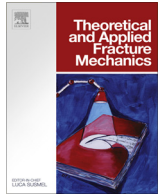




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## Fractal analysis of crack paths into innovative carbon-based cementitious composites

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## ABSTRACT

Fractal geometry has been widely used in literature to characterize the mechanical behaviour of quasi-brittle materials. In this work, innovative cementitious composites with carbon-based pyrolyzed micro-aggregates were tested until complete fracture and their fracture behaviour was studied in the light of fractal geometry. Images of the crack paths across the tested specimens were acquired by Scanning Electron Microscopy (SEM) and their fractal dimension was calculated via the box counting method. Results show that the pyrolyzed micro-aggregates, characterized by high strength and stiffness due to their significant carbon content, are able to alter the crack path by increasing its tortuosity, thus inducing toughening mechanisms in the cementitious composites. This favourable behaviour is explained by means of fractal geometry: it is found that, the greater the fractal dimension of the crack path, the higher the fracture energy.

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## 1. Introduction

The word *fractal*, from the Latin adjective *fractus*, meaning *broken*, was coined by Mandelbrot in his fundamental essay [1] to describe objects that were too irregular to fit into a traditional Euclidean geometrical setting, where points, lines, surfaces, and volumes have integer topological dimensions of 0, 1, 2, and 3, respectively. Falconer [2] developed the fractal theory into the mathematical field, giving prominence to the concept of fractal dimension, a measure of self-similarity and irregularity across scales, which can assume fractional values. Since then, fractal geometry has been widely used in material science and engineering to quantitatively describe complex non-smooth objects, such as rough surfaces [3] or grain boundaries [4], whose fractal dimension is usually greater than their topological dimension. Numerous fractographic studies have also reported that fracture surfaces [5] and crack paths [6] exhibit self-similar fractal nature and can be analyzed by fractal geometry in order to characterize their roughness and correlate it with mechanical properties [7]. This kind of approach has been successfully applied to cement-based materials [8]: the surface of hydrated Portland cement paste has been proved to be fractal in character [9,10]; Lange et al. [11] demonstrated a positive correlation between fracture surface roughness and frac-

ture toughness, reporting a fractal dimension of 2.087 for cement paste and of 2.117 for fine mortar; Saouma and Barton [12] showed that cracked concrete surfaces are fractal and determined the fractal dimension of their one-dimensional fracture profiles in the narrow range from 1.06 to 1.12. Fractal geometry has also been used for explaining the size effects on the mechanical properties of cementitious materials. In particular, a multifractal scaling law (MFSL) has been defined, according to which the decrease in the nominal tensile strength of concrete with the increasing size of specimen is due to the influence of the microstructure disorder [13–15]. Moreover, a similar approach has been used together with fragmentation theory to account for size effects on dissipated energy density in compression [16]. Issa et al. [17] analyzed the fracture surfaces of normal strength concretes with four different maximum aggregate sizes, highlighting their fractal characteristics and deriving a linear correlation between the fractal dimension of fracture surface, with values between 2.15 and 2.29, and the fracture toughness of concrete, represented by fracture energy; a similar result was reported in [18], although a simple relation between fractal dimension and fracture energy could not be established, and fractal dimension of crack paths was found to range from 1.03 to 1.25 depending on the nature of aggregates and the resolution of the processed images.

Following up previous studies concerning the mechanical behaviour and the manufacturing optimization of cementitious composites with carbon-based inerts [19–22], the aim of the present

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work is to apply fractal geometry to innovative composites with pyrolyzed hazelnut shells added as micro-aggregates, in order to explain the toughening mechanisms observed in their experimental fracture behaviour [23,24]. Three Points Bending (TPB) tests until complete fracture were carried out on prismatic specimens made of the innovative cementitious composites and of a plain cement paste. The addition of a small amount of carbonaceous micro-aggregates strongly influenced the mechanical performances of the cement paste, resulting in a significant increase in strength together with an increment in fracture energy, and then in ductility. Images of the crack paths across the tested specimens were acquired by Scanning Electron Microscopy (SEM), subsequently adjusted and binarized via image processing techniques and later examined via fractal geometry. The fractal dimension of the crack paths was calculated by implementing an algorithm based on the box counting method and a relationship with the values of fracture energy computed from the experimental loading curves was finally established. The analysis of the results shows that the increment of the fracture energy due to the addition of the carbonaceous micro-aggregates is strongly related to an increment of fracture surface tortuosity.

## 2. Materials and methods

### 2.1. Manufacturing of materials and specimens

Cementitious composites were manufactured by using ordinary Portland cement (Type I, CEM I 52, 5R, light grey color), deionized water and by adding to the cement paste carbon-based micro-aggregates produced from the pyrolysis of hazelnut shells.

Pyrolysis was carried out into a hermetically sealed reactor, where an inert environment was ensured by a continuous flowing of nitrogen. A heating ramp of 6 °C/min was held for 1 h, reaching a final temperature of 800 °C. After the pyrolysis process, the material was collected from the reactor, grinded for 10 min into a planetary mill to crush the bigger particles, and then milled for 1 h using ethanol and alumina balls of 2 mm diameter. At the end of the grinding cycle, the particle size was analyzed by laser granulometry using a Malvern Particle Sizer, obtaining an average value in the range of a few nanometers to 10 µm.

Mixing procedure is crucial for the performance of cementitious composites [25], hence the same procedure described here was followed to manufacture all the specimens. The pyrolyzed particles were dispersed in a solution of water and superplasticizer (Mapei Dynamon SP1), the latter intended for limiting as much as possible the re-agglomeration problem of the particles [26,27] and for keeping the water to cement (w/c) ratio equal to 0.35. The solution was sonicated in an ultrasonic bath for 15 min and then transferred into the mixing bowl of a homogenizer. The cement was gradually added to the solution while operating the homogenizer at 440 rpm for 2 min. Afterwards, the homogenizer speed was increased to 630 rpm for further 2 min, up to a total mixing time of 4 min. Various percentages of addition of pyrolyzed particles with respect to the cement weight were considered, in agreement with previous studies [28–30], and four different mixtures were obtained. The weights of the materials used for each mixture are reported in Table 1.

**Table 1**  
Weights of the materials used for each mixture.

Mixture ID	Cement (g)	Water (g)	w/c (-)	SP1 (g)	Pyrolyzed hazelnut shells (g)	Pyrolyzed hazelnut shells (%) <sup>a</sup>
PLAIN CEM	214	74.9	0.35	3.21	-	-
PY-HS_0.5%	214	74.9	0.35	3.21	1.07	0.5
PY-HS_0.8%	214	74.9	0.35	3.21	1.71	0.8
PY-HS_1%	214	74.9	0.35	3.21	2.14	1.0

<sup>a</sup> Percentage with respect to the cement weight.



**Fig. 1.** TPB test setup.

A total of 32 prismatic specimens of dimensions 20 mm × 20 mm × 75 mm were manufactured by casting the mixtures into acrylic molds and letting them mature for 24 h at room temperature in airtight plastic containers with 90% humidity level. After maturation, the specimens were removed from the molds and immersed in tap water for curing. At the end of a curing time of either 7 or 28 days, the specimens were notched by means of a Remet type TR100S abrasive cutter with 2 mm thick diamond cut-off wheel, realizing a 6 mm deep U-shaped notch.

### 2.2. Experimental tests

Each notched specimen was subjected to a crack mouth opening displacement-control Three Points Bending (TPB) test. TPB tests were performed using a single column Zwick Line-Z010 testing machine equipped with a 1 kN load cell and a clip-on strain gauge to measure the Crack Mouth Opening Displacement (CMOD), as shown in Fig. 1; a span length of 65 mm and a displacement rate of 0.005 mm/min were adopted.

Besides the mechanical tests, the chemical characterization of pyrolyzed hazelnut shells was carried out using the X-Rays Fluorescence (XRF) technique. The chemical composition reported in Table 2 shows that the pyrolyzed particles are almost exclusively made of carbon and present very low percentages of impurities.

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