proceedings

Proceedings

Mechanical Behavior of Gypsum Composites Reinforced with *Retama monosperma* Fibers †

Djamel-Eddine Aizi * and Meriem Kaid-Harche

Laboratory of Plant and Microbial Production and Valorization (LP2VM), University of Science and Technology Mohamed Boudiaf, BP 1505, El M'naouar Oran 31000, Algeria; kaidharche@yahoo.fr

* Correspondence: aizi_djameleddine@hotmail.fr; Tel.: +213-779922750

† Presented at the 14th International Conference on Interdisciplinarity in Engineering—INTER-ENG 2020, Târgu Mureș, Romania, 8–9 October 2020.

Published: 21 December 2020

Abstract: In this pioneering study, *Retama monosperma* fibers were used in the preparation of a plaster composite dedicated to the field of civil engineering in order to find a substitute for fiberglass as a reinforcement material. *Retama monosperma* (Rtam) is one of the plant species abundantly available in Mediterranean regions. The localization of fibers at the organic level, the extraction procedure, physical and mechanical properties were studied to compare them with other vegetable fibers currently used in the manufacture of biocomposites. The results obtained show the possibility of improving the mechanical properties of plaster by using the fibers of *Retama monosperma*. The purpose of this paper is to promote the fibers of *Retama monosperma* as a building material in civil engineering in order to boost researchers' interests in this Mediterranean plant.

Keywords: gypsum; fiber; stems; *Retama monosperma*; concretes plaster; mechanical characteristics

1. Introduction

Plaster is a white powder produced by the calcination of gypsum. This material is extremely old—it was discovered by humanity in antiquity. Gypsum is a very abundant rock with varied crystalline forms. Gypsum has the formula $CaSO_4$, $2H_2O$, and it is called calcium sulfate dehydrate, or, more simply, dehydrate. Due to its availability in subsoil, relatively low cost, ease of high usage and mechanical characteristics being suitable for many uses, plaster is a widely used construction material, and its use continues to grow, as does the use of prefabricated gypsum products such as tiles and plates. However, plaster appears to be permeable and too brittle [1]. Improving the crack resistance of gypsum is an essential problem which the science of composite materials has naturally responded to. The main idea is to introduce a material in the form of fibers into a binder, called a matrix. It is then possible to obtain extremely resistant materials from a fragile elastic fiber and matrix. Fiber glass first appeared in plaster, and a number of studies have shown that synthetic fiber reinforced plaster materials possess the best mechanical efficiencies [2,3], but it is an expensive reinforcement, and it makes the plates considerably heavier, making them less practical to use on larger sites; besides this, they have a harmful effect on the environment. Due to these kinds of fibers, scientists have been forced to look for a new material. Vegetable fibers are a potential source for a low-cost material from renewable resources, and are environmentally friendly, and they are less gluttonous regarding fossil energy. According to [4], the mechanical properties of plaster are enhanced by using natural fibers.

Nowadays, one of the current trends of the building industry is to develop "green materials": the use of natural fibers as reinforcement of lime plasters plays a leading role in this transition towards renewable materials [5]. Generally, the application of natural fibers is attractive for four main reasons: their specific properties, their price, their health advantages and their recyclability [6]. The fact that these natural fibers offer a low density and good specific properties is an important benefit. Furthermore, the fibers are renewable and have a CO_2-neutral life cycle, in contrast to their synthetic opponents.

Retama monosperma (Rtam) is a shrub 1 to 4 m tall [7], belonging to the Fabaceae family. It grows in coastal regions [8,9]. *Retama monosperma* is able to tolerate salt spray, high temperatures and nutrient-poor soils [9]. Rtam is known for its very fiber-rich stems, but unfortunately until now there has been no study on the valuation of *Retama monosperma* fibers in building construction.

In the present study, the main objective is the modification of the mechanical behavior of gypsum by *Retama monosperma* stem fibers. For this, we attempted to study the influence of proportion and length of *Retama monosperma* stem fibers on the mechanical properties of plaster concretes.

2. Materials and Methods

2.1. Fibers

2.1.1. Fiber Extraction

For this step (Figure 1), a procedure based on the combination of chemical and physical treatments was used [10].

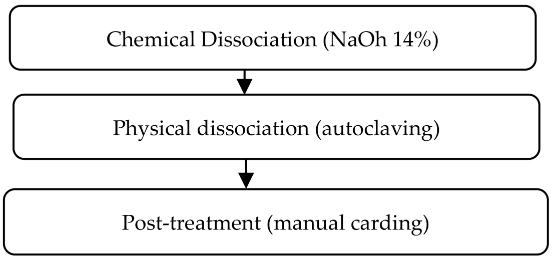


Figure 1. Fiber extraction procedure.

2.1.2. Location of Fibers within the Stem of *Retama monosperma*

Freehand cuts were made on freshly harvested stems of *Retama monosperma*, which were colored with congo red and methyl green in order to reveal the different parts of the stem.

2.1.3. Biometric Study of Fibers

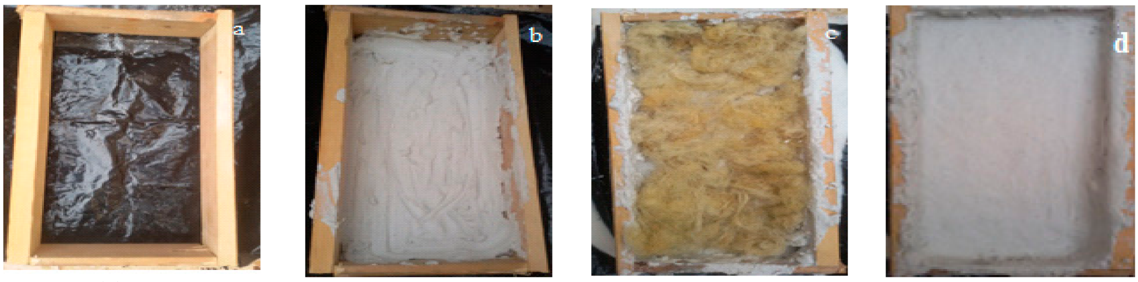
A biometric study requires the separation of elementary fibers from one another. This was carried out according to the protocol of [11] by putting fragments of 1 cm of young stems in a solution of hydrogen peroxide and acetic acid with equal volume, before putting them in the oven at 70 °C for 24 h. Once the fibers were separated, microscopic observations were made using a light microscope with a micrometer incorporated in the eyepiece.

2.1.4. Mechanical Characterization of Fibers

The mechanical properties (tensile strength, elongation and the modulus of elasticity of the fibers) were determined in accordance with standards NF EN ISO 5079(1996), under climatic conditions, T = 20 ± 2 °C, and a relative humidity, RH = 65 ± 2%, using an Instron universal testing machine equipped with a 250 N load sensor and two displacement transducers at a cross-head speed of 0.5 mm/min. Tests data were digitally recorded and the force/deformation curve was plotted.

2.1.5. Density

The density of fibers was determined by using a pycnometer (Figure 2).



(**a**) (**b**) (**c**) (**d**)

Figure 2. Illustration of the different steps of the composite preparation (plaster/fiber): (**a**) first step, (**b**) second step, (**c**) third step and (**d**) fourth step.

2.1.6. Thermogravimetric Analyses (TGA)

Thermogravimetric analyses (TGA) were carried out with an etaram TG/DTA 92 type device. The initial mass of the sample was approximately 10 mg. The heating rate was adjusted to 10 °C/min, with a temperature varying from 20 to 1000 °C.

2.1.7. Scanning Microscopy

A scanning electron microscope was used to examine the fibers' surface topographies and the section of fibers.

2.2. Plaster Concrete

The gypsum used comes from the Knauf factory of Oran in Algeria.

The steps of composite preparation:

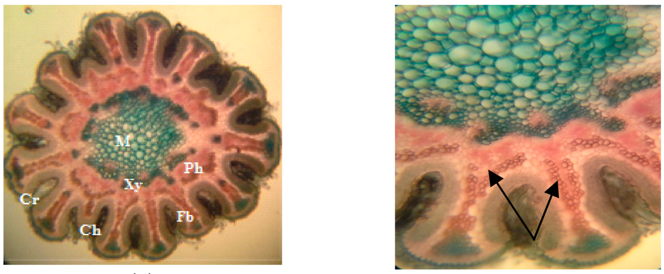
1. The powdered gypsum was mixed with water until a slurry was obtained, which was then spread out in a mold.
2. Production of the composite by the gypsum fiber blend; the proportion of fibers in the composite was 1%.
3. Finally, the surface of each plaster brick was smoothed to have a regular brick surface of 30 cm length and 15 cm width.
4. Mechanical properties relate to flexural strength as well as compressive strength; the results were obtained on MPa.

3. Results and Discussion

3.1. Fiber

3.1.1. Location of the Fibers at the Stems

The cross-section shows the distribution of fibers which go from the epidermis towards the medulla, grouping in clusters between each crypt (Figure 3). While the subepidermal fibers are colored green by methyl green, due to lignified walls, the fibers located in the direction of the phloem are colored in pink by the Congo red, which means that this cellulosic fibers are not lignified yet.



(**a**) (**b**)

Figure 3. Cross-section in a stem of *Retama monosperma*. (**a**) Cross-section observed at (×100), FB: Fibers, Ph: Phloem, Xy: Xylem, M: Marrow, Ch: chlorenchyma; (**b**) cross-section observed at (×400); the fibers are indicated by arrows.

3.1.2. Fibers Extraction

Using the protocol of [10] allowed us to obtain an average fiber yield of 10.5% after manual carding (Figure 4).

(**a**) (**b**)

Figure 4. *Retama monosperma* stem fibers. (**a**) Before manual carding; (**b**) after manual carding.

3.1.3. Physical and Mechanical Properties of *Retama monosperma* Fibers

Table 1 shows the density and mechanical properties of natural plant fibers commonly used in several sectors compared to *Retama monosperma* fibers. As can be seen from Table 1, the tensile strengths as well as Young's modulus of *Retama monosperma* are lower than other fibers mentioned in this table. However, elasticity of *Retama monosperma* is 4.6% higher than other fibers. On the other hand, the *Retama monosperma* fibers show an interesting density of 1.3 g/cm^3. The lower density is interesting where the weight of the structure needs to be reduced. Generally, the tensile strength and Young's modulus of plant fibers increase with the increasing cellulose content of the fibers [12]. Additionally, the structure and microfibrillar angle determine the overall properties of the fibers [13].

Table 1. Mechanical characteristics of some vegetable fibers [11,14–16].

Fibers	Tensile Strength (MPa)	Elongation at Break (%)	Young's Modulus (GPa)	Density (g/cm^3)
Retama monosperma	110	4.6–4.7	13.3	1.3
Flax	345–1035	1.3–3.3	27.6	1.5
Sisal	600	3	12	1.5
Jute	396–773	1.5–1.8	26.5	1.3
Hemp	690	1.6	30–60	1.15
Cotton	287–597	7–8	5.5–12.6	1.5–1.6
Raphia textilis	148–660	2	28–36	0.75
Raphia vinifera	500	4	12.3	-
Kenaf	700	3	55	-

Plant fibers are more ductile if the fibrils have a spiral orientation to the fiber axis. Fibers are inflexible, rigid, and have a high tensile strength if the fibrils are oriented parallel to the fiber axis [17]. However, the origin place and climatic conditions affect the physical-mechanical properties of natural plant fibers. It is also important to note that natural plant fibers with poor mechanical properties may be suitable for nonstructural applications [17].

3.1.4. Geometric Features Including Fiber Length and Width

The fibers extracted from the stems of *Retama Monosperma* have an average length of 155.7 mm and a width of 0.116 mm. Each fiber is microscopically made up of several unitary fibers.

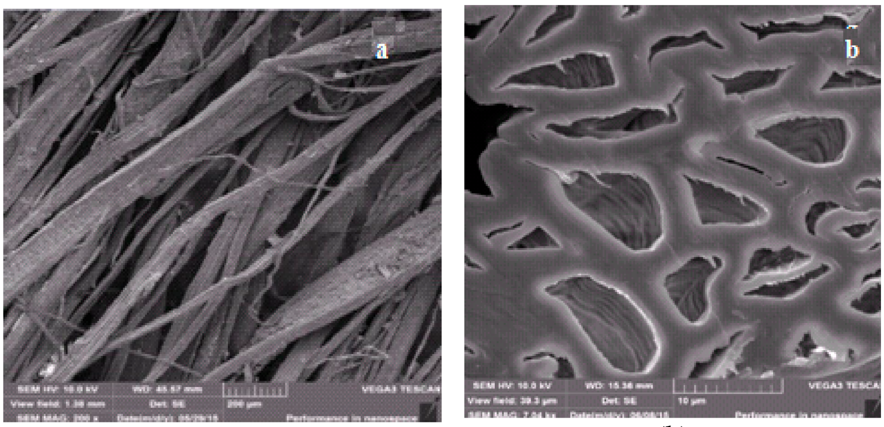
The *Retama monosperma* unitary fiber is 0.7 mm long and 20 µm wide. The comparison between the morphometry of *Retama monosperma* fibers and other fibers (Table 2) shows that the fibers of *Retama monosperma* are small.

Table 2. Morphological structure of some natural fibers [18–22].

Single Fiber	*Retama monosperma*	Cotton	Hemp	Kenaf
Length, mm	0.7	15–56	40–250	1.5–11
Width, µm	20	12–25	16–126	12–36

The morphological characteristics of the fibers, length and width, are important factors for the mechanical performance of fibers. In general, unitary fibers of short length give a fibrous bundle with limited resistance to mechanical traction, because the small unitary fibers need many interfibrillary bonds inside the fiber bundle compared to the long unitary fibers, which reduces the mechanical resistance of the entire bundle.

Microscopic observations with SEM show the appearance of the fibrous bundles of *Retama monosperma* (Figure 5a). The horizontal section of the fibers shows the presence of a triangular lumen in the center of each unitary fiber of *Retama monosperma* (Figure 5b), which gives the fiber a heat insulating capacity.



(**a**) (**b**)

Figure 5. Observation of Retama monosperma fibers using an electron microscope. (**a**) Bundles of fibers (×50,000); (**b**) horizontal section (×100,000).

3.1.5. Thermal Stability

The obtained results show the evolution of the loss of mass fibers as a function of the temperature. In this study, the degradation of fibers started at a temperature of 66.57 °C, with an energy release of 1.55 J and a weight loss of 6.859% of the initial weight of the fibers (Table 3). The first mass loss is linked to the evaporation of water trapped inside the fiber [23]. The degradation continued with the increase in temperature until it reached its peak at the temperature of 312.40 °C where the weight loss was 53.195%. More than half of the mass of the fibers was lost at 312.40 °C. This loss is linked to the cellulose mass loss; the numerous studies in the literature agree that cellulose degrades between 300 and 420 °C [24,25], while pectins and hemicelluloses degrade between 250 and 320 °C [24,25], and also indicate that breaks in glycosidic bonds in cellulose also occur from 200 °C. With regard to lignins, there remains a disagreement regarding their decomposition temperature which could be explained by the complexity of their chemical structure. [26] defined a very wide temperature range from 160 to 900 °C where the lignin degrades. The obtained results show a loss of 32.988% at 451 °C, before a total degradation of fibers at 568.21 °C.

Table 3. Fibers' weight loss and energy released during the thermogravimetry test.

Temperature	Energy Released	Weight Loss (%)
66.57	1.55 J	6.859%
312.40	38.64 J	53.195%
451	35.18 J	32.988%
568.21	6.29 J	2.404%

3.2. Plaster Biocomposites Reinforced with Retama Monosperma Fibers

Use of natural fibers as a reinforcement in a cement matrix has also been practiced for making low-cost building materials such as panels, claddings, roofing sheets and tiles, slabs and beams [27,28]. Sisal and coir are two of the most studied fibers, but bamboo, jute, hemp, reeds and grasses have also been studied for making sheeting materials [29]. Many studies focused on natural fiber composite reinforcement concluded that the tensile strength and modulus of the rupture of composites increased up to a certain fiber length and volume fraction; further increase in fiber length or volume fraction induce a considerable decrease in the composite strength. In this study, two parameters were taken into account—the fibers' lengths and their proportion in the plaster brick. Table 4 shows the obtained results.

Table 4. Mechanical properties of *Retama monosperma* fiber reinforced plaster composites.

Length (cm)	Test	Flexural Strength (MPa)	Compressive Strength (MPa)
0	control	0.7	7.51
10	0.5	1.33	8.75
	1	1.02	7.88
	1.5	1.04	7.91
15	0.5	1.09	10.49
	1	1.12	11.68
	1.5	1.01	9.21
20	0.5	0.51	6.54
	1	0.66	6.37
	1.5	0.47	5.98

Mechanical tests carried out on plaster samples reinforced with *Retama monosperma* fibers reveal that the fibers with a length of 15 cm and fraction of 1% gave the best performance in terms of the flexural strength, with an average of 1.12 MPa, as well as the compressive strength which was 11.68 MPa. According to these results, it is clearly noted that the flexural strength increases considerably with dosage and length of fibers. A clear improvement for different lengths was seen for a dosage of 1% fibers of 15 cm length, after which a loss of the flexural strength was recorded, owing to excess fibers and a bad distribution of the fibers in the matrix, which increased the porosity of the material and reduced the flexural strength. The plaster composite with a small amount of 0.5% *Retama monosperma* fiber has shown an improvement in the mechanical properties of the composite, this is in agreement with the results of [30] where it is reported that, even in small quantities, the behavior of the plaster changes.

Other studies have been conducted on different fibers, such as coconut fibers—Djoudi et al. [31] show that the best result is obtained with a volume fraction of 4% while Mathur [30] in a study on the strengthening of plaster concrete with date palm fibers has found that a fraction of 1.5% gives the best results in terms of tensile strength and bending. The length of the fibers and the optimal fraction in the composite are two parameters that differ from one fiber to another depending on their morphology and their chemical composition which are linked essentially to the organic fiber origin and of the plant itself. In general, the properties of a composite material result from the combination of several factors:

(1) the length of the fibers;
(2) fiber architecture;
(3) the orientation of the fibers;
(4) the fiber–matrix interface.

4. Conclusions

One of the most widely used construction techniques is the use of fibers as a reinforcement in building materials. In Algeria, imported oakum are currently used for the creation of false plaster ceilings. Glass fibers and sisal are the most used reinforcements to date. Finding a replacement for the imported oakum using natural resources and, in particular, the local flora is necessary. Using *Retama monosperma* fibers as reinforcements for plaster is an objective which was demonstrated as being possible in this study by the results obtained in terms of the mechanical properties of the composite plaster/fiber. In order to guarantee the quality of the composite material produced, further research is required to complete this pioneering study.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Eve, S.; Gominaa, M.; Gmouhb, A.; Samdib, A.; Moussab, R.; Orangec, G. Microstructural and mechanical behaviour of polyamide fibre-reinforced plaster composites. *J. Eur. Ceram. Soc.* **2002**, *22*, 2269–2275. [CrossRef]
2. Wu, Y.F. The structural behaviour and design methodology for a new building system consisting of glass fibre reinforced gypsum panels. *J. Constr. Build. Mater.* **2009**, *23*, 2905–2913. [CrossRef]
3. Çolak, A. Physical and mechanical properties of polymer-plaster composites. *J. Mater. Lett.* **2006**, *60*, 1977–1982. [CrossRef]
4. Dalmay, P.; Smith, A.; Chotard, T.; Sahay-Turner, P.; Gloaguen, V.; Krausz, P. Properties of cellulosic fibre reinforced plaster: Influence of hemp or flax fibres on the properties of set gypsum. *J. Mater. Sci.* **2010**, *45*, 793–803. [CrossRef]
5. Le Troëdec, M.; Dalmay, P.; Patapy, C.; Peyratout, C.; Smith, A.; Chotard, T. Mechanical properties of hemp-lime reinforced mortars: influence of the chemical treatment of fibres. *J. Compos. Mater.* **2011**, *45*, 2235–2347. [CrossRef]
6. Bos, H.L. The Potential of Flax Fibres as Reinforcement for Composite Materials. Ph.D. Thesis, Eindhoven University of Technology, University Press Facilities, Eindhoven, Germany, 2004.
7. Selami, N. Etude des Associations Symbiotiques de *Retama monosperma*: Approches Morphologique, Anatomique et Ultrastructurale, Caractérisation Moléculaire des Isolats. Ph.D. Thesis, Université Mouhamed Boudiaf, Oran, Algiers, 2014.
8. Benmiloud-Mahieddine, R.; Abirached-Darmency, M.; Brown, S.C.; Kaid-Harche, M.; Siljak-Yakovlev, S. Genome size and cytogenetic characterization of three Algerian Retama species. *Tree Genet. Genom.* **2011**, *7*, 987–998. [CrossRef]
9. Muñoz Vallés, S.; Gallego Fernández, J.B.; Cambrollé, J. The Biological Flora of Coastal Dunes and Wetlands: *Retama monosperma* (L.) Boiss. *J. Coast. Res.* **2013**, *29*, 1101–1110. [CrossRef]
10. Aizi, D.E.; Kaid Harche, M. Extraction and characterization of *Retama monosperma* fibers. *Afr. J. Biotechnol.* **2015**, *14*, 2644–2651. [CrossRef]

11. Harche, M. Contribution à l'étude sur l'alfa (*Stipa tenacissima* L.): Germination croissance des feuilles différenciation des fibres. Ph.D. Thesis, 3eme Cycle Univ. Sciences et Techniques, Lille, France, 1978.
12. Elenga, R.G.; Dirras, G.F.; Goma Maniongui, J.; Djema, P.; Biget, M.P. On the microstructure and physical properties of untreated raffia texilis fiber. *Compos. Part A* **2009**, *40*, 418–422. [CrossRef]
13. Komuraiah, A.; Shyam Kumar, N.; Durga Prasad, B. Chemical composition of natural fibers and its influence of their mechanical properties. *Mech. Compos. Mater.* **2014**, *50*, 359–376. [CrossRef]
14. Bledzki, A.K.; Gassan, J. Composites reinforced with cellulose based fibers. *Prog. Polym. Sci.* **1999**, *24*, 221–274. [CrossRef]
15. Sandy, M.; Bacon, L. Tensile testing of Raffia. *J. Mater. Sci. Lett.* **2001**, *20*, 529–530. [CrossRef]
16. Béakou, A.; Ntenga, R.; Lepetit, J.; Téba, J.A.A.; Ayina, L.O. Physico- chemical and microstructural characterization of Rhectophyllum. *Compos. Part A* **2008**, *39*, 67–74. [CrossRef]
17. Mohanty, A.K.; Misra, M.; Drzal, L.T.; Selke, S.E.; Harte, R.B.; Hinrichsen, G. Natural fibers, biopolymers, and biocomposites: An introduction. In *Natural Fibers, Biopolymers, Biocomposites*; Mohanty, A.K., Misra, M., Drzal, T., Eds.; CRC Press: New York, NY, USA, 2005; Chapter 1.
18. Djafari Petroudy, S.R. Physical and mechanical properties of natural fibers. In *Advanced High Strength Natural Fibre Composites in Construction*; Woodhead Publishing: Sawston, UK, 2017; pp. 59–83.
19. Ramaswamy, G.N.; Ruff, C.G.; Boyd, C.R. Effect of bacterial and chemical retting on kenaf fiber quality. *Text. Res. J.* **1994**, *64*, 305–308. [CrossRef]
20. Ramaswamy, G.N.; Craft, S. Uniformity and softness of kenaf fibers for textile products. *Text. Res. J.* **1995**, *65*, 765–770. [CrossRef]
21. Rowell, R.M.; Young, R.A.; Rowell, J.K. *Paper and Composites from Agro-Based Resources*; Lewis Publishers: New York, NY, USA, 1997.
22. Batra, S.K. Other long vegetable fibers. In *Handbook of Fiber Science and Technology, Fiber Chemistry*; Lewin, M., Pearce, E.M., Eds.; Marcel Dekker, Inc.: New York, NY, USA, 1998; Volume 4, pp. 505–571.
23. Reddy, N.; Yang, Y. New long natural cellulosic fibers from cornhusks: Structure and properties. *AATCC Rev.* **2005**, *5*, 24–27.
24. Deepa, B.; Eldho, A.; Bibin, M.C.; Bismarck, A.; Blaker, J.J.; Pothan, A.L.; Leao, A.L.; Souza, S.F.; Kottaisamy, M. Structure, morphology and thermal characteristics of banana nano fibers obtained by steam explosion. *Bioresour. Technol.* **2011**, *102*, 1988–1997. [CrossRef]
25. Thi Thu Loan, D. Investigation on Jute Fibers and Their Composites Based on Polypropylene and Epoxy Matrices. Ph.D. Thesis, Dresden University, Dresden, Germany, 2006.
26. Yang, H.; Yan, R.; Chen, H.; Ho Lee, D.; Zheng, C. Characteristics of hemicellulose, cellulose and lignin pyrolysis. *Fuel* **2007**, *86*, 1781–1788. [CrossRef]
27. Albano, C.; Gonzalez, J.; Ichazo, M.; Kaiser, D. Thermal stability of blends of polyolefins and sisal fiber. *Polym. Degrad. Stab.* **1999**, *66*, 179–190. [CrossRef]
28. Gram, H.E. *Durability of Natural Fibres in Concrete*; Swedish Research and Concrete Research Institute: Stockholm, Sweden, 1983.
29. Swamy, R.N. *Natural Fibre Reinforced Cement and Concrete. Concrete Technology and Design*; Blackle: London, UK, 1988; Volume 1.
30. Mathur, V.K. Composite materials from local resources. *Constr. Build. Mater.* **2005**, *20*, 470–477. [CrossRef]
31. Djoudi, A.; Khenfer, M.M.; Bali, A.; Kadri, E.H.; Debicki, G. Performance of date palm fibres reinforced plaster concrete. *Int. J. Phys. Sci.* **2012**, *7*, 2845–2853. [CrossRef]