards the development crack mediated self-healing phenomena in concrete to reduce the cost for structure maintenance.

Notations

WDC	Wet Dry Curing
DC	Dry Curing
P/C	Polymer – Cement ratio
UPV	Ultrasonic Pulse Velocity

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