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Hamza Allam, François Duplan, Jean-Pierre Clerc, Sofiane Amziane, Yves Burtschell. Carbon-fibred mortar: Effect of sand content and grain size distribution on electrical impedance. 13th fib International PhD Symposium in Civil Engineering, Aug 2020, paris, France. hal-03205971

HAL Id: hal-03205971

<https://hal.science/hal-03205971v1>

Submitted on 22 Apr 2021

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Carbon-fibred mortar: Effect of sand content and grain size distribution on electrical impedance

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Abstract

Self-sensing concrete, also known as “Smart concrete”, is obtained by including electrically conductive fibers in cement-based materials. These fibers may allow to reduce electrical resistivity and develop a piezoresistive behaviour. Smart Concrete could therefore be simultaneously both a structural and a sensing material, which eliminates the need for external instrumentation in Structural Health Monitoring.

By increasing the fiber volume fraction within the cement matrix, the electrical resistivity (or impedance) of the material is reduced once percolation threshold is reached. Above this percolation threshold, the fiber content is high enough to allow conductive particles to be in contact or very close to each other, thus creating a continuous conductive network within the insulative matrix.

Sand’s presence was stated to have an influence on resistivity in case of fibred mortar: a high sand content may prevent the network of conductive fibers from percolating. This phenomenon is referred to as “double percolation”: the cement paste needs to be a continuous phase between sand aggregates in order to allow fibers to maintain their efficiency in reducing the electrical resistivity of composites.

However, little attention has been given to the impact of the size of sand grains on the electrical percolation. This work intends to study the effect of the grain size distribution and volume fraction of sand within mortars containing various fiber volume fractions. The results confirm the “double percolation” phenomena: when the volume fraction of sand is close to its maximum packing density, the addition of fibres was not as effective in reducing the electrical impedance of mortar samples. In addition, sand’s grain size distribution proved an influence on impedance of mortar: fine sand showed higher impedance compared to standard sand, especially in case of high sand volume fraction. This could be related to the smaller maximum packing density in case of fine sand, where distance between particles would be in average reduced. This effect, combined with the higher number of insulative particles, could probably disrupt the continuity of the conductive network of fibres within mortar.

KEY WORDS: mortar, carbon fibers, electrical properties

1 Introduction

Self sensing mortar, developed by adding electrically conductive fibers to ordinary cementitious matrix, is an innovative construction material able to detect a stress or deformation in the material due to a mechanical or thermal action. Above the percolation threshold, conductive fibers are able to create conductive paths inside the insulative cementitious matrix, thus reducing its resistivity [1]. Starting even from very low fiber volume fraction, fibred mortar develops a piezoresistive behaviour, where strains caused by external solicitations may be monitored through the exploitation of their relationship with the electrical resistivity [2] [3] of the material. Potential applications of monitoring include the detection of actions such as mechanical loads in compression [4] or tension [5], or thermal variations [6], electromagnetic shielding and pavement’s heating have also been under investigation [7][8].

The relation between hydrating cement systems and AC impedance in absence of fibers was well studied previously [9]. The efficiency of conductive fibers as “doping agent” depends on fiber’s volume fraction (FVF) [1]. The evolution of mortar’s impedance (impedance is the ratio of applied voltage on the specimen to the current flowing through it in case of AC measurements) function of FVF consists in three main phases: an insulation zone, a percolation transition zone and a conductive zone. In the insulative zone, ie where fiber concentration is below the percolation threshold, the electrical current

cannot flow continuously within the fiber exclusively, and still have to go through the insulative matrix; therefore, the reduction of the resistivity of the material is not very significant yet. A strong transition zone is observed around the percolation threshold, when continuous conductive paths start to be established within the matrix, and most of the electrical current may be able to flow through this network. Therefore, the resistivity is being reduced drastically. Above the percolation threshold, increasing the content of fibers may still improve the density and connectivity of the conductive paths within the fiber network, but its beneficial effect on electrical resistivity may not be significant [10].

Various studies have treated the percolation of fiber-shaped inclusions within a homogeneous matrix medium. Balberg [11] proposed a model to estimate percolation threshold for thin spheroids depending on the complexion of their morphology. Celzard [12] developed it to be applied on thin fibers with high aspect ratio. Berhan [13] estimated percolation thresholds for high aspect ratio fibers considering two different possible approaches for fiber's percolation inside a hosting environment. Those studies could be applied to fiber's percolation in cement paste, considering cementitious matrix as a homogeneous phase. In case of mortar, the fibers are not able to penetrate insulative sand particles, and these theoretical approaches may not be valid.

In mortar, the connectivity of cement paste within sand particles has been reported to be a necessary condition for the percolation of fibers at low volume fractions [14]. This criterion is referred to as "double percolation". Garboczi et al. [15] has estimated in 1995 the connected fraction of sand's interfacial transition zone function of its volume fraction. The study showed that the phase surrounding sand particles starts to be connected around 33% and total percolation is reached around 50-55%. Baeza et al. [16] stated that in case of concrete, a multiscale "triple percolation" is necessary to allow the percolation of the fibers: within concrete matrix, mortar could be able to percolate through coarse aggregates, in combination of cement paste percolating through fine sand aggregates within the mortar phase.

While the effect of sand content on the resistivity of carbon fibred mortars has been treated in various works [14][16][17], there seems to be little information on the effect of its grain size distribution within the literature. This study intends to provide a contribution on the effect of sand grain size distribution and volume fraction on the electrical impedance of mortars containing carbon fibres. This study would provide, using AC impedance measurements, a better understanding of the interaction between sand grain size and volume fraction from one side, with the possibility of carbon fibers to create conductive paths through it.

It compares mortars produced with a Standard sand (well distributed sand) (diameter between 0 and 2 mm) and a finer grain size fraction of the same sand (diameter between 0 and 0.5 mm) with four sand volume fractions: 0, 15, 40 and 50%. Different sand volume fractions cover the various sand structure connectivity status. Real and imaginary impedances would be illustrated, permitting the assessment of of the several interactions in the composite.

2 Materials and experimental setup

Carbon fibers present a length of 6 mm and a diameter of 7 μm . Fibers surface is virgin from treatments. The cement matrix is made of: Cement CALCIA CEMI 52.5 R, Water-to-cement ratio (W/C) =0.4. Superplasticizer BASF MasterGlenium ACE 550 was increased proportionally to fiber's volume fraction to ensure workability, not exceeding 4% of cement weight to avoid bleeding effect. Two sand granulometries tested are illustrated in the figure below.

The standard sand (well distributed 0/2mm sand) is normalised in accordance with EN 196-1. The fine sand was obtained by taking the fraction 0/0.5mm of the standard sand, meaning that both sands have the same nature. The importance of 0/0.5 mm sand in this study is to physically provide a better understanding of changing grain size distribution on the efficiency of conductive fibers.

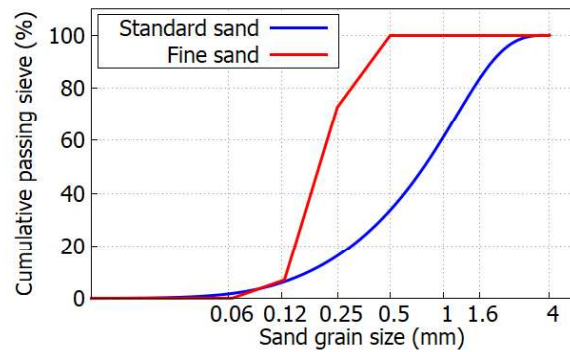


Fig 1: Grain size distribution of two used sands

Sand's volume fraction varies between 0%, 15%, 40% and 50% for both sands. For each of them, 6 carbon fiber contents ranging from 0 to 0.6 % of total volume. This range permits to have a reference mortar (0% fibers), low fibred mortars (0.1% & 0.2%) and High fibred mortar (0.3%, 0.4% & 0.6 %).

A Perrier mixer with 140 rpm was used to mix the components respecting the following procedure: 1 min mix for cement, carbon fibers with sand particles, after water was added with superplasticizer and all the components are mixed for other 2 minutes [18]. Mortar was then poured in a steel mold of 3 samples of 4 X4 X 16 cm. Samples were demolded after 24h then emerged in water for 28 days. They were then kept in stable conditions (20 °C, 50 % RH) for 120 days before measuring their AC impedance. Electrodes were made of copper plates with nickel conductive spray installed on the edges of the samples. AC impedance at 1 KHz was measured using a Hioki impedance analyzer IM3750 [], under a voltage of 0.1 V.

3 Results

Response of fibred cement paste and fibred mortar with different volume fraction of standard sand is illustrated in Fig 2. Real and imaginary impedances at 1 KHz are shown function of fiber's volume fraction. Due to the very high aspect ratio of carbon fibers, the percolation threshold in cement paste is very low and the typical insulative zone below percolation threshold does not show on the graph. The percolating transition zone shows a decrease of real impedance from $10E4$ at 0% FVF to $10E1 \Omega$ at 0.2% FVF. The conductive zone shows a slight decrease of real impedance from $10E1$ at 0.2% FVF to 5Ω . Capacitance decreased from $5*10E3 \Omega$ to 0.03Ω at 0.6 %. The very low value of capacitance above 0.2 % FVF are typical of an almost ideal resistive type behavior.

In presence of a EN 196-1 standard sand volume fraction (SVF) of 15%, real and imaginary impedances were almost identical to the ones of cement paste. With a 40% SVF, sand seemed to reduce the apparent percolation threshold of carbon fibers, with real impedance values of mortar lower than in cement paste below a FVF of 0.3 %. Beyond that point, real and imaginary impedance seemed to remain constant when the FVF was increased from 0.3 to 0.6% surpassing slightly cement paste impedance beyond 0.3% FVF. With a 50% SVF of standard sand, the real and imaginary impedance were higher than for cement paste or mortars with lower sand contents, regardless of the FVF, but followed a similar trend than previously described, with a significant decrease when the FVF was increased from 0 to 0.2 % FVF, while it remained almost constant with FVF ranging from 0.2 to 0.6%.

Real and imaginary impedance of mortar with fine sand measured at 1 KHz are illustrated in Fig 3. Presence of 15% of fine sand volume fraction did not seem to have any significant effect on impedance for all the range of fiber volume fraction. With 40% of fine sand volume fraction, real and imaginary were higher than for cement paste and mortar with a 15% SVF, especially with FVF above 0.3 %, when the SVF of fine sand was 50% of fine sand in mortar, real and imaginary impedance decreased slowly with increasing FVF, with a continuous decrease up to a 0.6% FVF. The real impedance decreased from $10E5$ to $10E3 \Omega$, while the imaginary impedance decreased from $10E4$ to $10E1 \Omega$. In comparison, for the highest FVF of 0.6%, the real and imaginary impedances of cement paste reached values of $10E1$ and $10E-1 \Omega$.

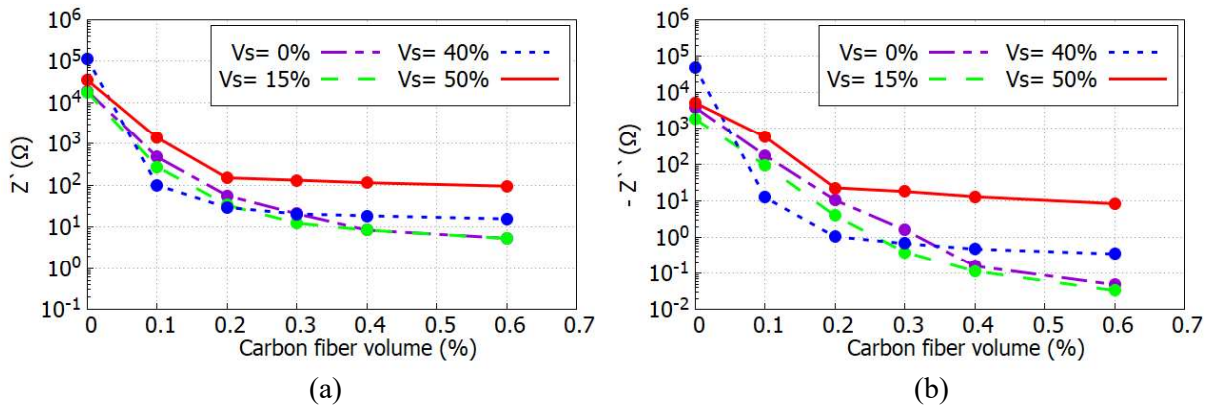


Fig 2: Electrical impedance at 1 KHz function of fiber's volume fraction for different normalized sand volume (a) real part (b) imaginary part

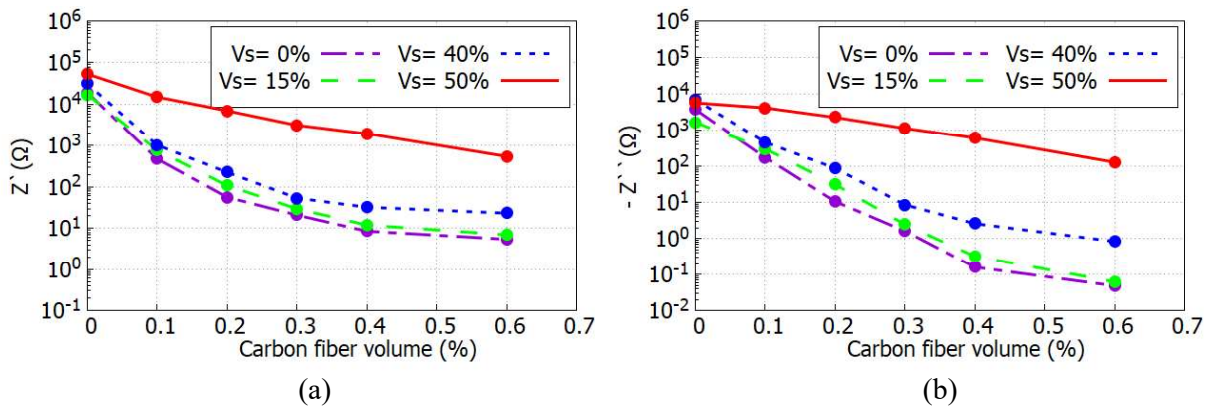
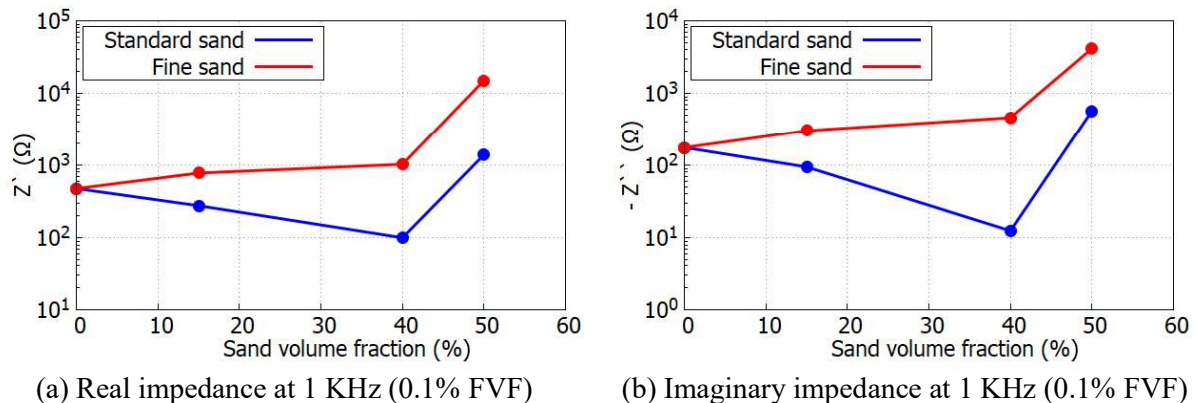


Fig 3: Electrical impedance at 1 KHz function of fiber's volume fraction for different fine sand volume (left) real part (right) imaginary part

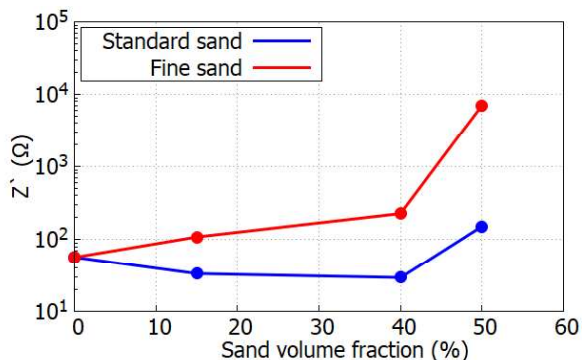
Fig 4. shows real and imaginary impedances of standard sand and fine sand mortars for a given FVF. For all FVF, the real and imaginary impedance of mortars with standard sand were decreasing when the SVF was increased to 15 and 40%, then increasing when the SVF reached 50%. For mortars containing fine sand the real and imaginary parts of impedance increased with an increasing SVF, regardless of the FVF. This increase, however, showed two clear phases, since it was way more significant when the SVF was increased from 40 to 50% than from 0 to 40%.

With 15% (theoretically diluted sand particles), results for both sands are in the same order of magnitude of cement paste as mentioned above. The deviation between mortars impedance with different sands is seen more clearly with 40% of sand volume fraction (theoretically sand volume is partially connected), where especially at low fiber volume fraction, standard sand seems to reduce mortar's impedance; on the other hand, fine sand tends to increase this apparent impedance, the gap is considerable between both sands at low FVF (< 0.2%).

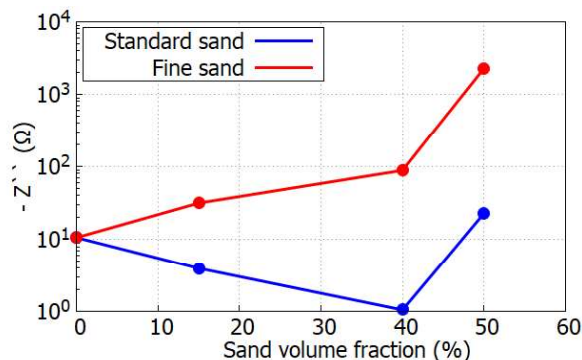


(a) Real impedance at 1 KHz (0.1% FVF)

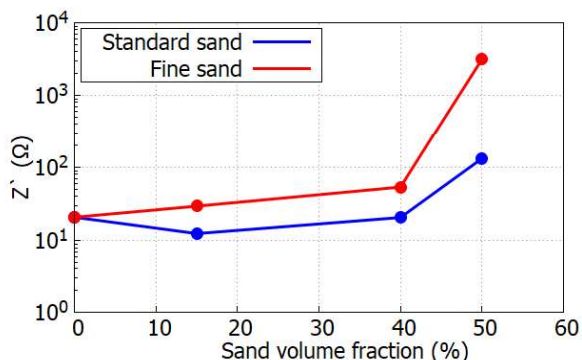
(b) Imaginary impedance at 1 KHz (0.1% FVF)



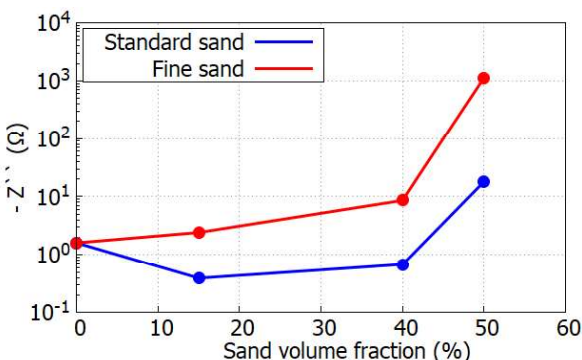
(c) Real impedance at 1 KHz (0.2% FVF)



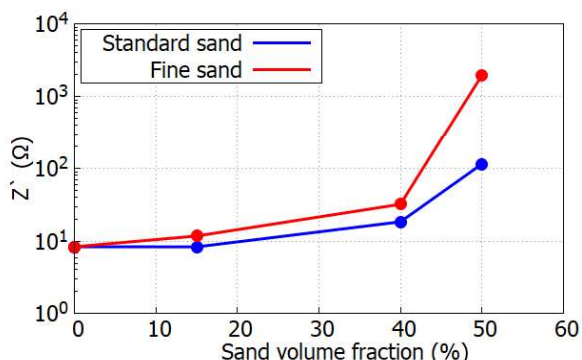
(d) Imaginary impedance at 1 KHz (0.2% FVF)



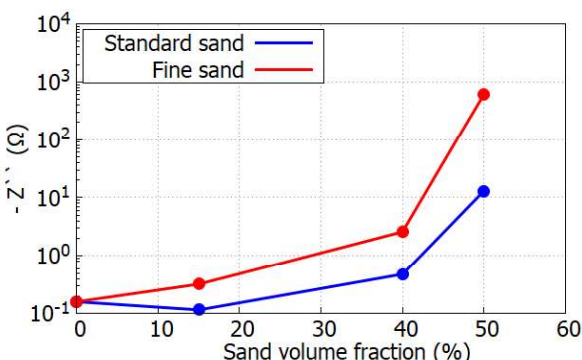
(e) Real impedance at 1 KHz (0.3% FVF)



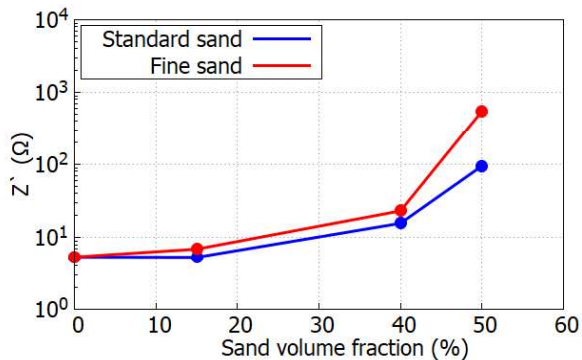
(f) Imaginary impedance at 1 KHz (0.3% FVF)



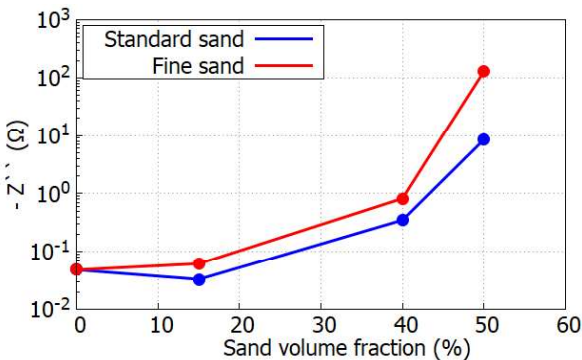
(g) Real impedance at 1 KHz (0.4% FVF)



(h) Imaginary impedance at 1 KHz (0.4% FVF)



(i) Real impedance at 1 KHz (0.6% FVF)



(j) Imaginary impedance at 1 KHz (0.6% FVF)

Fig 4: Electrical impedance at 1 KHz function of sand's volume fraction for well distributed sand and fine sand

4 Discussion

Equations developed in previous studies to estimate percolation threshold base their estimations on fibers morphology [11]. In the case of carbon fibers with a diameter of 7 μm and a length of 6 mm randomly dispersed in a homogeneous matrix, the percolation threshold would be approximatively of 0.14 % according to Celzard [12], and around 0.08% according to Helsing [19]. For cement paste, indeed, those estimations seemed to be relevant since percolation threshold was reached around 0.1 and 0.2% of FVF.

It is however crucial to note that for a given FVF, the relative volume fraction of carbon fibers within cement paste is higher in a mortar than in cement paste. For instance, with a 40% SVF and a 0.2% FVF, the actual volumic proportion of carbon fibers within cement paste would be $0.2/0.6 = 0.33\%$. As a matter of fact, for standard sand, the electrical impedance of mortar with a 40% SVF are lower than for cement paste when the FVF is below 0.33 %. In other words, sand aggregates do not allow carbon fibers to penetrate them and promote the percolation of fibers within cement paste, thus having a beneficial effect on the conductivity of the phase of fibred cement paste within the mortar material. Therefore, the overall electrical impedance of mortar samples was indeed lower than cement paste when the FVF was around the percolation threshold. At higher FVF, however, the insulative nature of the sand aggregates showed a slightly detrimental effect on the overall electrical impedance of the mortar samples.

With fine sand, the presence of sand aggregates did not seem to present any beneficial effect on the electrical impedance of mortars, thus showing that even if the relative volumic proportion of fibres in cement paste was higher, their percolation was not enhanced by the presence of sand at SVF of 15 and 40%.

The results were in clear accordance with the double percolation theory [14], since the only mortar matrices that seemed to hinder the effect of the FVF on the electrical impedance were the ones with the highest SVF of 50 %. This was clear for both standard and fine sand, the latter showing significantly higher values of impedance. Those observations are persistent with previous studies [15][20] stating that the double percolation may not be ensured when the SVF is above the range of 40-50% and sand aggregates might start to be in contact of each other, thus causing discontinuities within the conductive phase of fibred cement paste.

Presence of high standard sand volume fraction limited fibred mortar from reaching low values of impedance, values that were reached with cement paste and mortar with sand volume fraction lower than cement matrix percolation threshold. On the other hand, the effect of high fine sand volume was more radical on continuity of fibers conductive paths, where impedance persisted at high values even with fiber volume fractions that were efficient to reduce drastically impedance of cement paste.

Electrical impedance seemed to be significantly influenced by the grain size distribution as shown in the results above. Reducing grain size interval to finer sand with high volume fraction increased the electrical impedance and slowed percolation phenomenon. Maximum packing density of sand's arrangement inside the mortar could represent one of the key parameters. Experimentally, maximum packing density for both standard (between 0 and 2 mm) and fine sand (between 0 and 0.5 mm) were determined experimentally equal to $\Phi_{c1} = 64\%$ and $\Phi_{c2} = 54\%$ respectively. Mean diameters of both sands were calculated based on grain size distribution showed in figure 1 equal to $d_1 = 744 \mu\text{m}$ and $d_2 = 232 \mu\text{m}$ respectively. Spacing s between particles is calculated based on the assumption that for dense sand presence [21]:

$$\varphi \simeq d^3 / (s + d)^3 \quad (1)$$

Knowing that for $S = 0$, $\varphi = \varphi_c$, mean spacing between sand grains is estimated using Eq. 2:

$$\frac{\varphi}{\varphi_c} = \frac{1}{(1 + s/d)^3} \quad (2)$$

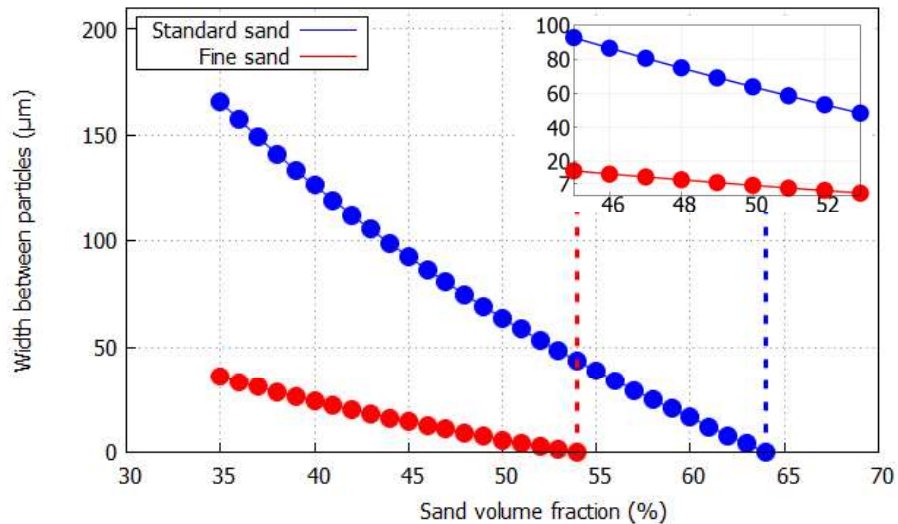


Fig 5: Distance between sand particles function of sand's volume fraction

Reducing sand's distribution interval minimises its maximum packing density. This effect, combined with the smaller diameter of sand particles, reduces the mean paste width between sand particles according to eq (2) and as shown in Fig 5. The reduction of mean distance between particles to small values of few μm s, in case of dense fine sand, would reflect a heavier presence of sand spacing width equal or smaller than fibers diameter. This effect combined with the increase in particles number (in case of fine sand), would multiply the narrow spacings between sand particles. therefore, having negative effect on the continuity of fibred cement paste phase. This effect of hindering the continuity of the conductive phase, more pronounced in case of fine sand, would increase the impedance value of mortar in case of high sand volume fraction. In such case, the presence of sand is expected to have a detrimental effect on the homogeneity of fiber dispersion within the cement paste phase by creating a strong disparity in the local volumetric concentration of fibers within cement paste

Further publications will be focused on showing the Nyquist plots of mortar with different sand volume fraction and different grain size distribution, and their sensing behavior under mechanical loads.

5 Conclusion

Fibers prove efficiency as doping agents to reduce resistivity by a factor of $10E4$ when the double percolation is ensured (percolation of fibers within cement paste and percolation of cement paste within mortar) in presence of enough conductive volume fraction to create conductive paths inside mortar. Percolation threshold of carbon fibers within a homogeneous matrix was estimated between 0.1 and 0.2% by percolation theories [12][19] and were in accordance with experimental results with cement paste.

Fibers efficiency was confirmed to depend on hosting matrix. Increasing sand's volume fraction proved significant influence on fibers percolation. This influence dependend on the grain size distribution of sand, as well as its volume fraction. For SVF up to 40%, the standard sand had a beneficial effect on the electrical impedance at FVF below 0.3%, and a very slight detrimental effect for FVF above 0.3%. For fine sand, no beneficial effect of sand aggregates on the impedance was observed. For both sands, the impedance of mortars with a 50% SVF was way higher than for cement paste, thus confirming the relevance of the double percolation theory [14].

The effect of grain size distribution of sand was illustrated. Finer sand proved higher impedance, i.e., less continuous conductive paths compared to cement paste, causing a slow decrease in resistivity function of carbon fibers. This could be related to its arrangement closer to monodisperse, reducing maximum packing density, and consequently for a given sand volume fraction, decreases mean spacing between sand particles. This effect, combined with the higher number of sand particles, could potentially cause more considerable discontinuities in fibred cement paste and consequently higher impedance values. The sand aggregates might have a negative effect on the dispersion of fibers within cement

paste phase, as well as creating insulative blocking points within the network of fibres, therefore mitigating the expected beneficial effect of the percolation of fibers on impedance reduction.

Further work is needed to study sand's effect on fibers tortuosity, that could have significant effects as well, especially with high aspect ratio fibers as well as as a better understanding of effect of fine sand effect on porosity of cementitious matrix.

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