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Toughness improvement of epoxy resin mortar by incorporation of ground calcium carbonate

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highlights and the second second

- The impact toughness of ERM had greatly improved with GCC addition.
- GCC induced lots of crazes and triggered extensive plastic deformation in ERM.
- The interface boundary between epoxy matrix and fillers (sand and GCC) was perfect.
- The high-performance repair mortar is obtained through orthogonal experiment.

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ARSTRACT

In this paper, ground calcium carbonate (GCC) was selected to improve the properties of epoxy resin mortar (ERM). The results indicated that the flexural strength and ratio of flexural strength to compressive strength of ERM got great increase by incorporation of GCC. At the same time, the impact toughness of ERM had greatly improved with GCC. To some extent, it increased with increasing addition of GCC. Scanning electron microscope (SEM) analysis illustrated the toughening mechanism of ERM filled with GCC. Through orthogonal experiment, the optimal combinations of the process parameters for a highperformance repair mortar are obtained.

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1. Introduction

To renovate and retrofit the old or impaired component, buildings and infrastructures with high quality repair materials is an economical and practical way to extend their service life. Herein increasing needs for enhanced performance of the repair materials are raised by the end user such as superior adhesion, higher compressive/flexural strength, high fracture toughness as well as great compatibility to underneath concrete substrate [\[1–3\]](#page--1-0). There are many kinds of materials available from the market ranging from traditional materials like cementitious material, epoxy, unsaturated polyester resin, polymer latex $[4]$ to emerging new type of reinforcing materials such as magnesium phosphate cement

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[\[5–8\].](#page--1-0) Among these materials epoxy repairing material (ERM) has become one of the most popular reinforcing materials for strengthening and repairing of buildings because of its excellent mechanical properties, interfacial adhesion and durability.

Epoxy is a highly crosslinking thermal setting material, this structure leads to high modulus, failure strength, thermal stability and so on. However, this material is regarded as a brittle material due to the same reason, this is quite undesirable when it is used as repair material because of its poor resistance to crack initiation and growth. Lots of efforts have been made in an attempt to enhance the properties of ERM with focus on the mechanical properties and durability by a variety of modification approaches. Polymers latex, redispersible polymer powder and water-soluble polymer have been developed by emulsification and particle dispersion technique to improve the performance of ERM $[4,9]$. Compared with dominant acrylic-based polymer modified mortar, modified mortar with epoxy emulsion developed by Aggarwal showed superior strength, better resistance to the penetration of chloride ions

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and carbon dioxide, it can be widely used for repair works in humid and industrial environments [\[10\];](#page--1-0) Luo et al. introduced PVA fiber into aqueous epoxy resin cement mortar and found PVA fiber had greatest influence on the fracture toughness (i.e., flexural/compressive strength ratio) and bond strength. The composition for the best performance is developed by orthogonal experiment as polymer/cement ratio at 0.20, water/cement at 0.35 and PVA fiber loading at 1% [\[4\].](#page--1-0) With different resin/sand, Nóvo studied the mechanical behavior of a cork which ranged from 0% to 45% of the total aggregate volume modified polymer mortar, the result showed the fracture toughness of cork-modified polymer concrete got improvement, the elastic modulus decreased, the brittle failure became less with increasing cork content [\[11\],](#page--1-0) Li studied the influence of nano-size calcium carbonate particle on the mechanical properties of epoxy resin. With the coupling agent added, the dispersion of nano-CaCO₃ in the resin matrix improved and therefore the compatibility between nano-CaCO₃ and epoxy resin were improved as well $[12-14]$, the results indicated the tensile strength increased by 39%, flexural modulus increased by 52.9%, flexural elastic modulus enhanced by 52.9% and the impact strength improved by 68.6% [\[15\]](#page--1-0). The enhancement of CaCO₃ with silane was confirmed again by He et al. $[13,14]$, they concluded that epoxy resin mixed with nano-CaCO₃ presented higher thermal stability and mechanical strength which was attributed to the surface modification of nano-particles. To explain the toughening mechanism by such kind of rigid filler, several theories have been developed, including crack pinning, crack deflection, interfacial debonding and plastic void growth. However, the real toughening mechanism for a specific material is still no that straightforward to clearly explain the observation [\[16–18,13\]](#page--1-0).

In the present study, GCC is introduced into ERM as the 5th ingredient in addition to epoxy resin, hardener, active diluent and sand in order to improve the toughness for ERM. Compared to sand, the size of GCC particle in micro-level is much smaller and it could be expected as rigid filler to bring about toughness improvement. Nevertheless, other properties other than toughness could also be affected such as rheology, strength development and so on. In order to achieve best material performance, orthogonal experiment is employed with 3 factors (diluent, curing agent and filler) and 4 responses (compressive strength, the flexural strength, the bond strength and the impact toughness). Fracture surface morphology of ERM is analyzed by means of SEM, the toughening mechanisms of ERM filled with GCC is discussed by these morphology analysis.

2. Experimental part

2.1. Materials

ERM includes epoxy resin, diluents, curing agent, fillers and other additive (i.e., coupling agent and defoamer). The epoxy equivalent of Epoxy resin CYD-128 (E-51) is 184–194 g/eq. 501 is the reactive diluent n-butyl glycidyl ether. The chemical structure of epoxy resin is shown in Fig. 1. The curing agent is triethylenetetramine. The coupling agent is γ -glycidylpropyltrimethoxysilane KH560 (A.R.). The chemical structure of KH560 is displayed in Fig. 2. The defoamer of BYK-A530 is a solution which composes of antifoaming polymer and polydimethylsiloxane. The sand is commercially available river sand, with fineness modulus 2.56 after oven-drying and 16 mesh sieve screening. Filler is ground calcium carbonate (GCC), the average particle size of GCC is 12.5 μ m and apparent density is 1.41 g/cm³.

Fig. 2. The chemical structure of KH560.

2.2. Preparation and characterization of specimens

Epoxy E-51 and diluent n-butyl glycidyl ether 501 were mixed with vigorous stirring, then adding silane KH560 and defoamer BYK-A530 with stirring again to obtain an even epoxy solution. GCC and sand were dry-mixed firstly with a mortar mixer, and then the dry mixture was mixed into the epoxy mixture solution with stirring again. Finally, epoxy hardener triethylenetetramine was added into epoxy/GCC/sand mixture at lower stirring rate for 2 min and higher stirring rate for 3 min. In the end, the well-mixed slurry was cast into three prism molds of 40 mm \times 40 mm \times 160 mm, which was put in the vibrator to remove the air bubble. The mold was brushed with release wax for easy demolding afterwards.

The slump flow and the impact toughness test of the ERM was measured according to the criteria DL/T 5193-2004 (China) [\[19\]](#page--1-0) (ACI 544.2R-89, reapproved 1999), as shown in [Figs. 3](#page--1-0) and [4](#page--1-0). The impact toughness is calculated by the following equation:

$f_{AT} = (9.8 G H n_I)/V,$

where n_l is the initial-crack impact cycle number of ERM, V is the volume of the specimen, and G is weight of impact hammer. H is the falling height of impact hammer.

The flexural and compressive strength of ERM were determined, as the criteria GB/T 17671-1999 (China) specified [\[20\]](#page--1-0) (ISO 679). The bond strength between ERM and concrete was tested with the bond strength tester (HC-6000C, Hichance, China) according to the criteria GB 50367-2006 (China) [\[21\]](#page--1-0) , as shown in [Fig. 5](#page--1-0).

After the mechanical testing, the fracture section morphologies of the samples were examined with scanning electron microscope (SEM, FEI Quanta 200, Holland) (see [Fig. 6](#page--1-0)).

2.3. Experimental design

Different amount of GCC was chosen as the variables in the mix design of ERM, as listed in [Table 1.](#page--1-0) The strength and toughness of ERM were analyzed with the compressive strength and flexural strength in different period (i.e., 1, 3 and 7 days), and the impact toughness at 7 days.

3. Results and discussions

3.1. Slump flow of GCC modified ERM

The slump flow test showed that the consistency of ERM decreased with increasing GCC amount when keeping the total filler and aggregate amount the same. It is obviously due to the total surface area increasing with GCC bearing smaller particle size to replace part of bigger particle sand. Increased internal fluid friction induced by epoxy-wrapped GCC is responsible for the reduced slump flow. Of course, the particle size of GCC is expected to be a factor to modify the slump flow. Too much of GCC replacement of sand is not good for flowability of ERM.

3.2. The fracture toughness of GCC modified ERM

The compressive strength, flexural strength and their development are plotted in [Fig. 7](#page--1-0)a and b. The ratio of flexural strength and compressive strength (f_b/f_c) is used as an indicator for flexural toughness, as showed in [Fig. 7](#page--1-0)c.

Fig. 1. The chemical structure of epoxy resin.

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