PERFORMANCE OF EPOXY-BASED SELF-HEALING MORTAR

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DEDICATION

Praise be to Allah s.w.t, the Lord of the Worlds

Who says (interpretation of the meaning):

“Give thanks to Me and to your parents. Unto Me is the final destination”

[Quraan, Luqmaan 31:14]

All glory and honor to Him

To my parents
Fadzillah Binti Idris and Ariffin Bin Sulaiman

To my greatest supporters
Nur Farhana, Mohd Syafiq Fauzan, Muhammad Syafiq Sazzuann, Nur Abyan Nabilah, Alif Dzulfieka and Qaireen Nur Amani

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ABSTRACT

Concrete is one of the oldest and versatile construction materials. After water, it is the most utilised material in the world. However, due to various reasons, the formation of micro-cracks can lead to a major problem such as corrosion of steel reinforcement and further deterioration of the structure. The cost of repairing deteriorated concrete structures is expensive as effective remedial requires special repair materials and skilled labours. Thus, the development of new technologies and material that can automatically repair cracks, consequently restore or even increase the strength of both mortar and concrete to prolong the service life is highly needed. Nowadays, the self-healing by using bacteria as a healing agent had gained interest among researchers. However, limited study had been conducted on the use of polymer as a healing agent. Thus, the aim of this study is to investigate the effects and behaviour of epoxy resin without hardener as a self-healing agent in mortar. The epoxy resin was used without hardener based on the understanding that it can harden inside the mortar through the reaction between the epoxy resin and hydroxyl ions produced from the cement hydration process. In this study, the 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. Two types of curing regimes were used namely dry-curing and wet-dry curing. Normal mortar was also prepared as a control sample. Various tests were carried out to determine the characteristics of materials including viscosity, Fourier transform infrared spectroscopy, X-ray fluorescence and sieve analysis. The performance of the hardened mortar containing epoxy resin without hardener was determined through compressive strength, flexural strength, splitting tensile strength, initial surface absorption, water absorption, apparent porosity, drying shrinkage and strength development. Furthermore, various techniques including X-ray diffraction, scanning electron microscopy and thermogravimetric analysis were used to study the microstructure of the hardened epoxy-modified mortar. The ultrasonic pulse velocity, permeability, damage degree and healing efficiency were conducted to determine the crack healing process by the epoxy resin inside the cracked mortar specimens. The experimental results show that the optimum percentage of epoxy resin used in the mix was 10% as it gives the highest engineering properties compare to other percentage. The wet-dry curing regime was found to give better performance of the sample containing epoxy resin compared to dry curing. The epoxy resin without hardener as a healing agent was found to perform effectively as the compressive strength, ultrasonic pulse velocity and permeability of the cracked mortar samples regain the initial reading with prolonged curing time. The microstructure study also revealed that the epoxy resin reacts with the hydroxyl ions to heal the micro-cracks in the mortar specimen. Overall test results together with microstructure study showed that epoxy resin without hardener can be used as a self-healing agent in repairing the micro-cracks.

ABSTRAK

Konkrit merupakan bahan binaan tertua dan serba boleh yang paling banyak digunakan di dunia selepas air. Walau bagaimanapun, disebabkan faktor tertentu, penghasilan retakan mikro boleh menyebabkan masalah seperti pengaratan tetulang keluli dan kerosakan kepada struktur. Kos membaikpulih struktur konkrit adalah mahal kerana kerja-kerja pemulihan memerlukan bahan terpilih dan tenaga kerja mahir. Oleh itu, teknologi dan bahan yang dapat membaiki retakan secara automatik dan mampu meningkatkan kekuatan mortar dan konkrit seterusnya memanjangkan hayat perkhidmatan adalah sangat diperlukan. Pada masa ini, konkrit penyembuhan automatik dengan menggunakan bakteria sebagai agen penyembuhan telah mendapat perhatian para penyelidik. Walau bagaimanapun, kajian menggunakan polimer sebagai agen penyembuhan masih tidak meluas. Oleh itu, tujuan penyelidikan ini adalah untuk mengkaji kesan dan perlakuan resin epoksi tanpa pengeras sebagai agen penyembuhan dalam mortar. Resin epoksi yang digunakan adalah tanpa pengeras berdasarkan pemahaman bahawa ia boleh mengeras di dalam mortar melalui tindak balas diantara resin epoksi dan ion hidroksil yang terhasil daripada proses penghidratan simen. Dalam kajian ini spesimen mortar telah disediakan dengan nisbah 1: 3 (simen: pasir), nisbah air-simen 0.48 dan 5% hingga 20% resin epoksi daripada kandungan simen. Dua jenis pengawetan digunakan iaitu pengawetan kering dan pengawetan basah-kering. Mortar biasa disediakan sebagai sampel kawalan untuk perbandingan. Pelbagai ujian telah dijalankan untuk menentukan ciri-ciri bahan termasuk ujian kelikatan, *Fourier transform infrared spectroscopy*, *X-ray Fluorescent* dan analisis ayak. Prestasi mortar keras yang mengandungi resin epoksi tanpa pengeras telah dikaji melalui kekuatan mampatan, kekuatan tegangan, kekuatan lenturan, penyerapan air dipermukaan, penyerapan air, jumlah keliangan, pengecutan kering and peningkatan kekuatan. Disamping itu, pelbagai teknik, termasuk *X-ray diffraction*, *scanning electron microscopy* dan ujian *thermogravimetric* telah digunakan untuk mengkaji mikrostruktur epoksi mortar. Halaju denyutan ultrasonik, ketelusan, tahap kerosakkan dan keberkesanan penyembuhan telah dilakukan untuk menentukan proses penyembuhan retakan oleh resin epoksi dalam spesimen mortar yang telah diretakkan. Keputusan kajian menunjukkan peratus optimum resin epoksi yang digunakan adalah 10% kerana ia memberikan kekuatan mampatan mortar yang tertinggi. Pengawetan kering-basah didapati telah memberikan prestasi yang lebih baik bagi spesimen yang mengandungi resin epoksi tanpa pengeras berbanding pengawetan kering. Resin epoksi tanpa pengeras juga didapati sangat efektif sebagai bahan penyembuh berasaskan kekuatan mampatan, halaju denyutan ultrasonik dan ketelusan air spesimen mortar yang diretakkan memberikan bacaan seperti bacaan awal pada jangkamasa pengawetan yang lama. Kajian mikrostruktur juga menunjukkan resin epoksi bertindak balas dengan ion hidroksil dan menyembuhkan keretakan mikro dalam spesimen mortar. Keputusan keseluruhan beserta kajian mikrostruktur menunjukkan resin epoksi tanpa pengeras boleh digunakan sebagai agen penyembuhan keretakan mikro.

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LIST OF ABBREVIATIONS

50EM	-	50% Epoxy-Modified
50NM	-	50% Normal Mortar
80EM	-	80% Epoxy-Modified
80NM	-	80% Normal Mortar
ASTM	-	American Society For Testing And Materials
BS	-	British Standard
CSH	-	Calcium Silicate Hydrate
C_3S	-	Tricalcium Silicate
C_2S	-	Dicalcium Silicate
C_3A	-	Tricalcium Aluminate
C_4AF	-	Tetracalcium Aluminoferrite
$Ca(OH)_2$	-	Calcium Hydroxide
EDX	-	Energy Dispersive X-Ray
SEM	-	Scanning Electron Microscopy
ISAT	-	Initial Surface Absorption Test
TGA	-	Thermogravmetry Analysis
UPV	-	Ultrasonic Pulse Velocity
XRD	-	X-Ray Differaction
XRF	-	X-Ray Fluorescence

LIST OF SYMBOLS

σ_c	Compressive strength
σ_f	Flexural strength
σ_T	Splitting tensile strength
P	Load
A	Area
t	Time
L	Length
b	Width
d	Thickness
H	Height
D	Diameter
π	3.142

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Polymer-modified mortar or concrete represents a modification technique or concept that had been used in order to enhance the properties of normal mortar or concrete. As in 1923, the first patent of polymer modified mortar and concrete had been submitted by Cresson. That patent referred to the paving mixture containing natural rubber latexes as a polymer addition and cement was used as filler. In following year (1924), Lefebvre patented the first concept of polymer modification (Ohama, 1998). Since then, the development of polymer modification for cement, mortar and concrete have been conducted in various countries for almost 70 years or more. Currently, many effective polymer modification systems for cement mortar and concrete have been developed and are used in various applications in the construction industry.

Concrete is a strong and relatively cheap construction material and is therefore presently the most widely used material in the construction industry. The main constituent that contributes to concrete strength is Portland cement. It is estimated that cement (Portland clinker) production alone contributes about 7% of the global carbon dioxide emissions due to the burning of limestone and clay at a temperature of 1500°C. During this process, calcium carbonate ($CaCO_3$) is converted to calcium oxide (CaO) and carbon dioxide is released (Worrell *et al*., 2001). From an environmental standpoint, concrete does not appear to be a sustainable material (Gerilla *et al*., 2007). Moreover, improperly manufactured concrete may experience shorter service life as it can easily develop cracks due to excessive loading applied and other environmental causes. A good quality of concrete is needed which not only prolongs its service life but would also reduce the production of cement. The main focus in this study is to develop a maintenance-free self-healing concrete that can curtail the need for cement production as well as reduce the carbon dioxide gas emissions in the atmosphere.

The major problem of concrete structures is cracking. A cracking of concrete can cause greater damage such as corrosion of steel reinforcement that can lead to the collapse of the structure. A number of cracks with different morphology and size will appear during the construction and the design life of the structure. In various environmental conditions, including temperature and humidity changes, the formation and expansion of the cracks will lead to reinforcement corrosion and reducing the load carrying capacity of the structure (Zhang *et al*., 2011). The allowable crack appears to be limited to micro-cracks with width of up to 0.1 to 0.2mm (Li and Yang, 2007).

Small cracks or micro-cracks need to be repaired before they become a major crack. There are many types of repair materials and methods that can be selected and used for repairing micro-cracks. However, in certain cases micro-cracks will still occur in the concrete structure. If the micro-crack cannot be repaired effectively, the performance of the structure is affected and service life will be shortened. The cost of repairing concrete is expensive as it requires many raw materials such as cement and skilled labour. Therefore, the development of new technologies and materials that can automatically repair cracks and restore/increase the strength of concrete giving longevity to the structure is essentially needed. Such concrete material will eventually reduce maintenance costs as well as fulfil the need for sustainability.

1.2 Problem Statement

In many concrete structures, tensile forces can lead to cracks and these can occur relatively soon after the structure is built. Repair of conventional concrete structures usually involves applying mortar which is bonded to the damaged surface. Sometimes, the mortar needs to be keyed into the existing structure with metal pins to ensure that it does not fall off. The bonding property between host concrete and new repair materials needs to be carefully checked to avoid another problem leading to extra repair cost. Repairs can be particularly time consuming and expensive; often it is very difficult to gain access to the structure to make repairs, especially if they are underground or at a great height.

The occurrence of autogenous healing or self-healing of cracks in concrete was recognized in a previous study by Reinhardt and Jooss (2003). In their study, they develop a high-strength concrete and tested on permeability and self-healing behaviour of cracked concrete. The self-healing performance was checked at the temperature between 20 °C and 80 °C and crack width between 0.05 and 0.20 mm. On the other hand, the research of Jonkers and Schlangen (2008) had developed a microorganism that can heal the cracks automatically. The concrete that can repair the crack automatically is called self-healing concrete. Self-healing concrete refers to the ability of concrete to automatically repair the cracks without any external intervention. This phenomenon has been adapted from the human skin healing process.

The bacteria used in some studies are known as microorganism that can survive in alkaline environment of concrete, as long as the air is available. In most of these studies, an ureolytic bacterium of the genus Bacillus was used as a healing agent for the biological production of calcium carbonate. The mechanism of calcium carbonate formation from the bacteria is based on the enzymatic hydrolysis of urea to ammonia and carbon dioxide (Hager *et al.*, 2010). This reaction causes pH to increase from neutral to alkaline conditions resulting in the formation of bicarbonate and carbonate ions which precipitate in the presence of calcium ions to form calcium carbonate minerals (Jonkers *et al*., 2010). This method proved promising for repairing cracks in concrete, allowing the concrete to regain strength and decrease permeability. Many research studies have been conducted using bacteria as a self-healing agent. Bacteria is a good self-healing agent but the acceptance of using it in the concrete is been a big issue among contractor. The perceptions of using bacteria in the concrete become a serious concern due to the bacteria itself that have a negative effect to human health.

The epoxy resin without hardener is introduced in this study as an additive material and self-healing agent in the mortar. Epoxy resin has been used as one of the repair materials to repair concrete for many decades. The use of epoxy resin which is an external repair process entails the injection of epoxy resin solution together with a hardener into existing concrete structure cracks. This method is well known and usually used for concrete structure repair suffering from cracks. Epoxy resin with different level of viscosity can repair cracks with different widths. Upon injection, the pressure applied leads the epoxy to flow through the cracks. In addition to using epoxy resin as a repair material, it can also be used as an additive or replacement material in concrete mixtures. The main advantage of epoxy resin is its amazing compressive strength, which can achieve 80 MPa or greater for most concrete (Kanerva *et al*., 1991). Usually, the use of epoxy resin with the hardener has been common practice. The hardener functions as hardening agent to the epoxy which strengthens the binders in concrete.

However in this study, the epoxy resin is used without hardener based on the understanding that epoxy resin can harden inside the concrete by reacting with hydroxyl ion. Hydroxyl ion is produced from cement hydration and reacts with epoxy resin to heal the cracks. The rationale for not using hardener was to let the epoxy resin stay in the same state for later when cracks occurred. The unhardened epoxy resin would then react with the hydroxyl ion to fill up the cracks. Quite limited studies have been conducted by using epoxy resin without hardener in concrete mixture and as a healing agent. This reported research focused on the engineering properties and self-healing performance of epoxy resin without hardener as an additive and self-healing agent as well as the microstructure studies.

1.3 Aim of the Study

The main aim of this research is to investigate the performance of epoxy resin without hardener as a healing agent in mortar.

1.4 Objectives of the Study

Several objectives can be drawn according to the problem statement. The objectives are as follow:

i. To characterize the properties of materials use in the study including epoxy resin, fine aggregates and Portland cement.

ii. To determine the optimum mix proportions of mortar using various epoxy-cement ratios.

iii. To investigate the engineering properties of self-healing epoxy-modified mortar.

iv. To investigate the mechanism of self-healing process of epoxy resin in hardened mortar.

v. To evaluate the self-healing performance of epoxy-modified mortar using microstructure study.

1.5 Research Question

Four major questions needed to be answered from this research:

i. What are the mechanical properties of mortar by using epoxy resin without hardener?

ii. How epoxy resin without hardener can contribute to the strength of the mortar?

iii. How does the mechanism of epoxy resin as a self-healing agent work to heal the cracks?

iv. What is the microstructure morphology of mortar with epoxy resin without hardener?

1.6 Scope and Limitation of Study

An epoxy resin with high viscosity namely Diglycidyl ether of bisphenol A type was used. Only one type of polymer was used since this polymer provides high reactivity even without hardener. The amount of epoxy resin without hardener was added in the mortar mixture was 5%, 10%, 15% and 20% of cement content in order to select the best amount of epoxy resin that can corporate as a healing agent inside the mortar specimens. The epoxy resin was added as an addition material in the mortar mixture so the mortar was categorized as a polymer-modified mortar or in this research was called epoxy-modified mortar. There are two types of curing regime applied to the mortar specimens which is dry curing and wet-dry curing. Various tests were carried out to determine the characteristics of materials including viscosity, Fourier transform infrared spectroscopy, X-ray fluorescence and sieve analysis. The performance of the hardened mortar containing epoxy resin without hardener was determined through compressive strength, flexural strength, splitting tensile strength, initial surface absorption, water absorption, apparent porosity, drying shrinkage and strength development. Furthermore, various techniques including X-ray diffraction, scanning electron microscopy and thermogravimetric analysis were used to study the microstructure of the hardened epoxy-modified mortar. The ultrasonic pulse velocity, permeability, damage degree and healing efficiency were conducted to determine the crack healing process by the epoxy resin inside the cracked mortar specimens.

The main function of epoxy resin without hardener was to serve as self-healing agent while producing high mechanical properties. The production of cracks was generated by 50% and 80% of the ultimate load and was checked by using the Ultrasonic Pulse Velocity (UPV) together with compressive strength test. the crack was produced in epoxy-modified self-healing mortar at the age of 28 days, 180 days and 365 days. The cube specimen of 70 mm × 70 mm × 70 mm size was used for compressive strength test, 100 mm × 100 mm × 100 mm for self-healing evaluation, 40 mm × 40 mm × 160 mm prism for flexural strength test and cylindrical specimen of Φ70 mm × 150 mm for splitting tensile test. Normal mortar was cast as a control specimen and water curing was applied. The tests were conducted based on American Standard Testing Method (ASTM) standard and British Standard (BS). Some testing methods such as degree of hardening of epoxy resin were adopted from previous researchers, since it was not stated in any other established standard.

1.7 Significance of Research

The programme of research significant is summarised in the flow chart shown in Figure 1.1. The significant findings of this study by using epoxy resin without hardener as a self-healing agent are as followed:

i. Enhance the service life of concrete structure by repairing micro-cracks automatically before the cracks become worse and as result, the service life of the concrete/mortar structure will be increase.

ii. Reduce the cost of repairs by self-healing repair phenomenon.

iii. Minimize environmental impact by reducing materials used for repair work especially the production of cement that produce carbon dioxide gases.

iv. Develop a self-healing agent by using existing materials instead of bacteria.

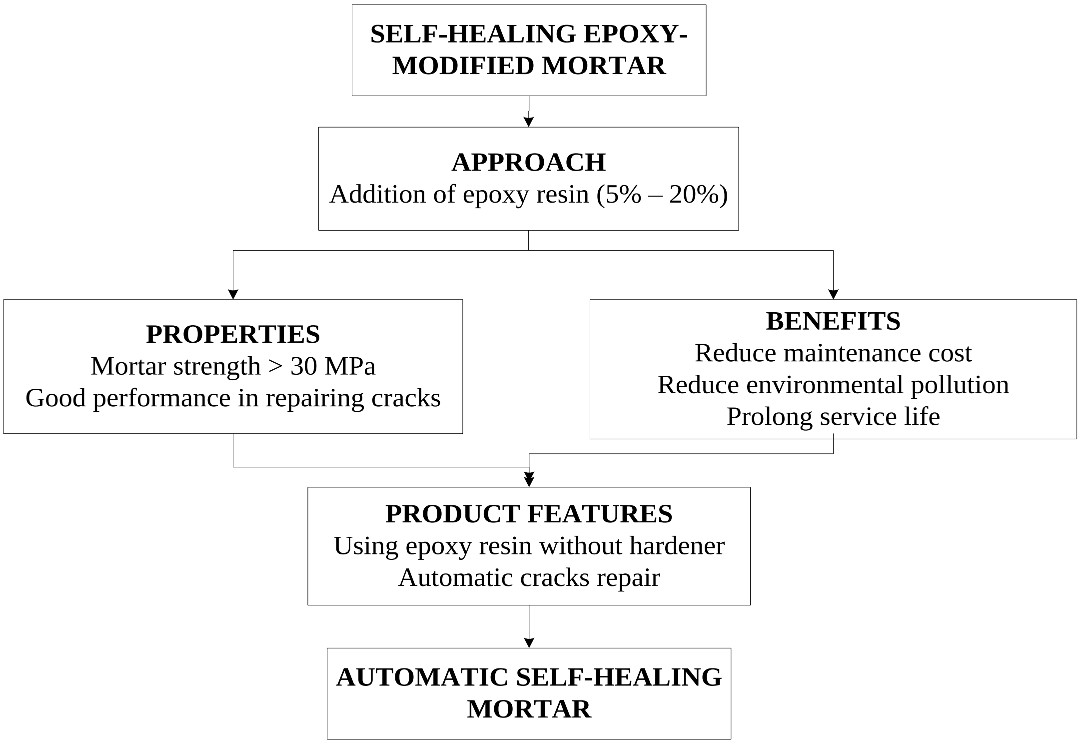

Figure 1.1 Research Significant

1.8 Thesis Outline

Chapter 1 (Introduction): Chapter 1 discusses the background of the research comprising the statement of problem, research objectives, research scope, the significance of the research, and limitations of study.

Chapter 2 (Literature Review): Chapter 2 initially discusses the past research from various researchers all around the world. The main focus is to identify important performance criteria and test parameters that had been done and compared with current research. This chapter then discusses the history, studies, science and different approaches of self-healing performance and evaluation. The differing self-healing performance evaluation methods are discussed by looking at the different healing agents that each researcher had reported.

Chapter 3 (Methodology): Chapter 3 focuses on testing method that had been conducted in order to evaluate the performance of epoxy-modified mortar. The methodology is separated into three parts: a) fresh properties of epoxy-modified mortar, b) hardened properties, and c) self-healing evaluation. All the testing methods and parameters are discussed in this chapter.

Chapter 4 (Engineering Properties of Epoxy-Modified Mortar): Chapter 4 reports and explains the analyses undertaken for achieving the first, second and third objectives of this research. The results of material properties and engineering performance of epoxy-resin without hardener are discussed in this chapter. Some sections from objective 5 based on microstructure morphology are also included.

Chapter 5 (Evaluation of Self-Healing Epoxy-Modified Mortar): Chapter 5 discusses the self-healing performance of epoxy-modified mortar as to achieve the

Objectives 4 and 5. The correlation analysis was carried out in order to evaluate an epoxy resin as a self-healing agent.

Chapter 6 (Conclusions and Recommendations): Chapter 6 concludes the findings of the overall research work that was undertaken. The contribution and implications of the findings toward the construction industry in general and performance evaluation in particular are explained. The limitations and possible improvements for future undertakings are also discussed. Suggestions for future research are conveyed in the final part of this chapter.

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