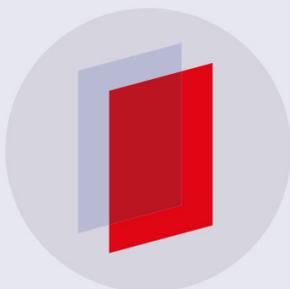


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Steel microfibres in fly ash geopolymer for multifunctional conductive composites

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Abstract. Steel fibres in construction materials not only enhance their mechanical and fracture properties but also increase their capability of electrical conductivity. This feature thus enables the material to obtain advanced multifunctional properties that can be utilized in self-sensing and other smart applications. Fly ash geopolymers with steel microfibres in 5–30% wt. were prepared to assess the influence of the fibres on selected electrical properties of the composite (resistance, capacitance and relative permittivity) with respect to mechanical performance. Microstructure and interfacial bonding of matrix and steel fibres was observed by electron microscopy imaging. Steel fibres caused an improvement in all assessed electrical properties but only above a certain frequency values. Furthermore, steel reinforced samples exhibited increase in both compressive and flexural strength.

1. Introduction

Development of smart conductive composites could support the growing efforts to produce multifunctional materials. Since the electrical properties of construction materials reflect the changes of external conditions, they can be implemented in self-sensing solutions for real time health monitoring of civil infrastructures, i.e. sense strain, detect cracks and other defects or observe the changes of temperature and humidity. Such applications eliminate or reduce the need for embedded or attached devices, which are expensive and limited in durability [1]. Moreover, this idea is adopted for design of advanced traffic monitoring systems to track the weight and speed of vehicles and traffic density [2]. Other applications include self-heating concretes for both indoor and outdoor structures, especially for de-icing and winter maintenance of roads, car parks or airport areas [3]. Electrically conductive materials with carbon filler also exhibit increased ability of electromagnetic shielding needed in protective structures for strategic communication and control systems [4].

Geopolymers are novel cementitious materials synthesized by the alkaline activation of solid aluminosilicate precursors, frequently industrial waste materials or by-products. Such raw materials with preferably high amorphous glassy content with reactive Si and Al are dissolved in alkaline activator solution, usually mixture of alkali hydroxides and alkali silicates [5]. The following geopolymerization process is described as a formation of highly coordinated polymeric structure of SiO₄ and AlO₄ tetrahedra balanced by Na⁺/K⁺ cations supplied by the alkaline activator [6]. Geopolymer materials produced from low Ca fly ash, metakaolin or other calcined clays have been widely investigated regarding their good strength, chemical resistance and exceptional performance in exposure to elevated temperatures [7]. Considerable savings of nonrenewable resources, energy and emissions make geopolymer binders more environmentally friendly alternative to Portland cement [8].



Alkali activated materials may represent promising materials for smart composites as they are considered to possess lower electrical resistivity compared with cement-based binders [9]. Direct piezoelectric effect in geopolymeric mortars was explained by a charge imbalance and the formation of local dipoles generated by the migration of the hydrated Na^+ cations [10]. According to Hanjitsuwan [9,11], the electrical conductivity of fly ash geopolymer matrix is affected by the NaOH concentration in activator solution, liquid activator/ash ratio and frequency spectrum.

The application of steel fibres in geopolymeric materials was mostly discussed in studies concerning their mechanical performance and fracture properties and they confirm significant improvement of reinforced geopolymer in flexural strength, fracture toughness and transition of failure mode from brittle to ductile [12]. The effect of steel fibres on the electrical properties is scarcely mentioned. Chung [13] compared steel fibre and carbon fibre (CF) reinforced cement pastes stating that CF paste is a better piezoresistive strain sensor than stainless steel fibre paste at a similar fibre volume fraction. Conductivity of fly ash geopolymers could be enhanced by a very low dosage of CNTs (up to 0.5%) but only in case of effective distribution within the matrix [14]. Recently, exceptional improvement of performance in both mechanical and electrical properties was observed in studies of fly ash geopolymer doped with graphene [15].

2. Materials and methods

Main source materials for the geopolymer binder was fly ash (FA) from black coal combustion with the chemical composition given in **Table 1** and commercially sold water glass (sodium silicate solution with molar ratio $\text{Na}_2\text{O}/\text{SiO}_2 = 1.6$). Brass coated steel microfibers DM 6/0,175 were used as a conductive admixture in 5–30 % wt. of the fly ash. We used standardized quartz sand as fine aggregate and a defoaming agent Lukosan S to diminish excessive pore generation in fresh mortars.

Table 1. Chemical composition of fly ash.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	S_{total}	Na_2O	K_2O
(%)	49.82	24.67	7.50	3.91	2.68	0.91	0.70	2.78

Each of the mixes was prepared according to mix compositions shown in **Table 2**, following these steps: first, mixing the sodium silicate solution with fly ash and water. After proper binder homogenization, quartz sand was added from fine to coarse fractions. Then the steel microfibers were added into the fresh mortar and the defoaming agent was added at the very end of the mixing procedure.

Fresh mortars were poured into prismatic moulds ($40 \times 40 \times 160$ mm) and covered with plastic sealant to protect the binder from undesirable moisture loss from the surface. After 2 hours in laboratory conditions (22 ± 2 °C, $\varphi = 45 \pm 5$ %), the samples were thermally treated at 40 °C for 24 h. Demoulded specimens were stored in laboratory till the age of testing.

Table 2. Mix design of geopolymer mortars.

	REF	SF 5	SF 10	SF 15	SF 20	SF 25	SF 30
Fly ash (g)	350	350	350	350	350	350	350
Sodium silicate (g)	280	280	280	280	280	280	280
Steel fibres (g)	-	17.5	35	52.5	70	87.5	105
Sand (g)	1050	1050	1050	1050	1050	1050	1050
Lukosan S 1% (ml)	-	2	2	2	3	3	3
Water (g)	35	35	35	35	35	35	35

Before the measurements of electrical properties, the prepared prismatic samples were dried at 105 °C to reduce the influence of moisture on assessed properties. The specimens were placed between parallel brass electrodes (30 × 100 mm) so that a distance between electrodes was 40 mm and characterized by impedance spectroscopy in the range of 40 Hz to 1 MHz using an Agilent 33220A sinusoidal signal generator and an Agilent 54645A dual-channel oscilloscope. The output voltage of the signal generator was 5.5 V, the input values for the electrical capacity and the resistance of the oscilloscope were 13 pF and 1 MΩ, respectively. Relative permittivity measurements were performed in the range of 100 to 3000 MHz using the vector analyser and DAK probe. Geopolymers were tested for their mechanical properties (compressive and flexural strength) and scanned by electron microscope (SEM) Tescan MIRA3 XMU to obtain the images of microstructure of the geopolymers.

3. Results and discussion

3.1. Electrical properties

The main purpose of conductive filler in smart materials is to enhance their conductivity and reduce electrical resistance. Electrical resistance of geopolymer mortars is depicted in **Figure 1** and decreased with higher frequency applied. Up to 600 Hz, the lowest resistance was unexpectedly attained by sample SF 5. Curve shapes of samples with 5–25% steel fibres seem to be somehow “inverted” from this certain point and increasing steel fibres content resulted in drop of resistance only above this frequency.

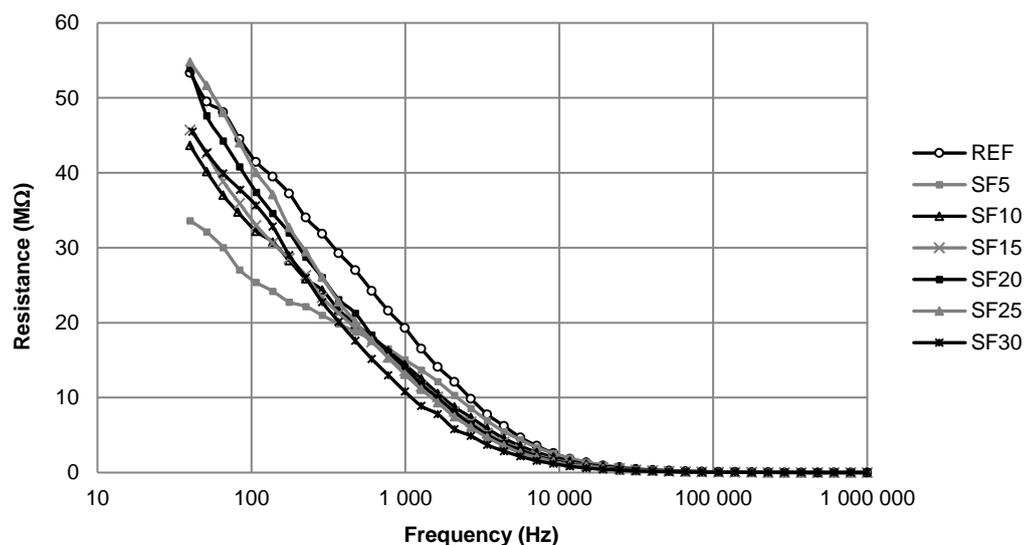


Figure 1. Electrical resistance of FA geopolymer mortars with different steel fibres content.

Similar behaviour was observed regarding the electrical capacitance (**Figure 2**). The capacitance decreased with frequency and increased with steel fibres content but this was noted only after the critical value, this time at frequency equalling to approximately 200 Hz. Capacitance of all samples exhibited minor differences at higher frequency. **Table 3** summarizes the values of resistance and capacitance at chosen reference frequency 1 kHz. Resistance and capacitance values above the points of frequency previously mentioned follow the expectations of reduced electrical resistance and growing capacitance with high steel fibres content.

As far as the output of electrical conductivity measurements was not applicable (due to a number of points with zero conductivity values measured across the frequency spectrum), the electrical properties at higher frequency up to 3000 MHz are represented by the relative permittivity (**Figure 3**). Relative permittivity increased with the steel fibres content across the spectrum and slightly declined with

higher frequency. Initial outlying values related to the transition between the electrode and the samples were omitted and the curves could be replaced by trend lines as the fluctuations are caused by the measuring device. Significant increment of relative permittivity in case of maximum steel fibres dose (SF 30) is probably attributed to the surface conductivity as the transmitting and receiving parts of probe were located on the same surface of the sample.

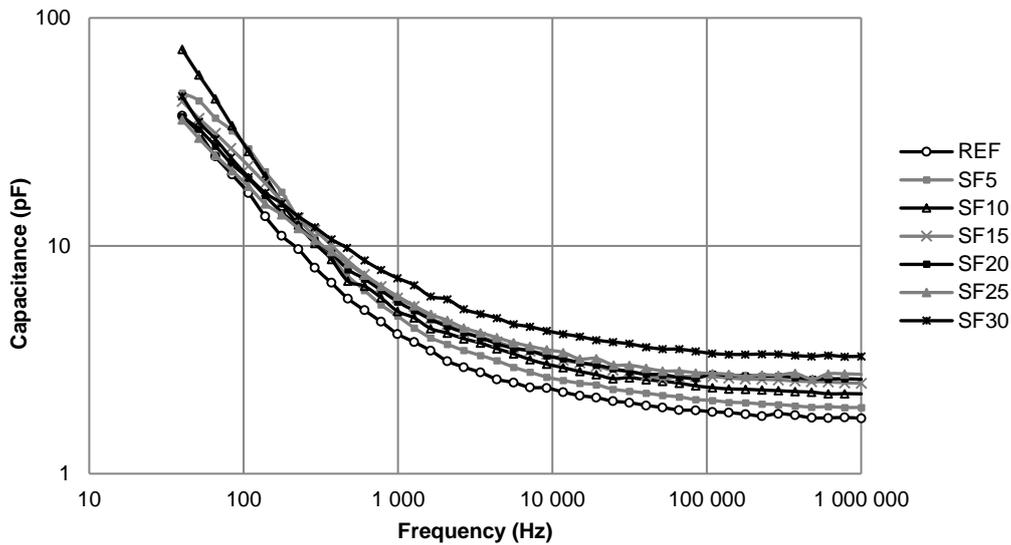


Figure 2. Capacitance of FA geopolymer mortars with different steel fibres content.

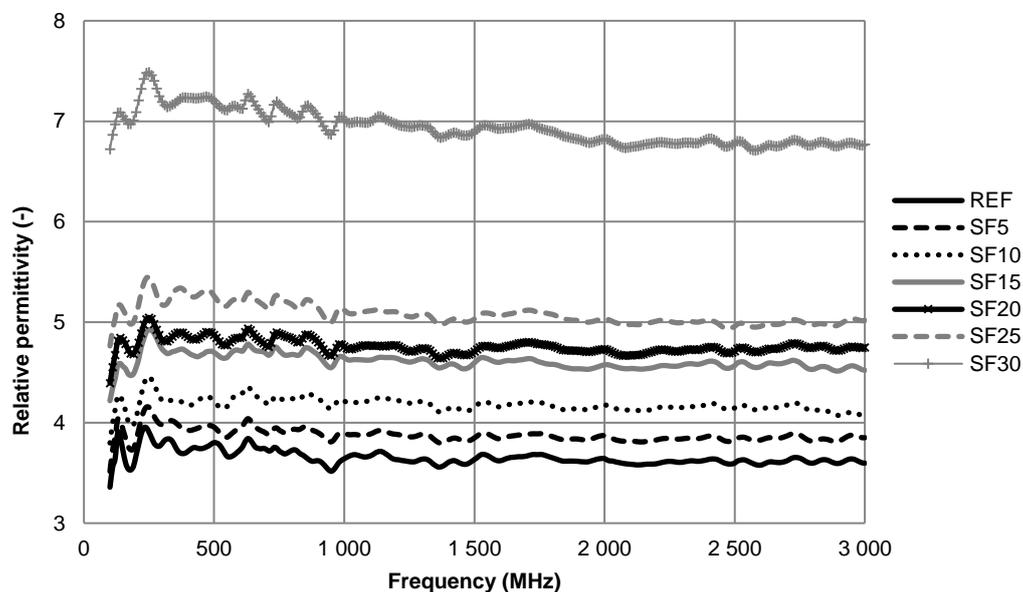


Figure 3. Relative permittivity of FA geopolymer mortars with different steel fibres content.

Table 3. Electrical resistance and capacitance at chosen frequency 1 kHz.

	REF	SF 5	SF 10	SF 15	SF 20	SF 25	SF 30
Electrical resistance (MΩ)	19.29	15.05	14.40	13.60	14.06	13.13	10.83
Electrical capacitance (pF)	4.11	4.92	5.17	5.90	5.68	6.02	7.22

3.2. Mechanical properties

Mechanical performance of steel fibre doped geopolymers is shown in **Figure 4**. Geopolymer with 5% of steel fibres exhibited similar strength values as the reference sample, slight reduction in compressive strength might be associated with possible increase in porosity of the material and insufficient bonding ability of the matrix and still relatively low dosage of fibres. Higher steel fibres content resulted in improvement in both flexural strength (by up to 80%) and compressive strength (by up to 20%).

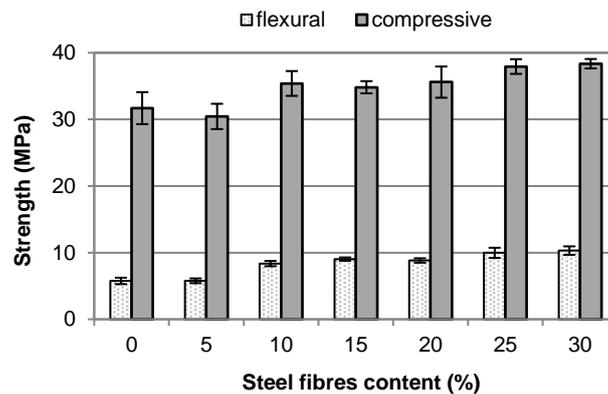


Figure 4. Mechanical performance of FA geopolymer mortars with different steel fibres content.

3.3. Microstructure

The nature of the interfacial zone between steel fibre and geopolymer matrix is shown in **Figure 5**. Steel fibre as received is depicted in picture A and as one can see in pictures B and C, the fibre in matrix is coated by a fine layer of geopolymer binding product. The fibre-matrix bonding is ensured by local geopolymer bond bridges (image C and D) because the matrix is not completely dense.

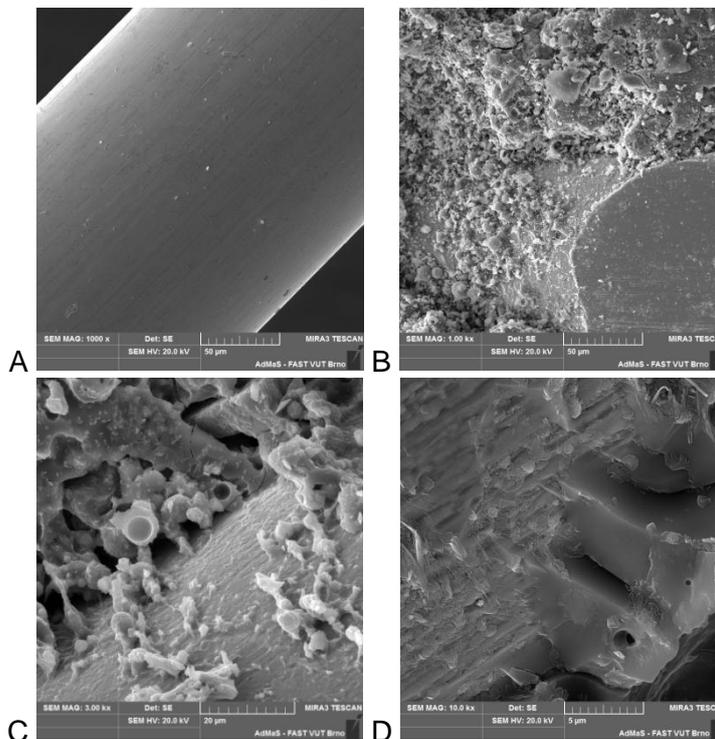


Figure 5. SEM images of steel fibres in geopolymer matrix. A: steel fibre as received, B: cross-section of the steel fibre, C: spot bonds of the matrix and binding product coated surface of the fibre, D: bonding in detail.

4. Conclusions

Steel fibres are usually applied in concrete to reduce shrinkage and increase flexural strength or fracture toughness. This research aimed to assess their contribution to the electrical properties of fly ash geopolymer intended to design advanced conductive composites. Geopolymer mortars with 5-30% of steel microfibers were analysed by means of impedance spectroscopy. Reduced electrical resistance and increase in capacitance with higher steel fibres content were registered only above certain points in frequency spectrum, approximately 600 Hz or 200 Hz, respectively. Samples with 15-25% of steel fibres exhibited similar behaviour in terms of electrical properties, electrical resistance and capacitance across the frequency spectrum did not considerably differ. Relative permittivity at 15% of steel fibres clearly increased and it was comparable with 20% dosage. As far as the differences among these samples were small, this concentration range might refer to percolation threshold or other change in microstructure that affects the transfer of electric charge. Concerning the mechanical performance, the steel fibres addition of 10% and higher resulted in improvement of both flexural and compressive strength. From the results obtained, 15% steel fibres concentration seems to meet the requirements for enhanced electrical and mechanical performance of fly ash geopolymer.

Acknowledgements

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