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Converting hollow fly ash into admixture carrier for concrete

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HIGHLIGHTS

- Perforated cenospheres were produced by chemical etching.
- Chemical etching removes the amorphous silicon-aluminum matrix on cenospheres.
- Large inner volume of cenospheres can be used to carry and release admixtures.
- Perforated cenospheres can survive the mixing of concrete.

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ABSTRACT

This study proposes a low-cost method to convert cenospheres into an admixture carrier for concrete manufacturing. Cenospheres are hollow fly ash particles generated in coal burning power plants, having an aluminosilicate shell with high strength and stiffness. The large inner volume of the cenospheres can be used to carry and release admixtures in concrete. However, directly using cenospheres as the carrier is not possible because the inner pore is not accessible to the admixture. To address this problem, chemical etching is employed to produce perforating holes through the shell. Liquid admixtures can be easily loaded into and later released from the produced perforated cenospheres (PCs). A series of characterization tests were carried out to understand the working mechanism of the chemical etching method and its effect on the properties of the PCs. It has been found that chemical etching dissolved a small amount of amorphous materials from the cenosphere shell. As a result, the cenoshpere shell was weakened, as indicated by the reduction of the bulk crushing strength of the PCs. Nevertheless, the bulk crushing strength of all produced PCs are sufficient to guarantee that they can survive the mixing and initial stress in fresh concrete. To experimentally confirm the feasibility of using the PCs as an admixture carrier for concrete, PCs were added into cement mortar as the internal water carrier, which successfully mitigated the autogeneous shrinkage in a low water-to-cement ratio concrete. Scanning electron microscopy analysis of the cement mortar confirms that PCs not only survived the mixing of concrete, but also were dispersed and bonded well to the cement mortar. This study suggests that PCs may provide a versatile tool to integrate various admixtures in concrete.

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1. Introduction

Although well designed concrete mixture (i.e. right proportions and sound materials) along with proper construction practices experience little or none deleterious effects, admixtures are essential ingredient of concrete for many reasons. They can greatly improve the performance of concrete when sound materials are too expensive or unavailable, or proportions is not optimal. More importantly, many high performance or multifunctional concretes [1–4] cannot be produced without proper admixtures. For example, most high performance concrete with low water to binder ratio, water reducer is a must. If we want to add new function to concretes, such as thermal energy storage, or self-healing, new admixtures such as phase change materials (PCMs) or self-healing agents (such as epoxy) will be necessary.

In current practice, admixtures are added into concrete at the time of mixing. However, undesired interaction between the admixtures and the hydration of cement may exist which limits the effects of the admixtures, and sometimes, prevents applications of the admixtures in concrete. For example, water reducers, the most commonly used chemical admixtures, can have undesirable interactions with the hydration reaction of cement. These can

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result in many undesirable side effects in concrete such as rapid loss of workability, excessive quickening/retardation of setting, reduced rates of strength gain, and changes in long-term behavior [5].

To minimize the undesirable interaction between the admixture and the concrete, we can isolate the admixture from the concrete through encapsulating it within a proper carrier. Two most commonly used admixture carriers for concrete are microcapsules and porous particles. Microcapsules are usually produced in-situ through physical or chemical methods, such as spray drying, coacervation, and polymerization methods [6]. The admixture can be encapsulated into the microcapsules and isolated from the surrounding concrete. For example, Choi et al. [7] synthesized a microcapsule with degradable shell using soluble acrylic resin-based compound, which can dissolve in concrete with time, releasing the admixtures (accelerator and retarder) to enhance the durability of the concrete. Microencapsulated phase change materials (PCMs) have also been extensively used in concrete to add thermal energy storage capacity to concrete [8]. However, existing microencapsulation method has yet seen successful applications in concretes because of high manufacturing cost of the microcapsules.

When porous particles such as light weight aggregates (LWAs) are used as the admixture carrier, the admixture is firstly directly impregnated into the pores of the porous particles through physical absorption, and then released into the concrete as needed. A typical example of this method is the internal curing technique developed for high-performance concrete (HPC), in which saturated LWAs are used as water carriers [9–11]. Since the admixture is only absorbed by the porous inclusions and no protective layer on the surface of the inclusions is provided, this method has little control on the release of the admixture and cannot be used to seal the admixture in concrete. In addition, porous inclusions used in this method either have low strength and stiffness (LWAs) or are soft materials (super absorbent polymers). The presence of these materials in the concrete can reduce the strength and stiffness of the concrete [12].

To overcome the drawbacks in existing admixture carriers, this study develops a low-cost but robust carrier, perforated cenospheres (PCs) for admixtures so that they can be added into concrete to achieve better performance and/or new functions. Cenospheres are hollow fly ash microspheres (Fig. 1(a)) collected from fly ash waste produced during the combustion of coal in thermal power plants. They enjoy many outstanding features, such as low density [13], strong filling ability, chemical inertness and thermal resistance [14]. The size of cenospheres ranges from a few to thousands of micrometers. As shown in Fig. 1, a typical cenosphere consists of a large inner pore and a porous aluminosilicate (Fig. 1(b)) shell. The large inner pore provides large capacity to carry admixtures. The aluminosilicate shell has high stiffness and strength, and a thickness in a few micrometers which can provide strong protection for admixtures stored in the inner pore.

However, it is not possible to directly use cenopsheres as the admixture carrier because the inner pore is not accessible to admixtures. Therefore, a method is needed to introduce admixtures into the cenospheres. As shown in Fig. 1(b), the shell of the cenosphere has a porous structure formed by gas inclusion and is covered by a glass-crystalline nanosize film. By removing this thin film, perforating holes can be produced on the shell, through which admixtures can be loaded into and released from the inner pore. This can be done by chemical etching, which can dissolve the amorphous nanosize film on the surface of the cenospheres to introduce perforating holes on the shell. The produced perforated cenospheres (PCs) are ideal candidate as the admixture carrier for concrete manufacturing.

In this study, the chemical etching process is first introduced in details, through which, PCs were successfully manufactured. A series of characterization tests were then carried out to understand the working mechanism of the chemical etching method and its effect on the properties of the produced PCs. These PCs were also added into cement mortar to demonstrate that they can be used as internal curing water carrier. Internal curing is a technology to mitigate the early age cracking induced by autogeneous shrinkage, which can cause significantly reduced strength and lifetime of concrete structures. This early age cracking due to autogeneous shrinkage is especially serious for HPC because of its low water to cement ratio. These cracking problems cannot be mitigated through conventional full water curing because of HPC's compact pore structure and very low permeability. To combat this problem, the internal curing method has been developed. In this method, curing water is continuously supplied inside concrete by a water carrier to replenish the empty pore volume that is created by self-desiccation. This will reduce autogenous shrinkage and also improve the curing of concrete at the early age.

2. Materials and methods

2.1. Materials

Reagent-grade hydrochloride (HCl) was purchased from ZhongShi Chemicals, China. Ammonium fluoride (NH_4F) and sodium bicarbonate ($NaHCO_3$) were purchased from Bodi Chemicals, China. All reagents in this paper were used as received without further purification. Cenospheres were purchased from five different sources in China, and were coded as C1 to C5. XRF-1800 sequential X-ray fluorescence spectroscopy (XRF) was used to determine the chemical compositions of these cenospheres. Particle diameters of cenospheres were analyzed by ASALD-7101 laser particle size analyzer (LPSA).

2.2. Manufacturing of PCs

As shown in Fig. 1(b), perforating holes can be produced on the shell by removing a thin film of amorphous materials from the cenosphere surface. Admixtures can be loaded into and released from the inner pore through these perforating

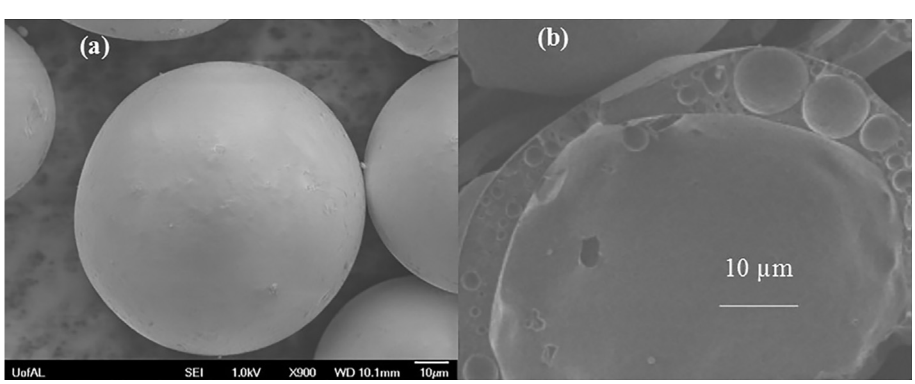

Fig. 1. Microstructure of cenospheres under SEM observation: (a) a cenoshpere with impermeable shell; (b) porous shell of the cenosphere.

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