



Lateral force microscopic examination of interfacial transition zone in ceramic concrete



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HIGHLIGHTS

- Effect of fine bone china ceramic aggregate on ITZ of concrete was studied.
- Lateral force microscopic technique was used to characterize ITZ.
- The ITZ of ceramic concrete was found to be superior to conventional concrete.

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ABSTRACT

The effects of fine bone china ceramic aggregate on the nanostructure properties of concrete were studied through Lateral Force Microscopy (LFM). The LFM tapping mode technique was used on concrete samples to observe the different phases of concrete such as CSH and pores. Roughness of the interfacial transition zone (ITZ) between aggregate and cement paste was also determined. The results of the LFM imaging show that incorporation of fine bone china ceramic aggregate resulted in denser CSH gel structure along with reduced roughness of the ITZ. The resulting concrete is therefore expected to have superior strength and durability properties.

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1. Introduction

The concrete industry can be a major contributor in the field of waste recycling and is a driving force towards the effective utilization of resources. A significant number of studies on use of different types of industrial waste in concrete have been done in the past two decades [1–6]. Ceramic waste is gaining ground as sustainable alternative for natural aggregates in concrete production. Limited studies have been carried out to investigate the behaviour of ceramic aggregates from various sources and their influence on the properties of concrete. The ceramic industry waste can broadly be divided into two parts: white ceramics and red ceramics. The raw materials used in the productions of ceramics provide its finish colour and properties [7].

Some specific studies of white ceramic waste as fine aggregate are discussed. Senthamarai and Manoharan [8] investigated the effect of ceramic electrical insulator based coarse recycled aggregates in concrete and reported a slight decrease in the compressive

strength of the resulting concrete mixes. Guerra et al. [9] reported that compared with the control concrete sample, concrete samples containing 5%, 7%, and 9% coarse recycled ceramic aggregate showed enhanced compressive strength at all curing ages. Torikitkul and Chaipanich [10] reported that the use of earthenware fine recycled ceramic aggregate results in higher compressive strength for Portland concrete and fly ash concrete mixtures. Medina et al. [11] found that utilisation of recycled coarse sanitary ware ceramic aggregate in concrete mixes resulted in increase of compressive strength due to pozzolanic behaviour of ceramic aggregates. Corominas and Etxeberria [12] reported higher compressive strength in concrete samples incorporating fine recycled ceramic aggregates. Zegardło et al. [13] concluded that ultra-high performance concrete containing recycled sanitary ware ceramic aggregate results in higher compressive and tensile strength. Siddique et al. [14] reported an increase in the 28 day compressive strength in concrete samples incorporating fine ceramic aggregate. Table 1 provides a brief summary of red ceramic waste used as fine aggregate in various studies highlighting the source of ceramic waste, replacement percentage and its influence on compressive strength.

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Table 1
Red ceramic waste used in different studies.

Authors	Source of red ceramic waste	Replacement	Influence on the compressive strength of concrete
Torgal and Jalali [15]	Ceramic brick	100%	Slight increase at 28 days curing period
Alves et al. [16]	Ceramic brick	20%, 50% and 100%	Slight decrease at all curing ages and replacement levels
Ettxeberria and Vegas [17]	Construction and demolition waste containing 70% ceramic brick	10%, 20%, 35% and 50%	Increase in compressive strength at all curing ages and replacement levels
Vieira et al. [18]	Ceramic brick	20%, 50% and 100%	Slight decrease at all curing ages and replacement levels

Study of available literature shows that there is a limited understanding of the complex nanostructure behaviour of ceramic concrete. However, knowledge about the micro/nano structure of resultant product is extremely important to overcome the shortcoming or to establish the desired properties responsible for the superior performance of such non-traditional concrete.

Calcium silicate hydrate (CSH) gel and calcium hydroxide are the two dominant products which are formed after the hydration process of cement concrete. The interfacial transition zone between cement paste and aggregate influences the properties of the hardened concrete at micro and Nano scale. Few studies have used scanning electron microscope (SEM) to report the microstructure properties of ceramic concrete [10,11,13,19,20]. There is a growing interest in exploring the Nanoscale properties of concrete and cement based materials. The most popular method that has been utilised is the Nano-indentation technique to study the toughness, modulus of elasticity and the fracture properties of alternative concrete [2,21–24].

The large scale tip of nanoindenters are not apt to measure the CSH formations which have smaller dimension in comparison to the tip. The AFM/LFM analysis incorporates tips of size 10 nm or below which can be suitable replacement for nanoindenters [25]. Few studies are available on the utilisation of atomic force microscopy (AFM) and lateral force microscopy (LFM) to describe the nanostructure characteristics of mortar [26–29]. Peled et al. [21] compared both AFM and LFM techniques to study the nanostructure properties of cement mortar. They concluded that LFM is a more sensitive technique to provide the nano-features of cement mortar surface. In lateral force microscopy (LFM) the sensor tip is positioned to remain in contact with the scan area and the tapping of the tip collects the lateral deflection signal. The LFM method measures the signal strength of the lateral deflection between the tip and scan area and is more sensitive to topographical variations and phase changes than AFM. Trtik et al. [22] quantified modulus of cement paste and concluded that AFM can be used to identify and quantify different phases present in cement paste.

2. Objectives and scope

The present study was designed to understand the influence of fine bone china ceramic aggregate on the nanostructure properties of concrete. The tapping mode LFM imaging technique was employed to examine the topographic and phase differences at the interfacial transition zone (ITZ) of cement paste and aggregate during the investigation.

3. Experimental details

3.1. Materials and sample preparation

Fig. 1 shows the fine bone china ceramic aggregate prepared by crushing tableware ceramic waste procured from local industry. The water absorption and specific gravity of the aggregate were 2.5% and 2.4 respectively. Ordinary Portland cement of 43 grade and Auramix 400 a polycarboxylic ether polymer based high range water reducer as super-plasticiser were used in the study. Table 2



Fig. 1. Fine bone china aggregate.

shows the mix proportions of the concrete samples prepared with a w/c of 0.35. The nomenclature followed for mix was CX where X stands for the percentage replacement of natural sand by fine bone china aggregate. Natural sand was replaced (by volume) by fine bone china ceramic aggregate up to 100% with 20% increment. Due to higher water absorption by fine bone china ceramic aggregate, excess water was added to maintain the w/c. After casting, the samples were demoulded and cured for 28 days. Table 2 also presents the compressive strength results of all the concrete mixes at 28 days. The chemical composition of fine bone china aggregate was obtained by XRF spectroscopy. Table 3 presents the chemical composition of fine bone china aggregate used in the present study along with the details of red and white ceramic waste aggregates used in various other studies. It was found, that fine bone china aggregate mainly contained SiO₂ (28.86%), Al₂O₃ (23.86%), CaO (24.15%), P₂O₅ (10.99%), Fe₂O₃ (5.41%), MgO (2.86%) and K₂O (1.58%) by weight.

The concrete specimens were cut with linear precision saw to achieve samples of 10 mm × 10 mm × 10 mm size. The samples were then cleaned with acetone and kept at 100 °C in an oven for 48 h. The polished smooth sample of size 10 mm × 10 mm × 2 mm were obtained by wet grinding on Buhler disks with paper grits of 120, 320, 800, 1000 and 1500 respectively. The samples were grinded for 10 min on 120 grit paper to reduce the depth and for 5 min on each grit paper to achieve smooth and plane surface. For polishing the surface of grinded samples, Buhler auto-polisher was used with velvet paper, diamond suspension liquid of 1 μm gradation and consistency paste. The rotation speed was kept in between 200 and 250 RPM (Revolutions per minute) and each sample was polished for 25 min. Epoxy impregnation was not done to maintain the undisturbed topographic characteristics of ITZ. The polished samples were then cleaned with acetone and were kept in oven for 72 h at 100 °C temperature.

3.2. LFM imaging technique

The atomic force microscope used to obtain LFM images was Multimode 8 HR AFM system (Bruker) with silicon tip attached

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