Crack path and fracture surface modifications in cement composites

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ABSTRACT. There is a tremendous increase in the use of high strength and high performance self-consolidating cementitious composites due to their superior workability and mechanical strengths. Cement composites are quasi-brittle in nature and possess extremely low tensile strength as compared to their compressive strength. Due to the low tensile strength capacity, cracks develop in cementitious composites due to the drying shrinkage, plastic settlements and/or stress concentrations (due to external restrains and/or applied stresses) etc. These cracks developed at the nanoscale may grow rapidly due to the applied stresses and join together to form micro and macro cracks. The growth of cracks from nanoscale to micro and macro scale is very rapid and may lead to sudden failure of the cement composites. The present paper reports the modifications in the crack growth pattern of the high performance cement composites to achieve enhanced ductility and toughness. The objective was accomplished by the incorporation of the micro sized inert particulates in the cement composite matrix. The results indicate that the incorporation of micro sized inert particles acted as the obstacles in the growth of the cracks thus improving the ductility and the energy absorption capacity of the self-consolidating cementitious composites.

KEYWORDS. Fracture energy; Toughness indices; Inert pyrolyzed particulate; Fracture process zone.

INTRODUCTION

Cement based composites i.e. paste, mortar and concrete are the most utilized materials in the construction industry all over the world and their quantities produced and utilized are rapidly increasing due the fast pace of development in the modern world [1,2]. Besides many advantages, there are few limitations in the utilization of the cementitious composites and their quasi-brittle nature is one of them. The cement composites possess extremely low tensile strength as compared to their compressive strength. Due to their low tensile strength, cracks develop in cementitious materials due to the drying, shrinkage, plastic settlements, stress concentrations (due to the external restrains and/or applied stresses) etc. These cracks developed at the nanoscale may grow rapidly due to the applied stresses and join together to form micro and subsequently macro cracks in the composite. The growth of cracks in the cementitious composites from nanoscale to micro and macro scale is very rapid and may lead to sudden failure. Generally the paths taken by the cracks for their growth in a composite structure determines whether the failure will be brittle and catastrophic or elastic and safe [3]. In the traditional cement composites, there is absence of crack trapping mechanisms such as fibers and hard inclusions at nano/micro scale and stress relieving mechanisms such as dislocations or crazing in metals and polymers. As a result, the stresses required to propagate the cracks are much lesser as compared to the stresses required for their initiation [4]. The large variations in the stresses required for crack initiation and growth in cement composites leads to unstable and rapid growth of cracks after their nucleation [5]. As it is a well-known fact that brittleness of cement composite is directly related to their strength so the issue of brittleness and crack susceptibility is more pronounced in the high strength cement composite, which have been developed to meet the modern world requirements [6]. From the viewpoint of high performance in terms of eco-efficiency, durability and sustainability, it is highly desirable to enhance the ductility and energy absorption capability of the cementitious composites. Literature indicates that researchers have investigated several types of materials/fibers for enhancing the ductility of the cement composites such as hemp, sisal, jute, cellulose whiskers, steel, polyvinyl alcohol (PVA), polypropylene (PP), carbon nanotubes (CNTs), carbon nano fibers (CNFs) and many others [7–17]. Recent studies also show the utilization of nano materials such as graphene, nano crystalline cellulose, calcium carbonate whiskers and nano SiO_2 particles for improving the ductility and strength of cement composites [18–21]. The fibers in the cement matrix behave as crack arresters and restrain their growth. The crack arresting mechanisms of fibers in cement composites impart toughness and enhance their energy absorption capacity. In the crack arresting mechanism of fibers, two crucial parameters limiting their performance are their dispersion in the host matrix and the interfacial bonding between the fiber and the composites. The uniform fibers dispersion in the cement matrix is usually a difficult task and requires special procedures. This factor greatly limits the fibers utilization in the cement composites [22–24].

In the present research, the objective of crack growth pattern modification in the high performance cement composites to achieve enhanced ductility and toughness has been accomplished by the incorporation of the micro sized inert particulates in the cement composite matrix. The inert micro sized inclusions increase the heterogeneity in the matrix as they help to reduce the void in the matrix, increasing the strength, but at difference of silica fume particle do not interact with the cement matrix. This results in complex crack tip stresses around the small particulates [25,26]. The presence of complex stress field at the growing crack tip restricts its usual in plane movement and the result is a crack deflection and sometimes crack contouring around the particulates. The random and multi-plane movement of the crack path in the cement composite matrix results in higher fracture surface area involving more and more material in the cracking process as compared to the cracking in a single plane. For that purpose, a novel inert material was synthesized by the carbonation of coconut shells and was subsequently used in the preparation of cement composites.

MATERIALS AND METHODS

Materials

In this experimental study the cement composites were prepared with Type-I, Ordinary Portland Cement (OPC) [27]. The chemical composition and the physical properties of OPC are reported in Table 1. High range water reducing agent (HRWRA) "Mapei Dynamon SP-1" which is based on the second generation of modified acrylic polymers was used to ensure sufficient workability of the cement composites at the given w/c ratio. Physical properties of the HRWRA are reported in Table 2 [28].

Chemical composition	CaO	Al_2O_3	SO_3	SiO_2	Fe_3O_4	MgO	K_2O
Content (wt.%)	44	26.5	12	9.5	2.5	1.3	0.6

Physical characteristics	Standard	Average values
Color	-	Light grey
Density	-	2,800 kg/m^3
Blain specific surface area	UNI EN 196-6	480 m^2/kg
Initial setting time	UNI EN 196-3	98 min
Final setting time	UNI EN 196-3	125 min

Table 1: Chemical and physical properties of cement [27]

Property	Appearance	Specific gravity	PH value	Solid content	Recommended dosage
Value	Amber	1.090	6.5 - 9.0	30.50%	1.5 wt% of cement

Table 2: Properties of super plasticizer [28]

In the present research, the micro inert carbonized particles were synthesized from coconut shells. The coconut plant belongs to the family of palm trees with heights ranges up to 30 m [29]. Coconut is found in abundance all over the world especially in the tropical regions of Asia, Africa and Latin America. FAO statistics shows that for the year 2012, the coconut plants covering area of 12.13 million hectares produced about 62.42 million ton of coconut fruit [30]. Based on the structure of coconut fruit, it is generally divided in to three parts i.e. the innermost part or the coconut meat also known as copra, thick hard shell surrounding the coconut meat and the outermost layer of coconut coir. The shell surrounding the copra is a waste material and it is generally used for burning and other such low-level applications. For the synthesis of inert micro sized pyrolyzed particles, the coconut shells were dried at 105±5°C for 24 h in oven to remove the absorbed moisture. The dried pieces of the coconut shells were pyrolyzed in an airtight quartz reactor under the constant flow of 99.9% pure argon gas under a pressure of 0.2 bar. The quartz reactor was heated with a constant rate of 1°C/sec up to 850°C and then the temperature was maintained there for 1 h. After the completion of pyrolysis, the pyrolyzed coconut shells were allowed to cool. Some quantity of the pyrolyzed coconut shell was annealed at 850°C for 2 h to obtain more pure carbonized material. The pyrolyzed coconut shell (PC) and the pyrolyzed and annealed coconut shell (PCA) were then grinded to reduce the particle sizes up to micron scale.

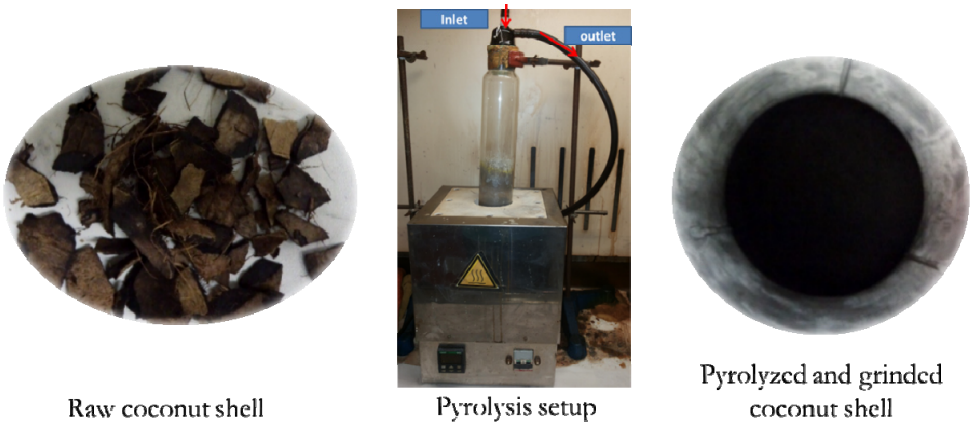

Figure 1: Synthesis of micro carbonized particles from coconut shell by pyrolysis

Preparation of cement composite samples

In total, three different wt% additions (i.e. 0.05, 0.08 and 0.20%) were studied for each type of carbonized particles and compared with the control mix. The details of the samples composition are reported in Table 3.

Notation	Pyrolyzed coconut shell particles	
	Weight (%)	Weight (mg)
CEM	Control mix	
CEM +0.05% PC	0.05	107
CEM +0.08% PC	0.08	171.2
CEM +0.20% PC	0.20	428
Notation	Pyrolyzed and annealed coconut shell particles	
	Weight (%)	Weight (mg)
CEM	Control mix	
CEM +0.05% PCA	0.05	107
CEM +0.08% PCA	0.08	171.2
CEM +0.20% PCA	0.20	428

*For each mix 214 g cement, 75 g water and 3.21 g HRWRA were used

Table 3: Composition of cement composite samples

In the preparation of cement composite samples initially, the pyrolyzed coconut shells particles were dispersed in the measured quantity of water and HRWRA solution by using sonication for 15 minutes. The water solution with thoroughly dispersed pyrolyzed coconut shells particles was then mixed with cement and mixing was carried out at 440 rpm for 2 minutes and then at 630 rpm for 2 more minutes [31]. The self-compacting fresh paste was then transferred into the acrylic molds of 20 x 20 x 75 mm^3. The samples were allowed to dry in an atmosphere with 90% humidity. The prism shaped samples were cured at room temperature for 28 day in water. After the completion of the curing period samples provided with 2 mm thick and 6 mm deep notches for carrying out flexural and subsequently compression tests.

Characterization of materials and composites

The characterization of the materials was carried out in two steps. Initially, the micro carbonized particles were analyzed to check their microstructure and morphology with the help of field emission scanning electron microscope (FESEM), for chemical composition by means of energy dispersive x-ray (EDX) analysis, for particles size analysis by using laser granulometer and for Raman spectroscopy by means of "Renishaw Raman-scope" equipped with laser beam of 514.5 nm wavelength. In the second phase, the cement composite samples were analyzed for their mechanical strength in flexure and compression. For flexural strength of the cement composites, the samples were tested according to the ASTM C348 standard in three point flexural by using "Zwick-Line Z010" testing machine in crack mouth opening displacement (CMOD) controlled mode. In the present study the CMOD rate was fixed at 0.003 mm/min [32]. The compression tests were carried out by using the broken pieces of the prism from the flexural tests according to ASTM C349 standard [33]. The compression test was carried out in displacement-controlled mode with a displacement rate of 0.5 mm/min. The broken pieces of the cement composites were analyzed by the means of FESEM to study the dispersion of the micro carbonized particles in the cement composite matrix and the fracture/crack growth properties of the cement composites incorporating micro carbonized particles.

RESULTS AND DISCUSSIONS

The FESEM analysis results of micro carbonized coconut shells particles at different magnifications are reported in Fig. 2. The FESEM observations revealed that the micro sized coconut shells particles possess smooth and glossy surfaces. The particles showed highly angular edges, which indicates their strength and toughness.

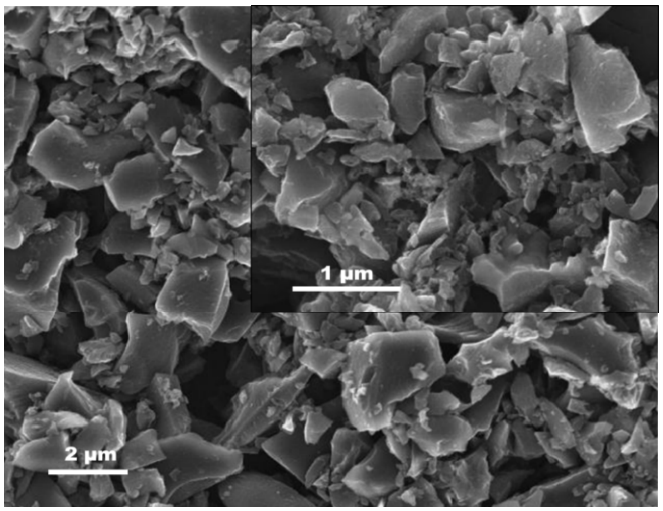

Figure 2: FESEM micrographs of pyrolyzed coconut shells particles.

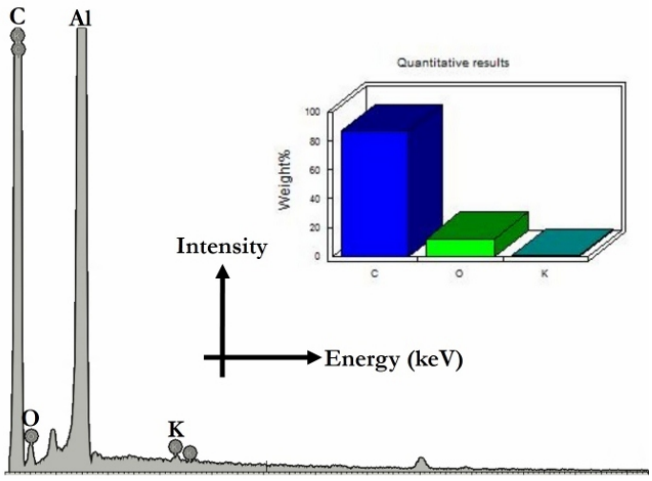

Figure 3: EDX analysis of pyrolyzed and ground coconut shells particles.

The chemical composition of micro inert pyrolyzed coconut shells particles was determined by means of the EDX analysis. The analysis results indicates the very high content of carbon about 87.29 wt.% and low amount of oxygen about 12.26 wt.% with few traces of potassium about 0.45 wt.% are present in the pyrolyzed coconut shell particles (see Fig. 3). The laser particles size analysis revealed that the grinded pyrolyzed particles possess a uniform distribution of the particles sizes ranging from 4.8 µm to sub-micro meter sizes. This observation was also confirmed with the FESEM observation of the pyrolyzed particles. Raman analysis of the pyrolyzed and ground coconut shell particles indicated two distinct peaks at 1346 cm^{-1} and 1595 cm^{-1} which corresponds to the defect peak (D) or disorder peak and the graphitic peak (G), respectively. A small peak was also observed at 3078 cm^{-1} that is due to the higher vibrational mode of the defect peak (2D) (see Fig. 4). The peak intensity ratio (I_D/I_G), which is the measure of the material's quality was evaluated as 0.8471 indicating that very low degree of structural defects are present in the pyrolyzed coconut shell particles [34,35].

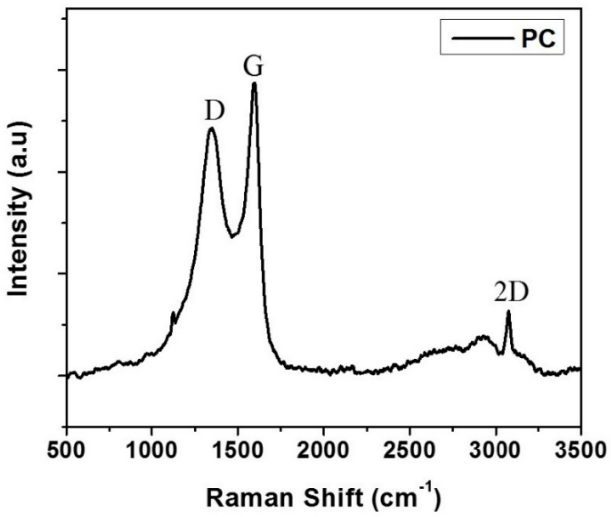

Figure 4: Raman spectroscopy of pyrolyzed coconut shell particles.

Mechanical testing of the cement composites were carried out to investigate the effect of the inclusion of pyrolyzed coconut shells particles in the cement composites. For each cement composite mix minimum of three notched prism

samples were tested in flexure and the broken pieces were tested for compressive strengths. Typical load-CMOD curves of the cement composite samples are presented in Fig. 5.

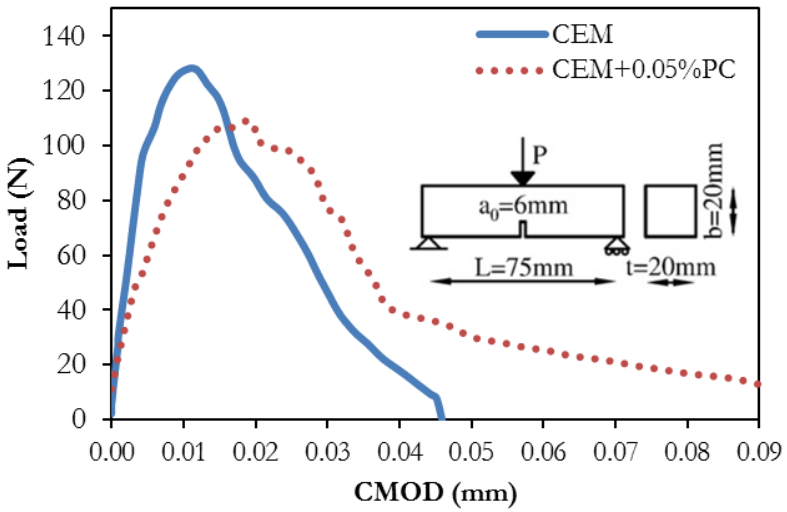

Figure 5: Typical load-CMOD curves for cement composites with and without pyrolyzed coconut shells particles.

The average modulus of rupture of cement composite samples and their compressive strengths are reported in Figs. 6 and 7 respectively. The results indicates that the flexural strength of the cement composites reduces with the inclusion of the pyrolyzed coconut shells particles, even a small amount of pyrolyzed coconut shell particles (i.e. 0.05%) may reduce the modulus of rupture up to 20% for both types of carbonized and carbonized & annealed coconut shell particles. This reduction may be attributed to the large number of inert inclusions in the composite matrix. In case of compressive strength, all the mixes showed increased strength as compared to the control mix but this increment showed no relation with the amount of inert particles inclusion.

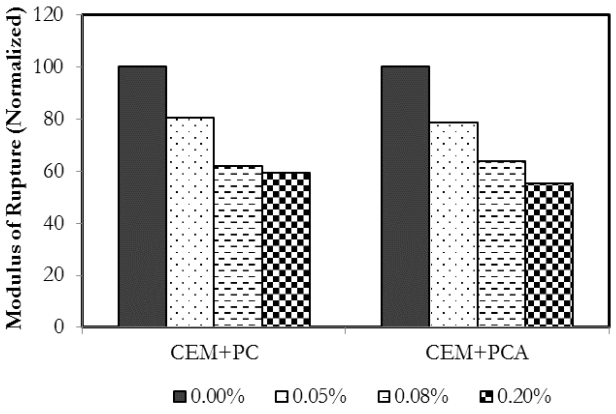

Figure 6: Modulus of rupture of cement composites with PC & PCA particles inclusions.

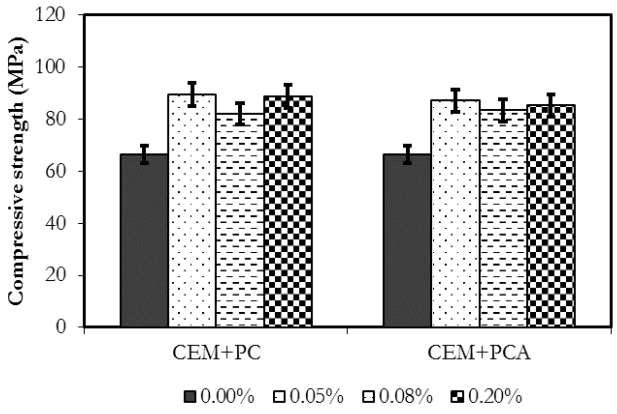

Figure 7: Compressive strength of the cement composites with PC & PCA particles inclusions.

The load-CMOD relations of the cement composite samples showed that the unstable cracks growth initiate around 0.010±0.002 mm CMOD in control mix and around 0.018±0.002 mm for the samples containing various amounts of inert pyrolyzed coconut shells particles. This indicates that the unstable crack growth as well as the final split or the braking of the cement paste composites is delayed by the addition of inert carbonized particles. The enhancement in the maximum CMOD varies from sample to sample, with an average trend of 80% to 100% enhancement. The result of this

enhancement in maximum CMOD is that the ductility and the fracture energy of the composites has improved substantially as depicted in the Fig. 8.

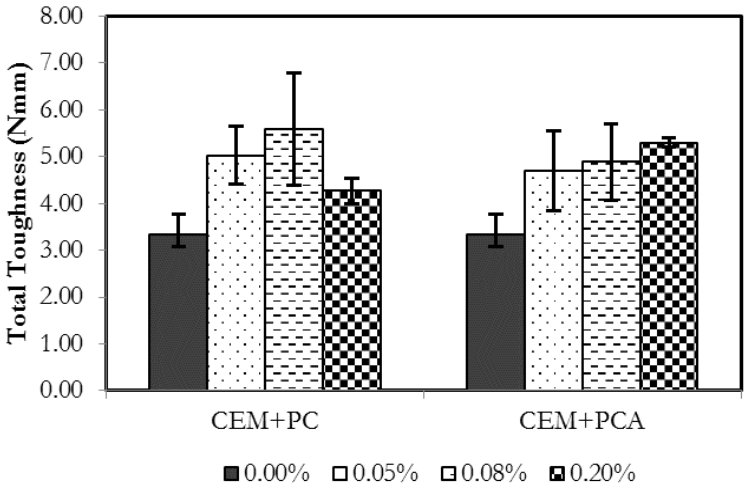

Figure 8: Toughness of the cement composites samples with various amounts of PC & PCA particles inclusions.

Typical fracture surfaces of the notched cement composite samples tested in flexure are presented in Fig. 9. The fracture surfaces of the control mix samples are smooth and sharp as compared to the cement composites containing various amounts of inert carbonized particles. The FESEM observations of the fracture surfaces revealed that the inert micro particles acted as an obstacle in the crack growth path, therefore the growing cracks have to deflect and contour around the inclusion. The most interesting thing about this modified cracking mechanism is that all the phenomenon of crack pinning, trapping, deflection and contouring is happening at the micro level, thus resulting in substantially improved performance in terms of energy absorption capacity (see Fig. 10, 11 and 12). The inert carbonized particles affect the cement matrix in two ways, one is that they provide the nucleation sites for the deposition of the calcium hydrated products and results in stronger matrix [36]; the second factor, which is more pronounced is that the inert carbonized particles affects the growing crack path. In pure cement matrix after the initiation of the crack, it propagates rapidly resulting in sudden failure while in other case the crack is trapped by the inert carbonized particles so the cracks have to divert their path involving more material in the failure process. Fig. 10, 11 and 12 shows the typical fracture surfaces of cement composites containing pyrolyzed coconut shell particles. The diversion of cracks due the toughening mechanisms led by the inclusion of inert carbonaceous particles results in the development of three-dimensional fracture surface of the cement composites. The development of torturous crack surfaces indicates that more energy is required by the composite for the growth of cracks. In FESEM analysis, it was also observed that sometimes the cracks propagated through the large particles inclusions but always contoured the small particles and the crack branching phenomena was also observed due the presence of inert pyrolyzed particles (see Fig. 11).

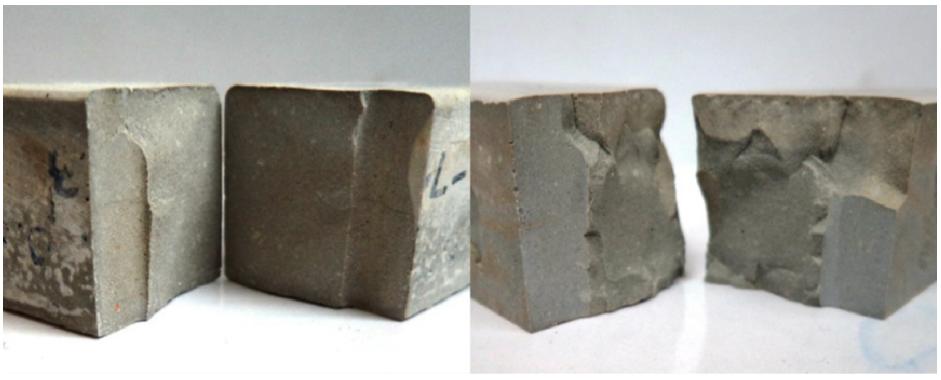

Figure 9: Typical fracture surfaces of the cement composites tested in flexure

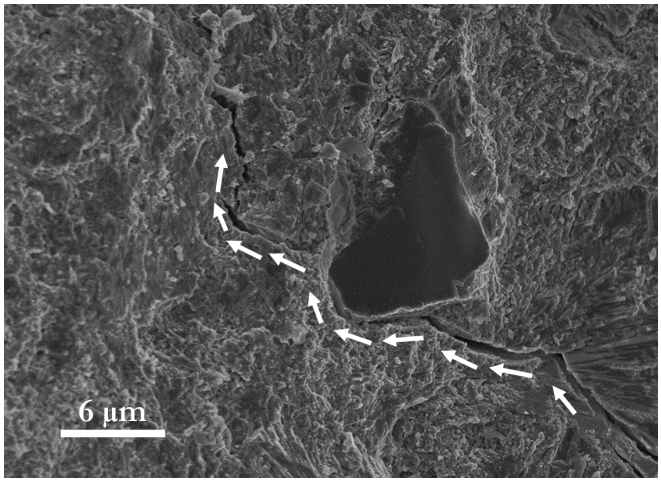

Figure 10: Crack contouring around a pyrolyzed coconut shells particles inclusion.

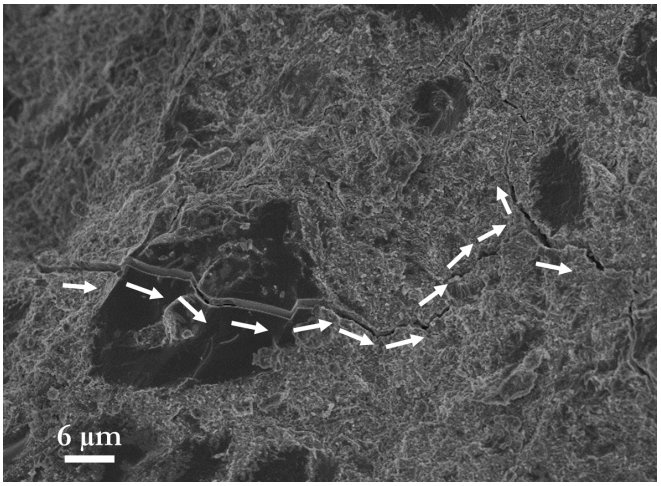

Figure 11: Crack passing through a large inert particle and crack branching.

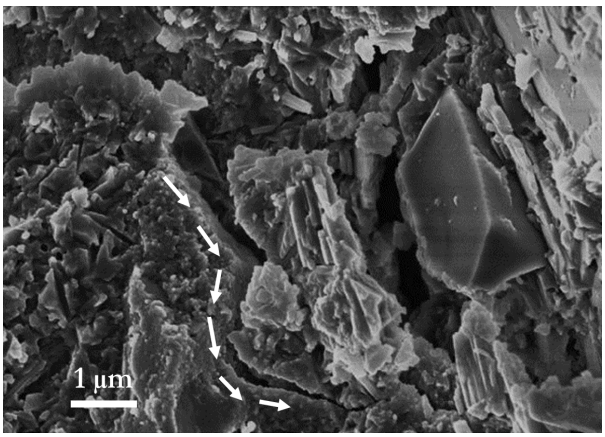

Figure 12: Crack propagation in cement composite matrix.

Conclusions

In this presented research work, the inert micro sized particles were synthesized from coconut shells by means of pyrolysis. The cement composites were prepared incorporating these micro inert particles and the resulting composites were studied in detail for their fracture properties and effects of these particles on the crack surfaces. The results indicate that the carbonized particles additions in the cement matrix successfully modify the fracture surface of the cement composites and results in improved fracture properties, ductility and toughness. It is believed that the crack contouring and the crack pinning are the mechanisms, which can explain the increase of toughness in the cement composite samples.

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