



## **COBEM-2021-1476 - Lignocellulosic residues as polymer composites reinforcements: a review on its manufacturing and characterization processes**

### **Carla Tainara de Oliveira Lima**

Federal Institute of Education, Science and Technology of Bahia - Emídio dos Santos St., s/n - Barbalho, Salvador/BA - Brazil,  
ZipCode: 40301-015  
e-mail: carlatainara.lima@gmail.com

### **Vitor da Silva Lacerda**

Federal Institute of Education, Science and Technology of Bahia - Emídio dos Santos St., s/n - Barbalho, Salvador/BA - Brazil,  
ZipCode: 40301-015  
e-mail: vitorslacerda85@gmail.com

### **Alberto Matheus Freitas Oliveira**

Polytechnic School, Federal University of Bahia - Prof. Aristides Novis St., 2 - Federação, Salvador/BA - Brazil, Zip Code:  
40210-630  
e-mail: matheusoliveira1341@gmail.com

### **Michel Giffoni Santos**

Federal Institute of Education, Science and Technology of Bahia - Emídio dos Santos St., s/n - Barbalho, Salvador/BA - Brazil,  
ZipCode: 40301-015  
e-mail: michel.giffoni13@gmail.com

### **Itana Cortes Vieira**

Federal Institute of Education, Science and Technology of Bahia - Emídio dos Santos St., s/n - Barbalho, Salvador/BA - Brazil,  
ZipCode: 40301-015  
e-mail: it-ana@hotmail.com

### **Victor Antunes Silva Barbosa**

Polytechnic School, Federal University of Bahia - Prof. Aristides Novis St., 2 - Federação, Salvador/BA - Brazil, Zip Code:  
40210-630  
e-mail: victor\_antunes07@hotmail.com

**Abstract.** *Polymer composites have been attracting the researchers' attention in the last few years because they can produce a material with high mechanical performance and low weight. Natural reinforcements present advantages to the composites due to their eco-friendly behavior. This work aims to produce a literature review about the use of natural fibers and residues as reinforcement or filler for the development of polymer composites. This paper was carried out through a systematic literature review in the main scientific databases, namely Scopus, Web of Science and Science Direct. The review showed the trend of an increasing number of published scientific papers referring to lignocellulosic residues in polymer composites. The results exhibited that most of the lignocellulosic residues used as reinforcement or filler were either fiber or particulate materials. This work also analyzed the most used polymeric matrices, plant-based fibers, manufacturing processes, and their main properties, through several tests review. This research helped to determine some fundamental parameters that should be used in the materials component's selection, the composite fabrication and its analysis and characterization. These results should be used as a foundation for further experimental researches that will develop new composite materials manufactured with the addition of lignocellulosic residues.*

**Keywords:** *Lignocellulosic residues, natural fibers, polymer composites, Mechanical Performance, water absorption.*

## **1. INTRODUCTION**

The use of composite materials demands the union of four factors: performance, competitiveness, sustainability, and application. The use of natural fibers in these materials has various advantages over synthetic fibers and, for these

reasons, natural fiber reinforced composites have been attracting the attention of researchers and becoming an alternative source with less environmental impact in comparison to synthetic fibers and at a lower cost (VENKATESHWARAN et al., 2013). Therefore, the use of natural fibers in polymer composites has great potential, since it is ecologically correct, recyclable, renewable and has low cost, in addition to good mechanical properties (VAISANEN, 2017).

In general, polymer composites have high mechanical strength / density ratio, having the ability to provide products that withstand high loads with low weight. For these reasons, these composites can meet the demands of industries such as marine, aerospace, packaging, automotive and construction. New polymer composites reinforced with plant based natural fibers have been developed, mainly in the last two decades, due to the large amount and variety of these fibers found in nature (WEARN et al., 2020 and MONTEIRO et al., 2011).

In this sense, this paper aims to present a systematic review of the literature, covering works from the last 10 years that studied the application of natural fibers as reinforcement in polymeric materials.

## 2. METHODOLOGY

The methodology applied in this research was a systematic review of the literature, determining procedures for selecting the papers to be analyzed. Search parameters were determined, such as the databases to be evaluated, the types of papers, period of time, and various filters for selecting a bibliographic portfolio, which addresses the most relevant papers in the area. Figure 1 shows a flowchart that covers all the steps for selecting papers.

A bibliographical research was carried out on the themes of natural fiber reinforced polymer composites, residues applied in this type of materials, mechanical properties, and water absorption. Searches were performed in scientific journals at the Scopus, Science Direct and Web of Science databases, investigating the works done regarding the subject in the last 10 years (from 2011 to 2021) to make it possible to find the most recent papers and in good quantity. Keywords such as lignocellulosic residues, natural fibers, polymer composites, mechanical performance and water absorption were used for data collection.

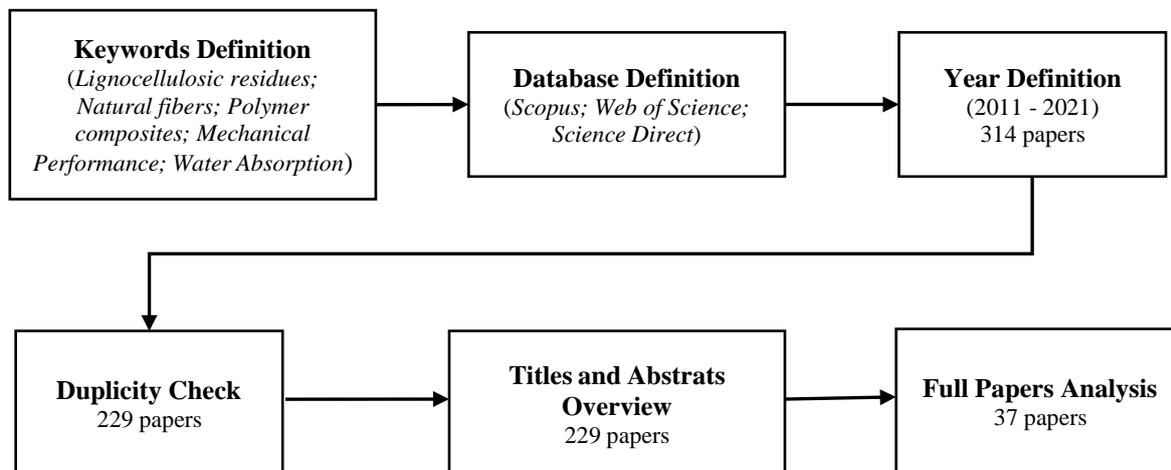


Figure 1. Methodological procedure applied to the object of study

A total of 314 papers were obtained from these filters. Then, the duplicity check and conformity of the papers from the three databases were verified, which resulted in 229 papers. Afterwards, titles and abstracts were read. Finally, the selection and complete reading of the works (2011 to 2021) were executed, resulting in 37 papers.

The outcome from this systematic review were sought through the evaluation of the results by database, year of publication, journals, and country of origin of the papers. Then, the main indicators were obtained regarding how the researches have been developing in the world about the use of lignocellulosic residues as reinforcement in polymer composites, and also the way they are replacing traditional materials. Subsequently, the final selection of papers was evaluated, seeking the main types of residues and matrices used, manufacturing methods and properties either chemical, physical or mechanical.

## 3. RESULTS

### 3.1 Analysis of the bibliographic portfolio

A systematic search was applied to select the most relevant papers to the topic in the main databases. Parameters filters for papers labeled research papers were defined, which led to the number of papers in the Scopus, Science

Direct and Web of Science databases, with a total of 111, 153 and 50 papers, respectively. However, there was a large number of repeated papers among the databases and with similar themes, but that did not match the purpose of this review. After removing these papers, a portfolio of 229 works was formed.

Next, the number of publications by year related to the proposed topic was evaluated. Figure 2 shows the number of publications from 2011 to 2021. It was observed that most publications occurred in the last 4 years (2018 - 2021). However, in 2019 and 2020, there was an increase in the number of publications referring to lignocellulosic residues in polymeric matrices, having a total of 13 and 15 papers, respectively. This indicates the importance the theme has been gaining recently, since it has been sought new ways to reduce and/or replace the use of plastics in products and their replacement by natural materials classified as renewable, clean, and low-cost sources. Thus, natural fiber reinforced polymer composites have been studied and increasingly occupying a significant position in the market.

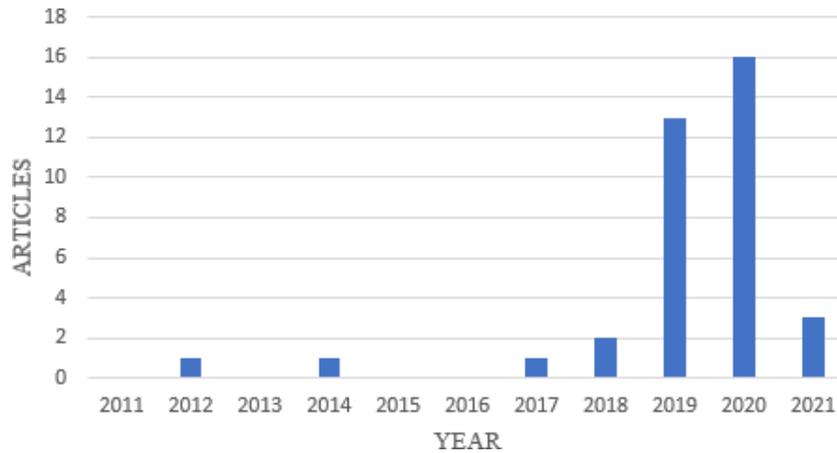


Figure 2. Number of papers published by year

It was verified that when it comes to publication, 6 journals excelled about this theme as it is shown in figure 3. They were Composites Part B: Engineering, Construction and Building Materials, Journal of Materials Research and Technology, Polymers, Journal of Polymers and the Environment, and The International Journal of Advanced Manufacturing Technology. The journal that published the most was Composites Part B: Engineering, with 8 papers.

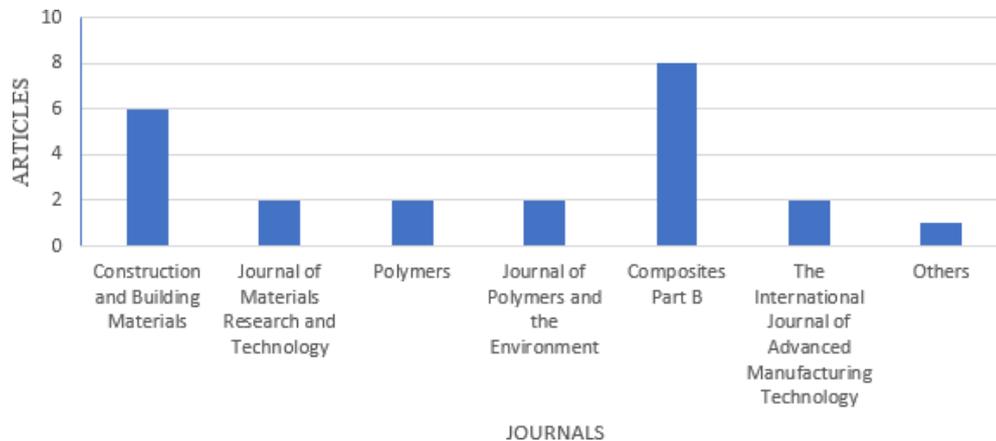


Figure 3. Number of papers published by Journal

The number of publications by country was analyzed, as shown in figure 4. The countries with the highest number of publications on the subject were India, Brazil, and China with 10, 7 and 3 publications, respectively. These 3 countries have an abundance and diversity of natural resources. Hence, these countries have a high production of lignocellulosic residues, a factor that drives studies related to its incorporation into polymeric matrices.

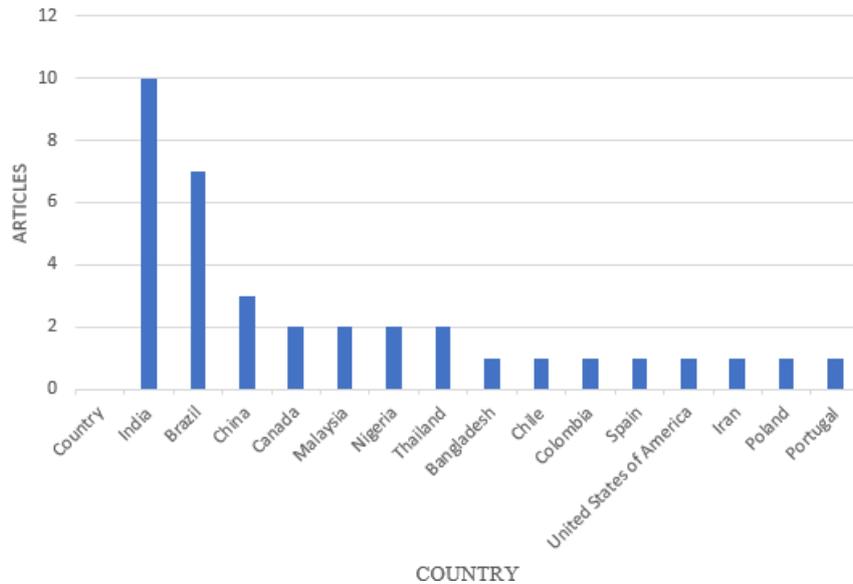


Figure 4. Number of papers published by Country

### 3.2 Analysis and discussion of residues, matrices, and manufacturing processes

It is shown in table 1 the type of fiber, the granulometry and percentage of replacement of the matrix regarding the applied residue. It is also shown the polymer applied in the matrix and the manufacturing processes used in the 37 papers selected from 2011 to 2021.

Table 1. Residues, matrices, and manufacturing processes applied in the papers

References	Residues			Matrix	Manufacturing Processes
	Type	Granulometry	%		
Liu et al. (2019)	Hemp fibers Bamboo fibers	-	50%	Epoxy	Compression molding
Santos et al. (2019)	Coir fiber	200 $\mu$ m	30%	Epoxy Polyester	Compression molding
Langhorst et al. (2019)	Agave fiber	0,297-0,841 mm	20%	Polypropylene	Injection molding
Moudood et al. (2019)	Flax fiber	2,5 mm	50%	Epoxy	Vacuum Infusion
Wechsler et al. (2019)	Peach pits Pine wood	2-0,42 mm	50 e 60%	Polypropylene	Extrusion
Hamdan et al. (2019)	Jute, Ramie and fiber Roselle	-	-	Polyester	Hand lay-up
Andrzejewski et al. (2019)	Wood flour Cork dust	70 a 150 $\mu$ m 0,2-0,5 mm	10, 20 e 30%	Polypropylene and Poly(lactic acid)	Injection molding
Ambrósio et al. (2019)	Wood flour	1 mm	25, 40 e 55%	Poly(vinyl butyral)	Extrusion
Kumar et al. (2020)	Himalayan Nettle fibers Bauhinia-vahlia fibers	-	0, 2, 4 e 6%	Epoxy	Hand lay-up
Ferreira et al. (2020)	Curauá, jute and sisal fibers	0,8 $\mu$ m, 6,7 $\mu$ m 8,2 $\mu$ m	-	Styrene butadiene latex	Hand lay-up

Table 1. Residues, matrices, and manufacturing processes applied in the papers

(to be continued)

References	Residues			Matrix	Manufacturing Processes
	Type	Granulometry	%		
Younesi-Kordkheili e Pizzi (2020)	Fiber from sugarcane bagasse	0,001 mm	60%	Recycled Polypropylene	Injection molding
Hao et al. (2020)	Jute fiber	1 mm	-	Polypropylene	Compression molding
Saikia et al. (2020)	Coir Fiber	-	65, 70, 75 e 80%	Polyethylene	Compression molding
Yadav et al. (2020)	Sugarcane dry leaves	-	2, 4, 6, e 8%	Epoxy	Hand lay-up
Rozyanty et al. (2021)	Kenaf fiber	-	10, 15, 20, 25, 30 e 35%	Unsaturated polyester	Hand lay-up
Ramlee et al. (2021)	Palm fiber Sugarcane bagasse	250-610 µm 120-700 µm	0, 15, 25, 35, 50%	Novolac type biophenolic resin	Hand lay-up
Haque et al. (2021)	Palm fiber	-	0, 5, 10 e 15%	Epoxy	Hand lay-up
Hidalgo-Salazar et al. (2019)	Colombian rice husk	0,420 mm	10, 20 e 30%	Polypropylene	Extrusion Injection
Zanini et al. (2019)	Macadamia Nutshell Fibers	35 mesh	5 e 10%	Polypropylene	Injection
Ahmed et al. (2017)	Coir fiber Jute fiber	3-5 mm	5, 10 e 15%	Polyethylene	Hot pressing
Neves et al. (2019)	Hemp fiber	-	10, 20, 30%	Epoxy Polyester	Hand lay-up
Srinivasan et al. (2020)	Banana fiber	0,8 mm	3 e 6% (CaCO <sub>3</sub> )	Epoxy	Compression molding
Shravanabelago la et al. (2020)	Limonia most acid shell powder	1 a 300 µm	0, 5%, 10%, 15% e 20%	Vinyl ester	-
Singh et al. (2014)	Walnut shell	40 e 80 mesh	15, 20, 25 e 30%	Epoxy	-
Sujin et al. (2019)	Coir fiber Wood Dust	600 µm	15, 20, 25, 30, 35 e 40%	Polyester	Hand lay-up
Oladele et al. (2019)	Palm kernel shell fiber and Cassava peel	< 75 µm	2, 4, 6, 8 e 10%	Epoxy	Compression molding
Suresh et al. (2020)	Bagasse, Rice husk, and Coconut shell	50 µm	5, 10, 15, 20 e 25%	Vinyl ester	Compression molding
Okonkwo et al. (2020)	Clay-Bambara nut shell	45 µm	8, 12, 16, 20 e 24%	Polyester	Compression molding
Hejna et al. (2020)	Wheat Bran	200 e 500 µm	2.5; 5; 10 e 20%	Low density polyethylene	Extrusion
Santos et al. (2018)	Mango's Seed Shell	<1mm	5, 13 e 20%	Green polyethylene	Extrusion Injection

Table 1. Residues, matrices, and manufacturing processes applied in the papers

(to be continued)

References	Residues			Matrix	Manufacturing Processes
	Type	Granulometry	%		
Montanes et al. (2018)	Thyme Herbs	25 µm	10 - 50%	High density polyethylene	Injection
Magalhães et al. (2012)	Heart-of- Peach Palm Sheath	0,1 mm	20 - 40%	Polypropylene	Extrusion
Junio et al. (2020)	Copernicia Prunifera Leaf Fiber	-	0,10%, 20%, 30% e 40%	Epoxy	Hand lay-up
Anidha et al. (2020)	Sisal fiber	3,5 mm	40%, 50% e 60%	Epoxy	Hand lay-up
Prakash et al. (2020)	Syngo- nanthus nitens fiber	10 mm	10, 20, 30 e 40%	Epoxy	Hand lay-up

Plant-based natural fibers have lignin, cellulose, and hemicellulose as constituents. Among all the papers analyzed, it was possible to highlight five kinds of fibers, namely: flax, hemp, coconut, sisal, and jute. These fibers were found in 16 papers.

Hemp fiber is obtained from the *Cannabis ruderalis* stem, a plant originally from Asia. This fiber length ranges between 1 and 3 meters and it is known for its high rigidity, tensile strength, and durability. The hems filament is five times stronger than cotton (Romão, 2003). Its use as reinforcement in composite materials was studied by Liu et al. (2019) and Neves et al. (2019). Liu et al. (2019) studied physical (density and water absorption) and mechanical (flexion) properties of epoxy/hemp and epoxy/bamboo composites. The authors reported that hemp fibers provided greater density, water absorption, and flexural strength in comparison to bamboo fibers when used as reinforcement due to its higher cellulose percentage, length-to-width ratio, and superior interfacial adhesion with resins. Neves et al. (2019) studied mechanical (tensile and bending tests), physical (density), bonding (FTIR), statistical (ANOVA) and microstructural (SEM) properties. FTIR showed evidence of good molecular interaction between hemp and epoxy, which contributes to its role as mechanical reinforcement. The mechanical tests showed the epoxy composite with 30 wt% of hemp fiber presented greater strength and modulus of elasticity compared to the other composites investigated, with statistically significant differences, according to ANOVA.

The coir comes from the coconut shell, scientifically known as *Cocos nucifera*, and its length ranges between 0.1 and 0.3 m. Coir stands out for its high lignin percentage which provides good stiffness, mechanical strength, and moisture resistance (Romão, 2003; Santos et al., 2019). This fiber has great use in the development of sustainable technologies, and in this study, it was found in 5 papers researched: Santos et al. (2019), Saikia et al. (2020), Ahmed et al. (2017), Sujin et al. (2019) and Suresh et al. (2020). Suresh et al. (2020) presented the most promising results when studying sugarcane bagasse, rice husk and coir as fillers in a biodegradable composite using vinyl ester as a matrix. The tensile and flexural strength increased 44% compared to other hybrid composites, due to coir, bagasse, and rice husk addition. Microstructural analysis (SEM) of the composite with reinforcement percentage of 20 wt% showed a compact surface without pores. This same percentage also provided greater wear resistance to the composites and consequently a longer material lifetime.

The jute fiber belongs to the Tiliaceae order, and it can be obtained from two species: *Corchorus capsularis* and *Corchorus olitorius*. The fiber is placed between the bark and the inner stem, reaching between 1.5 and 3.0 m in length. The main constituent of jute is the cellulose, which confers properties such as high flexural and torsional stiffness and good moisture resistance (Rosa, 2009; Romão, 2003). Due to its properties, jute is one of the most studied fibers as polymer composites reinforcement and it was studied in 4 papers: Hamdan et al. (2019), Ferreira et al. (2020), Hao et al. (2020) and Ahmed et al. (2017). Ahmed et al. (2017) used jute as reinforcement in a polyethylene matrix. While the increasing in fiber percentage reduced the composites tensile strength, it improved the flexural strength, flexural modulus, Charpy impact strength, hardness, and water absorption. The SEM analysis showed better adhesion between fiber and matrix for the composite with 15 wt% of fiber. Hence, the 15 wt% jute composite yielded the best set of mechanical properties compared to other composites.

The sisal fiber is obtained from the *Agave sisalana* plant leaves and its length ranges from 0.6 to 1.2 m. It has high tensile strength, durability, good thermal stability, and elasticity. Due to its properties, sisal is widely studied as reinforcement in composite materials (Romão, 2003). The present research found this fiber in 3 papers: Ferreira et al. (2020), Yorseng et al. (2020) and Anidha et al. (2020). Anidha et al. (2020) investigated the effect of adding 5%

of polyaramid fibers to sisal-reinforced epoxy composites. The objective was to give better adhesion to the composites, due to the polyaramid fiber hydrophobic nature. SEM analysis indicated better quality of interfacial adhesion when sisal was treated compared to the untreated fiber. A heterogeneous and dynamic structure was also verified, indicating the successful composite functionalization. Mechanical tests showed that epoxy/sisal composite properties increased with the use of treated fibers and the correct manufacturing method, thus demonstrating the quality of the adhesion between the sisal fiber and the epoxy matrix. The application of 5 wt% of polyaramid with 40 wt% of sisal improved the mechanical properties, such as resistance to traction, bending and impact.

### 3.3 Analysis of the studied properties

The papers studied have different objectives, requiring specific tests to characterize them and that allow finding answers related to the purposes of this research review. The figure 5 shows the main mechanical, residue's characterization, thermal, physical, microstructural, durability and dynamic tests, in addition to statistical analysis.

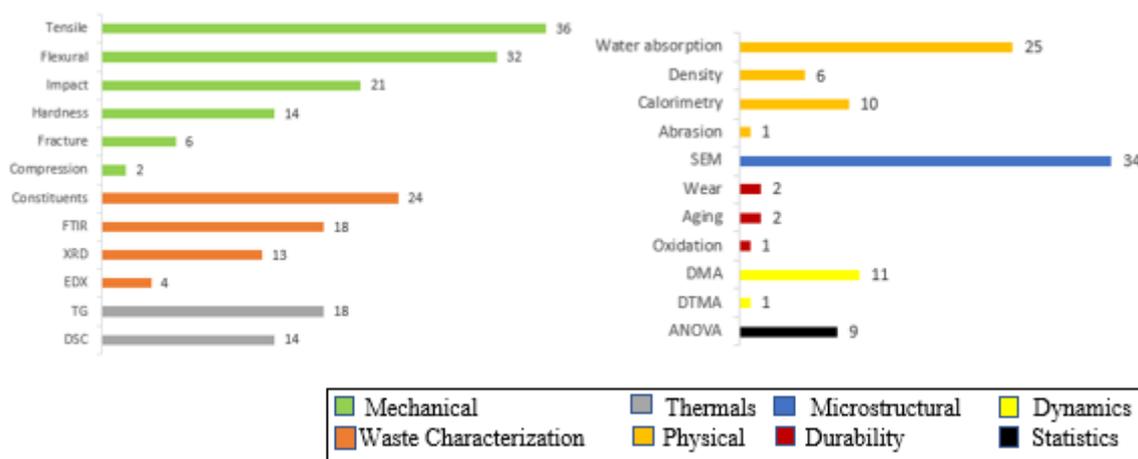


Figure 5. Main properties analyzed in the papers

In relation to mechanical properties, it was observed that the tensile test, identified in 36 papers, was the most applied test, followed by the bending test (32 papers), impact (21 papers), compression, hardness, and fracture. Through the mechanical properties, it is possible to obtain the tensile strength, the modulus of elasticity, specific deformation and tenacity of the composites.

The impact on the properties studied in natural fiber reinforced composites can be justified through the characterization of the constituents. According to the literature, natural fibers are basically composed of lignin, cellulose, and hemicellulose. Cellulose is the main constituent, being responsible for strength due to its high degree of polymerization and molecular orientation. The role of fibers as reinforcement in composite materials is to improve mechanical properties and that is exactly what we are looking for in this research. With regard to the papers analyzed, there is a reasonable number of studies related to the characterization of the reinforcement prior to its application in the composite. The most applied tests are the constituent's analysis (cellulose, hemicellulose, and lignin), followed by Fourier-transform infrared spectroscopy (FTIR) which performs the chemical analysis of the fiber surface (identification and/or determination of structural characteristics), X-ray diffraction (XRD) to assess fiber crystallinity and energy dispersive spectroscopy (EDS/EDX) to assess the chemical composition of the particles.

There is a reasonable application regarding thermal properties. The thermogravimetry test (TG), which checks fiber degradation (loss of mass) with increasing temperature, was studied in 18 papers, while the differential scanning calorimetry analysis (DSC) was studied in 14 papers. Those tests aim to evaluate the thermal stability of both the residue and the composite.

Regarding to the physical properties, many researches analyzed the water absorption and the huge influence of moisture in the composite materials. The density, calorimetry and abrasion were also studied.

As for microstructural tests, the scanning electron microscopy (SEM) had greater application in studies, being observed in 34 papers. Through this test it is possible to perform structural analyzes and obtain morphological information such as: orientation and quality of the fiber/matrix interface, particles size, presence of impurities, voids, irregular surfaces, cracks, and evaluation of the failure modes.

The durability tests had the lowest number of applications in the composites evaluated by the papers, being observed two papers, where one studied the degradation by aging tests and the other the degradation by oxidation. These tests aim to study the ability of materials to maintain not only their specific properties, but also their service conditions during the time for which they were designed and produced. Composites subject to aggressive agents and physical or thermal degradation mechanisms may suffer unfavorable changes in their properties.

Few studies used dynamic analysis to evaluate the composite materials. Eleven papers addressed the analysis under mechanical and dynamic load, while only one work evaluated the dynamic-mechanical thermal analysis. Finally, regarding statistical analysis, only 9 papers applied methods to test the equality of population average, based on the analysis and nature of sample variances. The method used by these 9 works was the analysis of variance (ANOVA).

#### 4. CONCLUSION

Currently, natural fibers are being widely used and can be easily applied as reinforcement in polymeric matrix for composite production. It was observed during this research that in the last 4 years there was a significant increase in publications related to lignocellulosic residues in polymeric matrix, indicating the growth in importance of these materials. Progressively these bio composites are replacing the traditional ones, because of the fact they are ecologically correct, renewable and a low cost.

Thirty-seven scientific papers using natural fibers as reinforcement in polymeric matrix were selected. Among these papers, the countries that stood out were Brazil and India, with publications in journals of high academic importance. Mechanical tests (tensile, bending and impact), physical properties (water absorption, calorimetry, density), thermal tests (TG and DSC), residue characterization (FTIR, XRF, EDX, XRD), microstructural (SEM) were studied, and they are of great importance in order to obtain more information about the characteristics presented by these composites. The polymer composites, in general, have a high mechanical strength / density ratio, having the ability to provide products that withstand high loads with low weight. For these reasons, these composites can meet the demands of industries such as marine, aerospace, packaging, automotive and construction.

According to the results obtained through a systematic literature review, it was possible to obtain important indicators to be used in future works on the application of lignocellulosic residues as reinforcement in polymeric materials.

#### 5. ACKNOWLEDGEMENTS

The authors would like to thank the Federal Institute of Bahia IFBA/PRPGI for the financial support during this research project.

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