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MATERIALS ENGINEERING | RESEARCH ARTICLE

Fique as thermal insulation morphologic and thermal characterization of fique fibers

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Abstract: Bio-insulations have gained great interest in recent years due to their potential to reduce energy consumption without negative environmental impacts. Fique is one of the most important crops in Colombia, it has shown a low thermal conductivity which makes it a potential replacement for the common insulation materials. However, there are few works that study its thermal properties. In order to enhance the understanding of fique as thermal insulation, in this paper a morphological analysis of raw fique fibers and Thermogravimetry (TGA) and Differential Scanning Calorimetry (DSC) tests of three fique samples are presented, namely, natural fique without treatment, fique washed with a commercial softener and fique after having been soaked for 24 hours in the same softener.

Subjects: Heat Transfer; Energy & Fuels; Biomaterials & Medical Devices

Keywords: thermal insulation; bio-insulations; natural insulations; fique; Thermogravimetry (TGA); Differential Scanning Calorimetry (DSC)

1. Introduction

Nowadays our society faces the great challenge of Global Warming, which has caused serious problems such as droughts, natural disasters, heat waves and global sea level rise (Haines,



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PUBLIC INTEREST STATEMENT

Buildings consume around 40% of world's energy, which greatly contributes to global warming, so it is essential to reduce that consume. One of the best ways to do it is by means of thermal insulators because it minimizes heat losses when heating or air conditioner systems are used. However, common thermal insulators cause environmental problems and serious human health problems, motivating the use of bio insulation materials made from natural and recycled products. Fique is a plant of great importance for Colombia, recent studies have shown that thermal conductivity of materials derived from the fique can be compared with that of the common commercial insulators, nevertheless, other important thermal properties have not been studied. In order to deepen the knowledge of the fique as a thermal insulator, in the present work a morphological, TGA and DSC analysis of different fique samples is presented.

Kovats, Campbell-Lendrum, & Corvalan, 2006). This increase in Earth's temperature is mainly due to increased emissions of greenhouse gases due to the use of fossil fuels in energy production. To face this problem, it is necessary to develop clean energy sources and reduce the energy consumption of society, which is directly related with the building sector since it is one of the sectors with the highest energy consumption worldwide. It is estimated that buildings consume around 40% of energy, 25% of water and 40% of the world's resources; besides accounting for 1/3 of the global greenhouse gas emissions (EERE, 2018). This situation has stimulated research aimed at improving the energy efficiency of buildings, which is evidenced by the increase in studies on the subject (De Boeck, Verbeke, Audenaert, & De Mesmaeker, 2015) and in the emergence of regulations about it (C. E. para A. L. y el Caribe, 2014; Schiavoni, D'Alessandro, Bianchi, & Asdrubali, 2016).

Within this context, thermal insulators are of great importance since its use is recognized as one of the most efficient ways to reduce energy consumption in buildings (Schiavoni et al., 2016; Tangjuank, 2011; Papadopoulos, 2005; Liu et al., 2017). By using them, energy losses can be reduced through the walls and ceilings of buildings, which result in a significant reduction in energy consumption when heating or air conditioner systems are used. As can be seen in the works of Nandapala and Halwatura (Nandapala & Halwatura, 2016) and Siggelsten and Said (Siggelsten, 2018). However, common thermal insulators are materials made from petrochemical products, such as polystyrene, or from natural sources processed with high-energy consumptions, such as rock wool and fiberglass. This causes environmental problems, due to the high energy consumption, pollutant emissions and waste generation, mainly in the production stage (Liu et al., 2017; Asdrubali, D'Alessandro, & Schiavoni, 2015) and serious human health problems caused by its harmful components (Binici, Aksogan, & Demirhan, 2016; Mati-Baouche et al., 2014; Mounika, Ramaniah, Ratna Prasad, Rao, & Reddy, 2012). These problems have motivated the use of bio insulation materials made from natural and recycled products, which could be developed without requiring high energy consumption and without risk to health. Bio insulations have been studied since 1974 but aroused great interest since 2010 to present, due to the increased use of air conditioning systems, greater environmental awareness and changes in the uses of biomass (Liu et al., 2017). A full review of this topic can be found in the works of Liu et al. (2017), Asdrubali et al. (2015), Lopez Hurtado, Rouilly, Vandenbossche, & Raynaud (2016), Madurwar, Ralegaonkar, & Mandavgane (2013), Ingrao et al. (2015) and Kymäläinen y Sjöberg (2008).

Fique (*Furcraea bedinghausii*) is a plant of great importance for Colombia, to the point of being the first producer worldwide with a production of about 30,000 tons/year (Navacerrada, Díaz, & Fernández, 2014). However, there are few works about its use as thermal insulation. In the reported studies it has been shown that thermal conductivity of materials derived from the fique can be compared with that of the common commercial insulators (Navacerrada et al., 2014; Muñoz & Cifuentes, 2007; Navacerrada et al., 2016; Navacerrada et al., 2013; Monsalve, Bolaños, Lopez, & Toro, 2014). Some of the mentioned works, the ranges of thermal conductivity obtained, the method used for its measurement and the sample characteristics are shown in Table 1. Nevertheless, other important thermal properties have not been studied, such as specific heat, which is crucial data for the evaluation of thermal dynamics properties. In order to deepen the knowledge of the fique as a thermal insulator, in the present work a thermal study of three fique samples is presented, namely, natural fique without treatment, fique washed with a commercial softener and fique after having been soaked for 24 hours in the same softener. It is presented a morphological analysis and the results of Thermogravimetry (TGA) and Differential Scanning Calorimetry (DSC) tests of the samples.

2. Materials and methods

When studying the performance of bio insulations, tests are usually carried out on samples manufactured by means of one of the following methods: bonding molding, pressing molding, hot-

Table 1. Thermal conductivity of insulations based in fique fibers

Study	Thermal Conductivity [W/m.K]	Method of Measurement	Sample Characteristics
Navacerrada et al. (2014)	0.037–0.078	High insulation box of 400 × 400 × 400 mm heated inside by means of a 100 W light bulb. This is not a standard method, however, it has an uncertainty of less than 10%.	Fique. Samples of different grammages constituted by short fibers arranged in a non-textured way with a superficial covering of polymer.
Muñoz y Cifuentes (2007)	0.028–0.075	Guarded-Hot-Plate Apparatus—ASTM C 177–63: Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission	Samples made of four commercial presentations of fique: chopped fiber (density: 285.416 g/m ³), fiber without spinning (density: 321.726 g/m ³), a commercial product called “teja de guata” (density: 103.437 g/m ³) and chopped fibers embedded in a thermosetting resin matrix (density: 336.384 g/m ³).
Navacerrada et al. (2016)	0.037	Heat Flow Meter—HFM 436 Lambda. ASTM C518 Standard Test Method for Steady-State Thermal Transmission Properties.	Non-woven fiber samples made by two methods: the manual method, that requires the use of a binder and a press, and by means a mechanism of needles, that allows linking the fibers together.
Navacerrada et al. (2013)	0.03–0.08 (woven)/0.13 (nonwoven)	High insulation box of 400 × 400 × 400 mm heated inside by means of a 100 W light bulb. This is not a standard method, however, it has an uncertainty of less than 10%.	Woven and non-woven fique fiber samples provided by a Colombian company.
Monsalve et al. (2014)	0.028–0.075	Guarded hot plate—UNE-EN 12.667:2002 Standard Test Method.	Tile sandwich consisting of three layers, the outer two made of a laminar composite cement matrix reinforced with fique fiber and aluminum oxide powder, and the middle layer made of the pulp of recycled newspaper.

pressing molding, injection molding, foaming molding, in natural (original/raw) form and others, e.g. needle-punching and hydroentanglement, aerosol processing method (Liu et al., 2017). In this work the performance of raw fique fibers was studied, therefore, tests were carried out with fique samples in natural form. In order to have a better knowledge of the constitutive form of the fique fibers some raw fibers were cut crosswise and observed in the Field Emission Scanning Electron Microscope (SEM) of the “Universidad Pontificia Bolivariana” (TESCAN—MIRA 3 FEG-SEM). Uncut fibers were also observed in the Microscope SEM.

In addition, tests were carried out on the fique washed with Soflan Suavitel®, a commercial fabric softener used to clean sediments deposited on the surface of the fibers, composed of a cationic surfactant type quaternary ammonium salts, fragrance, dye, preservative and water. In order to analyze the relationship between the thermal properties and the effect of the softener in the washing process, in spite of the raw fiber samples, two types of samples of fibers washed with softener were used: one removing the softener instantaneously (passed through softener) and the other after leaving them to soak for 24 hours. To determine the fiber weight changes with temperature, and thus to have a measure of the samples thermal stability, the Thermogravimetry (TGA) method was used, by means of the TA Instruments Q500 TGA test bench of the “Universidad Autónoma de Occidente” in Cali, Colombia. Sample masses were: 3.567 mg for raw fique and fique passed through a softener, and 3.276 mg for fique soaked in softener for 24 hours. All TGA tests were carried out at a heating ramp at 10°C/min, from 25 to 500°C. Scanning Difference Calorimetry (DSC) tests were also performed on the samples, in these tests the energy required for temperature change was measured. That tests were carried out in the TA Instruments DSC Q2000 test bench of the thermal analysis laboratory of the “Universidad Autónoma de Occidente” in Cali, Colombia. at a scanning rate of 10 °C/min.

3. Results

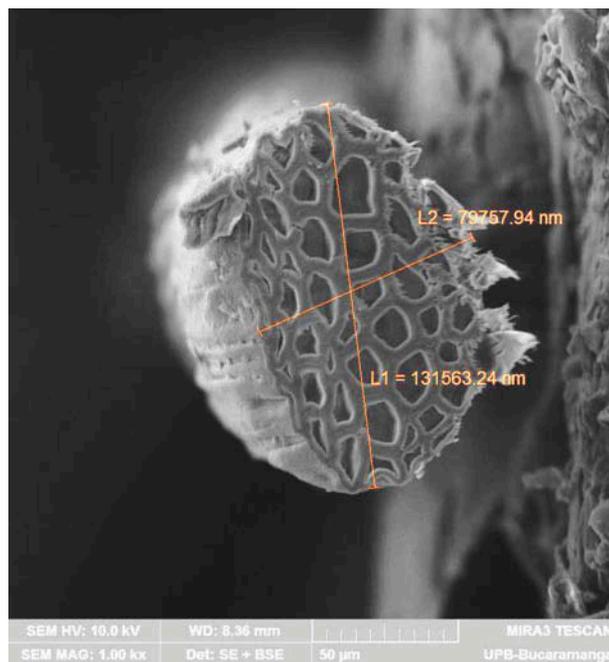
3.1. Fique morphology

Figures 1 and 2 show SEM images of raw fique fibers, Figure 1 shows images of fiber cross-section and Figure 2 an image of fiber longitudinal section. It can be seen that fibers have a hierarchical structure composed of vascular vessels of elementary fibers (fibrils) and cellulose micro-fibrils packages in a network of hemicellulose and lignin, microfibrils are wound helically along the fiber axis and form hollow cells. Each fiber cell is made up of four main parts, namely the primary wall, the thick secondary wall, the tertiary wall and the lumen. From the cross sections it was observed that the diameter of the fiber and the diameter of the lumen varies in size from 10 µm to 20 µm, but it generally has a geometry that approximates a well-defined circle. From the longitudinal images, it was observed that the fique fibers have an irregular shape, with variations of thickness and superficial damage (breakage), which can be produced in the mechanical defibering process carried out by farmers.

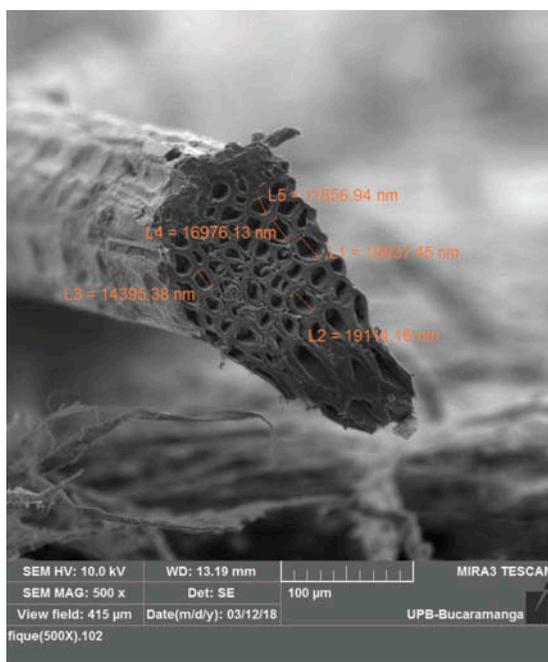
3.2. Thermogravimetry

Figure 3 shows the results of the TGA test for the samples of raw fique, fique passed through softener and fique soaked in softener for 24 hours, respectively. The green curve represents the weight loss of the fibers with temperature and the blue one represents the derivative of weight loss as a function of temperature. For all the TGA curves it was observed that all the samples present four loss of weight processes, whose percentages of loss can be observed in Table 2. The first process can be attributed to the loss of moisture from the specimen, in this stage, there is a small weight change of below 10% followed by a stable process without weight loss. The second and third loss of weight processes are due to the decomposition process of hemicellulose and cellulose, respectively; these stages present the maximum weight loss rate with two temperatures of appreciable weight loss (T1 and T2). Unlike hemicellulose, cellulose is a linear polymer without branches and a higher order, which gives it a higher thermal stability (Yang, Yan, Chen, Lee, & Zheng, 2007; Ovalle-Serrano,

Figure 1. Cross section of raw fique fibers. a) SEM imagen: 1000 x magnification, 10 kV HV and 129.85 nm sptosize. b) SEM imagen: 500 x magnification, 10 kV HV and 127.46 nm sptosize.



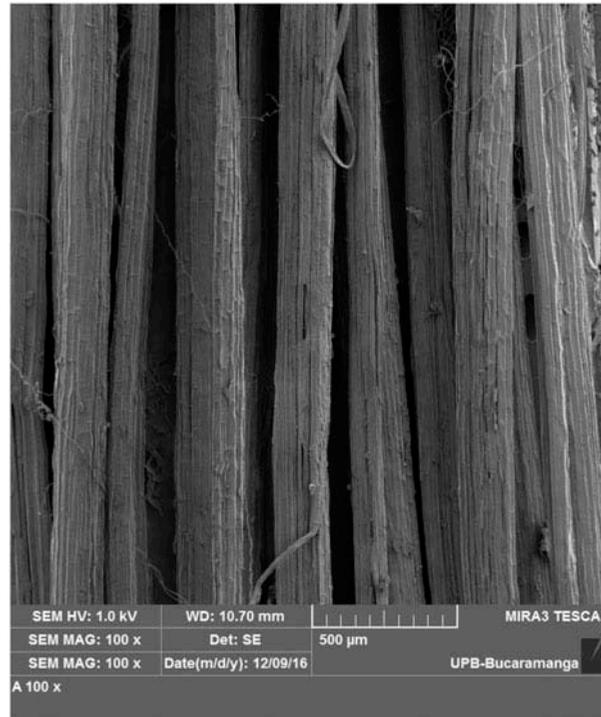
a)



b)

Blanco-Tirado, & Combariza, 2018), because of that, the point of maximum decomposition corresponds to the cellulose process (T2), reached at 365, 385 and 380 °C for the samples of natural fique, fique passed through softener and fique soaked in softener for 24h, respectively; which is similar to the point for maximum cellulose decomposition of other lignocellulosic materials (Ovalle-Serrano et al., 2018). Finally, the fourth process is due to degradation and combustion of the samples. As can be seen from the figure, material decomposition starts at 280°C for natural fique samples and 190°C for samples of fique washed with

Figure 2. Longitudinal image of raw fique fibers magnified at 100.



softener, which shows that fique could be used as thermal insulation of homes without suffering thermal degradation problems since buildings are subjected to temperatures below these values. Also, it can be observed a faster degradation when fique is washed with softener, that is because lignin gives resistance and rigidity to fibers and it is removed with washing, leaving cellulose fibrils exposed.

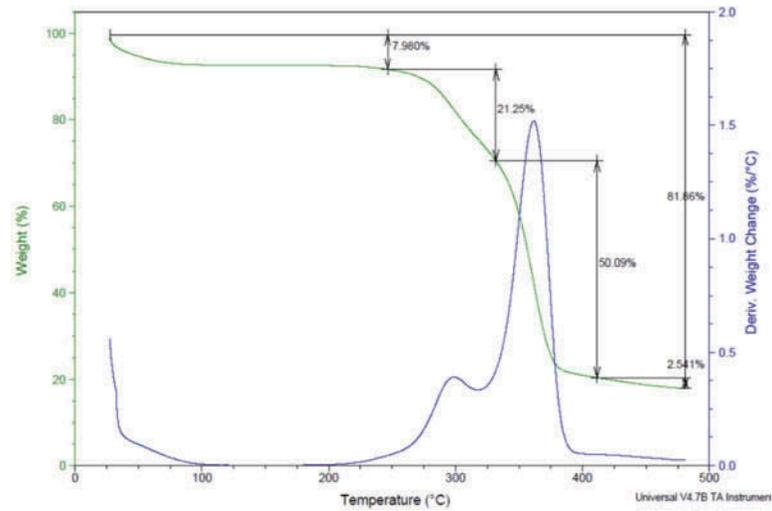
3.3. Differential scanning calorimetry

By means of the DSC tests, it was observed that all the samples present a step-type transition, due to the glass transition of the fiber, followed by an endothermic transition which can be attributed to fiber dehydration, and an exothermic transition which can be attributed to a possible combustion of the material due to the high temperature reached. In addition to the above, samples of fique passed through the softener and soaked in softener for 24 hours present a small transition after the main dehydration process that can be attributed to a small structural water release. Temperatures in which these transitions occur and the energy transferred vary for each sample. To illustrate the above, Figure 4 shows the DSC curve of the samples of fique passed through softener and Table 3 presents the comparison of enthalpies involved in the phase transitions of all the samples. It was observed that the energy requirements for the temperature change decrease when the fibers have been washed, which could be due to the degradation that the fiber suffers due to washing.

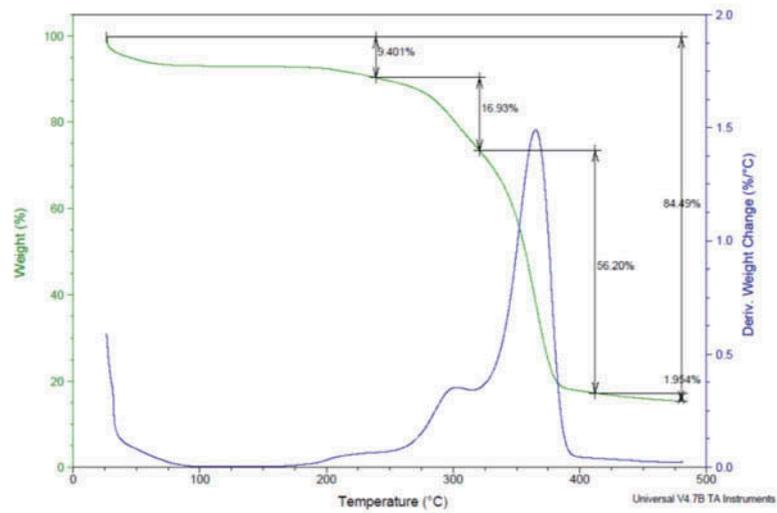
4. Conclusions

In this work, a morphological analysis of raw fique fibers was carried out and measurements of the weight change and absorbed energy were taken as a function of the temperature of three fique samples: natural, passed through the softener and soaked in fabric softener for 24 hours. all with the aim of deepening the knowledge of that fiber as a potential material for thermal insulation applications.

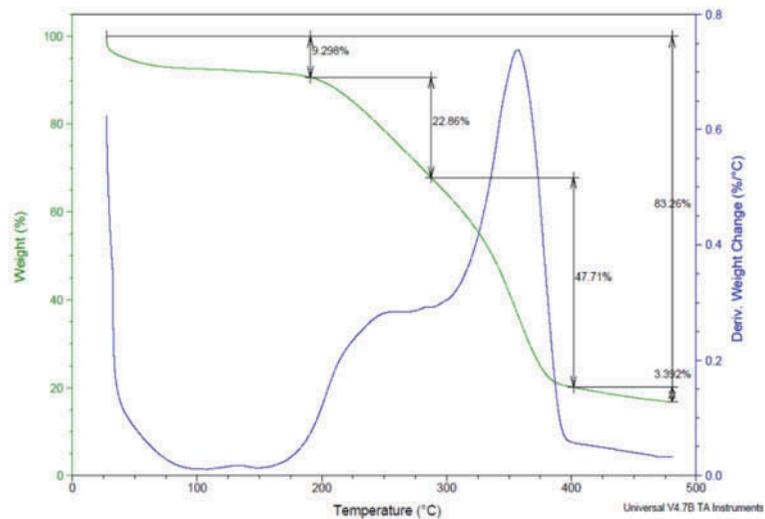
Figure 3. Weight variation with the temperature of fique samples. a) Raw fique, b) Fique passed through softener and c) Fique soaked in softener for 24 hours.



a)



b)

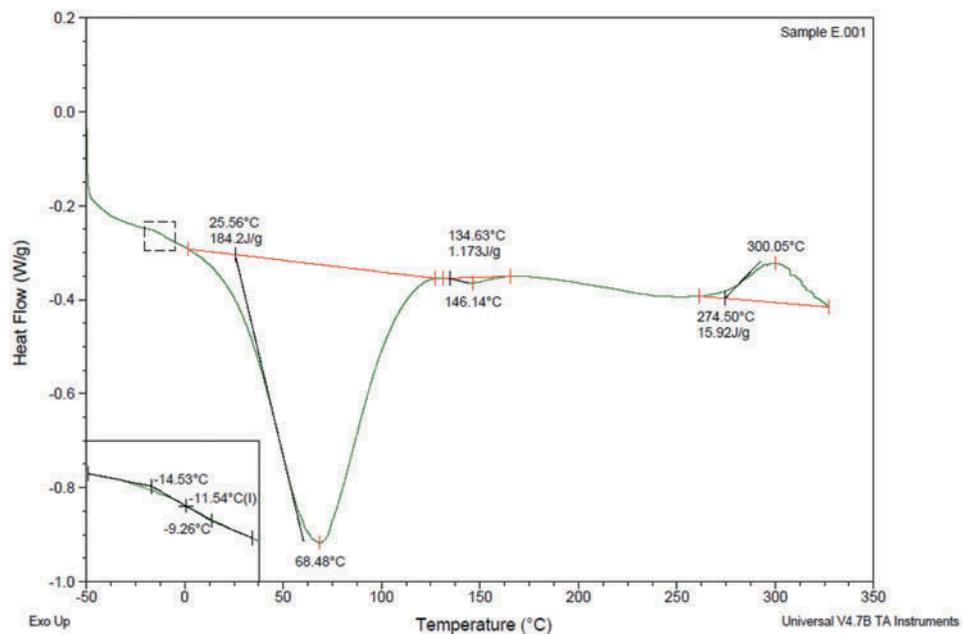


c)

Table 2. Temperatures of appreciable weight loss and weight losses of the samples in the TGA test

Sample	Natural fique	Fique passed through softener	Fique soaked in softener-24h
T1 (°C)	255	240	195
T2 (°C)	365	385	380
Weight loss 1 (%)	7.98	9.40	9.30
Weight loss 2 (%)	21.25	16.93	22.86
Weight loss 3 (%)	50.09	56.20	47.71
Weight loss 4 (%)	2.54	1.95	3.39
Total weight loss (%)	81.86	84.49	83.26

Figure 4. DSC curve of the fique samples fique passed through softener.



After visualizing the fiber cross-sections, it was observed that it has several hollow cavities in its structure, which can explain its low density and, due to the air inside cavities, the low thermal conductivity reached by the material in previous studies.

By means of TGA analysis, a material degradation temperature of 280 °C was observed for natural fique samples, and 190 °C for samples of fique washed with softener. With which it is concluded that fique can be used as thermal insulation of homes without suffering thermal degradation problems since buildings are subjected to a temperature below these values. It was also observed that the material loses resistance to degradation when washed with softener, that is because in this wash lignin is removed from the superficial part of the fibers. Lignin gives resistance and rigidity to fibers so when it is removed leave cellulose fibrils exposed and, as a result, make the fibers weaker.

Through the DSC tests, it was observed that the energy requirements for the temperature change decrease when the fibers have been washed, which can negatively affect its performance as a thermal insulator. This may also be due to the degradation that the fiber suffers due to washing.

Table 3. Comparison of enthalpies involved in the phase transitions of the samples

Fique Sample	Vitreous transition		Anomaly 1 (Endothermic)		Anomaly 2 (Endothermic)		Anomaly 3 (Exothermic)	
	Temperature (°C)	Enthalpy (J/g)	Temperature (°C)	Enthalpy (J/g)	Temperature (°C)	Enthalpy (J/g)	Temperature (°C)	Enthalpy (J/g)
Natural	-7,020	2,00	2,00°C—130°C	247,3	-	-	301,28	15,93
Softener	-14,53	1,50	2,00°C—130°C	184,2	134,63	1,173	300,05	15,92
24 h Softener	-11,22	0,90	2,00°C—130°C	176,6	148,81	5,171	293,75	8,99

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