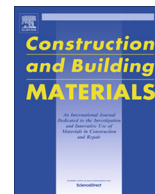




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## Bonding in cementitious materials with asphalt-coated particles: Part II – Cement-asphalt chemical interactions

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## HIGHLIGHTS

- Asphalt cohesion is the dominant failure mode between asphalt and cement.
- Chemical treatments to RAP can increase interfacial bond energy between asphalt and cement.
- Higher ITZ porosity is the reason for reduced modulus in concrete with RAP aggregates.
- Asphalt cohesion failure is the primary reason for reduced mechanical properties in concrete with RAP.

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## ABSTRACT

Reclaimed asphalt pavement (RAP), when used as an aggregate in concrete, will reduce bulk concrete strength and modulus. While Part I of this study investigated the properties of the interfacial transition zone (ITZ), Part II focuses on the nature of the cement-asphalt bond. Several chemical oxidative treatments of the asphalt were found to improve the interfacial cement-asphalt bond energy without affecting the ITZ porosity and size. Based on surface free energy measurements, the failure mode was estimated to occur preferentially as asphalt cohesion rather than cement-asphalt adhesion or ITZ cohesion. Based on the findings from Parts I and II, RAP aggregates reduce concrete strength and modulus because of: (1) the higher porosity in the ITZ, which produces a lower bulk modulus and allows for easier crack initiation, and (2) the preferential asphalt cohesion failure, which occurs rather than adhesive failure of the cement-asphalt interface or cohesive failure of the ITZ.

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## 1. Introduction

Reclaimed asphalt pavement (RAP) is recyclable by-product of roadway reconstruction that has seen somewhat recent interest as a partial replacement of aggregate in concrete pavements (see the Introduction in Part I [1] for an overview of RAP in concrete). Research has shown that the addition of RAP to concrete will decrease the strength and modulus properties, so the objective of the present study was to evaluate the microstructural causes for these decreases, particularly as it relates to bonding and the interfacial transition zone (ITZ). Considering that the ITZ is often referred to as the “weak link” in the failure of concrete – as a result of [2,3]: (1) the higher porosity in the ITZ, (2) the larger and preferentially-oriented CH crystals, and (3)

the potentially weak cement-aggregate bond – evaluation of the porosity and CH in the ITZ was examined in Part I, while Part II will focus on the chemical interactions at the cement-asphalt interface.

Based on the concrete mechanical property data by Brand and Roesler [4], the reduction in modulus was determined to not be a function of the asphalt coating thickness or stiffness. Rather, the reduction was reasoned to be a result of changes to ITZ composition and/or bond between the cement and asphalt [4–6]. Part I of this study found that the ITZ in cementitious materials at later ages (28 days) with RAP consisted of higher porosity, a larger ITZ size, and less CH and C–S–H contents near the RAP (asphalt) interface. However, the number of CH particles and distribution was not found to be different from the ITZ with dolomite aggregate, although the CH particles may be smaller near the asphalt interface, possibly because of a reaction with the asphalt. Despite the higher porosity at the RAP-cement interface, the cement-asphalt bond and potential treatment of RAP particles may have a significant impact on the bulk concrete properties.

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### 1.1. Hypothesis for bond improvement

There are two potential methods to improve the weakness of the ITZ: densification and physico-chemical interaction [7]. Densification is a process whereby very fine supplementary cementitious materials (e.g., silica fume) are added to the concrete in order to improve the packing density and reduce the wall effect, decrease bleeding, reduce the size of CH crystals, and continue densification by long-term pozzolanic reactions. A physico-chemical improvement is the process of pre-treating or pre-activating the aggregates. Part I of this study [1] indicated that silica fume only had a moderate effect on the densification of the ITZ of mortar with RAP, thus the primary focus of Part II considers the physico-chemical improvement by pre-treating RAP aggregates.

#### 1.1.1. Bond improvement by asphalt surface pre-treatment

Asphalt, a heterogeneous material consisting of large organic hydrocarbon molecules, inherently contains carbonyl (C=O) bonds, typically in carboxylic, phenolic, and ketonic functional groups [8]. Through oxidation (aging), additional carbonyl and sulfoxide (S=O) groups form as ketone, aldehyde, carboxylic acid, acid anhydride, sulfoxide, and sulfone functional groups [9,10], with carboxylic acid groups predominately forming under severe oxidation [11]. There is research suggesting calcium-based compounds, such as CH, can interact (associate) and/or bond with these carbonyl groups [11–14]. In addition, Plancher et al. [15] found that various functional groups (ketones, carboxylic acids, dicarboxylic anhydrides, 2-quinolone, and sulfoxides) were prevalently adsorbed on the surface of mineral aggregates and the carboxylic acids were the most prevalently adsorbed functional group. Based on these findings, it is hypothesized that an oxidized asphalt, particularly with more carboxylic acid groups, will chemically bond better with inorganic cementitious materials.

A number of chemical and other means have been found to rapidly oxidize asphalt. Wang et al. [16] showed evidence that sulfuric acid, nitric acid, and hydrofluoric acid may oxidize asphalt. Nitric acid in particular is a strong oxidizer, often used in industrial processes to form carboxyl groups [17], and has been shown to have an oxidizing and a nitrating effect on asphalt [18]. Phosphoric acid has also been shown to be an oxidizer of asphalt [19,20], as it is commonly applied to rapidly age roofing bitumens. Peroxide compounds, such as sodium peroxide and hydrogen peroxide, have been shown to oxidize asphaltenes [21]. Chromium-based compounds and potassium permanganate are other strong oxidizers used in industrial processes to form carboxyl groups [17] and have been shown to oxidize asphalt and asphaltenes [21–23]. Maleic anhydride (MAH) can affect the polarity of an asphalt by reacting with the double bonds in the asphalt in order to add a polar anhydride group [23], and Boucher et al. [24] found that asphalt treated with MAH also resulted in the presence of anhydride carbonyl groups. Finally, ultraviolet (UV) radiation has been known to oxidize asphalt by increasing the amount of carbonyl and sulfoxide groups in the asphalt [25–27].

Analogous to the objectives of this study has been the study of bond improvement in cementitious materials with crumb rubber, which is another organic heterogeneous material. Sulfuric acid and nitric acid treatments have been found to improve bonding with crumb rubber [28]. Sodium hydroxide has been shown by some researchers to improve bonding with rubber particles [29–31], but other studies have indicated little to no improvement [32,33]. Silane has also been used as a “coupling agent” to improve bonding with rubber particles [34–36]. In bond improvement with mineral aggregates, silane [37] and sodium silicate [38,39] have demonstrated some benefit.

#### 1.1.2. Bond improvement by chemical and mineral additives in concrete

Another solution to the bond improvement problem is to add a mineral or chemical additive to the concrete in order to alter the ITZ and/or improve bonding. Lagerblad and Kjellsen [40] suggested that any pozzolanic material with particle sizes finer than the cement can improve the ITZ properties through better packing density and subsequent pozzolanic reaction. Silica fume has been found to improve the ITZ-aggregate bond, while ground granulated blast furnace slag (GGBFS) has been found to have no effect and fly ash can decrease the width of the ITZ, depending on the fly ash properties [41]. Therefore, it is possible that the ITZ-aggregate bond may be enhanced through densification by adding silica fume. However, it is uncertain whether or not silica fume enhances the bond by actual chemical improvement or by simply densifying the ITZ [42]. Part I of this study [1] already found silica fume to only have a marginal effect on the ITZ of mortar with RAP, and the literature has already demonstrated that silica fume does not greatly improve the mechanical properties of concrete with RAP [5].

Polymeric additives have been found to improve bonding and/or improve the ITZ properties. Polyvinyl alcohol (PVA) has been found to significantly alter the ITZ microstructure, resulting in a denser ITZ with more C–S–H and less CH at the interface, possibly the result of the PVA having a superplasticizing effect on the cement [43–45]. The use of latex additives have also been shown to improve bond strength [46–49].

## 2. Experimental methodology

Based on the literature review, a number of asphalt pre-treatments and concrete additives were tested for potential bond improvement, as summarized in Table 1. The bond improvement treatments or additives in Table 1 were first evaluated through strength testing of mortar samples. If treatment improved the strength, additional testing was then conducted for functional group changes in the asphalt along with surface free energy of the component materials. An interfacial bond test was used to evaluate the strength the asphalt-cement bond on a subset of promising treatments. Finally, compositional backscattered electron microscopy studies were conducted on the mortar samples to determine ITZ phase contents and morphology.

### 2.1. Mechanical testing of mortar samples

A cement mortar mixture with 100% RAP was used to more rapidly evaluate the various bond improvement options. The mortar was created with an aggregate-to-cement ratio of 2.75 (by weight) and a water-to-cement ratio of 0.4 (by weight) using Type I portland cement (Table 2). To avoid gradations effects, all RAP particles were the same “monoparticle” size, passing the #4

**Table 1**  
Potential bond improvement asphalt pre-treatments or concrete additives.

Bond Improvement	Treatment or additive <sup>a</sup>
Asphalt oxidizer or pre-treatment	Nitric acid (HNO <sub>3</sub> ), sulfuric acid (H <sub>2</sub> SO <sub>4</sub> ), hydrochloric acid (HCl), hydrofluoric acid (HF), phosphoric acid (H <sub>3</sub> PO <sub>4</sub> ), chromium trioxide (CrO <sub>3</sub> ), hydrogen peroxide (H <sub>2</sub> O <sub>2</sub> ), potassium permanganate (KMnO <sub>4</sub> ), maleic anhydride (C <sub>2</sub> H <sub>2</sub> (CO) <sub>2</sub> O), ultraviolet (UV) light
Concrete additive	Silica fume, fly ash, GGBFS, PVA, latex, silane <sup>b</sup>

<sup>a</sup> All chemicals used in the study were reagent grade. The UV light source intensity was rated at 21.7 mW/cm<sup>2</sup> at a distance of 50 mm.

<sup>b</sup> The silane mixtures did not set, so the results are not included.

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