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Technical note Lateral force microscopic examination of calcium silicate hydrate in

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ABSTRACT

Lateral force microscopy was employed to study the influence of rubber ash on the microstructure of concrete. Tapping mode LFM images of microstructure were obtained to observe the calcium silicate hydrate gel in concrete mixes. The roughness parameters and roughness distribution of the scanned area were also determined. The results show that incorporation of hydrophobic rubber ash particles contribute to slight internal curing effect upto 10% substitution. However, at higher substitution (15–20%) the water to cement ratio of concrete gets disturbed and results in gel pores within the concrete matrix. The incorporation of rubber ash weakens the interfacial bonding between different hydration products which results in increased roughness.

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1. Introduction

For environmental and economic reasons the concrete industry is promoting the use of alternative raw materials obtained from different waste and by products of various industries [1–6]. A significant number of studies on using various forms of rubber waste (fibre, crumb and ash) in concrete have been carried out in the past two decades [7–15]. However, the rubber ash form of waste as aggregate is subjected to very few studies some of which are discussed here.

Al-Akhras and Smadi [7] studied the influence of rubber ash as fine aggregate on mechanical properties of mortar. They reported increase in compressive strength on replacing 10% of natural sand with rubber ash. They reported that filler action of rubber ash produced a more dense and homogenous interfacial transition zone. Taha et al. [16] observed the microstructure of concrete containing rubber particles. They reported that on pull out of rubber particles indentation marks are observed in cement paste which shows proper bonding between cement paste and rubber aggregate. Gupta et al. [12] reported that rubber ash concrete exhibited poor mechanical and durability properties at different water to cement ratios. They concluded that the light and smooth structure of raw rubber ash aggregate creates improper bonding with the cement paste causing decrease in the properties of concrete. Raffoul et al.

* Corresponding author. *E-mail address:* sandeep.nitjaipur@gmail.com (S. Chaudhary). use of crumb rubber aggregates results in poor bonding between aggregate and cement paste. They also observed micro cracks along the interfacial transition zone (ITZ) and concluded that hydrophobicity of rubber aggregates creates higher water content in the concrete mixes causing weaker microstructure. Wang et al. [18] studied the effect of treated and untreated crumb rubber fine aggregate on microstructure of high performance cementitious composites. They observed that the loose and smooth structure of untreated crumb rubber aggregate resulted in poor interfacial bonding and force transfer characteristics. The treatment of crumb rubber aggregate by 10% sodium hydroxide solution resulted in proper bonding between rubber aggregate and cement paste. Bing and Ning [19] observed the microstructure of crumb rubber fine aggregate concrete and reported presence of voids, interparticle spacing and gaps at interfacial transition zone of cement paste and crumb rubber aggregate. Gupta et al. [20] studied the effect of rubber fibre on the microstructural properties of concrete. They observed poor interfacial bonding between rubber fibre and cement paste. The smooth and loose structure of rubber fibre was reported to be the reason behind poor microstructural bonding. In previous two decades few authors have utilised atomic force

[17] investigated the effects of both fine and coarse crumb rubber aggregate on the microstructure of concrete. They reported that

In previous two decades few authors have utilised atomic force microscopic (AFM) investigations to better quantify the micro/nano structure of concrete. The AFM studies provide a qualitative and quantitative benefit over optical and scanning electron microscopy







as many modes of AFM provide values such as roughness profile, average roughness, and height deviations among many other parameters. Such advantages of AFM over other methods make it a useful technique to investigate a heterogeneous material such as concrete.

Papadakis et al. [21] investigated the influence of silica fume and fly ash on cement paste microstructure. They observed relatively smooth surface of the hydration products with pore and grain refinement. Peled et al., Peled and Weiss [22,23] investigated AFM and lateral force microscopic (LFM) images of cement paste and concluded that LFM mode of investigation provides a much clearer and precise observations than AFM. Trtik et al. [24] concluded that the different phases of cement paste can be quantified by AFM along with the modulus of cement paste. Xiao et al. [25] observed the roughness parameters of interfacial transition zone (ITZ) of recycled aggregate concrete. They concluded that higher roughness values of ITZ laver might be a probable cause for poor strength properties of concrete. Li et al. [26,27] compared nanoindentation, peak force mapping and modulus mapping modes of AFM and reported that peak force and modulus mapping provide a much suitable overview of the nano-mechanical properties of cement paste. Karthik et al. [28] reported that higher roughness values indicate better contact area between geopolymer paste and aggregate. They concluded that higher roughness values indicate improved ITZ characteristics of concrete. Siddique et al. [29] investigated the nano structure of interfacial transition zone in ceramic concrete. They observed that use of ceramic waste as fine aggregate leads to better ITZ characteristics of ceramic concrete than control concrete.

1.1. Research significance

The study on effect of rubber ash on the structure of calcium silicate hydrate (CSH) gel is of importance to overcome any shortcoming by suitable remedy methods. Since the CSH gel in concrete is formed at both nano and micro structure point. The gel pores in the CSH gel can be better quantified by the lateral force microscopy than optical or scanning electron microscopy.

1.2. Objectives and scope

The present study was designed to observe the nanostructure of CSH gel in rubber ash concrete mixes. Lateral force microscopy in tapping mode was employed to observe the formation of CSH gel pores, roughness of CSH gel and roughness distribution across the scan range of concrete.

2. Experimental programme

2.1. Materials, mix composition and sample preparation

Ordinary Portland cement of 43 grade was used as binder in this study. River basin sand and crushed stone were used as fine and coarse aggregates. Rubber ash was obtained from pyrolysis based incineration plant (Fig. 1). The particles of rubber ash varied from 0.15 mm to 1.9 mm. The EDX (energy dispersive X-ray spectroscopy) observations of rubber ash was done to identify the chemical elements. The rubber ash consisted of C (87.51%), O (9.23%), Zn (1.76%), Si (0.20%), S (1.08%), Mg (0.14%) and Al (0.08%). High range water superplasticiser was used as admixture to maintain a compaction factor of 0.90–0.95. Table 1 presents the physical properties of aggregates used in this study. The chemical composition of OPC and river basin sand are presented in Table 2.

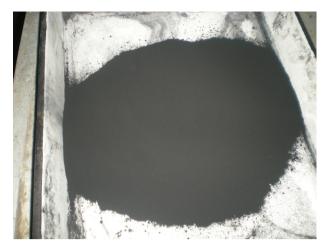


Fig. 1. Rubber ash used in the study.

Table 1

Properties of aggregates used in the concrete mixes.

Property	Natural	Rubber	Natural coarse
	sand	ash	aggregate
Specific gravity	2.56	1.33	2.59
Water absorption (%)	0.5	0.3	0.5

Table 2

Chemical composition of OPC, river basin sand and rubber ash.

Compound	OPC	River basin sand
Al ₂ O ₃	4.65%	9.81%
CaO	62.34%	1.15%
SiO ₂	20.14%	81.67%
MgO	2.23%	0.75%
Fe ₂ O ₃	3.29%	2.11%
K ₂ O	0.72%	2.52%
SO3	2.42%	-
LOI	1.96%	0.2%

Five concrete mixes were proportioned by replacing natural sand (volume replacement) by 0%, 5%, 10%, 15% and 20% rubber ash. Table 3 presents the mix compositions along with the 28 day compressive strength of concrete mixes (average of three 100 mm cubic specimens).

After 28 days of curing the samples were cut into cubes of 10 mm \times 10 mm \times 10 mm. The samples were then wet grinded and smoothened on 120, 320, 600 and 1000 paper grits to obtain a size of 10 mm \times 10 mm \times 2 mm. The samples were then polished on Buhler's auto polisher consisting of velvet paper, diamond suspension liquid of 1 μ m gradation and consistency paste. Each sample was polished for 25 min and was then kept in oven for 24 h at 50 °C temperature.

2.2. LFM imaging

Multimode 8 HR AFM system (Bruker) with silicon tip attached to cantilever arm was used to obtain LFM images of the concrete samples. The topographic images were obtained at a scan range of 10 μ m in tapping mode. The twisting cantilever movement of the probe in LFM mode is beneficial in providing both horizontal and vertical topographical characteristics of the scanned surface. The probe tip was placed in appropriate region with the help of optical microscope attached to the AFM system.

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