MAGNETIC ORIENTATION OF SISAL AND STEEL FIBERS IN CEMENTITIOUS MATRICES: EXPERIENCES OF SÃO JUDAS TADEU UNIVERSITY

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Abstract. This work presents results of experimental investigations focused on the magnetic orientation of steel and sisal fibers embedded in cementitious matrices. The work addresses the development of magnetic alignment methodologies and their influence on the mechanical and electrical properties of the resulting cementitious composites. In the first phase, the materials used in the research are presented, as well as two methods for magnetic alignment of fibers: neodymium magnets coupled to a computer-controlled industrial robot and a simplified magnetic circuit using a pair of coils wrapped in a ferromagnetic core. The second phase focuses on the analysis of the electrical and mechanical properties of the composites and assessment of the fiber orientation and positioning. X-ray computed tomography, electrical resistivity and bending tests were used in this phase. The results showed that the projected magnetic systems were able to generate a preferential orientation of the fibers, increasing the overall mechanical performance of the composites and generating anisotropy on their electrical properties.

Keywords: magnetic alignment, fiber orientation, mechanical behavior, electrical properties.

1 Introduction

Cementitious composites with aligned fibers are a new generation of materials that present significant improvements with relation to the mechanical performance [1]. The fibrous reinforcement, when aligned favorably with respect to the element tensile stresses, is able to provide the fragile matrix large post-cracking ductility, as well as generous increases in the load carrying capacity [2].

The main factors capable of changing the orientation of the fibrous reinforcement are: molding conditions (pouring processes, restrictions, formwork design, etc), consolidation procedures, gravity effects, fiber typology, and rheological properties of cementitious matrices. Above all, it is worth to mention the "presence of magnetic fields", focus of the present study.

Magnetic fields, in general, have a great effect on the orientation and distribution of steel fibrous reinforcement in a cementitious matrix [1,3,4]. As revealed by Mu et al. [4], the fibrous reinforcement is oriented along the magnetic lines, being affected mainly by the intensity of the field and rheological characteristics of the matrix.

Hybrid processes combining vibration and magnetic orientation have also shown promise [1,5,6]. Mu et al. [6] produced SFRC pipes using a solenoid (magnetic field with induction around 5×10^{-2} T) coupled to a vibration table (40 s). The orientation factors ($\eta\theta$) in the aligned case showed to be greater than 0.9 for all produced pipes (Vf: 0.8%, 1.2% and 1.6%). In the random case, however, it remained below 0.68. Fiber count also revealed that pipes with aligned fibers had about 24% more fibers in the fractured section than the random ones.

In this context, this research aims to present the influence of the magnetic alignment of steel and sisal fibers on the mechanical and electrical properties of different cementitious composites produced at São Judas Tadeu University. The results showed that the designed magnetic systems were able to generate a preferential orientation of the fibers, increasing the overall mechanical performance of the composites and generating anisotropy in their electrical properties.

2 Materials and methods

This item, as well as the others, will be divided between data referring to the study of the magnetic orientation of sisal fibers (with ferromagnetic coating) [7] and steel fibers [1], both carried out at Universidade São Judas Tadeu.

2.1 Components of the matrix

For the case of composites with sisal reinforcement [7], the employed matrix was produced from a High Early Strength Portland Cement CPV ARI (from Liz Cimentos Co.) and limestone filler (from Hocim Co.) using the following mass formulation: 1:1:1 (cement: sand: water). Such matrix, common in fiber cements, was produced with the aim of ensuring enough workability to provide the torque of the fibers under the action of the magnetic field.

For the composites reinforced with steel fibers, however, a high-performance matrix was used. The cementitious materials employed in this case were a commercially available high-early strength Portland cement (CEM I 52.5R), silica fume (920U from Elkem Co.) and metakaolin (Metacaulim HP ULTRA from Metacaulim do Brasil Co.). The choice of using two pozzolans was based on particle sizes, seeking to improve the packing density of the mixture. A quartz sand (Brasilminas Co.) with particle size distribution ranging from 0.075 mm to 2 mm was used as aggregate. A polycarboxylate superplasticizer (Adiment Premium from Vedacit Co.) with 30% of solid content was employed to provide adequate workability to favor magnetic alignment of the reinforcement within the matrix. The water/cementitious material ratio of the matrices was kept at 0.3. More details about the composition of the matrix can be found in the work of Brito et al [1].

2.2 Fibrous reinforcement

The sisal fibers used in the work of Frietas [7] were supplied by Sisal Sul Co. in packs of 1 kg and approximately 1 m long. Before mixing, all sisal fibers were subjected to an untangling process (Figure 1-a), giving rise to the fibers called, in this study, "in natura" (Figure 1-b). After this stage, part of the fibers was subjected to a surface treatment using an epoxy-based adhesive and metal cutting residue (Figure 1-c and Figure 1-d).

Figure 1. Surface treatment applied to the sisal fibers: (a) Untangling process, (b) fibers “in natura”, (c) adhesion of ferromagnetic residue to fibers and (d) treated fibers before cutting.

The length of the fibers and the fiber volumetric fraction (Vf) used in all composites were kept, respectively, at 10 mm and 0.56%. For surface treatment, only ferromagnetic particles smaller than 300 µm were used. This material was obtained through sieving and magnetic classification of metal cutting waste.

The adhesion of the ferromagnetic material to the reinforcement was performed through an epoxy adhesive applied manually to the surface of the fibers. After coated with adhesive, the fibers were gently covered with metallic particles for about 1 min. Once the adhesive was dry (~ 4h), the fibers were cut and packaged for later use as fibrous reinforcement. The mass ratio between ferromagnetic material (mFe) and fiber mass (mf) used in the surface treatment was ~11.

Analogously, the composites produced by Brito et al [1] were reinforced with short straight steel fibers (from Astra Co.) with tensile strength of 1100 MPa, elastic modulus of 210 GPa and specific weight equal to 7850 kg/m^3. The length, diameter and aspect ratio of the fibers were 12.5 mm, 0.5 mm and 25, respectively. The fiber volumetric fractions (Vf) used in the composites were 1%, 2%, 3% and 5%.

2.3 Nomenclature of the composites

The nomenclature and specific characteristics of each produced composite reinforced with sisal are presented, respectively, in Table 1 and Table 2. In the adopted nomenclature, the following abbreviations are used: CC for cementitious composite, S for sisal fiber, IN for in natura, RA for randomly arranged, T for treated (fibers with surface treatment) and OR for oriented.

Table 1. Nomenclature of the produced composites [7].

Nomenclature	Composite features
Matrix	Cementitious matrix without reinforcement
CC_S_IN_RA	Cementitious composite reinforced with sisal fibers (*in natura*) randomly arranged
CC_S_T_RA	Cementitious composite reinforced with treated sisal fibers randomly arranged
CC_S_T_OR	Cementitious composite reinforced with treated and oriented sisal fibers

Table 2. Fiber content present in each type of composite per specimen [7].

Nomenclature	Vf (%) *in natura*	Mass of fibers (g)	Mass of fibers + surface treatment (g)
Matrix	0	0	0
CC_S_IN_RA	0.56	0.78	-
CC_S_T_RA	0.56	0.78	15
CC_S_T_OR	0.56	0.78	15

In the study of Brito et al [7], five HPFRCC were produced (Table 3). One of them was the control mixture (M0), which did not contain steel fibers, and the additional four mixtures contained fiber volume fractions equal to 1%, 2%, 3% and 5% (M1, M2, M3 and M5, respectively). For comparative purposes, mixtures containing fibers were produced with random (RA) and aligned (AL) reinforcement. The nomenclature adopted to identify the mixtures is ‘Mfiber volume’_‘spatial arrangement of fibers’.

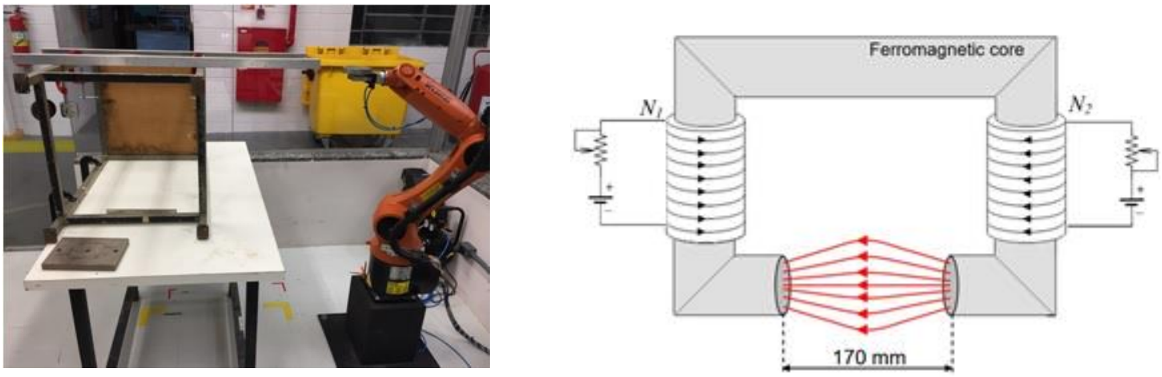
Table 3. Mix composition of the cementitious matrices analyzed [1].

Constituent (kg/m³)	Mixtures				
	M0	M1	M2	M3	M5
Cement	599.2	593.2	587.2	581.2	569.2
Metakaolin	257.6	255.1	252.5	249.9	244.7
Silica fume	65.9	65.2	64.6	63.9	62.6
Sand	1315	1302	1289	1276	1064
Water	179.7	177.9	176.1	174.3	170.7
Short straight steel fiber	0	78.5	157	235.5	392.5
Superplasticizer	25.7	25.5	25.2	24.9	24.4

2.4 Methods for fiber orientation

As reported on Table **1**, the composites reinforced with sisal were developed with and without magnetic fiber orientation. In order to guarantee uniformity in the production of the composites, the alignment of the fibers was carried out by neodymium magnets coupled to a computer-controlled industrial robot (see Figure 2 -a). The fiber alignment technique proposed in this study was based on the movement of the neodymium magnets under the molds (acrylic molds of 240 mm x 240 mm x 13 mm). For this, two neodymium magnets (model N50), supplied by New Imãs Indústria e Comércio Co., were used. The alignment process was controlled by a KUKA industrial robot, model KRC 900. The trajectory of the magnets was directed along the largest dimension of the specimens (240 mm). More details about the composite preparation and sisal alignment can be found in the study of Freitas [8].

The alignment of steel fibers within the matrix (mixtures AL) was conducted using the magnetic circuit presented in Figure 2 - b. The fresh cementitious matrix containing steel fibers was cast into acrylic molds measuring 40 mm x 40 mm x 160 mm (width x height x length). The casting process was carried out from the center of the molds leaving the mixtures free to flow towards the ends. After casting, molds were placed on a vibration table and positioned in the air gap of the circuit. Right after that, vibration (60 Hz) and magnetic field were simultaneously applied during 40 s. The magnetic field was generated with an electric current equal to 12 A.



(a) Freitas [7]

(b) Brito et al. [1]

Figure 2. Methods for fiber orientation: (a) Industrial robot coupled to neodymium magnets and (b) magnetic circuit containing two coils and a ferromagnetic material as nucleus (red line represents the spreading of the magnetic flux).

2.5 Experimental campaign

For the case of composites with sisal reinforcement [7], four-point bending tests were carried out in an EMIC universal testing machine, with 200 kN capacity, at 28 days of age. The span used between supports and loading point was respectively, 210 mm and 70 mm. The tests were controlled by the actuator displacement at a rate of 1 mm/min. The deflections of the plates at the mid-span were measured using an EMIC displacement transducer, up to 12 mm.

In addition, X-ray images were taken of all produced composites (largest specimen area, 240 mm x 50 mm) to assess the fiber orientation. The machine was adjusted for chest evaluation, with exposure time t = 0.05 s, at a current of 200 mA (milliampères) and a voltage of 68 kV. To determine the fiber orientation factor along the specimens, radiographic images of the composites CC_S_T_RA and CC_S_T_OR, were treated using the software AutoCAD. The angle established between the fiber and the magnets direction was then determined. The fiber orientation factor was calculated to be 0 for angles equal to 90° and 1 for angles equal to 0°.

For the study employing stell fibers [1], all specimens were subjected to three-point bending test at the age of 16 days. Tests were performed in a Kratos universal testing machine, model KE 20000MP, coupled to a 200 kN load cell. Prismatic specimens were supported by two steel cylinders (Ø 10 mm) 100 mm distant from each other. Specimens were loaded at midspan by another steel cylinder (Ø 10 mm) at a rate equal to 0.5 mm/min until the total displacement of 4 mm was reached. Load application rate was controlled by the actuator displacement. The toughness of all composites subjected to the three-point bending test was calculated as the total area under the load-displacement curve up to 4 mm.

Using the same specimens, two other techniques were adopted: X-ray computed tomography and electrical resistivity. X-ray computed tomography (XCT) was performed in hardened specimens of the composite M5_AL to assess the distribution and positioning of steel fibers along its volume. XCT imaging was conducted in a Carl Zeiss tomograph model METROTOM 800 (130kV). A current of 200 µA was employed while the integration point, Voxel and spot were equal to 500 ms, 56,06 µm and 40 µm, respectively. The 360 ° rotation images were then reconstructed to obtain the 3D volume.

Finally, the equipment used to determine electrical resistivity was the Resipod from PROCEQ Co, in the range from 1 kΩcm to 1000 kΩcm using a frequency of 40 Hz. The electrical resistivity was measured in all faces of the specimens (three per mixture) produced for flexural tests.

3 Results and discussion

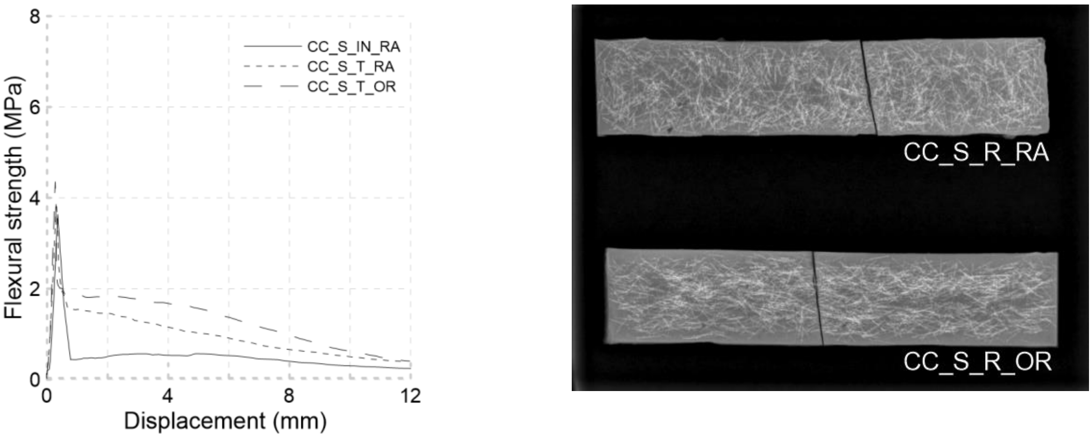
Figure 3 - a presents one typical curve obtained in the four-point bending tests for each composite produced with sisal reinforcement. MOR and toughness data of all composites can be found in Freitas [7]. All composites reinforced with sisal fibers presented a deflection softening behavior with a single crack formation. This behavior is in great part related to the low Vf employed as reinforcement on the studied composites (0.56%).

The lowest flexural performance was observed for the composite CC_S_IN_RA. This pattern results from the random orientation of the reinforcement as well as the characteristic low adhesion, and consequent interfacial debonding, occurred between natural sisal fibers and matrix under tension [8]. The hydrophilic nature of the sisal reinforcement is largely responsible for the latter effect. As a result, low post-cracking performance is observed.

The composite CC_S_T_RA, presented an increase of more than 80% on the accumulated toughness (up to 12 mm) when compared to the reference (CC_S_IN_RA). Such result indicates that the treatment using ferromagnetic particles was capable to improve fiber-matrix interaction. In addition to the obvious increase in specific surface area provided by ferromagnetic particles, the epoxy coating ends up shielding the sisal reinforcement, generating a water absorption 87% lower in the coated fibers. In this way, not only the adhesion characteristics are improved, but probably the drying shrinkage of fibers is reduced.

The highest flexural behavior was observed for the composite CC_S_T_OR with magnetically oriented fibers. The toughness computed as the total area under the load displacement curve showed to be, respectively, 1.2 and 2.2 times greater than that observed for the composites CC_S_T_RA and CC_S_IN_RA. This behavior results from the improvements related to fiber-matrix interaction and also the reinforcement orientation, clearly observed through the radiographic images presented on Figure 3.

In case of CC_S_T_OR, a preferential fiber orientation was clearly noticed (Figure 3 – b). Considering all specimens, the average orientation factor for the composite CC_S_T_OR was 0.794. In contrast, the composite CC_S_R_RA showed an average orientation factor of only 0.316.



(a) (b)

Figure 3. Results obtained for composites reinforced with sisal: (a) typical stress-displacement curves and (b) radiographic images of CC_S_T_RA and CC_S_T_OR specimens.

Moving on to the study with steel fibers [1], Figure 4 illustrates the distribution of the fibers along the volume of a M5_AL specimen after exposure to the magnetic field and vibration. As expected, fibers remained aligned in the direction of the magnetic flux. However, a clear concentration of fibers close to the edges of the air gap (point of maximum intensity of the magnetic field) and in the lower third of the specimen (bottom face) is observed.

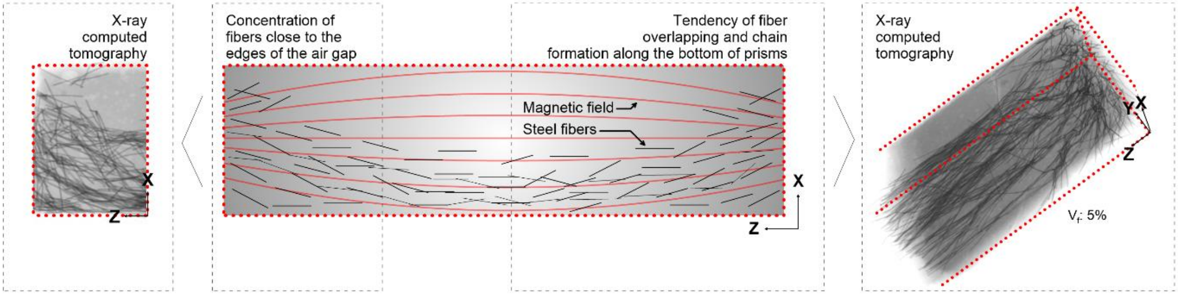
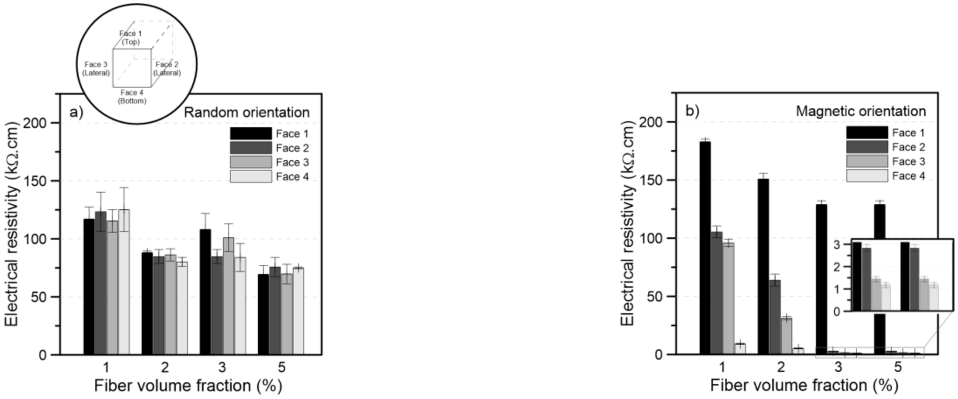


Figure 4. XCT images and simplified scheme of the fiber positioning along the volume of prisms [7].

The fibers under the action of the magnetic flux are magnetized, and may be considered as magnets, magnetic dipoles, which tend to align with the magnetic flux and suffer an attraction or repulsion among themselves. Such process results in a tendency of fiber overlapping and chain formation. As fibers are 3 times denser than the matrix, fiber magnetization allied to the gravity action, vibration process and wall effect, leads the fibers to the bottom of the molds.

Figure 5 depicts the results of the electrical resistivity of random (Figure 5 - a) and aligned (Figure 5 - b) mixtures as a function of the specimen faces.



(a) (b)

Figure 5. Electrical resistivity as a function of the faces of the specimens: (a) random orientation and (b) magnetic orientation.

A trend of reduction of the electrical resistivity with the magnetic alignment is observed. Specimens containing randomly oriented fibers present statistically equal values of electrical resistivity in all specimen faces (Figure 5 - a). Such isotropic characteristic demonstrates a uniform fiber distribution along the prismatic specimens containing 1%, 2%, 3% and 5% of fibers. In these specimens, a trend of reduction of the electrical resistivity with increasing fiber volume was observed.

Specimens containing aligned fibers present the opposite behavior (Figure 5 - b), and significant resistivity reductions are observed for the side and bottom faces of the specimen, which are regions with higher concentrations of fibers. As the top face of the specimen has few fibers (see Figure 4), the electric current is transported only by the ions in the matrix pore solution, increasing its electrical resistivity. The bottom face has the highest amount of fibers, as evidenced by XCT images (Figure 4). This is due to the dipole-dipole interactions that promote fiber agglomeration, leading the fibers to the bottom of the molds. In this situation, the bottom face is rich in metallic fibers, which reduces its electrical resistivity and makes it more conductive than the other faces.

Figure 5 shows typical stress–displacement curves obtained from the flexural tests grouped by fiber content. The red dashed curves represent the flexural behavior of mixtures with aligned fibers (mixtures AL). The continuous black curves represent the specimens with randomly oriented fibers (mixtures RA). The curves obtained indicate a deflection softening behavior for the mixtures M1_RA, M1_AL, M2_RA, M2_AL and M3_RA. All other composites presented a deflection hardening behavior. MOR and toughness data of all composites can be found in Brito et al. [1].

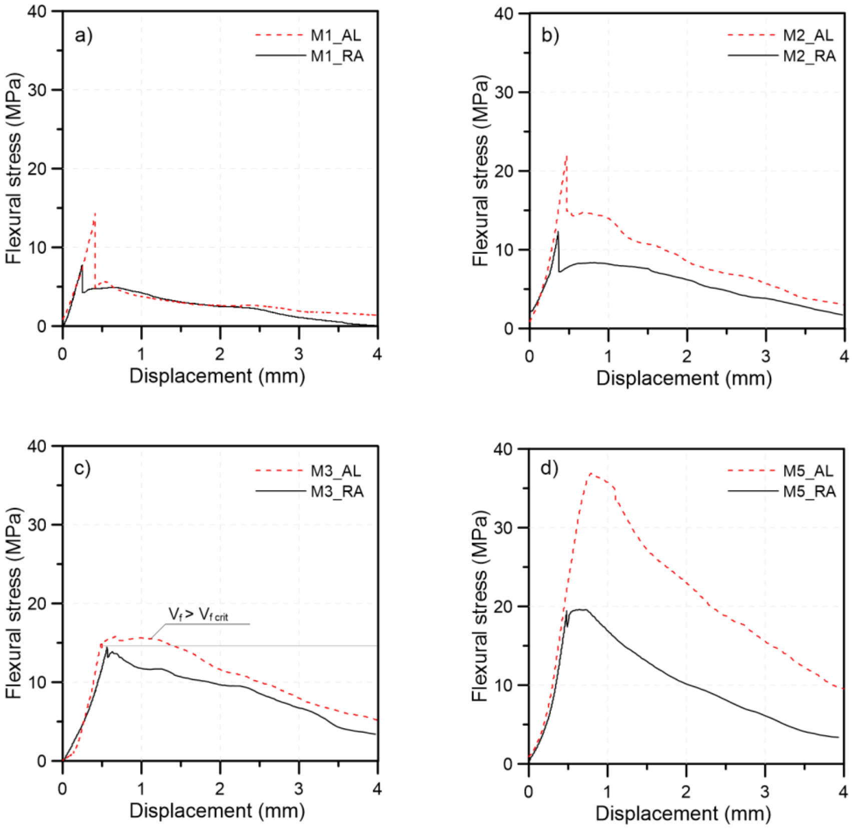


Figure 6. Typical stress-displacement curves for HPFRCC specimens reinforced with 1% (a), 2% (b), 3% (c) and 5% (d) of fibers (AL and RA).

As may be observed, the flexural performance in terms of MOR and toughness are higher in mixtures AL than in mixtures RA for all the fiber contents evaluated. This behavior was more pronounced for the composite M5_AL, which presented increases of MOR and toughness of, respectively, 72.6% and 114.6% in comparison to the M5_RA. It is likely that, in the case of the mixture M1_RA, the incorporation of random fibers has impaired the matrix compaction, generating MOR values lower than those observed for M0. It is also important to remark that fiber orientation was capable to reduce the critical fiber volume for bending, which may be clearly observed on Figure 6 - c.

These large increments obtained with magnetic alignment reflect the preferential orientation of the reinforcement, as well as the fiber concentration in part of the tensioned region of the prisms. It is important to remark that specimens were rotated 90° in relation to the casting direction before the bending tests. That is why the emphatic note that the fibers are deposited “in part” of the tensioned region of the prisms.

Figure 7 presents the Hoerl fitting to correlate toughness with the fiber volume employed for the HPFRCC production. It is possible to observe that the magnetic alignment leads to a remarkable reduction in the fiber volume necessary to reach a certain value of toughness. For example, using 2% and 3% of random fibers, toughness values of around 9.5 J and 13.2 J are obtained. In the case of specimens with aligned reinforcement, same toughness values are obtained with around 1.7% and 2.2% of fibers respectively, reducing the need of Vf in 15% and 26%.

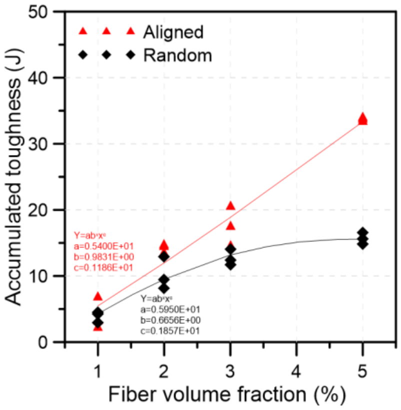


Figure 7. Relation between accumulated flexural toughness and fiber volume fraction for HPFRCC.

Another interesting point is that the toughness enhancement for an increase of 1% in fiber volume is significantly different depending on the orientation method. Comparing the average toughness gain between the M2 and M3 for both cases, RA and AL (data in Brito et al. [1]), is it possible to note values practically 30% grater for the AL case. This fact results from the greater amount of fibers crossing the fractured section in the AL case. Studies reported by Abrishambaf et al. [9] show that for composites reinforced with 3% of steel fibers (AL and RA), the amount of fibers/cm² in the fractured section of the specimens can be almost 50% higher in the case of oriented fibers.

It is believed that the greater distance between the toughness fitting lines RA and AL for higher fiber volumes of 3% and 5% is directly related to the dipole-dipole interactions. As reported by Wijffels et al. [10], the shorter the distance between the fibers, the greater the tendency of fiber overlapping and chain formation due to the dipole-dipole interactions. Therefore, higher concentrations of fibers will result in more fiber chains (i.e.: increased orientation factor), decreasing the randomness of the reinforcement and consequently increasing toughness. The reductions of the electrical resistivity in the AL specimens confirm these observations.

4 Conclusions

The following conclusions may be drawn from the experimental campaign on magnetic orientation of sisal and steel fibers conducted at São Judas Tadeu University:

- The magnetic alignment methodologies employed for the cementitious composites were capable to generate a preferential orientation of fibers.
- The magnetic orientation of the sisal fibers and the treatment using epoxy adhesive and ferromagnetic particles was capable to improve the flexural performance of the studied composites. The stress-displacement curves confirm this observation.
- The dipole-dipole interactions stablished between steel fibers resulted in fiber overlapping and chain formation along the bottom face of the specimens. In addition, a clear concentration of fibers occurred close to the edges of the air gap (point of maximum intensity of the magnetic field). The XCT analyses confirm these observations.
- As evidenced by the XCT analysis, the concentration of fibers at the extremities and bottom of the specimens containing aligned fibers resulted in a trend of reduction of the electrical resistivity on their side and top faces. However, the RA specimens presented isotropic characteristics, once similar values of electrical resistivity were obtained in all faces evaluated.
- Steel fiber alignment was capable to reduce the critical fiber volume for bending. The stress-displacement curves obtained for the composites containing 3% of random and aligned fibers confirm this observation.

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