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Ultrasonic characterization of GRC with high percentage of fly ash substitution

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ABSTRACT

New applications of non-destructive techniques (NDT) with ultrasonic tests (attenuation and velocity by means of ultrasonic frequency sweeps) have been developed for the characterization of fibre-reinforced cementitious composites. According to new lines of research on glass-fibre reinforced cement (GRC) matrix modification, two similar GRC composites with high percentages of fly ash and different water/binder ratios will be studied. Conventional techniques have been used to confirm their low $Ca(OH)_2$ content (thermogravimetry), fibre integrity (Scanning Electron Microscopy), low porosity (Mercury Intrusion Porosimetry) and good mechanical properties (compression and four points bending test). Ultrasound frequency sweeps allowed the estimation of the attenuation and pulse velocity as functions of frequency. This ultrasonic characterization was correlated successfully with conventional techniques.

1. Introduction

Glass-fibre reinforced cement (GRC) is a composite made of Portland cement mortar with low water/cement (w/c) ratio and a large amount of paste in relation to the aggregate quantity. Addition of a high proportion of glass fibres to the mortar matrix (3–5% by weight of mortar) improves the mechanical properties of the elements formed with this material, especially the toughness and ductility. This composite has an important role in non-steel reinforced pre-cast concrete elements, such as sheets, panels, and other slim shapes usually employed in building engineering and architecture, and also in cast-in situ sprayed-on surfaces [1,2].

The most important problem with this kind of material is the glass-fibre degradation. Exposure of the glass fibres to an alkaline environment (e.g.: the Portland cement matrix) leads to a rapid degradation process which involves strength and weight losses, and reduction in the filament diameter. This process can be attributed to the breaking of the Si–O–Si bonds in the glass network, by the OH^- ions which are present in high concentration in the pore solution of the cement matrix [3,2].

For that reason, research on this topic has focused on three main lines:

1. Fibre modification, making it more stable to a highly alkaline environment.
2. Matrix modification, adding moderate quantities of pozzolanic powders partially substituting for the Portland cement content.
3. Fibre and matrix modification at the same time.

As to fibre modification investigations, research has focused on modifying the chemical composition of the glass (e.g.: ZrO_2), which makes the system more chemically stable in alkaline solutions [4,3], and fibre-coating modification [5,6].

As to matrix modification, several authors have reported successful results by means of the partial replacement of cement by reactive mineral admixtures such as silica fume, fly ash, metakaolin or blast-furnace slag. These modifications of the matrix have resulted in an improvement of the fibre–matrix interface, partially inhibiting fibre degradation due to the reduction of the pH level and the $Ca(OH)_2$ content [7–10]. Thus the mechanical properties of the GRC specimens are improved significantly. Usually, the characterization of this kind of cement composite is carried out through conventional techniques such as X-ray diffraction, thermogravimetry, and mechanical tests [11,10].

Recently, several researchers have focused on non-destructive concrete and mortar characterization in order to obtain more reliable information and new aspects of cementitious composites without damaging the specimen under test. During recent years, non-destructive techniques (NDT) applied to concrete have been investigated, especially ultrasonics and acoustic spectroscopy, in

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order to obtain parameters related to the physical and mechanical properties of the material [12]. Some experimental studies have demonstrated that wave parameters such as the ultrasonic pulse velocity of P-waves are suitable to predict the dynamic elastic modulus of concrete, and also S-waves for its dynamic shear modulus. These parameters are proportional to the elastic and shear modulus of concrete and also to its compressive strength [13,14]. Other studies based on ultrasound propagation indicate that wave attenuation can be measured for various frequencies, in order to define different cementitious materials, distinguishing their microstructure, porosity, and other qualities in both the hardened [15–18] and the fresh state [19]. Some studies have shown interesting correlations between other properties of cementitious composites, such as their permeability or porosity, with ultrasonic parameters such as pulse velocity, signal attenuation, or reflection coefficients, using a specific test setup [20].

NDT techniques for characterizing GRC specimens have been applied: acoustic resonance techniques (Non-linear Impact Resonance Acoustic Spectroscopy, NIRAS) and ultrasonic guided waves have been used to obtain more information about the composite material and its ageing process than traditional tests on GRC [21]. The scope of this study is to characterize GRC specimens with high substitutions of the cement content by fly ash. As a novelty for fibre-reinforced cementitious composites, the characterization will be carried out by means of ultrasonic frequency sweeps that will allow obtaining ultrasonic parameters as functions of the frequency. The frequency response will be correlated with the porosity and the mechanical behaviour of this type of material. Thermogravimetry and Scanning Electron Microscopy will be used to assess the pozzolanic reaction and fibre integrity.

2. Experimental

2.1. Materials

Two different matrices with the same type of glass fibre and content were designed, as can be seen in Table 1. In both mixtures the proportion of Portland cement to fly ash was 40/60.

The raw materials and their brief description are summarized in Table 1. The main difference between both composites was the water/binder (w/b) ratio: fa-035 corresponds to 0.35 w/b ratio whereas fa-030 corresponds to 0.30 w/b ratio. The plates used for the ultrasonic tests and the four-point bending test were manufactured according to the BS EN 1170-5 standard [22]. In this experiment, a $400 \times 400 \times 20\ mm^3$ mother plate was chosen, according to the standard, seven specimens of $325 \times 50 \times 20\ mm^3$ were prepared by cutting the hardened mother plate. Three mortar specimens of each series ($40 \times 40 \times 160\ mm^3$) were manufactured for the compressive strength test. A sample of each series was taken for Mercury Intrusion Porosimetry (MIP). For the thermogravimetry tests, pastes with neither fibres nor sand were made. The plate manufacturing process was carried out on mixing the raw materials following the next procedure:

1. 60 s of mixing water + 2/3 superplasticizer + cement + fly ash.
2. 30 s of adding sand and mixing.
3. 30 s adding 1/3 superplasticizer.
4. 60 s mixing mortar.
5. 60 s adding fibre and mixing.

The mechanical and the non-destructive tests were performed 90 days after the plates were manufactured, in order to allow the completion of any hydration and pozzolanic reactions in the matrix. All the specimens were cured in a wet chamber at 20 °C and 100% HR.

Table 2 gives details of the cement and fly ash chemical composition. As can be observed, the fly ash contains a large amount of SiO_2 and Al_2O_3 (the sum of these oxides was higher than 75%), important components for the progress of a pozzolanic reaction.

2.2. Test methodology

Several tests were done on this study in order to compare the performance of ultrasonic tests and other characterization techniques. Mechanical tests (INSTRON universal testing machine, model 3382), Scanning Electron Microscopy (JEOL JSM 6300 aplying a 20 kV voltage), Mercury Instrusion Porosimetry (AutoPore IV 9500 of Micrometrics Instrument Corporation with a range of pressures between 13,782 Pa and 227.4 MPa) and thermogravimetry (Mettler TGA 850).

2.2.1. Description of the ultrasonic test equipment

An ultrasonic through-transmission setup was selected because it offers good penetration and good accuracy for velocity and attenuation estimation [23,24,15,25]. The disposition of the equipment is shown in Fig. 1. The transducers used were the *K1SM* (for transmission) and *K1SC* (for reception) from General Electric. Both are broadband transducers with a bandwidth centered at 1 MHz and a circular active surface of ∅28 mm. Due to the cross section of the specimens ($50 \times 20\ mm^2$), part of the active surface of the transducers (≈18%) keeps out of the samples. This fact will produce an identical reduction of the injected and received energy effectiveness for all measures.

The transmitter transducer was excited directly by a programmable signal generator (Agilent 33120*A*) while the reception transducer was connected to a 40 dB preamplifier (Panametrics 5600*B*). The received and amplified ultrasonic signal was captured by a digital oscilloscope (Tektronix *DPO*3014) with a sampling frequency of 25 MHz. Finally, a laptop was used to control the signal generator and to acquire and store the digitized signals by the oscilloscope.

Table 1
Dosages (in grams) used on GRC plate manufacturing.

Material	Type	fa-035 (g)	fa-030 (g)
Cement	CEM I-52,5 R	726	726
Fly Ash	F[a]	1089	1089
Water	-	635.25	544.50
Aggregate	Silica sand 0/2	1216	1216
Superplastizicer	Policarboxilate ether	3.50	7
Fibre[b]	Glass AR 12 mm ∅ 12 μm	73.50	73.50

[a] ASTM C-618.
[b] Cem-FIL® 62 82Tex.

Table 2
Cement and fly ash composition by % weight.

Component	LOI[a]	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	C_4AF	C_3A	C_3S	C_2S
CEM I-52,5 R	2.05	20.52	3.37	3.92	63.36	1.96	2.59	0.99	11.93	2.33	55.7	16.94
Fly ash type F	1.97	49.91	25.80	13.94	3.84	1.06	1.00	2.47	–	–	–	–

[a] Loss on ignition.