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Thermal performance, freeze-and-thaw resistance, and bond strength of cement mortar using rice husk-derived graphene

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HIGHLIGHTS

- Thermal performance of GRH was fair among other carbon-based materials.
- GRH mortar showed a better performance against freezing-and-thawing attack.
- Bond strength of thin GRH overlay exhibited better on 0.75 wt% specimen.

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ABSTRACT

We explored the thermal performance, bond strength, and freeze-and-thaw resistance of mortar composite incorporating rice husk-derived graphene-like material (GRH). These three features are key for road pavement application, especially against ice and snow on its surface. Groups of GRH mortar prisms (20 × 20 × 50 mm, 50 × 50 × 100 mm) were tested to examine heating and cooling performance, in comparison with other carbon-based materials: multi-walled CNT, graphene platelets, and carbon fibers. Additionally, thermal performance was assessed in terms of GRH wt% insertion. GRH cubes of 50 mm with different wt% were tested to capture freeze-and-thaw resistance for 300 cycles. XRD, Rietveld, and SEM-EDS analyses of calcium silicate hydrate (CSH) gels of undamaged/damaged cubes with/without GRH were compared at the age of 120 days after casting. A group of Φ 100 × 200 mm slanted concrete cylinders was tested with a 10-mm-thick overlay with different wt% GRH. The compressive failure load and fractured area were used to evaluate the bond strength of the GRH thin overlay.

1. Introduction

Traditional snow and ice control techniques focus mainly on chemical reactions between deicer (chloride) and water in many countries. Alternatively, a direct heat transfer method for rapid deicing using electricity and a conductive medium, such as copper wire, has been applied in some countries. Automatic spraying of chloride deicer has been used widely to prevent road freezing in an efficient and economical way. However, the dosage amount of deicer varies from year to year because, recently, various effects due to climate change have emerged. Additionally, deicer may induce a decrease in bond strength and cause surface scaling, as well as environmental concerns about the washed-out chloride. For these reasons, direct heat transfer methods have been considered, by introducing copper wire with electrical charges on the top of concrete pavements. Because the target temperature of the thin overlay in the melting process is 0 °C, thermally conductive materials can be mixed in a thin cementitious overlay onto the surface of a concrete pavement to enhance the thermal efficiency. However, this method has some drawbacks: (a) energy loss when electrical potential energy turns into thermal energy, (b) re-installation is needed when the thin overlay cracks and is damaged under mechanical loads and the harsh environmental load, and (c) normally, 30 min of pre-heating is required for a 5 °C temperature elevation [8]. To overcome these disadvantages, some researchers have mixed graphite powder [22] into concrete mix to enhance heat conduction. However, a large amount of this is required and it leads to a lower compressive strength than the normal mix. The reason of such low strength may be attributed to the graphite crystal structure itself: hexagonal and planar structure. The atoms are weakly-bonded to this graphite sheet above and below and all lie in a plane. Naturally, graphite is very lubricious because of this [26]. Some research has evaluated adding microsilica and meta-

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kaolin into concrete mixes, but this introduces a more complex production environment [5,24]. Previous research also examined the thermal properties of cement-mortar composites using multi-walled and single-walled carbon nanotubes (CNTs) [11,12,15,21] and graphene oxide and graphene nanoplatelets [9,10], in comparison with an ordinary mortar composite. Fundamental studies on the carbon material itself were reported to determine the multi-scale effects on strength and electrical and thermal conductivity [4,9]. Muramatsu et al. [14] proposed a new method for synthesizing graphene from rice husk. Low-cost graphene materials could be readily and cheaply synthesized on an industrial scale due to its abundance. Rice husk ash has already received much attention as an inclusion material for generating high-pozzolanic action inside cementitious materials. Previous studies on mechanical properties (compressive strength, electrical conductivity enhancement of cement mortar composite mixed with CNT, graphene platelets, graphene oxide [7,13,17–19], and rice husk-derived graphene [16,20]) showed ample enhancement, with the aid of fumed silica and a superplasticizer, compared with ordinary cement mortar. In this study, we sought to explore the enhancement of the thermal properties of cementitious mortar using rice husk-derived graphene (GRH) and then performed feasibility tests of GRH mortar in terms of bond strength between an existing concrete base and a thin GRH overlay and freeze-and-thaw resistance under cyclic environmental fatigue load. These three properties, conductive medium, bond strength, and freeze-and-thaw resistance of thin overlay, are the key to performance in actual application to a road pavement surface.

2. Rice husk-derived graphene materials

The graphenes used in this study was prepared via a chemical activation of a typical agricultural waste (e.g., rice husk) with the help of potassium hydroxide, as we demonstrated earlier [14,16,20]. A typical synthetic procedure is described as follows. Rice husk (10 g) was thermally treated at a temperature of 600 °C to obtain rice husk ash (RHA). Note that the yield of RHA from rice husk is ca. 30%. Then, RHA (2.5 g) was mixed with KOH (10 g) using a mortar and placed the mixture in an alumina pot, which was fully covered with ceramic fiber wool. To generate characteristics of graphenes from RHA, the alumina pot was encased in a silicon carbide melting pot covered with carbon powder and ceramic wool. The fully covered carbon powder was used to protect oxidation against air at high temperature. Then, the crucible was thermally treated at 850 °C for 2 h in an atmospheric condition. The resulting powder (ca. 1 g) was washed with distilled water for several hrs and then dried for 24 h under vacuum. Based on SEM observation, the sample we synthesized exhibited particle-like morphology with large sized holes (Fig. 1(a, b)), which is very similar to that of the conventional activated carbons. To identify the basic difference of our sample compared to the commonly known activated carbons, we carried out TEM and Raman studies. It is interesting to note that our sample consists of the large sized but few layered graphenes with corrugated paper-like texture (Fig. 1(c)). In addition, an intensified G' band around 2850 cm^{-1} also supports the two or three layered stacking nature of our graphene sample (Fig. 1(d)). Thus, our sample is relatively crystalline, but few layered graphene with a high specific surface area (ca.

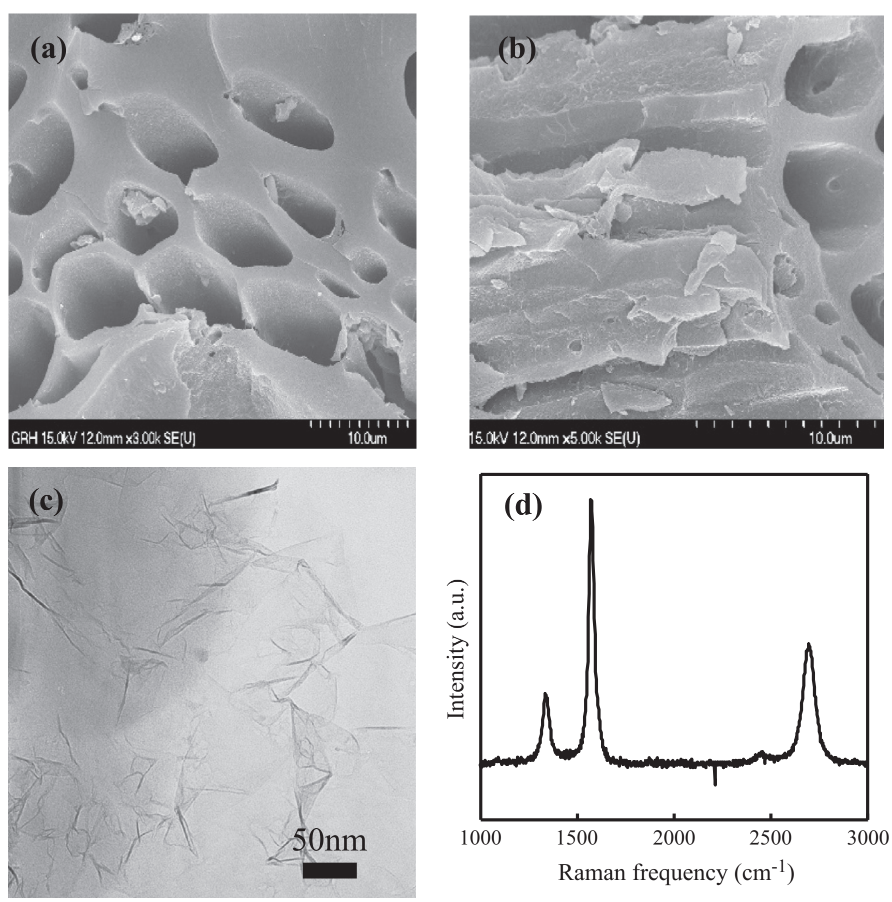

Fig. 1. (a), (b) Scanning electron microscopic (SEM) and (c) transmission electron microscopic (TEM) images of rice husk-derived graphenes and (d) its Raman spectrum 50 nm taken with a 532 nm laser line.