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Article

Influence of an Impregnation Treatment on the Morphology and Mechanical Behaviour of Flax Yarns Embedded in Hydraulic Lime Mortar

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Abstract: The increasing attention toward environmental aspects has led, also in the sector of construction materials, to the need for developing more eco-friendly solutions. Among several options, the employment of low energy raw materials appears as an efficient solution intended to enhance the sustainability of building structures. One of the applications moving in this direction is the use of plant fibers as a reinforcement in cement-based composites, hence named as natural textile reinforced mortar (NTRM) composites. Although representing a promising technique, there are still several open issues concerning the variability of plant fibers properties, the durability, and the mechanical compatibility with the mortar. This study aims at investigating the influence of an impregnation process on the thread's morphology and on the mechanical response. Therefore, the geometry of dry and impregnated flax threads is identified by using scanning electron microscope (SEM) images analysis, and their mechanical response in tension is assessed. In addition, the fibers-to-mortar bond behavior is investigated by means of pull-out tests. The proposed results show that the impregnation procedure employed, although not improving the fibers-to matrix bond, leads to a standardisation of the threads morphology and reduces the thread's deformability in tension, and paves the way for further investigations on a larger scale.

Keywords: plant fibers; flax fibers; natural composites; pull-out test

1. Introduction

The reduction of environmental impact represents one of the major challenges of our time that interests all the industrial sectors of our society [1]. In civil engineering, greater attention is being devoted to developing innovative procedures that, starting from the choice of raw materials and continuing with smart disposal techniques at the end of the structure's lifetime, may contribute to a more eco-friendly development in line with the main principles of a "circular" economy [2].

As for building materials, several strategies have been implemented with the aim of promoting life cycle assessment methodologies [3], the use of recycled sources [4,5], and, more generally, the adoption of "low embodied energy" raw materials [6]. With respect to the latter aspect, the use of plant fibers instead of industrial ones as reinforcement in composite systems appears to be an efficient solution to increase the sustainability [7]. Because of their mechanical, physical, and economical properties [8], the so-called bio-composites materials have gained a broad application field [9–11] in several sectors, such as the automotive [12], packaging [13], biomedical [14], infrastructural [15], and civil engineering fields [16].



The use of textile reinforced mortar (TRM) composite systems, conceived by embedding a textile in cementitious matrices, has gained a great deal of attention from the scientific community in the last decade [17–19], and nowadays represents one of the most efficient solutions to retrofit existing buildings. Aiming to improve the sustainability of these systems, several research studies focused on the use of vegetable textiles as reinforcement in cement-based composites, hence named as natural TRM [20,21], by adopting many types of plant fibers such as flax [22], curauà [23], sisal, jute [24], coir, and hemp [25]. These composites, although representing a promising solution, mainly due to the good mechanical properties of the fibers [26], still present several issues concerning the variability of the properties of the fibers themselves [27], the durability [28], and, more generally, the mechanical compatibility between fibers and inorganic matrix. Due to the uncertainty of plant fibers geometry, a morphological study is essential as a preliminary identification of both filaments and yarns. An efficient technique to deeply investigate these aspects consists in the analysis of scanning electron microscope (SEM) images [29].

Several studies have been carried out in order to analyze the fiber-to-matrix bond behavior by investigating the pull-out response of both plant fibers filaments [30] and yarns [31] embedded in mortar elements, or by putting tension in larger composite coupons [32]. As a matter of fact, the large deformability of plant fibers and their adherence with cementitious mortars appears to be the weak aspects influencing the whole mechanical behavior of the composite systems. In order to face these issues and to improve the composite system mechanical response, it is possible to apply fiber treatments mainly to hornification [33], alkali treatments [34], and coating procedures [35]. These treatments, specifically designed, may improve either the strength of the fibers and/or their durability once immersed in aggressive environments, as they are the inorganic mortar-based matrices.

This study aims to investigate the effect of flax fibers impregnation on their morphology, tensile strength, and mortar-to-fibers adherence behavior. To this purpose, flax yarns, extracted from a textile and coated by means of a polymeric material, are subjected to tensile and pull-out tests, and their response, compared with dry flax threads, is discussed, taking into account the yarns that were geometry assessed by means of scanning electron microscope (SEM) image analysis.

It represents a preliminary step to observe the influence of the fiber treatment on the local behavior at the fiber-to-matrix interface of a textile reinforced mortar (TRM) composite system characterized by a hydraulic lime-based matrix and flax fabric as reinforcement. Therefore, the study paves the way for further investigations performed on larger scales of analysis to verify the efficiency of the treatment on the behavior of the TRM composite system itself, and the mechanical response of structural systems strengthened by means of the application of TRMs reinforced by means of flax fabrics.

2. Materials and Methods

The flax fibers and the hydraulic lime mortar employed in the research study represent the components of a TRM composite system conceived to be applied on existing structures to enhance their strength. The methods employed, which are shown below, have been designed to analyze the bond behavior between the fibers and the mortar matrix.

2.1. Matrix

A hydraulic lime-based matrix has been employed for the production of the specimens as, in recent years, several studies have demonstrated the efficiency of using these kinds of binders for producing composite systems to be implemented as reinforcement in civil engineering applications [36]. Specifically, the pre-mixed mortar used in this research, labelled FIDCALX NHL5[®] [37], is commercialized by the Italian company "Innovation s.r.l." (Corciano, Italy) [38]. It consists of a pre-mixed mortar with pure natural hydraulic lime binder and selected inert materials with a maximum granulometry of 2.5 mm. Figure 1 shows its grain size distribution curve, obtained by means of a vibrating test sieve machine for the grains greater than 0.074 mm [39], and by means of a laser diffraction particle sizing machine for the finest particles.





Figure 1. Grain size distribution for lime-based matrix.

The mortar has been produced by mixing an amount of water equal to 19% (in weight) of the pre-mixed formula of binder and aggregates, as recommended by the manufacturer [38]. The water-to-binder ratio of the mixture is equal to 0.60. It is characterized by a consistency at the fresh state of 230 mm, according to the EN 1015-3 [40]. The mechanical performance of the hardened mortar was evaluated at 28 days, in accordance with the EN 191-1 [41] on prismatic samples exhibiting a flexural and a compressive strength equal to 2.75 MPa and 6.50 MPa, respectively.

2.2. Flax Fibers

Flax fibers are considered as one of the most effective plant fiber types, which have the potential to replace industrial fibers as reinforcement in composite systems in the construction sector due to their mechanical properties [42]. Table 1 summarizes the main physical and geometrical properties of the fabric employed. Herein, it is commercialized by the "Innovation s.r.l." company labelled FIDFLAX Grid 300 HS20[®] [43].

Property	Mean	Co.V. (%)		
filament diameter (mm)	22.52	76.00		
density (g/cm^3)	1.19	3.29		
linear density (Tex)	302	15.27		
n threads/cm	4.3	-		
thread cross-section (mm ²)	0.28	18.25		

Table 1. Flax textile properties. Co.V.: Coefficient of variation.

The plant fiber yarns are extracted from the textile consisting of a bi-directional woven flax fabric with plain wave, conceived to work as reinforcement in mortar-based composite systems. The yarn consists of two minor flax bundles twisted around each other that, in their turn, are a combination of filaments. As the filament is the elementary unit by which the textile is made, it is important to define its geometrical and mechanical properties in order to compare the material with other plant fibers used in similar applications [44].

Firstly, an estimation of the cross-section area of the filament was carried out by analyzing a scanning electron microscope (SEM) image representing the transversal section of a yarn. The mean

value of the cross section, derived by the measurement of 60 filaments, is equal to 698.30 μ m² with a coefficient of variation of 79%.

In addition, since one of the main issues related to the use of natural fibers in cement or lime-based composites is their adherence with the matrix, a carboxylated styrene butadiene rubber (XSBR, NITRIFLEX S/A, São Paolo, SP, Brazil) latex was used to coat part of the yarns under investigation. As a matter of fact, it has been demonstrated that the copolymer, largely used in the textile industry both with natural and synthetic fibers, can improve the bond between plant fibers and the surrounding matrices [30]. Specifically, the flax yarns were cut from the fabric (Figure 2a) and fully immersed in the polymer in environmental temperature conditions (Figure 2b), then the excess liquid was removed from the external surface (Figure 2c) and the treated fibers were dried at a controlled temperature of 38 ± 2 °C for 24 h. During the drying period, the yarns were arranged in a frame specifically studied to give them an elongated and regular shape (Figure 2d).



Figure 2. Fibers impregnation procedure: (**a**) flax yarn; (**b**) yarn immersion; (**c**) embedded yarn; (**d**) yarns drying.

Although no specific investigations have been carried out in this regard, it is worth emphasizing that further analysis is required for analyzing the XSBR coating sustainability impact of the entire system. However, as a first step, it is fundamental to quantify the benefits of the impregnation in terms of mechanical properties. Further durability and life-cycle assessment analysis may clarify if the eventual gap, in terms of environmental impact, is offset by the mechanical benefits, and if more sustainable coating materials may be employed.

2.3. Methods

The experimental program carried out at the laboratory NUMATS–COPPE of the Federal University of Rio de Janeiro was mainly conceived to investigate the adherence between the fibers and the hydraulic lime mortar by also considering the influence of the yarn impregnation.

As a preliminary step, a morphological analysis of the threads was carried out to get information about the yarn geometry [45]. Then, the single filaments constituting the thread were tested in tension to get an estimation of the flax tensile strength. Consequently, the strength of the entire yarn was assessed by means of tensile tests on both untreated and coated specimens. Finally, aiming to study the influence of the coating on the yarns-to-matrix bond, pull-out tests were carried out by taking out both untreated and treated yarns from mortar cylinders [30].

2.3.1. Morphological Identification

Concerning the morphological characterization of the flax yarns, high resolution SEM images were detected to analyze the cross section of both dry and impregnated fibers with a magnification of $60 \times$ and $80 \times$, respectively. All the samples investigated were extracted from the edge of the specimens subsequently subjected to mechanical tests, so that a value of the transversal area could be associated to each one of them. Specifically, 30 SEM images were analyzed concerning the non-treated yarns and 30 others concerning the impregnated ones.

The presence of voids within the yarns, due to the chaotic arrangement of the filaments, makes it difficult to get an accurate estimation of the threads cross section area. Consequently, an image manipulation software was used to analyze the SEM images. The contrast of each picture was modified so that a black-and-white image was obtained, in which the white part represented the filaments section. An estimation of the yarn cross section area was obtained by converting in square millimeters the number of white pixels contained in the modified picture. Moreover, by tracking the border of the white region, an estimation of the actual perimeter of each yarn was obtained as well (Figure 3).



Figure 3. Yarns' morphology identification: (**a**) non-impregnated yarn scanning electron microscope (SEM) image; (**b**) non-impregnated yarn manipulated SEM image; (**c**) impregnated yarn SEM image; (**d**) impregnated yarn manipulated SEM image.

Finally, in order to quantify the irregular geometric shape of plant fibers, the relative fiber intrinsic efficiency ratio (FIER_r) was assessed for all the specimens. The latter is defined as the ratio between the actual yarn perimeter, P_{f} , and the equivalent perimeter, P_{eq} , defined as the circumference of a circle having the same area of the actual yarn section [30].

2.3.2. Tensile Behavior

As mentioned before, the tensile strength of the flax fibers were assessed on both the single filaments constituting the threads and on the impregnated and non-impregnated yarns. The series of specimens considered in the study are as follow:

- filament: it consists of the smaller component of the textile. The samples, having a length of 30 mm, are randomly extracted from the fabric (Figure 4a);
- non-impregnated_Y_tensile: it consists of the yarn representing the main element of the fabric. The specimens, having a length of 70 mm, are randomly extracted from the textile (Figure 4b);
- impregnated_Y_tensile: it consists of specimens obtained by coating samples having the same characteristic of the dry-Y-tensile yarns (Figure 4c).

The series filament consists of 10 samples with a gauge length of 10 mm that were clamped at each edge for a length of 10 mm. The tests were performed by a microforce testing machine (Tytron 250, MTS System Corporation, Eden Prairie, MN, USA), with 50N load cell in displacement control by using a rate of 0.5 mm/min. The series dry-Y-tensile and impregnated-Y-tensile consist are characterized by 13 to 15 samples having a gauge length of 50 mm and clamped in each edge for a length of 10 mm. The tests were performed by a micro force machine, Tytron 250, with 500N load cell in displacement control by using a rate of 4 mm/min.



Figure 4. Flax specimens tested in tension: (a) filament; (b) non-impregnated yarn; (c) impregnated yarn.

2.3.3. Bond Behavior

Pull-out tests were carried out to investigate the bond behavior between flax fibers yarns and the mortar. Although the test was conceived on a local scale, the information obtained can be extended at the analysis at a greater scale of investigation aimed to describe the behavior of lime-based composite systems reinforced by plant textiles. The specimens consist of a hydraulic lime cylinder with a diameter of 25 mm and a height of 25 mm, representing the embedded fiber length. The latter was chosen by ensuring the tensile failure of the yarn was avoided and the debonding within the matrix was guaranteed. The series of specimens considered are as follow:

- non-impregnated_Y_pull-out: consists of uncoated flax yarns embedded in the mortar cylinders;
- impregnated_Y_pull-out: consists of impregnated flax yarns embedded in the mortar cylinders.

Both the series, characterized by the same geometry and test set-up, consist of 15 specimens. The yarns, having a length of 35 mm, were immersed in the mortar elements during their casting by ensuring it to be well-placed in the middle, embedded for the entire cylinder length, and in axis with it. The tests, conducted after 28 days of curing in environment conditions, were carried out by means of

a micro force machine, Tytron 250, with 500 N load in displacement control with a rate of 1 mm/min. On one side of the specimen, the cylinder was clamped by means of a steel device specifically conceived for this purpose, and on the other side the free edge of the yarn was clamped for a length of 10 mm by ensuring the clampers to be at the cylinder edge and to not record any fiber elastic elongation during the test (Figure 5).



Figure 5. Pull-out test set up.

3. Results and Discussion

3.1. Morphological Characterisation

The morphological characterization was carried out by analyzing 30 non-impregnated and 30 impregnated flax yarns. Figure 6 shows SEM images of some representative non-impregnated and impregnated specimens. Regarding the cross-section area, both the groups of fibers show the same average value equal to 0.28 mm².





Figure 6. SEM images for (a) non-impregnated and (b) Impregnated representative samples subjected to pull-out tests.

As both the groups of fibers derive from the same textile, this aspect shows that the coating procedure does not affect this geometrical parameter. This is also confirmed by analyzing the images proposed in Figure 7, where an impregnated yarn represented in different magnification scales shows that the coating polymer does not penetrate within the inner filaments of the yarn. In regards the perimeter of the flax bundles, the two groups of specimens show different average values equal to 4.89 mm for the non-impregnated specimens and 3.15 mm for the impregnated ones, respectively.



Figure 7. SEM images of an impregnated yarn in different magnification scales: 80×, 200×, 600×, 1200×.

Focusing on the value's relative frequency distribution, it can be observed that while in terms of cross-section area there is no discrepancy between non-impregnated and impregnated fibers (Figure 8a), on the other hand, in terms of perimeter, there is a significative difference not only in terms of average values but also in terms of probabilistic distribution (Figure 8b). In fact, when the impregnation is applied on the fibers, the distribution moves from a "multi-modal" to "quasi uni-modal" form, meaning that the coating system tends to reduce the heterogeneity of the fibers' geometry. This evidence is also confirmed by analyzing the results in terms of resulting relative FIER (Figure 8c). In fact, a significative reduction of around 35% is observed in terms of the FIER_r, the mean value of which moves from 2.61 (for non-impregnated yarns) to 1.69 (for the impregnated specimens). Moreover, the FIER_r value's relative frequency distributions in Figure 8c also shows a difference in terms of dispersion of the data, with a squeezed distribution for the non-impregnated fibers and a more stretched one for the coated yarns. As a matter of fact, the impregnation process significantly affects the geometry of the yarns by making them more regular in shape, with reduction in both lateral surfaces, due to a smaller space between the filaments, and variability of the properties.



Figure 8. Cont.



Figure 8. Frequency distribution for non-impregnated and impregnated yarns of the parameters: (a) area; (b) perimeter; (c) fiber intrinsic efficiency ratio (FIER_r).

3.2. Morphological Characterisation

During the tensile tests, both axial displacement and applied loads were recorded, and the parameters considered for describing the tensile behavior are:

- the maximum load (*F_{max}*);
- the displacement corresponding the maximum load (*disp_{max}*);
- the tensile strength (f_t) ;
- the strain corresponding to the maximum load (ε_{max});
- the Young's modulus, (*E*) calculated in the linear branch within the stress range from 50% to 90% of the maximum strength.

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The main values of the mechanical parameters concerning the filament, non-impregnated_Y_tensile and impregnated_Y_tensile series of specimens, together with the respective coefficient of variation, are reported in Table 2. Meanwhile, Figure 9 shows the tensile response of the filaments in terms of stress–strain.

	F _{max}		disp _{max}		f _t		ε _{max}		Е	
Series of Specimens	Mean	Co.V.	Mean	Co.V.	Mean	Co.V.	Mean	Co.V.	Mean	Co.V.
	Ν	(%)	mm	(%)	MPa	(%)	%	(%)	GPa	(%)
filaments	0.29	35	0.27	18	411.39	35	2.66	18	15.14	21
non-imp_Y_tens	99.8	9	3.04	10	341.25	20	6.09	10	9.06	19
imp_Y_tens	79.3	15	1.82	18	295.76	18	3.63	18	9.11	18

Table 2. Mechanical properties of specimens tested in tension.



Figure 9. Tensile test results on filament series.

The mechanical parameters that arose from the experimental tests on the filaments assume values slightly out of the range of values already found in the literature for flax, and more generally, plant fibers [46]. Specifically, the Young's modulus presents lower values. This could be due to the manufacturing process of the filament roving from which the samples are extracted. In fact, the filament could have been subjected to rotation. By comparing the mechanical response assessed on the filaments with the one obtained on the non-impregnated yarns, it is clear that by increasing the size of the sample, a reduction in terms of both strength and stiffness is observed, and an increase of up to more than two times of the maximum strain is detected. This variability of the mechanical properties with the size of the specimen is typical of plant fibers and can be due to a non-homogeneity of the geometry deriving from the chaotic arrangement of the filaments within the yarns [47]. Figure 10 shows the tensile responses, in terms of stress–strain, for both non-impregnated (i.e., non-impregnated_Y_tensile) and impregnated (i.e., impregnated_Y_tensile) yarns.



Figure 10. Tensile test results of non-impregnated_Y_tensile and impregnated_Y_tensile series of specimens.

Both the series of specimens, in line with the literature [25], are characterized by a lower initial stiffness that increases up to a constant value before the failure. Although no significative differences in terms of Young's modulus (evaluated in the linear branch) are observed, the coated yarns present a maximum strain smaller than the one exhibited by the non-impregnated ones, and a lower strength. As a matter of fact, the impregnated yarns behave as "polymeric composite", in which the amount of polymer strongly affects the failure mode. The lower initial stiffness observed in the non-impregnated specimens can be due to the fact that, during the first phase of the tensile tests, the yarns are stretched. Meanwhile, the application of the coating system tends to reduce this effect, resulting in a more "rigid" behavior characterized by a quasi-constant value of the stiffness from the beginning of the test. The resulting lower deformability of the impregnated yarns represents an important aspect in view of the application of the textile as reinforcement in cement-based composites, in which one of the limits is characterized by the large difference in terms of deformability between mortar and plant fibers [20].

3.3. Bond Behavior

The pull-out behavior of the non-impregnated_Y_pull-out and the impregnated_Y_pull-out series of flax yarns is shown in terms of load-displacement curves in Figure 11. In all the specimens, the failure mode is characterized by the slipping of the bundle within the matrix up to the complete ejection of the bundle form the mortar cylinder. Both the groups of fibers exhibit a response typical of the cases in which the debonding occurs [30]. This response is generally characterized by a first elastic branch, named as the bond adhesive phase, and a frictional bond phase following a drop of the peak, characterized by a quasi-constant value due to the residual force. In some cases, between the two phases another stage occurs, in which the peak load is achieved after a non-elastic hardening branch, named as mechanical bond phase. In most of the non-impregnated samples, the peak load occurs after a non-elastic hardening phase, while the impregnated specimens mainly show an elastic behavior up to the achievement of the maximum load (Figure 11).



Figure 11. Pull-out test results of non-impregnated_Y_pull-out and impregnated_Y_pull-out series of specimens.

Regarding the post peak phase, the non-impregnated samples are characterized by a softening behavior in which a mechanical bond component, still present, makes the test last up to the complete ejection of the yarn from the mortar. Conversely, the softening branch exhibited by the coated yarn specimens is mainly characterized by a residual frictional component that, in most cases, gets to a null value of the load before the complete slipping of the yarns from the mortar. In terms of maximum load, the mean value achieved by the non-impregnated specimens, equal to 40.96 N, is two times higher than the one reached by the impregnated yarns, the latter being equal to 20.23 N. This experimental result highlights that the impregnation procedure employed in the study reduces the performance in terms of adherence between the fibers and the mortar. Although this aspect may be attributed to a loss of adherence due to the presence of the coating, it also depends, in part, on the different morphology of the impregnated fibers that are characterized, as shown above, by a smaller fiber-to-mortar interface surface.

To make the analysis independent from the geometry, the results are also expressed in terms of adherence shear stress, τ_{max} , assessed for each sample by dividing the maximum load for its interface surface. By looking at the interface shear stress relative frequency distribution (see Figure 12), the two groups of data, having a similar dispersion, still show a difference in terms of average values.

By comparing the higher average maximum shear stress values, 0.37 MPa and 0.25 MPa for the non-impregnated and impregnated specimens respectively, it is obvious that the discrepancy, although still significative and consisting in a reduction of the mean maximum stress of about the 30%, results to be lower with respect to the case in which the comparison is made in terms of load. This last aspect brings to light that the coated fibers are characterized by a local bond behavior performing less than the one exhibited by putting the fibers directly in contact with the mortar. It may be attributed to the specific polymer material or coating procedure used to impregnate the fibers. In fact, the impregnation creates a weak layer between the fibers and the mortar that reduces the adhesion of the reinforcement-to-mortar system. The work is a result of a preliminary study aimed at analyzing the efficiency of the coating procedure adopted. Clearly, further investigations are needed, perhaps by adopting a different coating

material and/or amount or impregnation treatment, in order to get an impregnation process that may confer mechanical benefits to the system in terms of adherence response. Moreover, since one of the main issues related to the use of natural-based reinforcement in cement- or lime-based matrix is the alkaline environment in which the fibers are embedded, it is worth mentioning that the application of impregnation processes could also mitigate these durability-related issues. A more comprehensive study is foreseen for further analysis of this fundamental aspect.



Figure 12. Frequency distribution of shear stress of non-impregnated_Y_pull-out and impregnated_Y_pull-out series of specimens.

4. Conclusions

This study reports the experimental results of a campaign intended at investigating the influence of flax fibers impregnation on their morphology, mechanical properties, and adherence with a hydraulic-lime based mortar. It represents a preliminary study in view of future applications concerning the use of flax textile as reinforcement in cement-based composite systems. The investigation shows that the impregnation of the fibers by means of XSBR latex affects the fibers properties, hence the bond with the mortar.

The main findings of the research study are summarized as follows:

- the analysis of the SEM images shows that the impregnation significantly affects the flax yarns shape, with a reduction of the FIERr of the 35%; moreover, the dispersion in the measurements of geometric properties of the impregnated yarns is much lower than the dispersion in measurements of the non-impregnated fibers;
- similarly, a good influence of the impregnation on the mechanical response in tension has been observed: despite a small reduction in the tensile strength, the impregnated fibers exhibit an ultimate strain much smaller than the one observed by testing non-impregnated yarns;
- however, as results of the impregnation, a decrease of the bond between fibers and mortar is
 observed with a reduction of the maximum shear stress of about 30% because of a polymeric weak
 layer between the fibers and the mortar created by the impregnation.

Finally, the deformability of fibers and the fiber-to-mortar adherence represents crucial aspects strongly influencing the mechanical performance of textile reinforced cement-based composites. Therefore, further analysis may be needed, perhaps by testing composite coupons on a larger scale in order to clearly get the influence of the impregnation on the mechanical response of the composite system

itself and of structural systems strengthened by it. Moreover, an investigation focused on the influence of the coating on the durability of the composite system may complement the analysis conducted with the present study. In regard to the fiber-to-mortar bond behavior, additional experiments may be planned aimed at identifying more suitable coating substances which may improve this specific aspect.

Author Contributions: All the authors contributed to the conceptualization and the methodology of the research. G.F. performed the experimental activities; G.F. and M.P. analyzed the data and prepared the manuscript under the supervision of E.M. and R.D.T.F.; All the authors reviewed and edited the final version of the paper. Finally, the funding acquisition was coordinated by E.M.

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