

Statistical Analysis and Mechanical Behavior for Polypropylene Composites Reinforced with Benzoylated Sugarcane Fibres

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Abstract

Sugarcane fibres, in the form of bagasse, are abundant by-products from the sucro-alcohol industry and are commonly used as reinforcement in composite materials. The main objective of this work concerns the modification of the sugarcane bagasse fibers (*in natura* and pulps rich in cellulose) by benzoylation. Unfortunately, the natural fibres have hygroscopic properties, causing poor fibre/matrix interaction. In order to encourage non-polar properties in the fibres, this current work is concerned with the feasibility of aromatic group insertion in natural fibre structures using benzoylation reactions. Sugarcane fibres were soaked in aqueous NaOH and mixed with benzoyl chloride. After chemical treatments, the modified sugarcane fibres were characterized by weight gain, Fourier Transformer Infrared-FTIR, Scanning Electron Microscopy SEM, thermal analysis, mechanical tests and statistical analysis. Results show that there was significant weight gain during the first hour of the reaction. Esterification reaction efficiencies were confirmed by FTIR. SEM characterization revealed that the chemical treatment changed the morphology of the fibres. Thermogravimetric-TG curves illustrated different behaviour between modified and non-modified fibres. Mechanical properties show that the benzoylated fibres insertion caused an increase in the tensile strength in regard to non-modified fibres. Finally, the results for strength obtained from the uniaxial tensile tests demonstrated the effectiveness of treating the fibres with benzoyl groups, with condition C6 providing the best results, followed by condition C7.

Keywords: Polypropylene (PP); Sugarcane bagasse fibres; Mechanical properties; Statistical analysis

Introduction

Sugarcane bagasse is an abundant agro-industrial by product in Brazil. Although it is used in many different applications [1], the world still produces an excess of approximately 54 million tons annually. This agricultural material therefore represents an abundant, inexpensive, and readily available source of renewable lignocellulosic biomass [2]. Sugarcane straw fibre is also a readily available lignocellulosic material due to the recent mechanization of harvesting, and is now being evaluated. The estimated annual world supply of sugarcane straw is approximately 30 million tons [3].

Sugarcane bagasse and straw fibres are lignocellulosic materials predominantly composed of cellulose, hemicellulose and lignin. The two former components are hydrophilic and the latter is hydrophobic [2]. The hydroxyl groups, which contribute greatly to the hygroscopicity of the materials, are attached to cellulose, lignin and hemicellulose and can be chemically modified to possess hydrophobic properties. Overall, natural fibres have proved to be an excellent reinforcement in polymers [4,5]. However, the natural fibres hydrophilic nature is a significant disadvantage when they are being used as reinforcement in polymers for structural applications [6,7]. Many methods for fibre surface modifications, such as mercerization, latex coating, gamma irradiation, silane treatment, isocyanate treatment, acetylation and peroxide treatment, have been tested as methods to reduce the hydrophilicity and decrease water absorption [8-10].

Cellulose is very susceptible and reactive in chemical reactions due to the presence of hydroxyl groups [11]. The three hydroxyl groups in the glucose unit may be partially or completely replaced by other functional groups: one at the primary carbon and two at the secondary carbons [11,12]. This work use chemical modifications by esterification with benzoyl chloride, respectively, as illustrated in Figure 1. These

chemical modifications are accompanied with a pretreatment in aqueous NaOH to activate the hydroxyl groups, transforming them into alkoxides and facilitating the subsequent reaction [13]. The insertion of aromatic rings in the fibres chemical structure promotes stronger interaction between the fibre and matrix in composite materials. We chose to use benzyl and benzoyl groups because of their reaction facility and non-polar structure, similar to that of lignin.

Lignin is the major non-carbohydrate found in plant cell walls. Chemically, it is different from cellulose and hemicellulose, and it is more difficult. It has cross-connections and a three-dimensional macromolecular structure formed from phenolic units. The aromatic nature of the phenolic units gives lignin its apolar and hydrophobic characteristics [5,10].

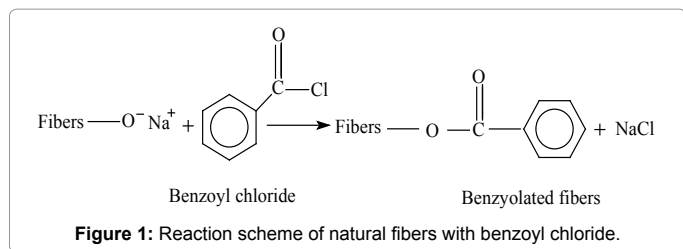
The extent of the reactions was monitored by weight variation as a function of time. After the reaction, benzoyl groups substituted the hydrogen in the OH group and the resulting weight difference between the initial and final polymers was calculated. Infrared spectroscopy was used to determine possible modifications in the lignocellulosic materials [14]. Characteristics of the modified sugarcane fibres were further investigated by FTIR, as developed by [15]. We used two

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different thermal analysis techniques to study the modified sugarcane (DTG) provide information about the nature, extent of degradation and thermal stability of the materials [16].

The use of lignocellulosic fibres and its components as raw material in the composites has been considered as a technological progress in the context of sustainable development. Composite materials have received a crescent attention, particularly in regard to applications in industry segments, e.g., automotive and furniture [17].

The search in turn of new alternatives for materials that offer advantages to the environment has led to growing research on the application on natural fibres as reinforcement of polymers, mainly due to their high stiffness, low density and because the fibres are also highly favorable from the standpoint of the modulus when compared to other polymeric materials. In addition to these characteristics, natural fibres have other advantages, such as biodegradability, low cost, low abrasiveness, ease in processing, recyclability and low power consumption during preparation [4,18]. In addition, the natural fibres are derived from renewable resources; they are available in large quantities, contributing even further to the rising interest in studying these materials. The use of natural fibres in composites presents some disadvantages, such as high moisture absorption, low decomposition temperature and poor adhesion between the fibre and matrix. Despite these drawbacks, the use of these fibres in composites has been growing due to the possibility of circumventing these problems [18]. Polypropylene is a semi crystalline polymer obtained by polyaddition, and it is widely used as a thermoplastic in engineering applications. The addition of fibres in polymer matrices, such as polypropylene is known to modify the mechanical properties of the resulting composite [5,10].

In this work, polypropylene was used as the matrix, and as reinforcement non-modified and modified sugarcane bagasse fibre. This work describes an investigation of mechanical behavior of polypropylene composites reinforced with benzoylated sugarcane bagasse fibres. The insertion of high modulus natural fibres in thermoplastic polymer matrices (low modulus) can contribute to the obtaining of materials with remarkable properties. Therefore, the mechanical behavior of composites reinforced with fibres shows that these materials can perform structural and non-structural applications. The synergetic characteristics of composite materials depend on the initial characteristics of the reinforcement or the matrix [5,10].

The modulus of elasticity in tension and bending are directly influenced by the high modulus of the fibres [19]. Gassan and Bledzki [20] found that the flexural strength of jute/ polypropylene composites increases when the amount of fibres grows. The method of experiment factorial planning was also used. And then, scanning electron microscopy was employed to evaluate the morphological aspects of the fibres after the modification process [17].

The main objective of this work concerns the modification of the sugarcane bagasse fibres (*in natura* and pulps rich in cellulose) by benzoylation. We study the modified fibres by weight gain, FTIR characterization and thermal analysis, with also to do, statistical

analysis and mechanical behavior for polypropylene composites reinforced with benzoylated sugarcane fibres.

Experimental

Chemical modification of sugarcane fibres by benzoylation

Two grams of lignocellulosic material were added to a 50 mL Becker flask and mixed with 20 mL of aqueous NaOH (18% w/v). After 2 hours, aqueous NaOH (10% w/v) and distilled water (total of 50 mL) were added. The mixture was then transferred to a 500 mL round flask with the introduction of 10 mL of benzoyl chloride. The temperature was raised to 55°C, and the reaction proceeded for 1 to 5 hours.

Characterization of modified materials by FTIR

Samples of modified materials were analyzed using FTIR spectroscopy. The samples (2 mg) were ground with KBr (200 mg) and formed into pellets. The spectra were recorded with a Perkin Elmer Spectrometer (Spectrum One) in the 400-4000 cm⁻¹ range, using 16 scans and a resolution of 4 cm⁻¹.

Thermogravimetric analysis (TGA)

TGA were carried out using a Perkin Elmer (TGA 7) instrument, in a N₂ atmosphere, from 30 to 650°C with a 10°C.min⁻¹ heating rate.

Composite preparation, Scanning Electron Microscopy (SEM)

The composites were prepared in batches of 50 g in a thermo kinetic mixer (model MH - 50H) rotating at 5250 rpm with fibre fractions of either 10, 15 or 20% by weight (% wt.) / PP. Morphological analysis of modified fibres was performed using a JEOL JSM - 7001F instrument (SEM). The fibre surfaces were coated with gold and then imaged using the SEM operated at 20 keV.

Mechanical tests

For all standard tests, composites were analyzed in an “Instron” universal-testing machine (model 4301), equipped with pneumatic claws. In the tensile tests, five specimens of composites were analyzed, with dimensions in agreement with the ASTM D 638. In the flexural tests, five specimens were analyzed with dimensions in agreement with the ASTM D 790 standard [21,22].

Statistical analysis

Statistical analyses of different composites, considering fibre content and treatment of fibres were performed. Tensile strength (MOR_t) and flexural strength (MOR_f) analysis was performed, leading to a full factorial design type 2⁴, generating eight experimental conditions (EC), laid out in Table 1.

Analysis of variance was employed to verify the effect of the treatment on the fibres, the fibre content, and the interaction between them on the investigated mechanical properties [23].

EC	Treatment	Fiber Fraction (% wt.)
C1	non-modified	0
C2	non-modified	10
C3	non-modified	15
C4	non-modified	20
C5	Benzoylated	0
C6	Benzoylated	10
C7	Benzoylated	15
C8	Benzoylated	20

Table 1: Experimental conditions (EC) in terms of fiber content and treatment.

Results and Discussion

Chemical modification of sugarcane fibres by benzoylation

As mentioned previously, the procedures of surface modification were applied to sugarcane bagasse. About the benzoylation treatment, it is observed a consistent weight gain as a function of time, as shows in the Table 2.

It is interesting to note that from 1 h reaction with benzoyl chloride was observed slight weight variations (loss of 1.5% to 4.6%). This small change in all the reaction time, it can be related to the benzoylation prevent hydrolysis of the cellulose, leaving the cellulose fibres better preserved. It is possible to estimate that there was incorporated group benzoylated on the cellulose structure. For the experiments in 4 h, the blank presented a weight loss of 47.5% and for benzoylated materials and an increase of weight of 4.6% resulting in an total increase in weight (relative to blank) 120.2%. Thus, it is possible the incorporation of up to two groups per benzoylated cellulose glucose unit.

Characterization of modified materials by FTIR

Products resulting from the benzoylation reactions were analyzed using an FTIR spectrometer. The lignocellulosic materials, in the form of bagasse pulps and bagasse *in nature* were modified separately for different reactions times and each obtained product was analyzed. During the chemical modification of bagasse, there may have been a concurrent reaction between lignin and hemicellulose with cellulose, due to presence of OH groups.

Benzoylated pulps that contain approximately 80% cellulose provide the best medium to investigate the chemical modification. Figure 2 compares the FTIR spectra of bagasse pulp modified by benzoylation to those of non-modified bagasse pulp. The insertion of benzoyl groups in the cellulose structure through the esterification reaction can cause further changes in the absorption properties in the infrared region. We observed decreases in OH band intensity (3400 cm^{-1}), typical of intermolecular hydrogen bonds (large strip of C-H stretching of cellulose - 2900 cm^{-1}). In C, we observe the appearance of a carbonyl band from ester. D and E delimit the in-plane angular deformation of carbonyl band from $1100 - 1300\text{ cm}^{-1}$. At a wavenumber of 718 cm^{-1} (F), a band appeared, attributed to a mono-substituted aromatic ring [24].

Thermogravimetric analysis (TGA)

The modified materials and the respective controls were analyzed by Thermogravimetric (TG) analysis. TG analysis was used to evaluate the thermal stability of the samples. Figure 3 shows the curve behavior of thermal degradation on derivatives, compared to the control. The curves show that the materials without chemical modification present a higher initial weight loss than the derivatives.

Table 3 shows the percentage of weight loss between 100°C and 500°C , as well as the degradation peak temperatures calculated from the digital data of TG. The weight loss of modified materials after 400°C was higher than that of the non-modified materials. This suggests that the chemical modification of the fibres result in structures that are less stable at high temperatures.

The weight loss behavior for benzoylated bagasse pulp was similar to non-modified materials at 200°C . Between 400°C and 500°C , however, the weight loss was more accentuated for modified pulps, reaching 98.7%. Nevertheless, the benzoylated bagasse pulp had a higher degradation temperature 361.5°C than the other analyzed materials did.

Reaction time (h)	Variation of weight (%)
Branco (4h)	-47.5
1	-1.4
3	-1.5
4	4.6
5	2.0

Table 2: Variation of weight as a function of reaction time.

Samples	Weight loss (%)				Degradation temperatures ($^\circ\text{C}$)	
	100°C	200°C	400°C	500°C		
Benzoylated bagasse pulp	3.2	3.8	90.8	92.1	45.3	361.5
Bagasse pulp	3.1	5.3	71.3	75.4	56.3	324.4

¹Sets obtained by DTG curves; ²Materials modified after 4 h reaction; ³Control of chemical modification (5 h reaction).

Table 3: Weight loss at different temperatures and temperatures of degradation peaks temperatures of several materials from chemical modification of pulps.

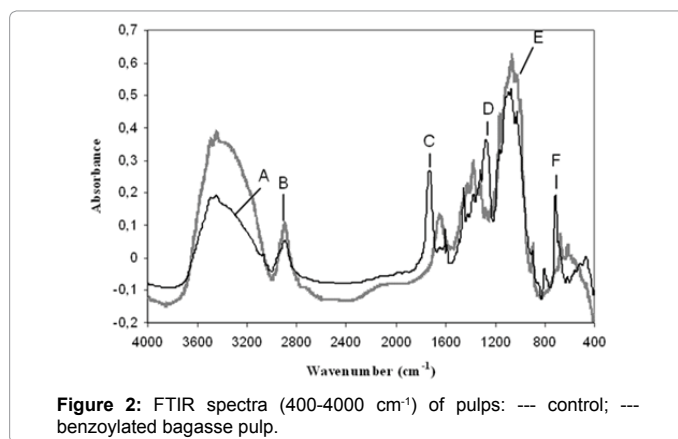


Figure 2: FTIR spectra ($400-4000\text{ cm}^{-1}$) of pulps: --- control; --- benzoylated bagasse pulp.

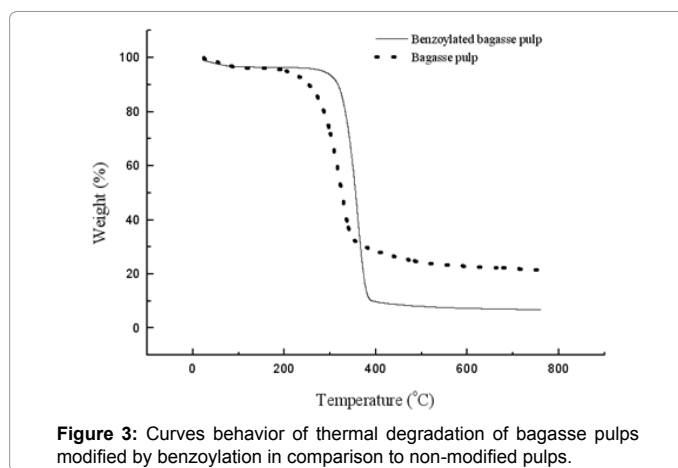


Figure 3: Curves behavior of thermal degradation of bagasse pulps modified by benzoylation in comparison to non-modified pulps.

Composite preparation, Scanning Electron Microscopy (SEM)

A solubilization in part of the lignin was also observed. The treatment effectiveness was evaluated by SEM, and the Figure 4 shows the morphological aspects of the fibres. SEM characterization revealed that the chemical treatment changed the morphology of the fibres.

The *in nature* and modified fibres showed distinctions, as the presence of pits (bagasse) and plasticization (benzoylation) caused by fibre degradation. There was a decrease in lignin, hemicellulose and extractives content after treatment, improving the compatibility

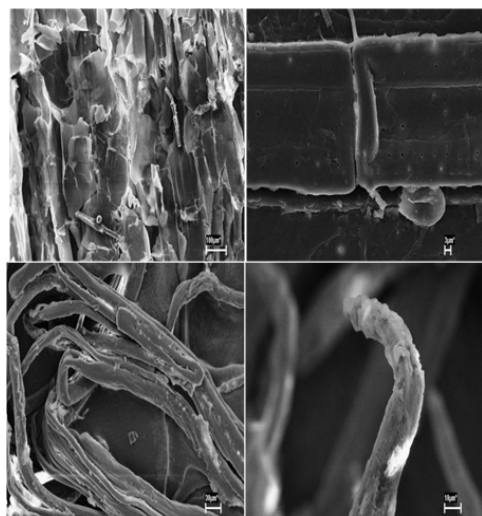


Figure 4: SEM micrographs of sugarcane (A, B) bagasse fibers *in nature* and (C, D) benzoylated fibers.

between the fibres and the matrix [25]. The chemical modification of the fibres was confirmed through SEM. That is, this treatment causes changes to the surface structure of the fibre.

After the chemical modification, the polypropylene (PP) composites reinforced with 10, 15 or 20% by weight of non-modified and benzoylated sugarcane bagasse fibres were obtained throughout a thermo kinetic mixer turning at high speeds to process fibre and PP batches.

The mixing between the polymer matrix and the fibre is promoted by PP melting, due to the heating of the system. Mixing time production for the fibre and the PP matrix varied according to fibre type and content. It was observed that the mixing time for preparation of composites with 10% by weight of fibres reaches to 200 s and for 20% by weight, 750 s. The increased mixing time creates a greater amount of fibres because of the longer fibre/ fibre contact. Hence, the matrix does not easily enter into direct contact with the wall of the bipartite capsule. This causes the fusion of the matrix and subsequent incorporation of the fibre. Furthermore, long mixing times may cause excessive breakdown and thermal degradation of fibres, which will negatively affect the composite properties [17,18].

Mechanical tests

Various composite systems were obtained, as described in Table 4, using polypropylene as matrix and both *in nature* and chemically modified fibres as reinforcements.

Table 4 shows that the composites containing benzoylated fibres could be adhering more to the polymer because of the presence of the polar groups (benzoyl), which increases the values for tensile strength. Thus, the values for tensile strength increased from 8.7% for 10 wt.%, 13% for 15 wt.% and 9.3% for 20 wt.% for benzoylated fibres when compared to the non-modified fibres. Flexural strength, on the other hand, is impaired in the composites reinforced with benzoylated fibres, although the rigidity of the material leads to the rupture of all BB composites independent of the composition. Something that was not observed for any composite reinforced with sugarcane bagasse *in nature*.

Statistical analysis

Table 5 shows the results for the mechanical properties of the eight investigated experimental conditions. Table 6 shows the analysis

of variance of the eight conditions of the full factorial design on the investigated mechanical properties. Underlined are the P-values less than or equal to 0.05 (5%), which indicate significance up to 95% [23,26]. These values are presented in conjunction with their corresponding R² coefficients of determination.

In analysis of variance both samples are assumed to be extracted from independent populations, which can be described by a normal distribution. The Anderson-Darling normality test was therefore employed and the normal distribution of the investigated responses was established with all P-values being greater than 0.05 and falling in the interval between 0.127 and 0.604 [23,26].

The factors investigated in Table 5, treatment was the only one considered remarkable, showing a P-value less than 0.05 for the flexural strength modulus. This implies that treatment, fibre content and their interaction produced comparable results in almost all investigated responses. Figure 2 shows the interaction between the factors for the investigated responses.

The introduction of non-modified fibres (*in nature*) caused a decrease in the strength of composites when compared to the results of the material produced from 100% polypropylene (Figure 5a).

The lowest values were found for the materials consisting of 15% wt. fibres (C3), which were 11.52% worse than the condition of the reference materials (C1). With the addition of 0% to 10% wt. of fibres, the reduction in tensile strength was 2.23%. With 10% to 15% wt. of fibres, tensile strength was reduced by 9.51%. And with 15% to 20% wt. of fibres, the strength of composites increased by 4.20%. Treating the bagasse fibres provided excellent results for the tensile strength

Reinforcement (% wt.)	Tensile strength (MPa)		Flexural strength (MPa)	
	BN'	BB''	BN'	BB''
0	26.9 ± 1.3	26.9 ± 1.3	25.4 ± 0.1	25.4 ± 0.1
10	26.3 ± 0.2	28.6 ± 0.3	30.9 ± 0.1	29.8 ± 0.0
15	23.8 ± 0.5	26.9 ± 0.3	31.3 ± 0.4	30.7 ± 0.2
20	24.8 ± 0.2	27.1 ± 1.1	35.1 ± 1.2	31.3 ± 0.2

BN (bagasse *in nature*); BB'' (benzoylated bagasse).

Table 4: Mechanical results for tensile and flexural properties for PP composites reinforced with non-modified and benzoylated fibers.

EC	MOR _t (MPa)	MOR _f (MPa)
C1	26.9 ± 1.3	25.4 ± 0.1
C2	26.3 ± 0.2	30.9 ± 0.1
C3	23.8 ± 0.5	31.3 ± 0.4
C4	24.8 ± 0.2	35.1 ± 1.2
C5	26.9 ± 1.3	25.4 ± 0.1
C6	28.6 ± 0.3	29.8 ± 0.0
C7	26.9 ± 0.3	30.7 ± 0.2
C8	27.1 ± 1.1	31.3 ± 0.2

Where (MOR_t): tensile strength and (MOR_f): flexural strength.

Table 5: Mechanical properties from tensile and flexural tests for neat PP and composites reinforced with BB.

Treatment/Fiber	MOR _t (MPa)		MOR _f (MPa)	
	P-value	R ²	P-value	R ²
Treatment	0.405	77%	0.002	86%
Fiber fraction	0.064	73%	0.200	81%
Treatment × Fiber fraction	0.154	83%	0.256	75%

Table 6: Analysis of variance in terms of fiber content and treatment.

of all composites when compared with materials that did not receive benzoylation treatment (Figure 5). The addition of 10% wt. of bagasse fibres caused the tensile strength of materials to increase by 6.32%. When 10% to 15% wt. was added, the tensile strength was reduced by 5.94%. And it increased by 0.74% for a 15% to 20% wt. percentage of fibres. The progressive increase of the fraction of *in nature* and treated fibres led to successive increases in the flexural strength of the composites (Figure 5b). The highest values were observed for condition C4, which was 38.19% superior in this regard when compared to materials made from 100% polypropylene (C1). For all investigated composites, the treatment of the fibres yielded lower values for flexural strength when compared to the untreated conditions. The largest difference was obtained for the composite with 20% wt. of fibres, with the composites with non-modified fibres having 12.14% superior results in comparison to those made with treated fibres.

Conclusions

During the benzoylation reaction of bagasse, it was observed a constant weight gain as a function of time. The chemical modification was necessary to reduce the hydrophilicity of the fibres and to increase the interfacial adhesion of polar fibres with the non-polar matrix. SEM allowed us to evaluate the morphology and dimensions of the modified and unmodified fibres and indicated that there were considerable morphology changes after the chemical treatments. Spectra from FTIR confirmed that esterification reactions had taken place. The newly added groups resulted in decreases in the OH bands and the appearance of aromatic groups. Thermal analysis revealed that the lignocellulosic material modified has a higher thermal stability than the without modification. The benzoylated fibres insertion caused an increase in the tensile strength in regard to non-modified fibres. However for flexural strength, the chemical modification decreased this mechanical property. Already, the flexural modulus values increase

when compared to neat PP (15 and 20% by weight of fibres). Among the employed experimental factors, treatment of the fibres proved to be the most significant, directly affecting the flexural strength of the composites, indicating that the compositions of the fibres and the interaction between the two factors had little influence on the evaluated mechanical properties. The results for strength obtained from the uniaxial tensile tests demonstrated the effectiveness of treating the fibres with benzoyl groups, with condition C6 providing the best results, followed by condition C7. The progressive use of sugarcane bagasse fibres provided successive increases in flexural strength of the composites for both treatments and the composites with the four non-modified fibres yielded the best results. This indicates that, when flexural strength was concerned, treatment was ineffective. In general, the treatment produced higher values for the material in two of the three investigated fibre composites and proved to be efficient in this regard.

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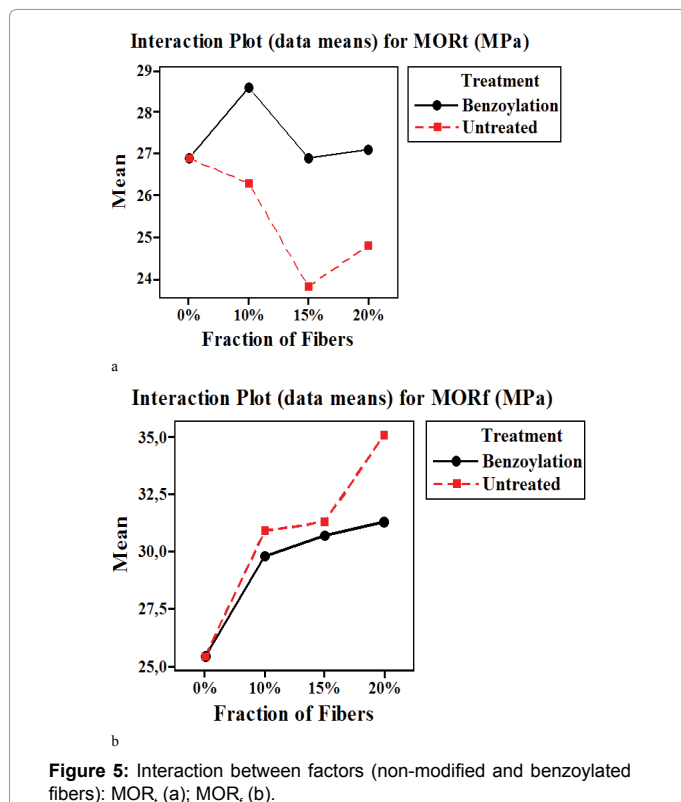


Figure 5: Interaction between factors (non-modified and benzoylated fibers): MOR_t (a); MOR_f (b).

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