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## Original Article

# Study of the reuse potential of the sisal fibers powder as a particulate material in polymer composites



Kátia Moreira de Melo<sup>b</sup>, Thiago Felix dos Santos<sup>a,\*</sup>, Caroliny Minely da Silva Santos<sup>a</sup>, Rubens Tavares da Fonseca<sup>a</sup>, Nestor Dantas de Lucena<sup>c</sup>, José Ivan de Medeiros<sup>b</sup>, Marcos Silva de Aquino<sup>a</sup>

<sup>a</sup> DET - Department of Textile Engineering, Labtex - Textile Engineering Laboratory, UFRN- Federal University of Rio Grande do Norte, Natal, Rio Grande do Norte, Brazil

<sup>b</sup> Textile Engineering Post Graduate Program (PPGET), Federal University of Rio Grande do Norte, Natal, Rio Grande do Norte, Brazil

<sup>c</sup> Federal Institute of Rio Grande of Norte, IFRN, Rio Grande do Norte, Natal, Rio Grande do Norte, Brazil

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## ABSTRACT

The present work studied the reuse of sisal fiber powder, contaminated by industrial oil, resulting from the activities of a Brazilian textile industry. With the objective of reusing the contaminated sisal fiber powder residue, harmful to the environment, this work evaluate the potential of the use this sisal powder as reinforcement in composites. Natural fibre reinforced polymer composites became more attractive structural materials due to their high specific strength, lightweight and biodegradability properties. The mechanical behaviour of polyester resin based natural polymeric composites, made by using experimental planning (granulometry of sisal powder, fiber content and with or without styrene). Thus, combination of analysis of variance (ANOVA), response surface methodology (RSM) and experimental methods allowed evaluated, analyzed and validated the mechanical properties. The results obted showed that the waste from the sisal industry exhibits potential application in reinforcing composite materials Thus, different values of mechanical properties can be obtained by different interactions of the entry variables. Thus, the condition that exhibited high results of mechanical behavior independent of the addition of styrene were the particle size and fiber content 1680  $\mu\text{m}$  and 2.5%, respectively. Therefore, the best results were stress (0.59 MPa), strain (5.68%), tenacity (2.01 E-05 MJ/m<sup>3</sup>) and energy at break (14J) for the composite reinforced with sisal powder.

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\* Corresponding author.

E-mail: [thiagotextilufrn@hotmail.com](mailto:thiagotextilufrn@hotmail.com) (T.F. Santos).  
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## 1. Introduction

The sisal (Agave Sisalana Perrine) is a plant originated from Mexico. Currently Brazil is the largest producer of sisal in the world. In recent years, sisal has been used as a reinforcement alternative in composites. The main products derived from this fiber are biodegradable yarns used in handicrafts, ropes of various utilities, production of upholstery, pulp for cellulose industry, production of tequilas, decorative carpets, medicines, biofertilizers, animal feed, organic fertilizer and bagging. In addition to these strong utilities, sisal has shown important features for other somewhat recent applications in asbestos-based civil construction and in asphalt blankets, in the base of carpeting, and in the automobile industry in panels and seats [1-4].

Because it is a biodegradable fiber, sisal has been attracting the attention of many researchers because of increased interest in environmentally sound materials. The sisal fiber has been used and studied as reinforcement in thermoplastic, thermoplastic and elastomers [2,5,6]. For this reason, recently, new technologies of applicability have appeared in several sectors for the use of these solid residues, among them the polymeric composites [6-8].

When we talk about polymer composites, we soon think of synthetic materials to be used as reinforcement. The most commonly used are carbon and glass fibers. However the use of natural fibers as a substitute is receiving great attention due to its cost and comparable properties in high volume applications. Natural fibers are generally lignocellulosic, consisting of fibrous wrap cellulose microfibrils in an amorphous matrix of lignin and hemicellulose. A high content of cellulose and low angle microfibrils are required properties in a fiber to be applied as a reinforcement in polymeric composites [9]. The interest of lignocellulosic materials in the application as reinforcement in polymeric composites has been growing rapidly. As advantages to its use it is worth mentioning the low cost, low density, good flexibility during processing, the ease of being modified by chemical agents, availability, non-abrasiveness, biodegradability, etc. [10].

Some researchers verified that there is a great amount of residues generated during the process of cultivation, extraction, processing and industrialization of sisal fibers [11]. Northeast Brazil has been showing a strong decline in sisal activities, both in the reduction of planted area and in production. One of the main factors for this decline is the low fiber utilization, because of all the sisal defibration process, only 4% of the leaf is destined for fiber, 16% is solid waste and 80% is liquid waste. What does not compensate the high cost of production with the low value paid by the fiber [12]. Therefore, the possibility of an economical and sustainable application for solid waste has been studied. Normally some producers leave this waste heaped in sisal fields, or they are destined to the sustenance of animals, which can cause problems of bloating. However, presently, new applicability technologies for these wastes have appeared in several sectors, such as in the manufacture of plastic composites [13-16].

Therefore, the present work investigates the mechanical behavior of polyester matrix composites reinforced with sisal powder. In addition, to understand the effects of the interac-

**Table 1 – Variables and experimental conditions.**

Name	Units	Type	Low	High
A - Fiber content	%	Numeric	2,5	5
B - Styrene		Categoric	NS	WS
C - Particle size	$\mu\text{m}$	Numeric	841	1680

tions produced by the composite manufacturing variables and conditions the mechanical properties of the polymer matrix composites (polyester) were analyzed via statistical factorial design.

## 2. Experimental methods

The experimental design was entirely performed using surface response method (RSM), based on a three-particle Sisal powder residue substrate as shown in Fig. 1 (841  $\mu\text{m}$ , 1190  $\mu\text{m}$  and 1680  $\mu\text{m}$ ), these were obtained by the process of particle separation through the granulometric strainer.

During the production of polymer composites were used as study variables, powder fiber content, particle size and a condition of use or not of styrene as shown in Table 1. The composites materials that received addition of styrene, the nomenclature used is the abbreviation (WS) and for the composites that did not receive addition of styrene the abbreviation used is (NS) as shown in Table 1.

The design power is very important to analyze therefore, this has the purpose of specifying the power of the response. In this way, establishing a relationship between useful information (response) and unwanted data (noises in the data). Therefore, higher power of response percentages (approximately 80% or greater) signify that the effects to be detected will have a better specification. In Table 2 is exposed the power of the design of this work.

Factorial Planning ( $2^3$ ) of variables with 5 central points and 2 replicates The statistical software Design Expert 11 was used to manipulate the data using the design of experiment (DoE), analysis of variance (ANOVA) and response surface methodology (RSM). Samples with dimensions of 250 mm in length, 25 mm in width and 3 mm in thickness were made in gypsum molds with a layer of carnauba wax to facilitate the demoulding of the samples. Tension tests were performed using the Tensolab 3000 dynamometer from MESDAN, according to the standard ASTM D3039 [17]. The samples were tested at a rate of 10 mm/min and distance between claws of 125 mm. Then, the results were treated and inserted into the Software Design Expert 11, following each variable presented in Table 1.

## 3. Results and discussions

The stress of the samples of polymeric composites reinforced with sisal fiber powder with and without styrene were shown on the Fig. 2. In the curves presented in Fig. 2(a) and (b), it was observed that the addition of styrene (WS) promotes modification of mechanical behavior, evidencing that styrene exerts influence on stress properties of polymeric composites compared to those without styrene (NS). Can also be observed, that in the polymer composites with styrene (WS) the relation

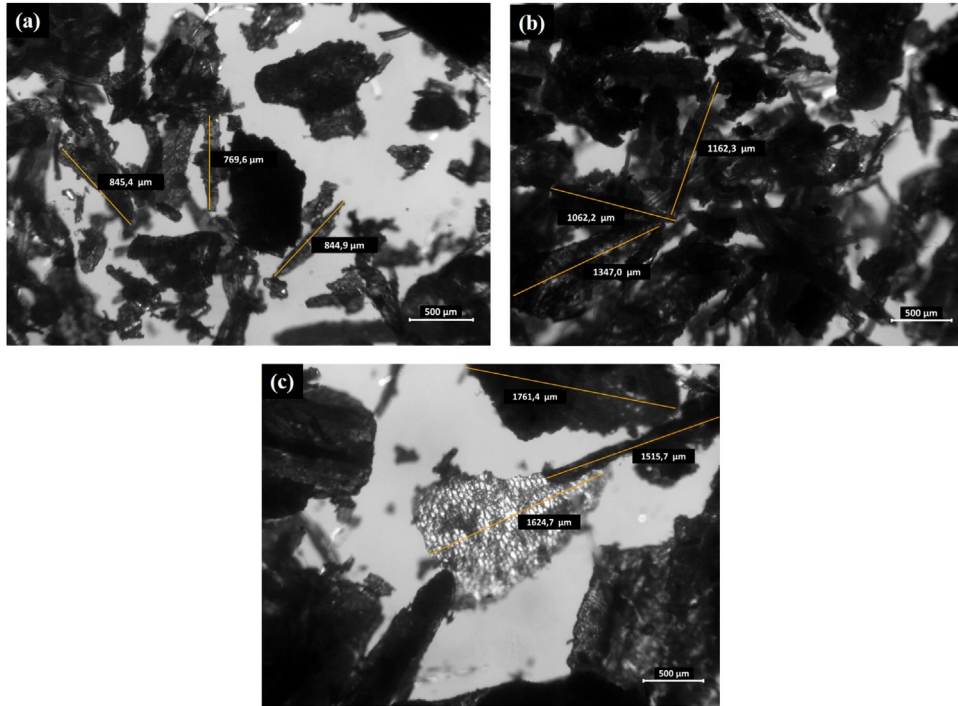


Fig. 1 – Particles size of crushed waste (a) 841 μm, (b) 1260 μm and (c) 1680 μm.

Table 2 – Design of the power of response.

Name	Units	Delta (Signal)	Sigma (Noise)	Signal/Noise	Power for A	Power for B	Power for C
Stress	Mpa	2	1	2	96,9	99,8	96,9
Strain	%	2	1	2	%	%	%
Tenacity	MJ/m <sup>3</sup>	2	1	2			
Energy break	J	2	1	2			

between particle size and fiber content is inversely proportional. Thus, the variables with significantly higher stress properties were, respectively, particle size and fiber content

1680 μm and 2.5% (0,59 MPa) and 841 μm and 5% (0,53 MPa). In the samples of without styrene (NS) composites it was possible to observe that only for the condition 2,5% of fiber content

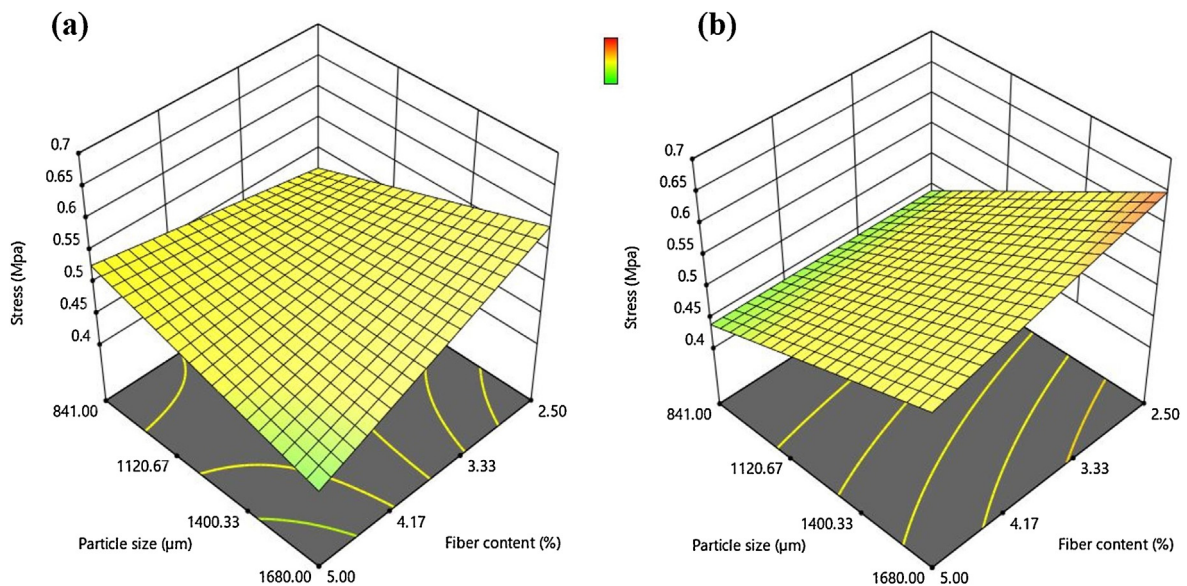
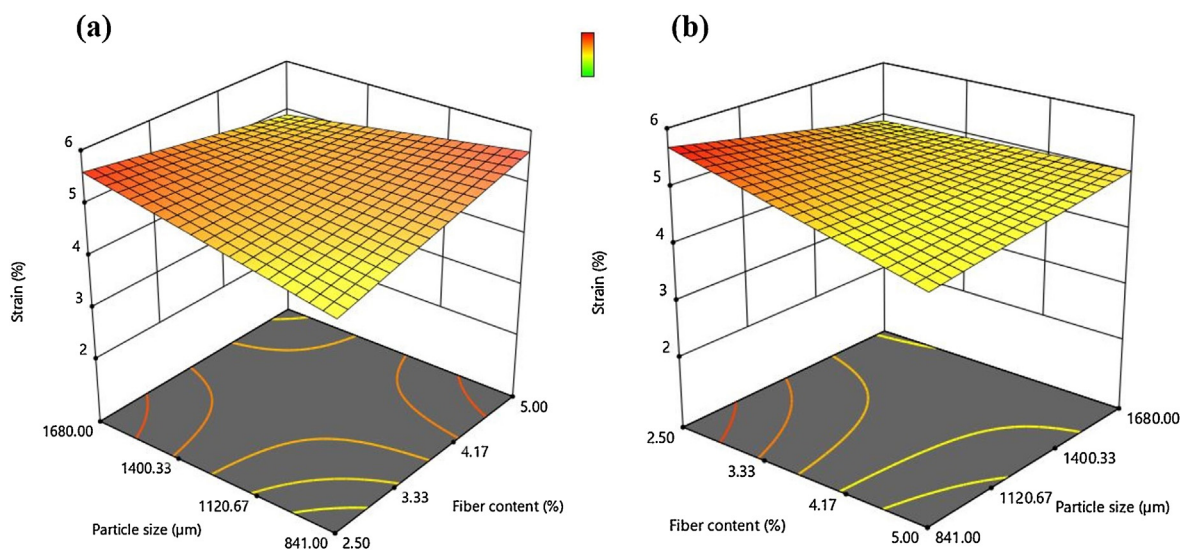


Fig. 2 – Result of Stress (a) with styrene WS and (b) NS of the samples without styrene.



**Fig. 3 – Result of Strain (a) with styrene WS and (b) NS of the samples without styrene.**

and 1860  $\mu\text{m}$  of the particle size (0,65 MPa) high stress values, however this result is the highest stress value obtained [18,19]. Finally, among the studied variables, it can be concluded that in the stress property the particle size has a greater influence than the styrene condition (with or without), but, the fiber content has greater influence when compared to all the variables of study. Because the stress increased significantly with the decrease in particle size, as well as, the particle size decreases it is necessary to increase the fiber content [20,21].

In the Fig. 3 show strain of the samples of polymeric composites with and without styrene. In the curves presented in Fig. 3, it was observed that the addition of styrene (WS) promoted change and improvement of mechanical behavior of the strain, justifying that styrene influences on strain properties of polymeric composites compared to those without styrene (NS). It was possible to observe, that in the polymer composites with styrene (WS) the relation between particle size and fiber content is inversely proportional and still in WS samples was obtained two similar strain peaks (5,68%). Thus, the variables and conditions with significantly higher strain properties were, respectively, particle size and fiber content 1680  $\mu\text{m}$  and 2.5% (5,68 %) and 841  $\mu\text{m}$  and 5% (5,68 %) [19,22,23]. In the samples of without styrene (NS) composites it was possible to observe that only for the condition 2,5% of fiber content and 841  $\mu\text{m}$  of the particle size (5,68 %) high strain values. Finally, among the studied variables, the strain property the fiber content has a greater influence than the styrene condition (with or without), but, the particle size has greater influence when compared to all the variables of study. Because the stress increased significantly with the decrease in particle size, as well as, the particle size decreases it is necessary to increase the fiber content and the result 5,68 % is the highest strain value obtained independent of the addition of styrene [24,25].

The Fig. 4 shows the tenacity of samples of polymeric composites reinforced with sisal fiber powder with and without styrene. Were observed that the addition of styrene (WS) promoted improvement of tenacity mechanical property, evi-

dencing that styrene influences on tenacity properties of polymeric composites compared to those without styrene (NS). It is observed that the fiber content factor exerts less influence, because the increase in fiber volume in the composite reduces tenacity, while the particle size factor causes a significant increase of this mechanical property behavior [19,26]. Thus, the variables and conditions with styrene were significantly higher tenacity, respectively, particle size and fiber content 1680  $\mu\text{m}$  and 2.5% ( $2,01 \text{ E}^{-05} \text{ MJ/m}^3$ ) and for the composites without styrene 841–1680  $\mu\text{m}$  and 2,5% ( $1,73 \text{ E}^{-05} \text{ MJ/m}^3$ ) and the result  $2,01 \text{ E}^{-05} \text{ MJ/m}^3$  is the highest tenacity value obtained [27,28].

The mechanical behavior of the energy at rupture was diversified between samples of composites with and without styrene as shown in Fig. 5. In the samples with styrene (WS), the increase of the powder content causes increase of energy at break, but, significantly lower, when compared to the particle size [29,30]. And the composites produced with larger particle size (1680  $\mu\text{m}$ ) and lower fiber powder content (2.5%) promote a significant increase in energy at break (14J) [31,32]. The mechanical behavior of the composites without styrene (NS) is very similar and presents a result of 12J for the same conditions of production (1680  $\mu\text{m}$  and 2.5%), however, in the WS composites showed an inflection (minimum point) and a model of exponential mechanical behavior while NS has a linear model as shown in the Fig. 5.

It is a methodology based on statistics and another discipline to arrive at an efficient and effective planning of experiments with a view to obtain a valid conclusion from the analysis of experimental data. The design of experiments determines the pattern of observations to be made with a minimum of experimental efforts. More specifically, the use of orthogonal matrices (OA) for DOE provides an efficient and effective method to determine the most significant factors and interactions in a given design problem [33,34]. The value- $\rho$  is greater than or equal to 0,05 ( $\rho \geq 0,05$ ). The value of “F” corresponds to how much the means of the groups of analyzed samples are different. How much extreme the  $\bar{F}_i$ ; the more sig-

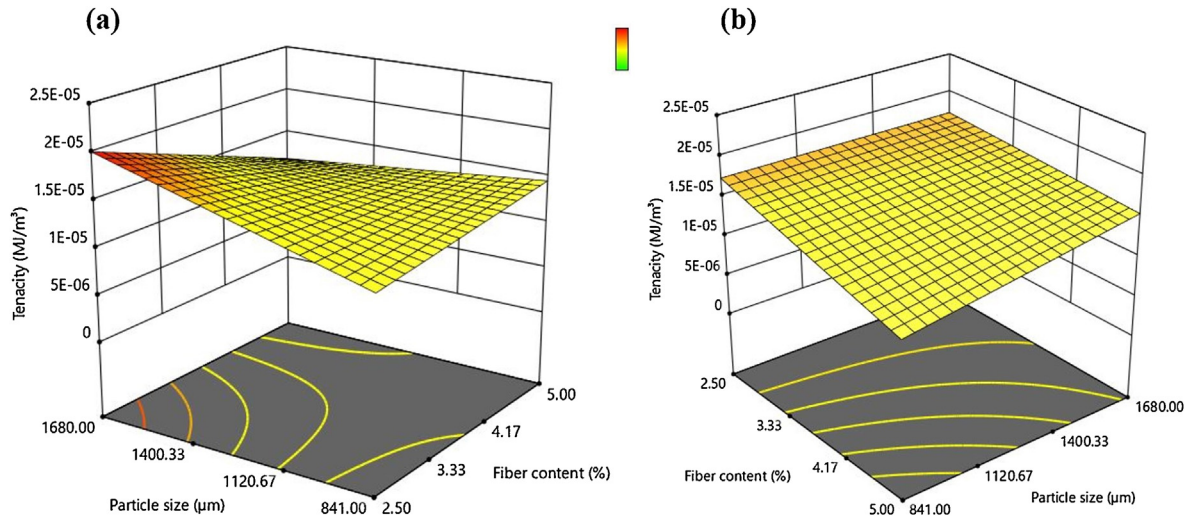


Fig. 4 – Result of Tenacity (a) with styrene WS and (b) NS of the samples without styrene of the samples without styrene.

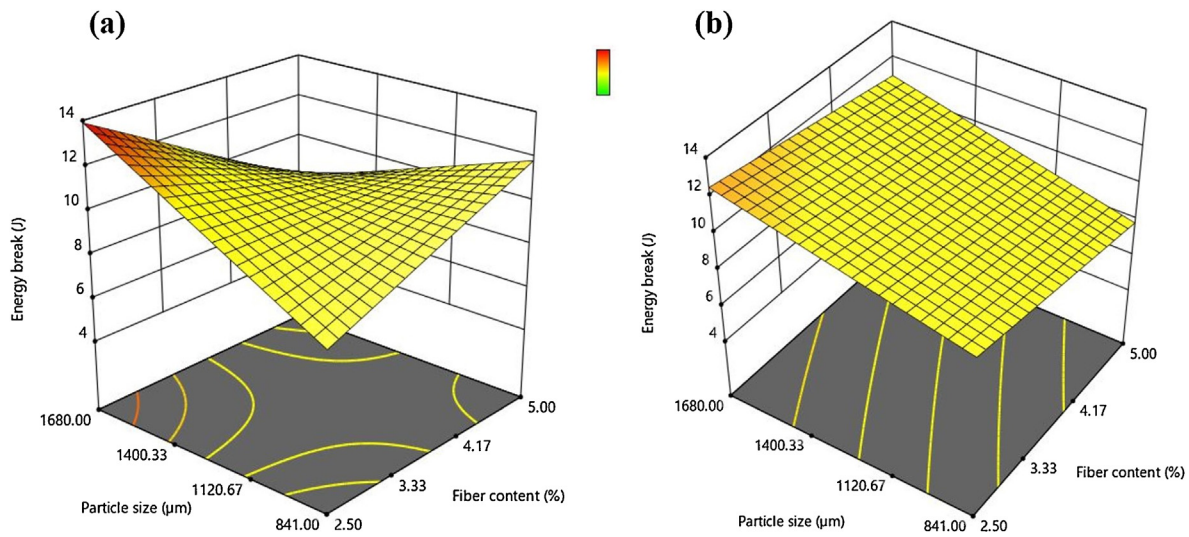


Fig. 5 – Result of energy at break (a) with styrene WS and (b) NS of the samples without styrene of the samples without styrene.

Table 3 – ANOVA of results of stress, strain, MOE and tenacity of the sample studied.

	Stress		Strain		Tenacity (MJ/m <sup>3</sup> )		Energy break (J)	
	Value of F	Value - p (Prob > F)	Value of F	Value - p (Prob > F)	Value of F	Value - p (Prob > F)	Value of F	Value - p (Prob > F)
Model	247,30	<0.0001	307.26	<0.0001	163,10	<0.0001	12584,08	<0.0001
A-Fiber content	112,81	<0.0001	68.45	<0.0001	233,59	<0.0001	5281,98	<0.0001
B-Styrene	15,31	0.0012	48.05	<0.0001	8,84	0.0090	39,47	<0.0001
C-Particle size	382,81	<0.0001	0.45	0.5119	117,35	<0.0001	13575,1	<0.0001
AB	0,31	0.5839	186.05	<0.0001	8,19	0.0113	382,7	<0.0001
AC	300,31	<0.0001	14.45	0.0016	51,04	<0.0001	18671,7	<0.0001
BC	300,31	<0.0001	36.45	<0.0001	2,11	0.1657	2760.9	<0.0001
ABC	52,81	<0.0001	1022.45	<0.0001	285,76	<0.0001	21671	<0.0001
Standard deviation	0,01		0,04		4,47 E-07		0,0350	
Mean	0,52		4,36		1,28 E-05		9,04	
C.V. %	1,71		1,03		3,50		0,39	
R <sup>2</sup>	0,99		0,99		0,99		0,99	
R <sup>2</sup> adjusted	0,99		0,99		0,98		0,99	

nificant the value- $\rho$  for the factors and interactions. In this study stress, strain, tenacity and energy break the values of  $\rho$  were significant for practically all factors and interactions as shown in Table 3. The factors studied as well as their interactions can significantly improve the stress, strain, tenacity properties, as well as the energy break of sisal fiber powder reinforced composites [35,36]. The  $R^2$  is a statistical tool that varies from 0 to 1 and indicates, in percentage, how much the model can explain the input values of the stress, strain, tenacity and energy break. It can be observed the value of  $R^2$  is 0.99 for the variables as studied variables that show the high quality between the developed model and the experimental results. In addition, it allows to state that the experiments results validate the developed model [37-39].

#### 4. Conclusions

The present study investigated the mechanical behavior of polyester matrix composites reinforced with sisal powder. For this, a series of static experimental tests were carried out in order to evaluate the successful manufacture of composites, through the tools ANOVA and RSM used to analyze and validate the models developed. Thus, it was concluded from these data that:

- ✓ The results obtained for the stress, strain, tenacity and energy at break response variables for the composites are able to validate that the residue of the sisal fiber powder presented significant potentials for use as reinforcement.
- ✓ The variables content of fiber (%) and particle size ( $\mu\text{m}$ ) independent of the use of styrene are predominant factors that significantly influence the mechanical behavior of the composite (stress, strain, tenacity and energy at break). Otherwise, the use of styrene was shown to improve mechanical behavior except stress and was the variable that showed the least influence among the three entry parameters.
- ✓ The best results obtained from the studied properties (stress, strain, tenacity and energy at break) independent of the styrene variable were obtained in the conditions of particle size and fiber content 1680  $\mu\text{m}$  and 2.5% respectively.
- ✓ The fiber content (%) is the variable that has the greatest influence on the mechanical behavior of the composites.
- ✓ The lower the fiber content, the higher the stress, strain, tenacity and energy at break of composites reinforced with sisal powder.
- ✓ Waste from the sisal industry has great potential for application in composite materials for application on pallets, development of tiles, partitions, revetment and etc.
- ✓ Response surface methodology (RSM) describes and provides better understanding of the data and analysis of variance (ANOVA) strongly shows the behavior of the mechanical properties with an appropriate precision of 99%.

#### Conflicts of interest

The authors declare no conflicts of interest.

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