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Geopolymers Reinforced with Natural Fibers: A Comparison among Different Sources

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Featured Application: Geopolymers formulated with different types of natural fibers with improved mechanical proper-ties can be obtained and used in civil engineering applications.

Abstract: The performance of different natural fibers (hemp, kenaf and bamboo) used to formulate composites with an alkali-activated matrix based on metakaolin is evaluated. Short fibers were randomly dispersed up to about 3% of the binder weight, and the fresh and cured properties of the derived composites were determined. Up to the investigated fraction, it is still possible to obtain adequate workability without the supply of additional water or additives. Upon modification with fibers, the mechanical behavior changes from completely brittle to pseudoplastic with increased toughness. The flexural strength increases by up to 80% at the highest bamboo amount and up to 20% for kenaf. Hemp fibers have a negligible effect on flexural strength but strongly improve the materials' toughness. Moreover, the addition of fibers does not change the manner in which the material interacts with moisture. Indeed, the water uptake of the modified samples was comparable to that of the unmodified samples, and the composites showed a decreased rate of water diffusion as the amount of fiber increased.

Keywords: geopolymer; natural fibers; mechanical properties; pseudoplastic behavior

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1. Introduction

Geopolymers represent a possible way to reduce the environmental impact caused by building materials based on traditional binders. While they possess promising mechanical properties, i.e., compressive strength, they suffer from extreme brittleness and from limited dimensional stability. These features, as well as the absence of a well-established standardization, presently limit the application of these materials in civil engineering, although some commercial applications have already been reported [1]. In order to overcome this problem, a common strategy is to modify the material through the addition of fibers [2–5]. Synthetic fibers, either organic (PVA, PP, carbon) or inorganic (basalt, steel), have been used. The possibility to insert recycled fibers has also been tested in order to increase the environmental benefits [6,7]. A further alternative is to insert renewable natural fibers of different origins [8–11]. Among these, flax has been frequently studied [12–15], as has bamboo [16–19]. Other types have received less attention, such as cotton [20,21], hemp [22,23] and kenaf. The use of natural fibers may cause some problems related to their hydrophobicity, porosity and the presence of incoherent material on their surfaces. In some cases, pretreatments have been made in order to overcome these problems, although this increases the overall economic and environmental impact of the process [24–28]. Another problem related to the use of natural fibers as substitutes for synthetic fibers is the variability of the chemical composition, which affects the final mechanical properties. In the present paper, for the first time, the effects of three different types of natural fibers inserted in an alkali-activated matrix based on metakaolin on fresh and cured properties have been compared. Bamboo, kenaf and hemp fibers have been selected to modify a geopolymeric matrix based on metakaolin. The fibers were not submitted to any specific surface treatments, and the composites have been cured at a low temperature, which is the most favorable condition to minimize the environmental impact. Their effects on the workability, microstructure, mechanical properties and dimensional stability have been investigated. A comparison among the different types of fibers is performed, and an attempt to link the results to the fibers' characteristics is made. Moreover, concerns related to the stability of the natural fibers in relation to moisture have been expressed. Indeed, durability is a key aspect in view of the final application of these materials. Accordingly, a test based on moisture permeation has been carried out to get a preliminary evaluation of the mortars' durability.

2. Materials and Methods

2.1. Materials

2.1.1. Binder

Metakaolin (Argicem, France) with d_{10} = 4.815 µm, d_{40} = 40.778 µm, d_{90} = 117.829 µm was used as binder. The chemical composition (wt%) is the following: SiO_2 68.8, Al_2O_3 27.0, Fe_2O_3 2.6 and CaO 1.2.

2.1.2. Activators

An 8 M water solution of sodium hydroxide (NaOH reagent grade Carlo Erba, Milano, Italy) and sodium silicate solution (Na_2SiO_3, Ingessil, Verona, Italy), a viscous liquid produced for the cement industry with a water content of 57 wt% and a SiO_2/Na_2O ratio of 3 were used as alkaline activators.

2.1.3. Fibers

Bamboo fibers (*Phyllostachys iridescens* species from Italy) deprived of the parenchymal fraction by a mechanical method and by soaking in a 0.1 N NaOH solution, 6 ± 1 mm long and 0.8–2.0 mm thick were prepared. Bast kenaf fibers (*Hybiscus cannabinus* from Liberia) with a length of 7 ± 2 mm and thickness between 0.2 and 0.8 mm, as well as bast hemp fibers (*Cannabis sativa* L. from the Netherlands), were used as the other types of reinforcement. Figure 1 reports the morphology of the three fibers.

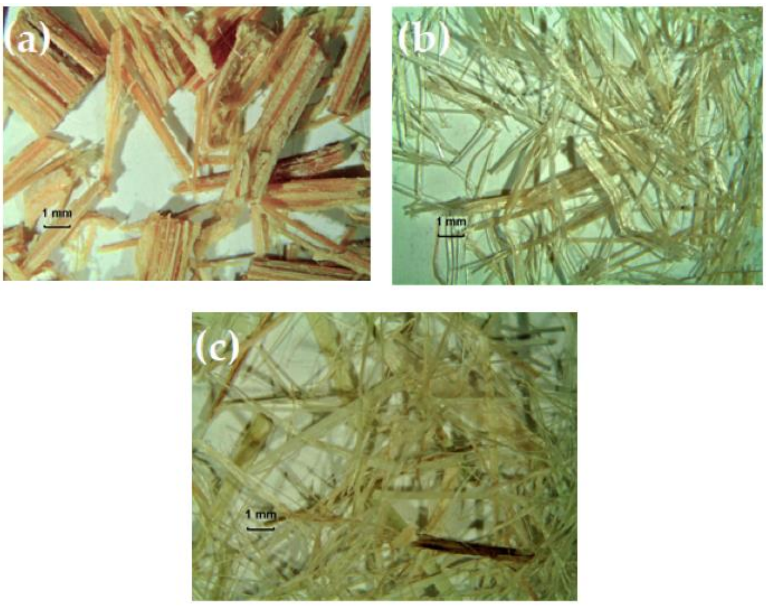

Figure 1. Morphology of the bamboo (**a**), kenaf (**b**) and hemp (**c**) fibers.

The bare fibers were submitted to a thermogravimetric analysis in a nitrogen atmosphere at 10 °C/min, from room temperature to 850 °C, using a PerkinElmer TGA7

(Perkin Elmer, Waltham, MA, USA) apparatus (gas flow 40 mL/min). Before testing, samples were kept overnight at 50 °C. The test was performed to investigate the composition of the investigated reinforcements in order to try to explain their different behaviors.

2.2. Composites Preparation

The mix compositions are reported in Table 1, where the labelling code, hereafter used to classify the composites, is also included. Mixing was performed by first adding sodium hydroxide to the metakaolin and subsequently pouring the silicate into the vessel. After 3 min of stirring, fibers were added to the paste, followed by deionized water. The whole procedure lasted for 10 min. Composite workability was obtained according to a mini-slump test. Mixed components were cast in a truncated conical mold with a circular base (40 mm of diameter at the bottom, 20 mm of diameter at the top and 60 mm of height). After mold removal, the area of three collapsed, uncured composites was evaluated for each composition. The amount of deionized water used in all formulations was kept constant. Consequently, the workability was changed upon the addition of fibers. However, the rheology of the mortars still allowed specimens to be cast in the molds without the creation of macroscopic defects. Prisms (15 × 20 × 75 mm) were prepared for flexural tests. Steel jolts were applied to the same specimens in order to evaluate their dimensional stability. Squared specimens (50 × 50 × 50 mm) were used for compression tests, and cylinders (25 mm diameter, 60 mm high) were used for water absorption and water permeability tests. Composite specimens were kept in polyethylene bags at 23 ± 2 °C for 24 h. After demolding, they were stored in sealed polyethylene bags at 23 ± 2 °C and 90 ± 5 RH% until testing.

Table 1. Composition and labelling of the investigated composites.

Sample	Code	Metakaolin	Na_2SiO_3 + NaOH	Water	Fibers	wt% *
Metakaolin	MTK-0	100	15 + 15	5	0	0.00
Metakaolin + 1%Kenaf	Ke1	100	15 + 15	5	1	0.73
Metakaolin + 2%Kenaf	Ke2	100	15 + 15	5	2	1.46
Metakaolin + 3%Kenaf	Ke3	100	15 + 15	5	3	2.17
Metakaolin + 1%Hemp	He1	100	15 + 15	5	1	0.73
Metakaolin + 2%Hemp	He2	100	15 + 15	5	2	1.46
Metakaolin + 3%Hemp	He3	100	15 + 15	5	3	2.17
Metakaolin + 1%Bamboo	Ba1	100	15 + 15	5	1	0.73
Metakaolin + 2%Bamboo	Ba2	100	15 + 15	5	2	1.46
Metakaolin + 3%Bamboo	Ba3	100	15 + 15	5	3	2.17
Metakaolin + 4%Bamboo	Ba4	100	15 + 15	5	4	2.88

All amounts are expressed in grams. * Last column shows the wt% fiber amount in the composite.

2.3. Characterization Tests

2.3.1. Water Absorption

The amount of absorbed deionized water at room temperature and atmospheric pressure was determined following the EN 772-21 Standard on cylinders (six samples, 25 mm diameter, and 60 mm high) at 28 days of curing. A Sartorius balance Model CP225D (Sartorius, Gottingen, Germany) was used.

2.3.2. Mechanical Tests

Three-point flexural strength tests as well as compression tests were performed at a speed rate of 0.5 mm/min with a Wolpert 4000 kN test machine (Wolpert, Neu Ulm, Germany), at 23 ± 1 °C and 65 ± 10 RH%. The span length for the flexural tests was 70 mm with a support radius of 2 mm. At least three samples were tested in flexural mode and six for compressive mode after 28 days of curing at 23 ± 2 °C in sealed plastic bags. Apart from the reported parameters, the procedure followed the instructions of the EN 196-1 Standard, which is formulated for Portland cement composites.

2.3.3. Water Permeability

The water permeability of the composites was evaluated, following the EN 15801 Standard, on three cylindrical specimens with 25 mm diameters and 60 mm lengths, cured for 28 days at 23 ± 1 °C. A Sartorius balance Model CP225D (Sartorius, Gottingen, Germany) was used to evaluate moisture absorption as a function of time.

2.3.4. Dimensional Stability

The dimensional stability testing was performed according to the ASTM C1012/C1012M Standard on three prisms (25 × 20 × 75 mm) cured at 25 °C and 35 ± 5 RH%.

2.3.5. Microstructure

A microstructural analysis was performed by means of scanning electron microscopy (SEM XL20 type, FEI Instruments (FEI, Hillsboro, Oregon, USA)) on unperturbed fractured samples obtained after the flexural test and metallized under vacuum with aluminum. Operating conditions were set at 20 kV and the vacuum condition was below 10^{-4} Torr.

3. Results and Discussion

Table 2 reports the composition of the different fibers, as derived from the TGA reported in Figure 2. The results enable the investigation of the fibers' compositions. After a first weight loss deriving from the loss of water molecules, two overlapping peaks appear from 180 °C to 350 °C, deriving from hemicellulose and cellulose decomposition. Afterwards, at higher temperatures, the degradation of the lignin fraction takes place [29–33].

Table 2. TGA data of the fibers.

Fibers	T_{max} (°C)	Moisture (%)	Pectin and/or Hemicellulose(%)	Cellulose (%)	Lignin (%)	Residue (%)
Kenaf	388	≈4	≈10	≈74	/	12
Hemp	363	≈3	≈12	≈48	≈14	23
Bamboo	339	≈3	/	≈60	≈10	26

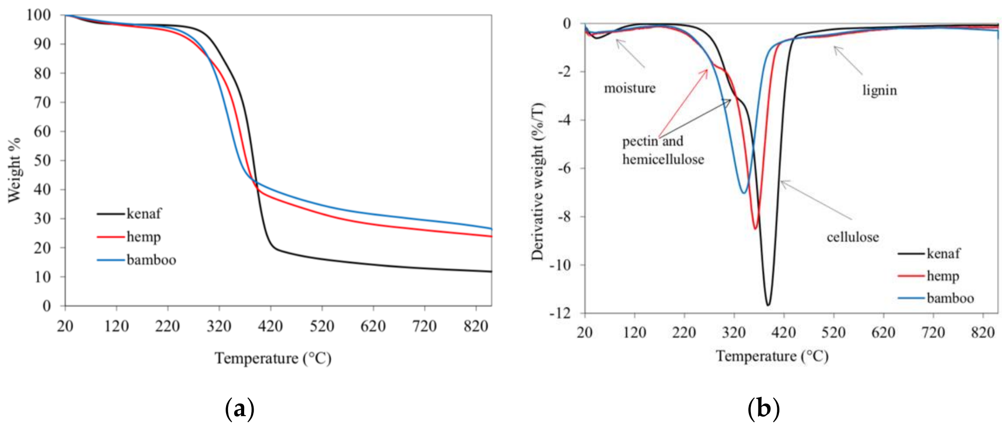

Figure 2. TGA (**a**) and dTGA (**b**) profiles of the fibers.

Figure 3 reports the relative workability of the fresh composites after the mixing stage, i.e., the ratio between the slump of the composite and the slump of the bare matrix. Since the water amount was unchanged, the addition of fibers reduced the slump values. As can be seen, the behavior of bamboo fibers is different from those of hemp and kenaf. A limited decrease in workability takes place as the amount of fibers increases, and consequently, a higher amount of added fiber was achievable (4 wt%). In kenaf and hemp modified materials, after a slight decrease in workability at the lowest amount (1 wt%), a large drop takes place at 2 wt% loading. This may be related to the formation, at this amount, of a 3D structure, i.e., a connected medium among the single fibers that constrains the matrix flow induced by the slightly different morphology of the fibers. Nevertheless, it should be underlined that despite the reduced workability of the composites, it was still possible to cast all of the specimens without the visible creation of macro-porosities.

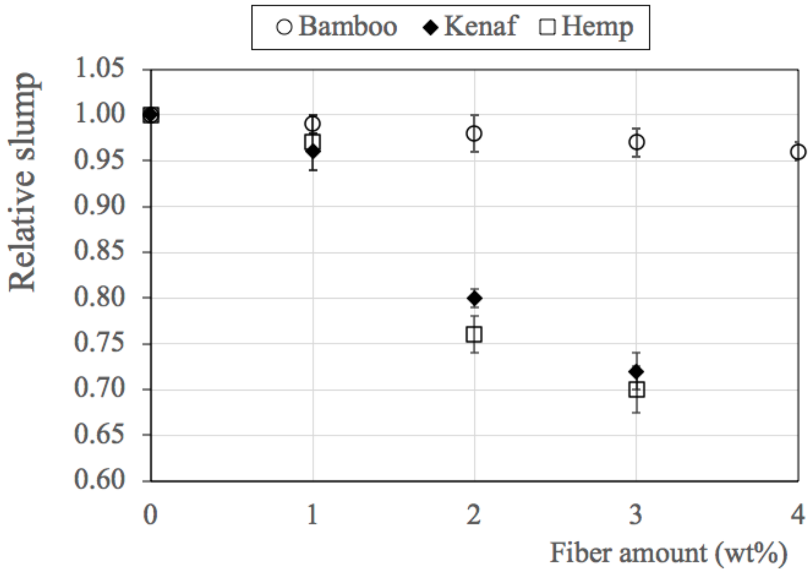

Figure 3. Relative workability of the investigated composites.

This last macroscopic evidence was further supported by the results of the water absorption test reported in Figure 4.

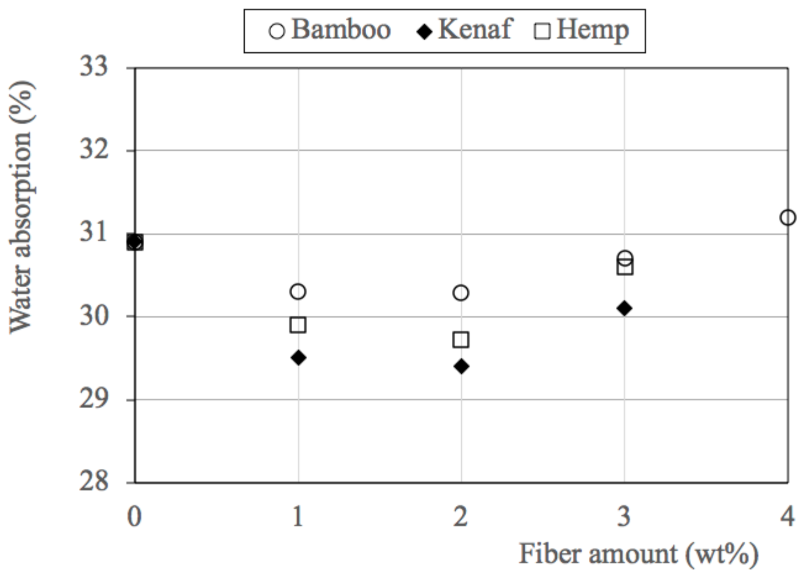

Figure 4. Absorbed water as a function of the fiber amount.

Water absorption is generally accepted as an indication of the overall open porosity of materials. Indeed, all samples, except Ba4, show a comparable or even lower amount of absorbed water (and, therefore, of the overall porosity) as compared to the metakaolin matrix. It should be considered that the matrix is a porous medium, as determined by mercury intrusion porosimetry [7]. The substitution of a porous phase, i.e., the matrix, with a less porous one, the fiber, decreases the overall porosity. In theory, a contribution to the overall porosity could derive from the scarce adhesion of the organic fibers to the inorganic matrix. However, this effect, if present, should be limited. Consequently, a positive interaction of the fibers with the matrix, leading to the creation of a quite continuous transition zone between the two phases, can be proposed based on the mutual presence of hydroxyl groups in both phases. The progressive increase may derive from air bubble entrapment.

Figure 5 reports the flexural strength of all investigated materials. A linear increase is found for bamboo and kenaf samples, the positive effect being stronger for the bamboo samples (about 80% for bamboo and about 20% for kenaf at the highest amount). Hemp samples show, instead, an almost constant value with a slight positive effect at the highest content (5%). Although it is difficult to compare these data with the existing literature, *ceteris paribus*, in similar metakaolin systems, [34] a comparable increase was obtained by the addition of sisal and pineapple fibers.

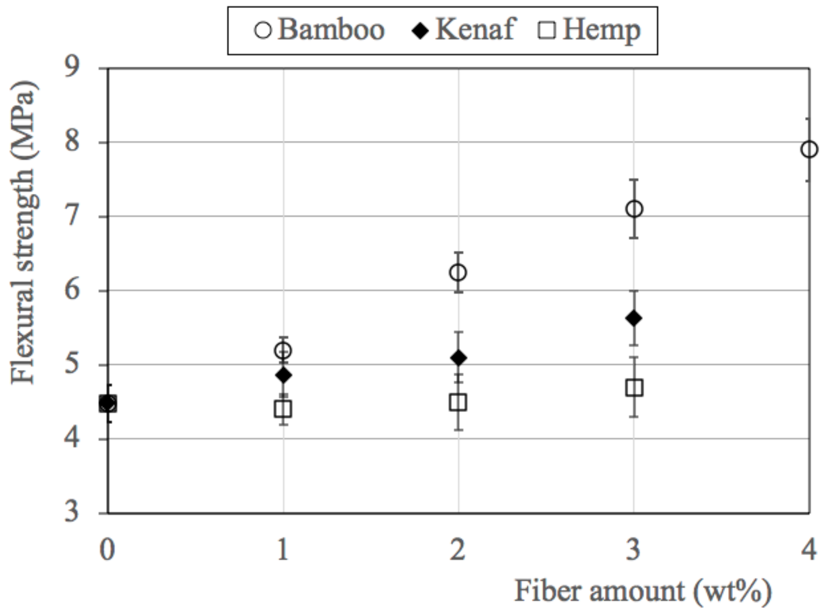

Figure 5. Flexural strength of the investigated materials.

Figure 6 shows the fracture surfaces of the broken samples, highlighting the morphology of the fibers. All of the fibers appear to be quite embedded in the matrix without the presence of large porosities at the interface or spherical-shaped porosities deriving from entrapped air during the mixing stage. Bamboo and kenaf fibers show some matrix fragments still adhering while the surface of hemp fibers is rather clean. Bamboo fibers also show evidence of disaggregation caused by the tensile breaking.

The plot of the applied force vs. the midspan deflection for the reinforced 3 wt% samples is shown in Figure 7. From the completely brittle behavior of the unmodified sample, after fiber addition, the mechanical behavior becomes pseudoplastic. The effect is more intense in the kenaf and hemp samples. Indeed, the average absorbed energy for the specimens is reported in Figure 8 as a function of fiber content.

As to the compression tests, the addition of fibers does not significantly alter this parameter (Figure 9). A slight increase in compressive strength takes place at the lowest wt%, but as wt% increases, the positive effect levels off and He3 specimens show a 20% decrease in compressive strength. This is, however, lower than the decrease in strength reported elsewhere [35], although with a higher fibers' content. A similar trend has been found in other papers concerning the modification of natural fibers [17,36,37].

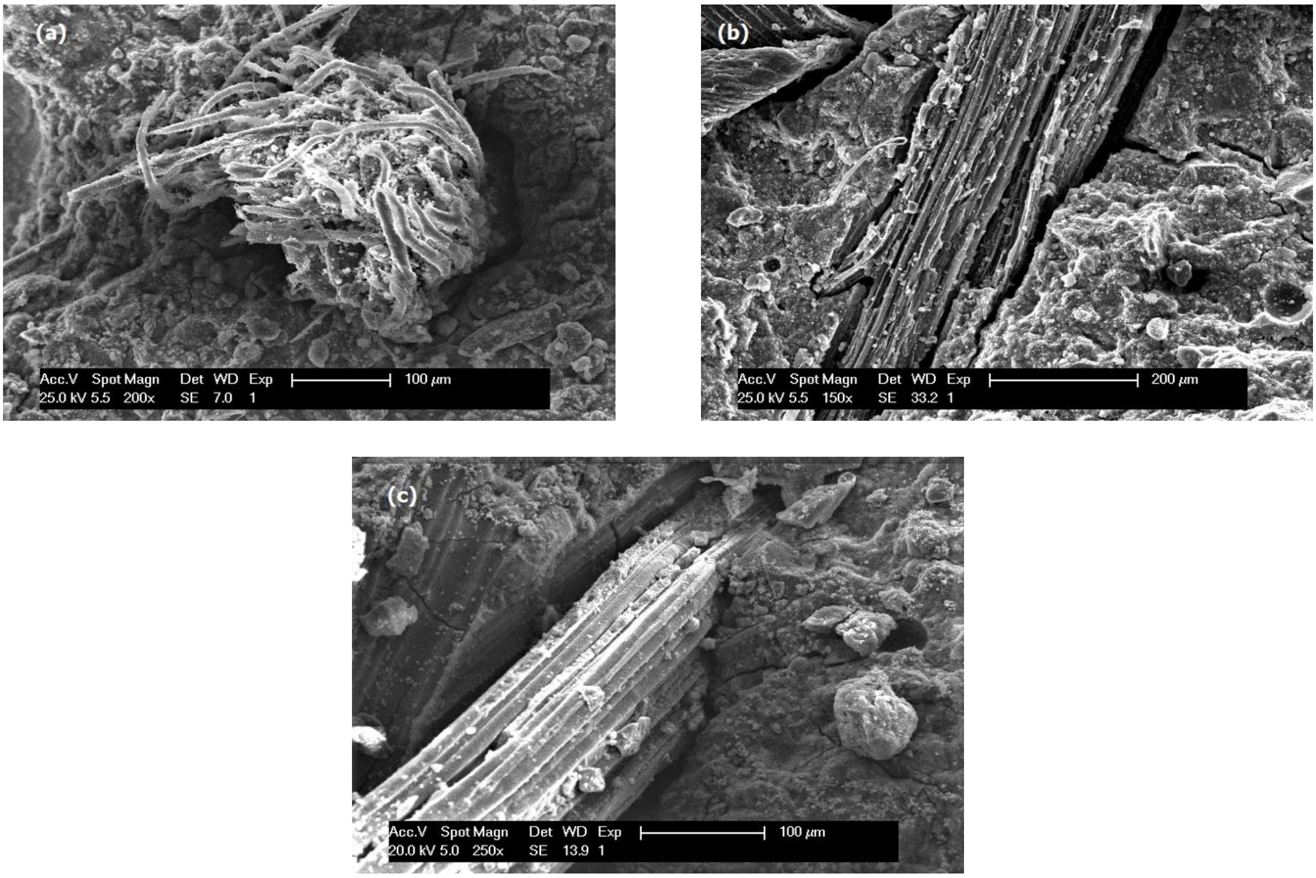

Figure 6. Morphology of the different fracture surfaces of the composites: (**a**) bamboo, **(b**) hemp and (**c**) kenaf.

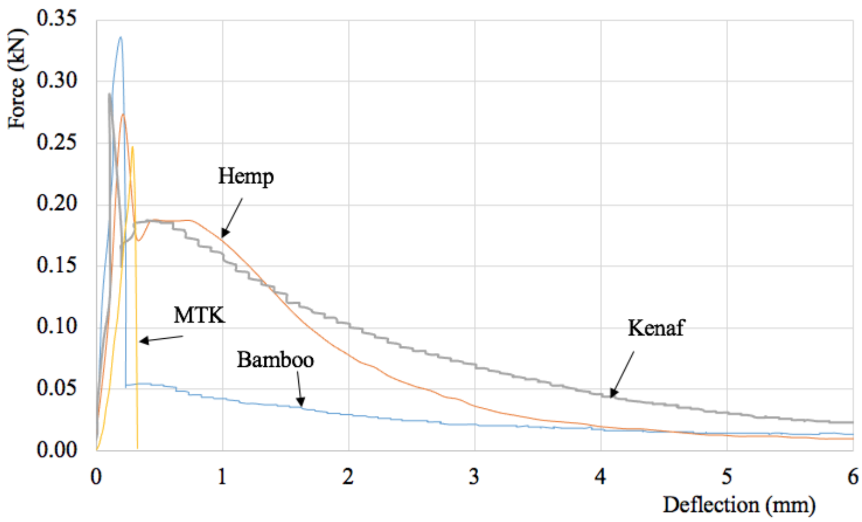

Figure 7. Force vs. displacement for 3 wt% samples and the unmodified matrix.

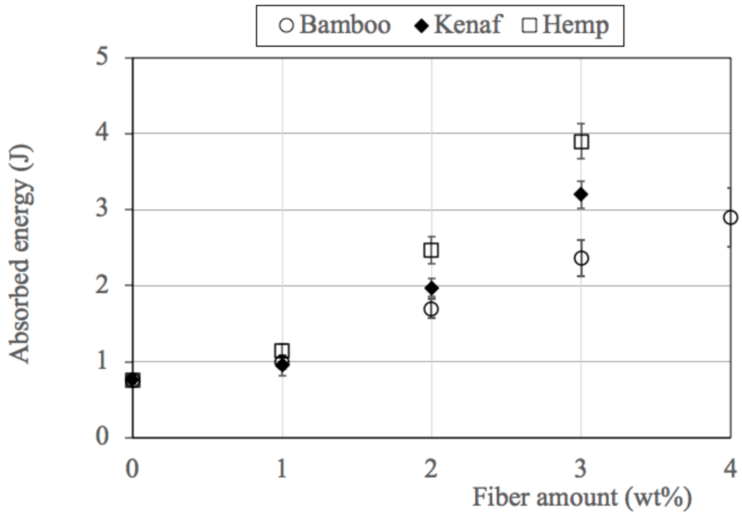

Figure 8. Absorbed energy for the different composites during the flexural test.

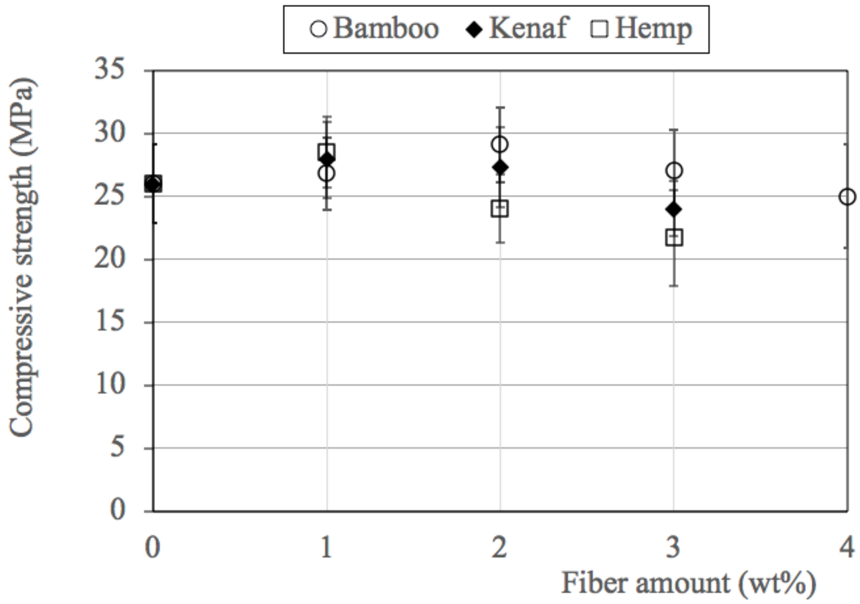

Figure 9. Compressive strength of the investigated materials.

The dimensional stability of the unmodified material and of all the samples containing a 3 wt% of fibers is reported in Figure 10. As can be seen, an increase in stability is obtained, and this effect is independent of fiber type. Since measurements began 24 h after mortar casting, dimensional stability on the fresh state was not included in the experiment. Thus, the positive effect may be even higher than the reported one.

Finally, the results concerning water permeability are shown in Figure 11. The plot concerns all of the 3 wt% samples, and the square root of time is reported on the x-axis. After an initial linear trend, common to all samples, an asymptotic value is reached. At the longest investigated times of the experiment (20 days, not reported in the figure), the values were still constant, confirming Fickian behavior of all materials, i.e., no swelling phenomena taking place, leading to a continuous increase in the absorbed water. Contrary to what could be expected, the modified samples have a lower absorption rate than that of the bare matrix.

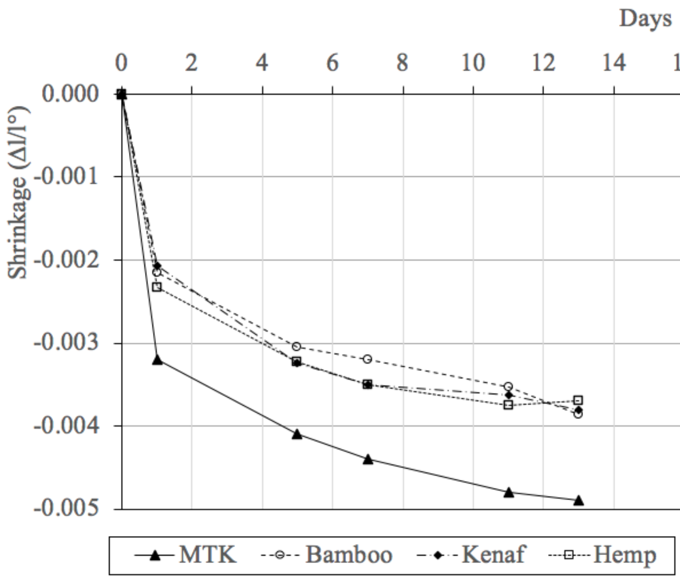

Figure 10. Dimensional variation vs. time for the 3 wt% fibers.

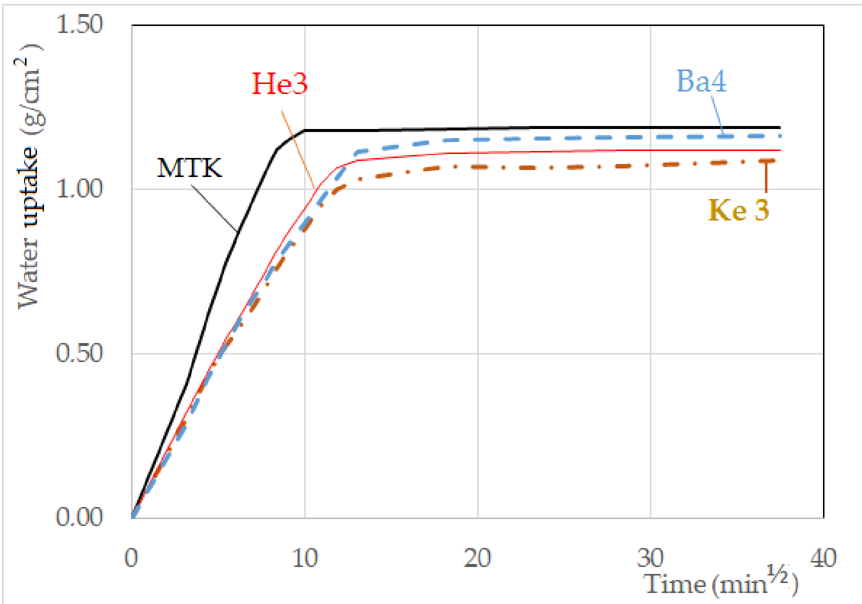

Figure 11. Water uptake of the plain matrix and the samples at the highest amount of fibers.

This result confirms again that the fibers and the interface between the fibers and the matrix do not create a preferential path for water diffusion. Indeed, the subtraction of a small volume of porous matrix by the fiber substitution creates a slightly impervious path to water diffusion. The final water uptake of modified samples is lower than that of the MTK sample, and this agrees with what was previously found in the water absorption test (Figure 4).

As a general consideration, the addition of all of the investigated fibers has, apart from workability, a positive effect on the investigated properties, or they at least do not compromise the geopolymer performance. However, the influence is different when the type of fiber is considered. Bamboo fiber-modified composites have stronger mechanical properties than hemp ones, a feature reflected by the flexural and, albeit partially, compressive strength. Contrarily, hemp fibers provide the highest toughness but do not increase compressive and flexural strengths. Kenaf samples have intermediate behavior. The adhesion between fiber and matrix plays a fundamental role. Strong interaction between the two phases increases the maximum sustainable load, but the energy dispersion should derive mainly from the pull-out effect after the matrix failure. Hemp has the highest amount of the fraction containing pectin. This incoherent matter may decrease the interlocking between fiber and matrix, favoring energy dissipation through the pulling-out process instead of the breaking event that should affect bamboo fibers. A correlation between the mechanical properties and the amount of cellulose is, however, not evident. This feature is lacking, also, in the existing literature but deserves further investigation by techniques able to characterize the chemical composition of the external surface of the fibers, such as the infrared technique. Moreover, a quantitative investigation of surface roughness by atomic force microscopy (AFM) could improve the understanding of the pull-out mechanism in the different fibers.

4. Conclusions

The addition of all of the investigated fibers has positive effects on the materials' properties. Although the mortars' workability is decreased, samples' casting can be obtained without the use of additives. The flexural strength of samples modified by kenaf and bamboo increases up to 80% and 20%, respectively, in the best conditions. A general increase in toughness and dimensional stability is observed, regardless of fiber type. The contribution to the compressive strength is not relevant. Moreover, fiber addition does not increase the permeability of water, a feature that should not compromise the durability of the composites. Further studies are needed to establish a robust correlation between fibers' composition and the mechanical contribution to the composite materials as well as long-term tests to evaluate the materials' durability as a function of time and environmental conditions. Indeed, all types of investigated fibers contribute to making up for the main problems related to geopolymer matrices in civil engineering, which are brittleness and low dimensional stability.

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References

1. Korniejenko, K.; Łach, M.; Mikuła, J. The influence of short coir, glass and carbon fibers on the properties of composites with geopolymer matrix. *Materials* **2021**, *14*, 4599. [CrossRef]
2. Rashad, A.M. The effect of polypropylene, polyvinyl-alcohol, carbon and glass fibres on geopolymers properties. *Mater. Sci. Technol.* **2019**, *35*, 127–146. [CrossRef]
3. Shaikh, F.U.A. Review of mechanical properties of short fibre reinforced geopolymer composites. *Constr. Build. Mater.* **2013**, *43*, 37–49. [CrossRef]
4. Ranjbar, N.; Zhang, M. Fiber-reinforced geopolymer composites: A review. *Cem. Concr. Comp.* **2020**, *107*, 103498. [CrossRef]
5. Shanthini, D.; Grija, S.; Abinaya, S.; Devaki, R. Fibre reinforced geopolymer concrete—A review. *Int. J. Civ. Eng. Technol.* **2016**, *7*, 435–438.
6. Saccani, A.; Manzi, S.; Lancellotti, I.; Lipparini, L. Composites obtained by recycling carbon fibre/epoxy composite wastes in building materials. *Constr. Build. Mater.* **2019**, *204*, 296–302. [CrossRef]
7. Manzi, S.; Lancellotti, I.; Masi, G.; Saccani, A. Alkali-activated binders from waste incinerator bottom ashes and metakaolin reinforced by recycled carbon fiber composites. *Front. Mater.* **2020**, *7*, 583400. [CrossRef]
8. Yan, L.; Kasal, B.; Huang, L. A review of recent research on the use of cellulosic fibres, their fibre fabric reinforced cementitious, geopolymer and polymer composites in civil engineering. *Compos. Part. B Eng.* **2016**, *92*, 94–132. [CrossRef]

9. Silva, G.; Kim, S.; Aguilar, R.; Nakamatsu, J. Natural fibers as reinforcement additives for geopolymers—A review of potential eco-friendly applications to the construction industry. *Sustain. Mater. Technol.* **2020**, *23*, e00132. [CrossRef]
10. Camargo, M.M.; Taye, E.A.; Roether, J.A.; Redda, D.T.; Boccaccini, A.R. A review on natural fiber-reinforced geopolymer and cement-based composites. *Materials* **2020**, *13*, 4603. [CrossRef]
11. Korniejenko, K.; Łach, M.; Dogan-Saglamtimur, N.; Furtos, G.; Mikuła, J. The overview of mechanical properties of short natural fiber reinforced geopolymer composites. *Environ. Res. Technol.* **2020**, *3*, 28–39. [CrossRef]
12. Alzeer, M.; MacKenzie, K. Synthesis and mechanical properties of novel composites of inorganic polymers (geopolymers) with unidirectional natural flax fibres (phormium tenax). *Appl. Clay Sci.* **2013**, *75*, 148–152. [CrossRef]
13. Lazorenko, G.; Kasprzhitskii, A.; Yavna, V.; Mischinenko, V.; Kukharskii, A.; Kruglikov, A.; Kolodina, A.; Yalovega, G. Effect of pre-treatment of flax tows on mechanical properties and microstructure of natural fiber reinforced geopolymer composites. *Environ. Technol. Innov.* **2020**, *20*, 101105. [CrossRef]
14. Assaedi, H.; Shaikh, F.U.A.; Low, I.M. Influence of mixing methods of nano silica on the microstructural and mechanical properties of flax fabric reinforced geopolymer composites. *Constr. Build. Mater.* **2016**, *123*, 541–552. [CrossRef]
15. Assaedi, H.; Alomayri, T.; Shaikh, F.U.A.; It-Meng, L. Characterisation of mechanical and thermal properties in flax fabric reinforced geopolymer composites. *J. Adv. Ceram.* **2015**, *4*, 272–281. [CrossRef]
16. Wang, X.; Yuan, Z.; Zhan, X.; Li, Y.; Li, M.; Shen, L.; Cheng, D.; Li, Y.; Xu, B. Multi-scale characterization of the thermal—Mechanically isolated bamboo fiber bundles and its potential application on engineered composites. *Constr. Build. Mater.* **2020**, *262*, 120866. [CrossRef]
17. Sá Ribeiro, R.A.; Sá Ribeiro, M.G.; Sankar, K.; Kriven, W.M. Geopolymer-bamboo composite—A novel sustainable construction material. *Constr. Build. Mater.* **2016**, *123*, 501–507. [CrossRef]
18. Sankar, K.; Sá Ribeiro, R.A.; Sá Ribeiro, M.G.; Kriven, W.M. Potassium-based geopolymer composites reinforced with chopped bamboo fibers. *J. Am. Ceram. Soc.* **2017**, *100*, 49–55. [CrossRef]
19. Da Costa Correia, V.; Ardanuy, M.; Claramunt, J.; Savastano, H. Assessment of chemical and mechanical behavior of bamboo pulp and nanofibrillated cellulose exposed to alkaline environments. *Cellulose* **2019**, *26*, 9269–9285. [CrossRef]
20. Zhou, B.; Wang, L.; Ma, G.; Zhao, X.; Zhao, X. Preparation and properties of bio-geopolymer composites with waste cotton stalk materials. *J. Clean. Prod.* **2020**, *245*, 118842. [CrossRef]
21. Alomayri, T.; Shaikh, F.U.A.; Low, I.M. Synthesis and mechanical properties of cotton fabric reinforced geopolymer composites. *Compos. Part. B Eng.* **2014**, *60*, 36–42. [CrossRef]
22. Galzerano, B.; Formisano, A.; Durante, M.; Iucolano, F.; Caputo, D.; Liguori, B. Hemp reinforcement in lightweight geopolymers. *J. Comp. Mater.* **2018**, *52*, 2313–2320. [CrossRef]
23. Simonova, H.; Kucharczykov, B.; Topolar, L.; Kersner, Z.; Merta, I.; Dragas, J.; Ignjatovic, I.; Komljenovic, M.; Nikolic, V. Crack initiation of selected geopolymer mortars with hemp fibers. *Procedia Struct. Integr.* **2018**, *13*, 578–583. [CrossRef]
24. Valášek, P.; Müller, M.; Šleger, V.; Kolaf, V.; Hromasova, M.; D'Amato, R.; Ruggiero, A. Influence of alkali treatment on the microstructure and mechanical properties of coir and abaca fibers. *Materials* **2021**, *14*, 2636. [CrossRef]
25. Gierarda, W.; Rojewski, S.; Rozanska, W. Effectiveness of silanization and plasma treatment in the improvement of selected flax fibers' properties. *Materials* **2021**, *14*, 3564. [CrossRef] [PubMed]
26. Malinowski, R.; Raszkowska-Kaczor, A.; Moraczewski, K.; Głuszewski, W.; Krasinskyi, V.; Wedderburn, L. The structure and mechanical properties of hemp fibers-reinforced Poly(ε-Caprolactone) composites modified by electron beam irradiation. *Appl. Sci.* **2021**, *11*, 5317. [CrossRef]
27. Asim, M.; Jawaid, M.; Abdan, K.; Ri. Ishak, M.R. Effect of alkali and silane treatments on mechanical and fibre-matrix bond strength of kenaf and pineapple leaf fibres. *J. Bionic Eng.* **2016**, *13*, 426–435. [CrossRef]
28. Sanjay, M.R.; Siengchin, S.; Parameswaranpillai, J.; Jawai, M.; Pruncu, C.I.; Khan, A. A comprehensive review of techniques for natural fibers as reinforcement in composites: Preparation, processing and characterization. *Carbohydr. Polym.* **2019**, *207*, 108–121. [CrossRef]
29. Azwa, Z.N.; Yousif, B.F. Characteristics of kenaf fibre/epoxy composites subjected to thermal degradation. *Polym. Degrad. Stabil.* **2013**, *98*, 2752. [CrossRef]
30. Sisti, L.; Kalia, S.; Totaro, G.; Zanaroli, G.; Celli, A. Enzymatically treated curaua fibers in poly(butylene succinate)-based biocomposites. *Environ. Eng. Chem. Eng.* **2018**, *6*, 4452–4458. [CrossRef]
31. Sisti, L.; Totaro, G.; Vannini, M.; Fabbri, P.; Zatta, A.; Celli, A. Evaluation of the retting process as a pre-treatment of vegetable fibers for the preparation of high-performance polymer biocomposites. *Ind. Crop. Prod.* **2016**, *81*, 56–65. [CrossRef]
32. Dhakal, D.H.N.; Ismail, S.O. *Sustainable Composites for Lightweight Applications*; Woodhead Publishing by Elsevier: Amsterdam, The Netherlands, 2020; pp. 75–76. [CrossRef]
33. Zakikhani, P.; Zahari, R.; Sultan, M.T.H.; Majid, D.L. Thermal degradation of four bamboo species. *BioResources* **2016**, *11*, 414–425. [CrossRef]
34. Correja, E.A.; Torres, S.M.; Alexandre, M.; Gomes, K.C.; Barbosa, N.P.; Barros, S. Mechanical performance of natural fibers reinforced geopolymer composites. *Mat. Sci. Forum* **2013**, *758*, 139–145. [CrossRef]
35. Mastali, M.; Abdollahnejad, Z.; Pacheco Torgal, F. Carbon dioxide sequestration of fly ash alkaline based mortars containing recycled aggregates and reinforced by hemp fibres. *Constr. Build. Mater.* **2018**, *160*, 48–56. [CrossRef]

36. Kim, D.J.; Naaman, A.E.; El-Tawil, S. Comparative flexural behaviour of four reinforced cementitious composites. *Cem. Concr. Compos.* **2008**, *30*, 917–928. [CrossRef]
37. Da Fonseca, R.P.; Rocha, J.C.; Cheriaf, M. Mechanical properties of mortars reinforced with Amazon Rainforest natural fibers. *Materials* **2021**, *14*, 155. [CrossRef]