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Rheological, hydration and mechanical characteristics of microsilica fibre reinforced cement combinations with incremental fly ash contents

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HIGHLIGHTS

- Properties of fibre cement systems were studied at varying FA contents of up to 60%.
- Mixes with FA up to 30% yielded comparable or slightly lower performance to the REF.
- On FA contents 30% onwards, changes in microstructural properties were more drastic.

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ABSTRACT

Cement combinations are usually manufactured by partially replacing Portland cement with fly ash (FA) due to its pozzolanic properties and low carbon emissions. The challenge, however, is selecting an optimum FA content that is viable in terms of the sustainability, cost and performance of the combination for a given application. This paper examines the properties, microstructure and hydration of fibre reinforced microsilica cement combinations with varying FA contents. Results indicated that the addition of FA prolonged the initial and final setting times of the combinations, decreased the viscosity peaks and lowered the ettringite, C-S-H and $Ca(OH)_2$ contents. At low FA contents, permeation and mechanical properties were still comparable to those of a reference sample. It was observed that combinations with 30% or more FA contents exhibited significant reductions in alkalinity, performance and Ca/Si ratios. Furthermore, the performance of the combinations seemed to reach a plateau regime between FA contents of 15% and 30%.

1. Introduction

There is currently a growing demand in the use of fibre reinforced cement products as a construction material, especially in tiling industries. Designing such products to deliver an optimum balance between performance, low carbon footprint and overall cost has been the main challenge for these industries to tackle. In 2017, 4.1 billion tonnes of Portland cement (PC) were manufactured globally [1], and considering that the current overall embodied CO_2 (eCO_2) for PC production in the UK is more than 913 kg of CO_2 per tonne of PC produced [2,3], the incorporation of industrial by-products in cement currently the most commonly established route to minimizing PC contents within cement products.

Although blast-furnace slag and silica fume have commonly been used as additions in cementitious systems, the use of fly ash (FA) is becoming more popular because of its low eCO_2 of just 4 kg/tonne [2]. The effect of FA on the properties of cement and concrete has been indeed extensively studied over the last few decades, with studies showing that it enhances concrete durability, permeability and long term strengths [4–10]. The key properties of FA that are known to directly affect the overall performance of cement and concrete –and that essentially constitute the quality of the material- are the particle size, calcium oxide content, fineness, surface to volume ratio, and mineralogical composition [11]. As part of a pozzolanic reaction, the FA reacts with available $Ca(OH)_2$ amounts produced by alite and belite hydration to generate additional C-S-H, which contributes to the density and strength development of the system [10–13]. Apart from this reaction, FA can offer additional benefits when introduced at optimum contents, such as aiding in particle packing, improving workability and accelerating hydration patterns [10–13]. However, as many studies have shown, the variability of the FA content as an aluminosilicate can also lead to varying performance of a combination, which may be better or poorer than that of ordinary PC system [14–16]. Barbuta et al. [17] is more recently studied the effect of fly ash, glass and polyester fiber quantities on the mechanical properties of concrete. Their results indicated that a 10%

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replacement of cement with the fly ash and the fibers improved the compressive strength of the concrete, although higher doses lead to a strength reduction. Also, the polyester fibers were found to be more beneficial than the glass fibers in increasing the flexural strength for the same fly ash content in the concrete. In another study conducted by [18], the replacement of cement by 15% fly ash and 0.15% coconut coir fiber (cellulose based) was found to exhibit compressive strengths that were comparable to ordinary PC concrete. Chore and Vaidya [19] investigated the variation of both fly ash and polymers quantities and its effect on the unconfined compressive strength (UCS) and Brazilian tensile strength (BTS). It was surmised from their results that the a replacement of 20% fly ash for cement and 1% addition of polypropylene yielded optimum values of the UCS and BTS. Zeyad and Saba [20] also examined the influence of higher fly ash contents and polypropylene fibers on the fresh properties of self-compacting concrete, including the segregation, bleeding, slump flow and V-funnel tests. The results showed that the best workability of the concrete was achieved with a 20–40% fly ash replacement.

Due to such degree of variability, the potential of drawing conclusions on how to achieve the best balance (i.e. developing products chemically optimized, cheap and high performing, all at the same time) in fibre reinforced cements is reduced. Therefore, an important challenge is to determine a beneficial regime of amounts that a given FA may be incorporated in a cementitious combination to produce environmentally friendly and cheap products without sacrificing the chemical stability or the performance of the combination. Furthermore, this challenge involves tailoring the properties of the developed combination to suit the demands and needs of a given application, e.g. in the tiling industry.

This paper investigates the properties of fibre reinforced microsilica cement combinations by partially replacing PC with FA contents by mass. Microstructural and hydration aspects of the cement systems containing up to 60% FA by mass were studied using x-ray diffraction, thermogravimetric analysis, setting time determinations, pH measurements and viscosity profiles. The mechanical properties that were studied included flexural strengths up to 28 days, apparent densities and water absorption rates due to capillary rise. The effect of the FA content on these properties of the combinations is discussed.

2. Materials and methods

The materials and their abbreviations used in the experimental procedure of this study are given in Table 1, and the respective mineralogical compositions are shown in Table 2. Particle size distributions were obtained using a Malvern Mastersizer 2000 particle size analyser. Particle densities of the materials dispersed either in kerosene (PC) or in water (LF, FA, MS) and these were determined using the pycnometric method as in ASTM C188 [21]. Oxide and phase compositions were obtained by a combination of XRD (D8 ADVANCE diffractometer), Rietveld analysis and thermogravimetric analysis (SETARAM TG-92 thermogravimetric analyser). The mix proportions of the investigated combinations are shown in Table 3. It should be noted that the reference combination (REF) contained (by mass) 80% PC [22], 12% LF, 0% FA [23], 5% MS, 1% FB, and 2% CL. The rest of the combinations were produced by increasing the FA content by 5% increments until FA reached 30% within the combination at the expense of the PC content; then the FA content was increased by 15% increments reaching up to 60% by mass of the combination. Incorporating MS in all combinations at a fixed content was done on the basis of the known effect that the MS offers in the enhancement of cement performance [24], whereas the incorporation of LF at all combinations aimed to resemble a CEM II/ B-L Portland limestone cement as in EN 197-1:2015 [22], which is to a fair extent a commonly utilized cement in tiling products. The addition of FB and CL at a fixed content was done on the basis of the known effect that fibres have on enhancing the durability of cementitious materials [25]. The above constituents within the combination were maintained at the same percentage by mass in order to be able to conduct valid comparisons following the experimental programme. The w/c ratio used to prepare all combinations was 0.5 [26], except for the case of testing the initial and final setting times to EN 196-3, in which the consistency found was 0.46 [27].

Initial and final setting times of fresh cement pastes were determined in accordance to EN 196-1 [27] using the Vicat apparatus. Apparent viscosity curves immediately after mixing were obtained at 20 °C and 65% RH by using a VT 550 rotational viscometer at increasing shear rate from 0 to 60 s^{-1}. The mixing and casting of the prismatic 40 × 40 × 160 mm cement samples were carried out in accordance to [26]. The samples were demoulded after 24 h and then cured at maintained conditions in water (20 °C, 65% RH) until specified ages of testing.

For obtaining the apparent density, 40 × 40 × 160 mm samples were tested in accordance to [28], wherein fully saturated samples were weighed both in air and water, then fully dried and re-weighed. The flexural strength of 40 × 40 × 160 mm samples were determined at 1, 3, 7, and 28 days in accordance to [26] on a load-control basis with a 10 N/s load rate.

Initial water absorption rates, S_i, were determined by using the same method described in [29], except that the dimensions for the tested three samples per mix were 50 × 100 × 100 mm, which were air-cured in maintained conditions of 20 °C and 65% RH for 28 days. The samples were periodically immersed at specified time intervals within a 3 mm water depth and the mass changes were measured to

Table 2
Mineralogical composition of the materials in the experimental study.

Compound	Notation	Mineralogical composition, % by mass			
		PC	LF	FA	MS
Calcium oxide	CaO	65.32	55.58	7.48	–
Silicon Dioxide	SiO_2	17.36	0.54	58.35	98.51
Aluminium oxide	Al_2O_3	8.01	–	26.67	–
Ferric Oxide	Fe_2O_3	3.29	–	5.02	–
Magnesium oxide	MgO	1.07	–	–	–
Sulfur Trioxide	SO_3	3.04	–	2.1	–
Sodium Oxide	Na_2O	–	–	0.38	–
Carbon dioxide	CO_2	–	43.88	–	–
Loss on ignition	–	1.6	–	–	1.49
Insoluble residue	–	0.31	–	–	–

Table 3
Mix proportions of the combinations used.

Combination	Percentage by mass					
Nomenclature	PC	LF	FA	MS	FB	CL
REF	80	12	0	5	1	2
FA5	75	12	5	5	1	2
FA10	70	12	10	5	1	2
FA15	65	12	15	5	1	2
FA30	50	12	30	5	1	2
FA45	35	12	45	5	1	2
FA60	20	12	60	5	1	2

Table 1
Materials used in the experimental study.

Material	Abbrv.	Particle density (kg/m^3)	Mean diameter (μm)	Particle size distribution	
				d_{10}	d_{90}
Portland cement CEM I 42.5N to EN 197-1:2011	PC	3010	33.4	2.2	67.3
Commercially available limestone filler	LF	2720	29.6	1.9	58.4
Fly ash to BS EN 450-1:2012	FA	2280	37.6	2.4	81.6
Undensified microsilica	MS	2150	0.13	0.06	0.30
PVA fibres 6 mm	FB	–	–	–	–
Methyl cellulose 400cPs	CL	–	–	–	–