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Evaluation of self-healing epoxy coatings for steel reinforcement

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HIGHLIGHTS

- Rebar with self-healing coatings outperforms controls in corrosion testing.
- Self-healing coatings on rebar outperform in all damage cases.
- Self-healing coatings on steel coupons were less consistent.
- Addition of microcapsules did not significantly affect coating adhesion after 28 days of testing according to ASTM D3359.

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ABSTRACT

Epoxy coatings are currently the most popular corrosion protection mechanism for steel reinforcement in structural concrete in North America. However, these coatings are easily damaged on worksites, negating their intended purpose. This study investigated self-healing coatings for steel reinforcement to introduce an autonomous healing mechanism for damaged coatings. Coatings were applied to steel coupons and rebar, intentionally damaged, and introduced to a corrosive environment via salt-water aeration tanks and accelerated corrosion testing. Performance of the experimental coatings was evaluated qualitatively and quantitatively. Adhesion strength and effects of coating thickness were also studied. Results from pre-corroded coated steel coupons submerged in salt-water aeration tanks exhibited improved corrosion resistance performance with self-healing coatings, although self-healing and conventional coatings performed similarly under other conditions. Steel reinforcement with a self-healing coating embedded in concrete and subjected to accelerated corrosion testing lasted longer than the conventionally coated counterparts. Self-healing coatings had comparable adhesion to the substrate as do conventional coatings. With numerous avenues for future research towards the adoption of self-healing coatings for steel reinforcement, this paper shows preliminary results demonstrating the potential benefits of their use.

1. Introduction

The American Society of Civil Engineers (ASCE) assesses infrastructure in the U.S. every four years based on key criteria including condition, operation and maintenance. Since 1998, American infrastructure has averaged a "D" grade. This score represents that "...infrastructure is in poor to fair condition and mostly below standard, with many elements approaching the end of their service life (sic). A large portion of the system exhibits significant deterioration. Condition and capacity are of serious concern with strong risk of failure" [1]. The ASCE estimates that $4.59 trillion are needed by 2027 to rehabilitate infrastructure to acceptable conditions. Furthermore, the ASCE recommends solutions including "support[ing] research and development into innovative new materials, technologies, and processes to modernize and extend the life of infrastructure, expedite repairs or replacement, and promote cost savings" [1].

Steel-reinforced concrete, also known as structural concrete, was first adopted in the U.S. in the late 1800s. Since then, structural concrete has become the most widely used infrastructure material. Currently, 9.2 billion yd^3 (7 billion m^3) of structural concrete is in place in the U.S. and 497 million yd^3 (380 million m^3) more are added each year [2]. Of the 607751 bridges in the U.S. as of 2013, roughly 66% are constructed with structural concrete [3].

In the mid 1970s, researchers recognized that the deterioration of bridge decks coincided with locations of deicing salt application and attributed this degradation to corrosion of the steel reinforcement. Corrosion of steel in concrete is a three-step process. First, a depolarization reagent and an electrolyte, such as oxygen and

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water, respectively, diffuse through concrete and arrive at the surface of the metal. Then, electrochemical reactions occur at the interface between steel and concrete. These reactions create a rust byproduct that accumulates on the surface of the metal [4]. The deposition of rust on the rebar causes the volume to increase within the confinement of the concrete, leading to internal stresses that crack through the concrete cover and cause spalling. The rust, it should be noted, does not adhere strongly to the original portion of steel, flaking off and causing an overall reduction in cross section, leading to reduced structural capacity. The presence of deicing salt greatly increases the rate of electrochemical reactions at the surface of the steel.

Many methods have been employed to delay the onset of corrosion. The most common corrosion protection method is the application of epoxy coatings to rebar [5]. Epoxy coatings have proven successful in delaying the onset of corrosion; however, the brittle coatings are easily damaged, which negates their usefulness. Self-healing coatings have the potential to be an improved mechanism, compared to conventional epoxy coatings, to inhibit deterioration of modern infrastructure. Self-healing coatings autonomously "heal" at damage locations and continue to provide protection to the substrate from corrosive media. Coatings that initiate self-healing in response to external damage have seen substantial research for anti-corrosion applications [6–8], but have only recently been investigated for structural concrete systems [9].

Chen et al. first reported the use of self-healing coatings containing microencapsulated tung oil for steel reinforcement within structural concrete [9]. Samples incorporating self-healing coatings lasted 300% longer than the conventional epoxy coatings. These results showed the potential to extend the service life of steel rebar placed in real world settings. However, in the study by Chen et al., the experimental damaged samples performed similarly to undamaged samples in accelerated corrosion testing. This suggests that the damage protocol did not induce sufficient coating damage to investigate the efficacy of the coating's self-healing ability.

The purpose of this work was to further assess the effectiveness of self-healing coatings under harsher damaging conditions and evaluate the coating's self-healing capacity. To this end, self-healing coatings were produced on steel coupon samples that were damaged and submerged in aerated salt-water tanks. The experimental self-healing coatings were also used to coat sections of rebar that were embedded in mortar and subjected to accelerated corrosion tests.

2. Methodology

2.1. Materials

Urea, formaldehyde, tung oil, ammonium chloride, resorcinol, and sodium chloride were obtained from Sigma-Aldrich. Ethyl maleic anhydride was received from Vertellus. Steel reinforcement was purchased from Sullivan Metals. Portland cement and aggregate were obtained from Bond Sand and Gravel. Steel coupons were purchased from Q-Lab Corporation. Two-part epoxy resin and activator were obtained from Rust-Oleum.

2.2. Microcapsule synthesis and characterization

The procedure used to encapsulate tung oil in a poly (urea-formaldehyde) shell was based on the work of Samadzadeh et al. [10] and is described in greater detail in Chen et al. [9]. Briefly: water, ethyl maleic anhydride (a surfactant), resorcinol (a stabilizer), ammonium chloride, and urea were mixed in a beaker. The pH of the solution was then adjusted from 2.7 to 3.5, and stirred in a water bath at 77 °F (25 °C). Tung oil was then added and stirred rapidly to form a stabilized emulsion, after which formaldehyde solution was added and the solution was stirred at 140 °F (60 °C) while microcapsules formed. Capsules were recovered using vacuum filtration, and were washed with deionized water and acetone before air-drying. Microcapsules were imaged using Scanning Electron Microscopy (SEM) in order to determine their size distribution.

2.3. Self-healing coating preparation

Self-healing coatings were prepared containing 10 wt% microcapsules by combining microcapsules with a two-part epoxy. It was observed during early mixing that the pot life of the self-healing coatings was shorter than that of the unmodified coatings, possibly due to heat generated as a result of the frictional forces between microcapsules that accelerated the curing reaction. For this reason, the epoxy resin and microcapsules were first mixed in a planetary centrifugal mixer at 200 rpm for two minutes, followed by degassing at 400 rpm for 30 s, and then combined with the activator. The coating was then applied to both steel coupons and rebar specimens.

2.4. Steel coupon coating and damage

Coatings were applied to steel coupons so that the mechanical properties and corrosion resistance of the coatings could be studied on a flat surface. 3 in × 5 in × 0.032 in (76.2 mm × 127 mm × 0.8 mm) steel coupons, designed to comply with ASTM B117, were used. Prior to coating, all samples were taped around the edges using water resistant tape, paying particular attention to avoid entrapping air and ensuring a tight bond to the steel substrate. This border was measured using calipers to confirm a consistent tape thickness on all samples. Coatings were applied to the steel substrates and leveled with a plastic squeegee, using the tape as a guide for thickness control. Only one side of the coupon was coated at a time and left to cure for 72 h under ambient conditions.

Since coating thickness could affect corrosion resistance, measurements were made to determine whether there was a statistically significant difference between the unmodified and modified epoxy coatings. For each sample, ten thickness measurements were taken per side after the coating cured. For the first coated side, the confirmed manufacturer certified thickness of the substrate was factored out of the thickness measurements. After both sides were coated and cured, another ten thickness measurements were taken per sample and the average of the previous measurement was factored out. A one-way analysis of variance (ANOVA) was then conducted using 20 coating thickness measurements per sample for each of the different coatings. Three sample sets with different coatings were created for this study; two unmodified coatings with varying coating thickness and one with a self-healing coating. The coatings will be referred to as 0 wt% thin, 0 wt% thick, and 10 wt%, respectively.

Cut samples were created using a utility knife to make 3 in (7.62 cm) cuts on one side of the sample. Impact damaged samples were damaged with an 11 lb_m (5 kg) weight dropped from a height of 3 in (7.62 cm). The weight had a spherical tip with a 1 in (2.54 cm) diameter. Coatings were given three days to heal before adhesion and corrosion resistance testing.

Pre-corroded samples were created to assess the coatings' ability to inhibit further corrosion on samples that had already begun to corrode, mimicking a situation in which a coating is applied over an unnoticed corrosive area. To create these samples, one drop of 5 wt% sodium chloride solution (approximate volume of