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Viscoelastic Performance of Biocomposites

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Additional information is available at the end of the chapter

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Abstract

The viscoelastic behavior and performance to creep of biocomposites made from fique natural fiber and low-density polyethylene-aluminum (LDPE-Al) obtained from recycled long-life packages were studied. A relationship was observed between the creep mechanical responses of biocomposites with respect to natural fibers. Additionally, the four and six parameters of the mathematical model were calculated from the creep curves. A very good agreement between the experimental data and the theoretical curves was obtained in the fluency region. The relationship between interfacial fiber or filler and the polymer matrix is an indicator of mechanical performance of biocomposite, regardless of the application that you want to give. It is known that the mechanical and viscoelastic properties depend on the application time of loading, the type of load, temperature, micromechanics relationship between the natural fiber and the matrix, the type of anchor prevailing for the transfer effort to micro- and nano-levels and cannot be treated mathematically only by the laws of solids or fluids, viscoelastic behavior of biocomposites. It is possible to obtain mathematical models that fit different rheological phenomena; for example, creep and stress relaxation can be modeled and correlated with biocomposite experiment using dynamic mechanical analysis (DMA).

Keywords: biocomposites, DMA, natural fiber, fique fiber, viscoelastic behavior, mathematical models

1. Introduction

One biocomposite is a material formed by a polymer matrix and a filler or reinforcement, with the characteristic that both the matrix and the filler or reinforcement, one should at least be of biological origin. Now, it is known that the concept of sustainability has managed to motivate industries to seek alternative and sustainable materials using natural fibers that can reinforce or fill materials for different applications. Similarly, efforts are made to the development of

compound parts that could become an option to supply the irregular use of wood, and 100% the use of synthetic polymers and origin of oil. Biocomposites are already accepted, viable, and sustainable alternative, are characterized by one of its phases, and are of biological origin may be fibers or natural fillers or polymer that can obtained from renewable resources such as sugar cane, corn, and among others. Various natural fibers have been used for strengthening plastic matrices due to their low cost compared to synthetic fibers. In this context, we have already conducted several investigations that have developed new composite materials using natural fibers from different sources [1–7], the composites of thermoplastic matrix reinforced with natural fibers or fillers, they can mainly improve the mechanical performance of the original polymers, besides obtaining benefit in lowering the density; so it may be possible to obtain lighter, economical, and resistant products, as it is already known applications to the automobile industry where natural fibers are replacing the synthetic fibers in different parts of automobiles due to their light weight and low cost [8–13]. Cellulosic fibers such as sisal, fique, coir, jute, palm, bamboo, wood, and among others, in their natural condition, and several of cellulosic wastes such as shells, wood flour, and pulp are used as reinforcing agents or filled thermoplastic and thermosetting resins in different years.

In Colombia, researcher are working on the development of different biocomposites with natural resources that have already been prepared and cultivated for different uses, one of these resources is the fique. This natural fiber grows on the leaves of the plants and furcraea in Andes; it is native monocotiledón xerofítico of the Andean regions of Colombia, Ecuador, and Peru. These plants are grown from Venezuela to the east coast of Brazil. The common names of these plants are fique, cabuya, pita, penca, penco, maguey, cabui, chuchao, or cokes. Mainly in Colombia the fique has been used as an alternative to develop compounds with ceramic and polymeric matrices. Investigations have been carried out with the aim of finding alternatives to the use of short fiber waste fique [14]. They have evaluated the flexural properties and voltage matrix composite with high-density polyethylene (HDPE), and fique fiber reinforced percentages are found between 7 and 55% (v/v). Similarly, we have investigated the effect of composition on the mechanical properties by incorporating 20% of calcium carbonate, in order to stiffen the material for use in construction, especially for the manufacture of plates or rectangular profiles for manufacturing pallets. Because of the interest to include sisal in other manufacturing processes, we have studied the influence of different surface fibers of sisal treatments, in the case of alkanization, chemical modification with maleic anhydride, acrylic acid, and silane was carried out in order to improve the mechanical behavior of a compound of unsaturated polyester resin matrix. It was possible to analyze the mechanical behavior of the composite material through bending tests, where it was observed that the best properties are presented in both compounds with fibers subjected to alkanization, as those in which the alkali treatment was a preprocessing of other modification surface [15]. We also studied the behavior of the hydrolysis of compounds of epoxy matrix, in which two types of surface treatments were analyzed in which fique fibers used as reinforcement (alkalinization and silanization). The authors tested specimens by immersing in tap and distilled water for obtaining decreased flexural mechanical performance, and also reduction in weight due to the presence of water in the material [16, 17].

Now, it is possible to develop new alternatives for fique, especially to develop new composite materials, particularly fique arranged as blanket, which uses short fibers in two-dimensional random arrangement. This material is susceptible to be used in manufacturing of various products and various thermoplastic matrices, especially for structural applications or products such as pallets or similar products, which are typically subjected to withstand loads varying time intervals and temperature changes. This chapter presents, as an example, a study of the mechanical and viscoelastic performance biocomposite low-density polyethylene, filled with aluminum and reinforced with natural fibers sisal which is called LDPE-Al-Fique. It was possible to obtain a formulation of biocomposite, especially based on analysis of micromechanical interactions between the continuous phase and the dispersed phase, including study parameters such as surface treatments and the arrangement of the fibers in the composite. Also, a study of the effect of fibers on sisal treatments alkalization, silanization, and impregnation regarding the effect on the strain rate, and viscoelastic properties, such as the module TOR-AGE, loss modulus, and tangent delta was performed. The behavior of the compliance function of time, compared to other biocomposites reinforced or filled bagasse and wood, was analyzed [6, 7].

2. Fillers or natural fibers reinforced biocomposites

The biocomposites are composite materials consisting of one or more phases of biological origin. They could be made from natural fibers, or natural fillers such as wood flour, and combined with various common polymers such as polyolefins. Biopolymers can also be used for its formation, which identify different types of polymeric materials, in the case of common polymers such as polyethylene, but based on renewable raw materials, for example, from cultures of sugarcane, or biodegradable polymers such as the PLA or PHB. The design of a biocomposite material arises from the intention to optimize the mechanical performance and/or physical materials, or fill, to achieve the improvement of different properties, among which are: thermal, water absorption, tribological, viscoelastic behavior, stress relaxation, slowing flame, energy absorption, and among others, in summary seeks to improve physical and mechanical properties and/or thermochemical, studying various effects of mechanical, chemical and/or physical treatments specially made of fibers to improve the properties of biocomposites of these fibers or work as fillers. The formation of the phases of the biocomposites determines their properties, usually, the formed fibers or fillers, phase aims at strengthening properties such as increased stiffness, or increasing the breaking load and achieving a low density [7, 18–26]. In general, polymeric materials, especially thermoplastics, are transformed by injection molding, extrusion, and thermoforming; these materials allow a dispersion of fibers that can be used for obtaining new materials and products, which can be fiber reinforced or fillers. The function of the fibers in the compounds is directly related to the applied stress resistance, while the matrix is responsible for the transmission of efforts to the reinforcement; conjugation of the two functions results in a better response of the reinforcement, which in turn can lead to an increase in the rigidity and strength of the material. The fiber-reinforced thermoplastic materials should be considered important factors in the

theoretical study of the properties of the matrix, fiber characteristics, matrix content, fibers or fillers, relationship, and response of the interface between the matrix fibers and fillers, and reinforcing fiber content is presented in terms of volume or weight [18, 27–29]. Applications of biocomposites reinforced with natural fibers or fillers are being investigated as a result of the increasing demand for sustainable and environmentally friendly products, the most common applications are: construction, automotive industry, and of packaging. Applications for the production of housing roofs, structural panels, beams, and door frames have been investigated. There are other applications in the construction in which the NFCS could be an economic and ecological choice. Now, we can find several applications for the development of new packaging and interior automotive parts, which are mainly developed by extrusion, injection molding, and thermoforming [30–33]. The use of renewable for designing biocomposite materials obtained from sources such as palm, flax, sisal, sisal, jute, and among others, these cellulose fibers can be classified into bast fibers and seed fibers such as cotton, coir fibers cane, rice bran, and wheat, as well as all other types such as wood and roots [34]. A global approach of the annual production of these fibers can be seen in **Table 1**.

Fiber source	World production (10 ³ Ton)
Bamboo	30,000
Sugarcane bagasse	75,000
Jute	2300
Kenaf	970
Flax	830
Grass	700
Sisal	375
Hemp	214
Coir	100
Ramie	100
Abaca	70
Fique*	22

Table 1. A global approach of the annual production of natural fibers [35].

It is known today for automotive applications, such as the German automotive industry companies such as Volkswagen, BMW, Ford, Audi, Daimler Chrysler, Mercedes, and among others. The applications of biocomposites are also in the construction industry, packaging, sports, and others, applied to the design of various products [36], some physical and mechanical properties of these natural fibers currently used for production biocomposites as shown in **Table 2** [37, 38].

Logistics problems for the collection of agro-industrial waste, emissions of greenhouse gases generated by incineration and subsequent problematic waste volumes continue to motivate

the use of natural fibers from various sources for the development of biocomposites for various applications. These new composite materials using wastes of agricultural and industrial processes are a sustainable alternative, provided that the production volume of these residues is not greater than the volume of use in the development of biocomposites for various applications [39]. Currently, applications in the development of structural products are also sought, which require a more precise understanding as to their physical and mechanical behavior, especially when trying to develop products that claim to have a long shelf life of months or years, which required to have a comprehensive understanding of the behavior or performance biocomposite in time, under different stress conditions and temperature mainly. By the technique of dynamic mechanical analysis (DMA) plus a good mathematical approximation behavior viscoelásto-plastic of biocomposites, it is possible to predict the behavior, physical, and mechanical modified biocomposite or reinforced with natural fibers based on experiments using this technique assays laboratory short-term (hours, days, or weeks) and align them with mathematical models that can predict behavior for longer periods, months or years [7, 40]. For structural applications, where biocomposites are subjected to cyclic or constant long periods of time loads, natural fibers such as bamboo, jute, sisal, sisal, hemp, and among others, by its constitution at the macro-level, have better mechanical performance and viscoelasticity when supporting loads over time [6, 10]. In the case of south American sisal, especially in the Andean region (Colombia, Ecuador, and Peru), it promises be a fiber with an acceptable reinforcing materials for various applications and manufacturing processes, also nowadays cultivation is sustainable, and widely used in South America for the production of textiles blankets, bags for packing coffee mattresses, and among others.

Fiber	Density (g/cm ³)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation (%)
Fique	1.47	132.4	8.2–9.1	9.8
Flax	1.4	88–1500	60–80	1.2–1.6
Hemp	1.48	550–900	70	1.6
Jute	1.46	400–800	10–30	1.8
Ramie	1.5	500	44	2
Coir	1.25	220	6	15–25
Sisal	1.33	600–700	38	2–3
Abaca	1.5	980	—	—
Cotton	1.51	400	12	3–10
Kenaf (bast)	1.2	295	295	—
Kenaf (core)	0.21	—	—	—
Bagasse	1.2	20–290	19.7–27.1	1.1
Henequen	1.4	430–580	—	3–4.7
pineapple	1.5	170–1672	82	1–3
Banana	1.35	355	33.8	53

Table 2. Physical and mechanical properties of natural fibers.

2.1. Biocomposites fillers or reinforced with fique fibers

Fique, commonly called Cabuya, Fique, Motua in the countries of the Andean region and its scientific name is *Furcraea bedinghausii*, is a large plant stem erect; its height varies from 2 to 7 m, with green leaves, long (1–3 m), narrow (10–20 cm), pointed, ribbed, and thorny; in some varieties, it has faint lines or stripes of about 3 mm long. Young plants consist of a rosette of thick fleshy leaves bluish-green; as the plant grows, it develops at the base of a short stem carrying 75–100 sheets whose length and width ranges from 150 to 200 cm and 15 to 20 cm, respectively, in the widest part near the middle, tapering to 10 cm near the base, having a thickness of 6–8 cm and can be seen in **Figure 1** [41].



Figure 1. Plant of fique in the Andean region.

Some characteristics and properties of fique fiber are shown in **Table 3**, the great variability of diameters can be obtained from fibers of the same batch, and even along the same fiber is highlighted, as is usual in the natural fibers [42].

Characteristic	Fique	Average
Equivalent diameter (mm)	0.16–0.42	0.24
Bulk density (g/cm ³)	0.72	–
Specific density (g/cm ³)	1.47	–
Water absorption (%)	60.00	
Water (%)	12.00	–
Effort last tension (MPa)	43.00–71.00	132.40
Last elongation (%)	9.80	
Modulus of elasticity (GPa)	8.20–9.10	–

Table 3. Characteristics of fique fiber [42].

Table 4 shows the chemical characterization of fique leaf, and composition of the fiber and bagasse juice leaf. They have been reported thermal analysis of fiber properties by thermogravimetry, which shows that the fiber supports fique at 220°C without degradation. The authors reported a bulk density of 0.87 g/cm³ density important in terms of specific properties [43].

Fiber		Juice	chaff	
Ashes	0.70%	Chlorophyll	Cenizas	12.20%
Cellulose	73.80%	Carotenoids	E.E.	3.64%
Resins, ceras and fats	1.90%	Saponins	Proteína	9.84%
Lignin	11.30%	Resins	Calcium	21.65%
Pentosan	10.50%	Flavonoids	Phosphorus	0.09%
		Orgánic acids	Magnesium	0.2%
Total	98.20%	Tras	Phosphorus	1.81%
		Water	Sodium	0.04%
		Lignin	Copper	14 ppm
		Calcium	Iron	647.00 ppm
		Lipoides	Manganese	33.00 ppm
		Phosphorus	Zinc	17.00 ppm

Table 4. Chemical composition of leaf sisal [41].

Position	Fiber	Country	Tons/year	Participation
1	Jute	India	1,900,000	57%
2	Jute	Bangladesh	800,000	24%
3	Fique	Brazil	191,103	6%
4	Abaca	Filipinas	70,356	2%
5	Jute	China	68,000	2%
6	Fique	México	41,856	1%
7	Abaca	Ecuador	27,194	1%
8	Jute	Myanmar	26,169	1%
9	Fique	Kenya	25,000	1%
10	Fique	Colombia	22,000	1%

Table 5. World production of natural fibers [45].

Table 5 shows some data are presented worldwide in the production of fibers, which leaves observe the position of women Colombia. It should be noted that in many countries have industrialized the use of compounds based on natural fibers that are available in their regions,

showing a very positive outlook for the industrial production of compounds based on fique fiber [14]. At present, there are very positive reports on an international level studies fique, where it has been used as reinforcement for polymer matrix composites with PE, PP, and among others [14, 17, 44].

The fibers of fique, regarding mechanical properties, have an approximate tensile strength of 237 MPa, a modulus of elasticity of 8.01 GPa resistance, and a strain of 6.02% at break [37, 42].

Fiber	Density (g/cm3)	Strain (%)	Tensile strength(MPa)	Young's modulus(GPa)
Cotton	1.50–1.60	7.00–8.00	287.00–597.00	5.50–12.60
Jute	1.30	1.50–1.80	393.00–773.00	26.50
Linen	1.50	2.70–3.20	345.00–1035.00	27.60
Fique	1.47	9.80	43.00–571.00	8.20–9.10
Hemp	-	1.60	690.00	-
Ramie	-	3.60–3.80	400.00–938.00	61.40–128.00
Fique	1.50	2.00–2.50	511.00–635.00	9.40–22.0
Coconut	1.20	30.00	175.00	4.00–6.00
Soft wood	1.50	-	1000.00	40.00
E Glass	2.50	2.50	2000.00–3500.00	70.00
S Glass	2.50	2.80	4570.00	86.00
Aramid (normal)	1.40	3.30–3.70	3000.00–3150.00	63.00–67.00
Carbon (standard)	1.40	1.40–1.80	4000.00	230.00–240.00

Table 6. Comparison of natural and synthetic fiber properties [11, 37, 42, 47, 48].

This has facilitated fique understand that is a good alternative to reinforce a thermoplastic materials to develop different products and different manufacturing processes. Fique is a natural plant that is used in ancient as fiber in the manufacture of packaging and others, which led to its establishment as permanent cultivation in the Andean region countries. However, currently, it is recognized as a vegetable product with different craft and agro-industrial applications and with immense potential in generating environmental benefits, employment, and income. The cultivated area fique in Colombia is distributed along 13 national departments: 98% of the 21,445 tons of fique produced are concentrated in 4 Colombian departments (Cauca, Nariño, Santander, and Antioquia); about 60% of the total production is in Nariño and Cauca. Fique fiber is used in products like ropes and sacks of seeds, grains, and coffee [37, 45]. The presence of synthetic fibers such as polypropylene has gradually made inroads in these markets. To develop products based on natural fibers de-

manding structural rigor required mainly improving mechanical properties and viscoelastic biocomposites who wish to develop. Previous studies in the field show that the viscoelastic performance of biocomposites varies with the type of filler, fiber, coupling treatment, and types of polymer matrices [6, 7, 10, 46]. Several modeling techniques have also been applied to analyze the flow behavior (CREEP) [6, 10–13]. **Table 6** shows a comparison of the most important properties of some natural and synthetic fibers, including fique.

In **Table 6** it can be seen that the novel compounds manufactured from natural fibers have advantages over the weight of the end products compared to glass fibers with an average of 2.7 g/cm^3 against $1.2\text{--}1.6 \text{ g/cm}^3$ of natural fibers. Natural fibers like fique other natural fibers can be processed in different ways to produce reinforcing elements with different mechanical properties. Depending on the type of reinforcement produced and its method of production, the modulus of elasticity and resistance may vary. Among others, cellulose fibers are obtained from wood by a chemical pulping process, they could have a modulus of elasticity of 40 GPa. These fibers can be subdivided to obtain microfibers by the hydrolysis process, reaching moduli of 70 GPa. Finally, by theoretical calculations of modulus of elasticity they were obtained up to 250 GPa predictions for cellulose chains (crystallites). The properties and structure of fibers also are affected by conditions and growth parameters, such as growth area, climate, and plant age [34].

Fique shows that it is susceptible to develop new materials used for different magnifications, but have similar disadvantages of any natural fibers in the world. For example, the fiber quality is variable, depending on unpredictable influences such as weather, moisture absorption, which causes swelling of the fibers, the maximum processing temperature is restricted, there is uncertainty in the viscoelastic performance over time but treatments fiber can greatly improve the price of the fiber that may vary from the results of the crop or agricultural policy and natural fibers are less durable and less resistance than glass fibers. In the research context, the above disadvantages are considered as opportunities to deepen their study and facilitate disinherit its applicability, as well as motivating their uses. At the same time, they have advantages employ: thermal recycling, where the glass causes problems in combustion furnaces, low specific weight, which results in greater strength and specific stiffness than glass, a renewable resource is possible; production requires little energy, carbon dioxide is used as oxygen is returned to the environment, can be used with virgin polymer matrix, recycled, as fillers, producible at low cost, processing and handling are friendly; low tool wear, no skin irritation occurs, and having good thermal and acoustic insulation. The hydrophilic nature of fique for its high cellulose content is one of the most important problems when trying to reinforce polymer matrices, because the vast majority of polymer matrices in the market are hydrophobic thermoplastic; this difference in physicochemical properties occurs as a result of an incompatibility between the natural fiber and the thermoplastic matrix, and this is reflected in poor stress transfer and mechanical behavior depends on the micromechanical interfacial relationship matrix fiber, also it affected the viscoelastic performance and general structural products to be manufactured with materials using natural fibers Fique without any surface treatment that improves the performance micromechanical compound and therefore the product is designed.

3. Behavior of viscoelastic biocomposites

The biocomposites inherit the behavior of the matrix with which they were manufactured, making their mechanical properties strongly dependent on the ratio of applied strain; therefore the mechanical behavior and viscoelastic structural products that are designed as sustainable products, which can be applied to the construction industry and automotive, mainly be affected by the dependence of applied stress and temperature conditions at the time. When biocomposites are processed and take the desired shape, e.g., extruded beams, molded housings, or any product which may be subjected to a constant load, will be generated on these products efforts, bending or tension or combinations thereof, constants, the effect of a constant effort on a product manufactured in biocomposites can be seen reflected in unwanted time warps, inclusive could produce the product failure. In this context, the durability of biocomposites can limit their applications, and implement risk of all the efforts of previous research, in order to develop sustainable materials for sustainable products. Studying the behavior of NFPCs under constant load conditions, where the deformation increases in time, that is, the material flows under the load (effect of creep), can understand that in a system biocomposite load between redistributes the matrix and natural fibers during deformation, when these materials are subjected to constant loads, can be affected by various effects of creep, the matrix, fiber, and the interface. There are several applications that have achieved biocomposites for extrusion with an addition 40–60%, mixed with thermoplastics, such as HDPE, PP, PVC, and materials [6, 49]. Compounds where manufacturers use natural fibers from different sources, as one of the fillers or reinforcements. These thermoplastic biocomposites can be used as tables for decks, fences, railway sleepers, etc. When used under these requirements, the CREEP or viscoelastic deformation becomes a problem, because the application of the effort takes the material to work under load long periods of time (months and years). This has been studied extensively in the case of advanced thermoset composites, and nowadays the investigations on biocomposites observed that the viscoelastoplastic behavior can lead to failure sustainable product, when subjected to large deformations and long periods of time, under conditions of dynamic or static load and temperature variations. These materials progressively accumulate deformation, causing internal damage occurs due to creep and/or fatigue, both cause cumulative damage [7, 50, 51]. There have also been efforts to correlate effects at smaller scales, relating effort plastic flow [52–54], according to the nonlinear response which it is due to permanent deformation. Investigations of some thermoplastic compounds have focused on deformation patterns, and have shown the strain-fluence compounds with particulate wood plastic, with alteration of the compositions and components of compound [49, 55, 56]. It has also been observed that with increased fiber content, the effect of creep decreases. Agro compounds used to develop products for structural construction, often requiring improved mechanical properties, particularly creep performance. It has been shown that the fluence of biocomposites varies with the type of filler and content, coupling treatment, and types of polymer matrices [6, 10]. Several molding techniques have also been applied to analyze the behavior CREEP [7, 10–12, 57].

At present, it is of interest to develop new thermoplastic biocomposites for sustainable products, and it is about the future course of implementation and sustainability over time of

biocomposites. There are estimates based on theoretical predictions, especially validated parameters obtained from accelerated tests, using the technique of dynamic mechanical analysis, DMA; this is the case study of creep behavior of composite materials based on different fibers such as bagasse, bamboo, and wood flour as matrix polyvinyl virgin and recycled vinyl and high density polyethylene. They tried to develop and adjust different theoretical models during all stages of CREEP to help predict long-term behavior. And observing different treatments and source matrices, they observed that models fit well in the linear zone CREEP, difficulties in predicting the primary and tertiary CREEP, referring difficulties in predicting the adjustment parameters, on lacking. Experimental long-term break in the tertiary CREEP, for the limited number of experiments that can be done using only accelerated techniques with DMA, especially those of CREEP long term [6, 10]. At constant load level a biocomposite has better creep resistance than ordinary polymer systems at low temperatures. However, biocomposites usually show higher temperature dependence. Various models of creep (Burgers model, model Findley power law, and a model of simple power law two parameters) have been used to adjust the data flow. The principle of time-temperature superposition (TTS) is typically used for predicting long-term creep, where it is important to understand that this method is valid mainly in the linear viscoelastic region of the biocomposites, however this method suffers from a prediction of the aging of natural fibers, including an error in time, which has now become complex including models for developing sustainable biocomposites. It has been shown that the four elements of the model Burgers and the power law with two parameters, adjust flow curves of biocomposite [6, 7]. Other authors have shown that PP-agglomerate compounds show different behaviors yield according to the processing conditions, i.e., with increasing fiber content, fluence compounds for example with wood fibers decreases [11, 56], studies are not derived from expressions that clearly include the flow properties of the matrix and fiber in their models, nor aging, or other factors associated with the nature of natural fibers. Therefore, the constant creep of these mathematical models is fully specified biocomposites, and are only valid for those compounds in particular and the conditions imposed nowadays. There is no complete method that can predict with high accuracy the viscoelastic performance of biocomposites, however, estimates that they are possible to perform with the use of the DMA, achieving reasonably guide the industry to seek applications for the development of new sustainable products, using biocomposite.

3.1. Linear viscoelasticity

Biocomposites have a typical response to mechanical loads, and can be studied as materials in some cases behave as elastic solids, and other, as viscous fluids. It is known that the mechanical and viscoelastic properties depend on the application time of loading, the type of load, temperature, micromechanics relationship between the natural fiber and the matrix, the type of anchor prevailing for the transfer effort to micro- and nanolevels, and cannot be treated mathematically only by the laws of solids or fluids, as viscoelastic behavior of biocomposites has high temperature dependence, especially if the work environment exceeds the glass transition temperature of the biocomposite, from the foregoing, the biocomposites in working conditions at constant load can be considered as super cold fluids. The above findings were mooted at the time of Boltzmann and others, but it is now clear that the vision of Boltzmann

was the right approach. As the understanding of the physical nature of the biocomposites and matured techniques has increased they have been developed many biocomposites. Since these materials are motivating the development of sustainable products, it is essential to analyze and understand from an engineering perspective, the response of biocomposites when load is applied and other environmental variables such as temperature and humidity. The difference between an elastic solid biocomposite and a viscous liquid is not an absolute difference, the ability to detect the elastic or viscous responses biocomposite object of study often depends on the time scale of the experiment and the conditions required recreate. Thus, from a strict point of view, all biocomposites have a viscoelastic behavior, i.e., depends not only on the state of stress to which the material is subjected, but also the history of preloading the material and all condition biocomposite that may affect the macrolevel, micro and nanolevel. The biocomposites are complex viscoelastic systems for manufacturing and high dependence on renewable raw materials, such as natural fibers. Viscoelastic behavior can be investigated using various methods; the use of dynamic mechanical analysis (DMA) is the most common nowadays. For example, in an experiment fluence (CREEP) a constant σ effort applied to a sample and the deformation ε is observed as a response function of time t . Normally effort increases with time and the flow curves (as a function of time) may exhibit three regions (**Figure 2**): primary creep in which the curve is concave downward; the secondary creep deformation in which it is proportional to time; and tertiary creep where the deformation is accelerated until the creep rupture occurs. Strain rate, which would be represented by the derivative of the deformation curve, also exhibits three regions.

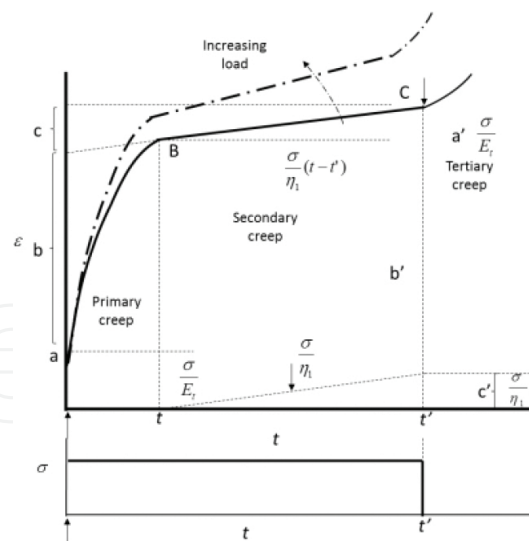


Figure 2. Schematic yield curve (CREEP).

In the yield curve (CREEP) high, the material has a linear viscoelastic behavior, so it is possible to apply the principle of superposition time TTSP temperature. However, nonlinearity presents high deformation speeds. In other words, the stress curves as a function of strain rate could show the transition from linear to nonlinear behavior in flow experiments (**Figure 2**).

A composite thermoplastic polymer matrix subjected to constant loads for periods of time and prolonged temperatures above the glass transition of the matrix work, regardless of the direction of load application, the material works to tension, bending, compression, or some combination of these efforts; its response to deformation over time is a combination of deformation, micromechanics, elastic, and viscous, which can be expressed in terms of compliance creep D , as:

$$\varepsilon_{(t)} = \sigma_o D(t, T, \sigma_o) \quad (1)$$

The creep compliance $D_{(t)}$ is the ratio of stress and strain generally as a function of time, as seen in Eq. (1). When considering the case of the response to creep as linear material, creep deformation is independent of the level of effort, which makes it look as a property of the material between different systems of composite materials, taken under similar environmental conditions. The total deformation at any instant of time $\varepsilon_{(t)}$ in a creep test of a biocomposite can be represented as the sum of the instantaneous elastic deformation ε_E (i.e., the initial deformation when the constant voltage is applied) and ε_V viscoelastic deformation. Similarly, compliance can be divided with elastic and viscous component. By submitting the biocomposites constant loads, regardless of your work address, the response of the material is creep or creeps. Where compliance depends on the deformation function in the time and effort that is subjected in Eq. (2) shows an expression for compliance:

$$D_{(t)} = \frac{E_{(t)}}{\sigma_o} \quad (2)$$

For the design and manufacture of products based on biocomposites require to define the CREEP as the change in function of time in the dimensions of a product polymer or composite when subjected to constant stress in different working conditions, which may include, temperature, environmental, cyclic loading, and among others. The biocomposites usually have CREEP behavior at room temperature; which is due mainly to its micromechanical relationship fiber-filler with the matrix, and the combined efforts to which the material may be subjected to some cases also the flow behavior may be generally negligible. Therefore, design procedures are simpler because the module can be considered constant (except at high temperatures). However, the modulus of a polymer or composite material is not constant (as shown in Eq.(2)), because the deformation is a function of time, and compliance is directly related to the stiffness of the material. Whenever variation is known, the behavior CREEP of biocomposites can be compensated by the precise use and well-established design procedures, or by modifying the composition of the biocomposite, using reinforcements and/or fillers to correct their mechanical performance and viscoelastic. For biocomposites, the aim of the design methods to determine the stress values does not cause permanent deformation intolerable products or fractures. Excessive deformation becomes a limiting factor in the selection of work effort, leading to the conclusion that it is essential to qualify and specifically quantify the

deformation behavior of the biocomposites, depending on time and temperature. A schematic diagram of flow behavior (creep) can be seen in **Figure 3**; given load shows a configuration of four point bending biocomposite. The weight or load, along with gravity, provides a constant effort in biocomposite. After 5 days in this condition no significant unfavorable deformation occurs. However, after 7 months deformation caused by the effort has increased, and deforms further after 2 years.

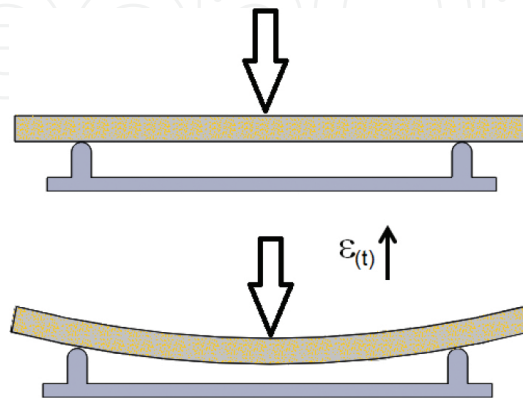


Figure 3. CREEP response of a biocomposite beam subjected to bending at four points, an illustrative estimate given by the author.

The biocomposites polymer matrix has significant sensitivity to use a function of time and temperature, resulting in a limited use of structural applications demanding applications or in dimensional stability value. When the biocomposite is subjected to high stresses, it can result in the material, and excessive deformations that may cause the product to lose its functionality, one could get the material to the tertiary region where creep occur until fracture. This is called the upper region and is also known as the acceleration phase of CREEP. The importance of the tertiary region for normal operation and design to CREEP is also important, since parts of polymer or compound should be designed to avoid this area; safety factors must ensure away from this region over the lifetime of the products developed with biocomposites.

3.2. Mathematical models

The experimental response of a dynamic test to tension, bending, or compression creep compliance of a biocomposite can be modeled with provisions of springs and dampers, where the springs represent the elastic solid behavior and cushion the behavior of a viscous liquid. It represents the Hooke spring deformation force that is proportional to the applied stress and the damper flow proportional to the strain rate Newtonian. To model mathematically one biocomposite, the stress, strain and time, you can relate to the constant characteristics of the mechanical elements [57]. The mechanical model mimics the actual behavior of biocomposites, although the elements themselves may not have direct analogies with real material. However, these models represent a mathematical understanding of the problems of viscoelastic performance of biocomposites, studied by accelerated tests in the laboratory, which can easily be articulate studies of continuum mechanics means to solve even more complex models with the

help of numerical methods and approach to more realistic models that include visco-elastic-plastic. It is emphasized that mathematical models presented in this chapter are classical performances already studied by several authors, that when applied to biocomposites, approximate their behavior and allow you to compare and study relationship deformation at short times and long-term predictions that might suggest designers, which are the most desirable when applying for the development of sustainable products made from biocomposite materials.

3.3. Maxwell, Kelvin, and four parameter models

Figure 4(a) shows the Maxwell model, which is represented by a spring connected in series with a damper and **Figure 4(b)** shows Kelvin model (or Voigt), which is represented by a spring connected in parallel with a damper; in both cases an approximation of a system characterized by time dependent and the ratio of η viscosity (damper) with the modulus of elasticity E represented by the spring is obtained, which they can be approximated to describe the viscoelastic behavior of a biocomposite. The parameter η leads to model a related response delay time for the Kelvin model, and the relaxation time for the Maxwell model.

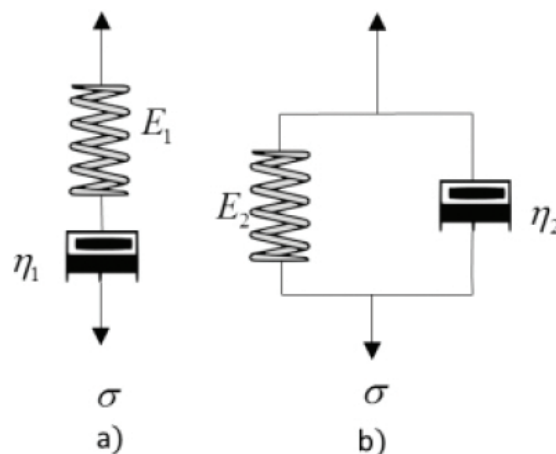


Figure 4. Models: (a) Maxwell, (b) Kelvin.

To analyze the flow behavior (CREEP) of biocomposites, it is possible to apply the model of four parameters successfully, which is derived from a series combination of the Maxwell model and Kelvin model, as shown in **Figure 5**.

The four-facing model fits the response obtained experimentally for the controlled creep test, based on the technique of dynamic mechanical analysis (DMA). **Figure 6** depicts a curve of biocomposite creep subjected to a three-point bending at a constant effort in the linear viscoelastic region. The fraction of O to A shows the rapid response of the initial deformation on the flow curve, i.e., it occurs an instantaneous elastic response. This behavior is followed by a region of creep from A to B, where the shear rate decreases at a constant rate introduced in Section B to C. Once the stress is removed, the instantaneous elastic O to A response is fully retrieved from C to D, i.e., the distance $a' = a$. Then the curve drops from D to E in a slower

recovery. However, this recovery is not complete due to the initial state by increasing $c' = c$. This response is completely unrecoverable, and is a measure of plastic flow [58].

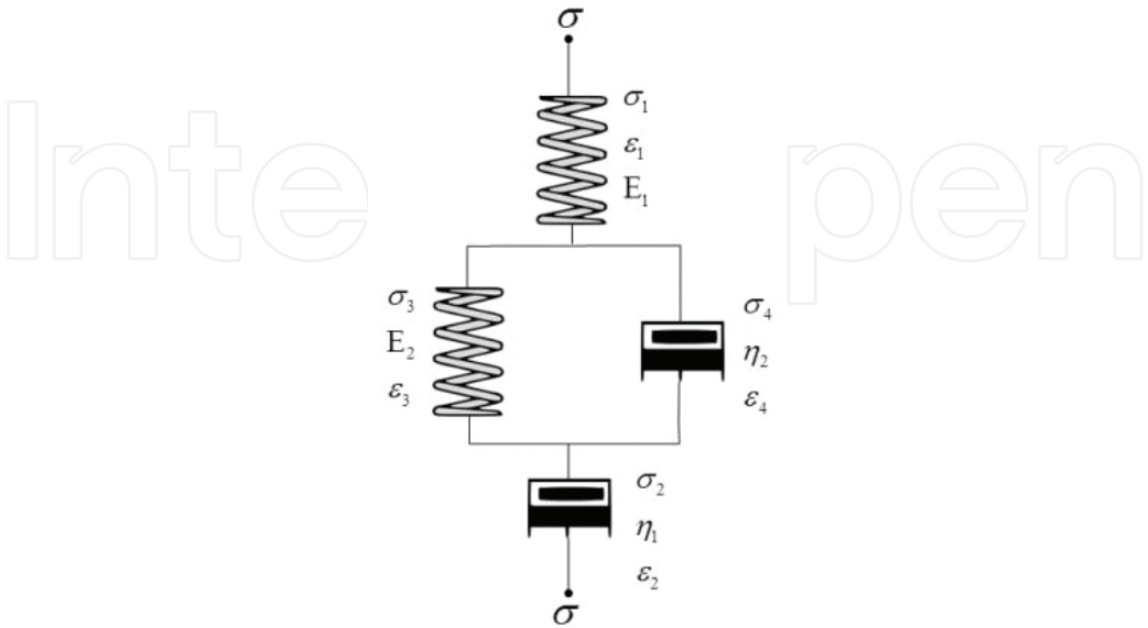


Figure 5. Four-parameter model.

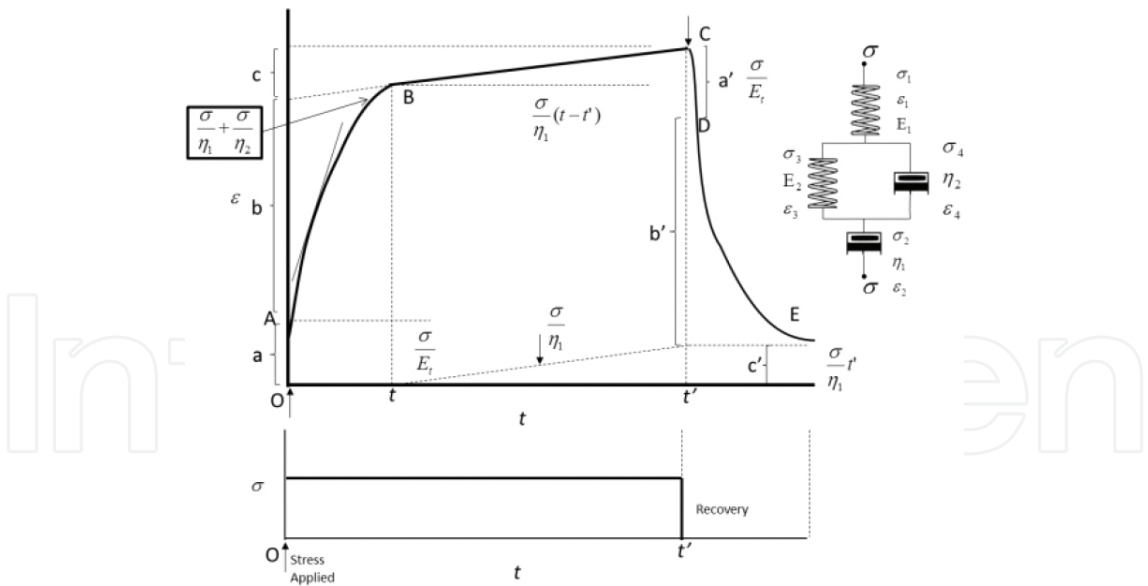


Figure 6. Four-parameter model and yield curve (CREEP).

Figure 5 represents the elastic response changes in A and A'. A convenient to model this response element is the Hooke law. The Kelvin-Voigt model corresponds to changes in b' (change symbol) in the region of creep; it represents a damping plastic flow c'. **Figure 6** shows the mechanical models for the purpose of better illustrating the appearance of the flow curve.

The four-parameter model is an assembly of a Maxwell element and Kelvin-Voigt element, where the latter component is time dependent. **Figure 7** shows that (zone 1) the system is idle effortless. When it exposed to constant stress to three-point bending, in **Figure 7** (zone 2), the spring system with a constant amount of E_1 extends instantaneously to “a” $\sigma/E_1 = a$. Then, in **Figure 7** (zone 3), the fluence rate decreases with a gradual increase in load bearing spring E_2 , until fully extended and the damping η_2 no longer carry any load. As the spring is now E_2 fully extended, the creep ratio to a solid phase, corresponding to the plastic flow of the linear viscoelastic region represented by the constant η_1 damper. The damper deforms until the load is removed, as illustrated in **Figure 7** (zone 4), leaving permanent deformation. Now, the spring retracts quickly E_1 to a' and the recovery period is b' . During this time, the damper η_2 is forced to retreat to its initial position by spring E_2 representing a delayed or anelasticity elastic response. The damper position η_3 remains in the extended state, since the spring cannot influence its final position; this can be seen in **Figure 7** (zone 5). Thus, the nonrecoverable plastic flow is equal to $c' = \sigma t/\eta_3$. The model fully represents the elastic, inelastic, and viscous behaviors of biocomposites, indicating that if possible with fillers or natural fibers affecting the interfacial relationship, fiber matrix polymer or cross-linked polymer, the variable η_1 increase, which it is reflected in a decrease in permanent deformation c .

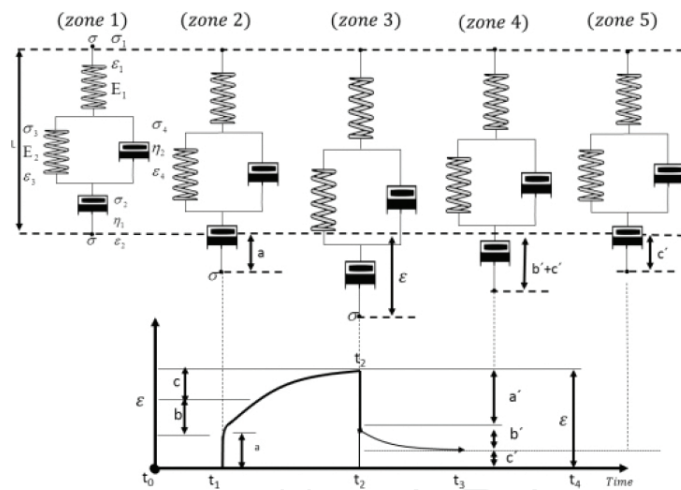


Figure 7. Response of four-parameter model adjusted to fit a creep curve.

Given the balance of forces occurring in the four-parameter model, we can write the following expression, with respect to the effort and deformation:

$$\sigma = \sigma_1 = \sigma_2 = \sigma_3 + \sigma_4 \quad (3)$$

$$\varepsilon = \varepsilon_1 + \varepsilon_2 + \varepsilon_{3,4} = \varepsilon_1 + \varepsilon_2 + \varepsilon_k \dots \dots \dots \varepsilon_3 = \varepsilon_4 \quad (4)$$

where ε_k is the strain response of the Kelvin-Voigt model, the equations representing different relationship stress-strain are:

$$\sigma_1 = E_1 \varepsilon_1, \sigma_2 = \eta_2 (d\varepsilon_2 / dt), \sigma_3 = E_3 \varepsilon_3, \sigma_4 = \eta_4 (d\varepsilon_4 / dt) \quad (5)$$

And the equation that relates and models the behavior of biocomposite visco-elastic-plastic holistically can be arranged as follows:

$$\frac{\eta_1 \eta_2}{E_1 E_2} \left[\frac{d^2 \sigma}{dt^2} \right] + \left[\frac{\eta_1}{E_1} + \frac{\eta_1 + \eta_2}{E_2} \right] \frac{d\sigma}{dt} + \sigma = \frac{\eta_1 \eta_2}{E_2} \frac{d^2 \varepsilon}{dt^2} + \eta_1 \frac{d\varepsilon}{dt} \quad (6)$$

Equation (7) can be solved for flow conditions and/or relaxation; response to deformation is:

$$\varepsilon(t) = \frac{\sigma_0}{E_1} + \frac{\sigma_0}{\eta_1} t + \frac{\sigma_0}{E_2} \left[1 - e^{-\frac{E_2 t}{\eta_2}} \right] \quad (7)$$

Response to creep recovery is:

$$\varepsilon(t) = \frac{\sigma_0}{\eta_1} t + \frac{\sigma_0}{E_2} \left[e^{-\frac{E_2 t_1}{\eta_2}} - 1 \right] e^{-\frac{E_2 t}{\eta_2}} \dots t > t_1 \quad (8)$$

where $\varepsilon_{(t)}$ is the yield strength, σ_0 is the initial applied stress, t is time, E_1 and E_2 are the elastic modulus of the springs of Maxwell and Kelvin, respectively, and η_1 and η_2 are the viscosities of the Maxwell and Kelvin dampers. η_2/E_2 is usually denoted as τ , the delay time required to generate 63.2% strain on the Kelvin unit [59].

In Equation (7), the first term is the instantaneous elastic strain. The second term is the early stage of creep deformation, and is due to mechanisms such as relaxation, extension of the molecular chain, and biocomposites closely related to the performance of the fiber matrix micromechanic relationship. The last term represents the long-term creep deformation, and is due to the overall performance of the biocomposite. The parameters E_1 , E_2 , η_1 , and η_2 can be obtained by adjusting the equation to the experimental data and can be used to describe the creep behavior. The strain rate of the linear viscoelastic region of the biocomposite is possible to calculate, if we derive Eq. (7), and obtain the strain rate:

$$\dot{\varepsilon} = \frac{\sigma_0}{\eta_1} + \frac{\sigma_0}{\eta_2} e^{-\frac{E_2 t_1}{\eta_2}} \quad (9)$$

For all models of linear viscoelasticity, it is important to note that the response of deformation creep is independent of the level of effort, giving the opportunity to study and compare with

other systems evaluated under the same environmental conditions, must obtain curves and models of creep. The equation for calculating the compliance $D_{(t)}$ is defined by:

$$D_{(t)} = \frac{1}{E_1} + \frac{t}{\eta_1} + \frac{1}{E_2} \left[1 - e^{-\frac{E_2}{\eta_2} t} \right] \quad (10)$$

The viscoelastic behavior of a system biocomposite presents different delay times; therefore, it is possible to model more precisely repeating n Kelvin-Voigt models, which are particularly adjusted in the delayed elastic region or annalistic, which has default a typical nonlinear region. In **Figure 8**, you can see a diagram showing this behavior.

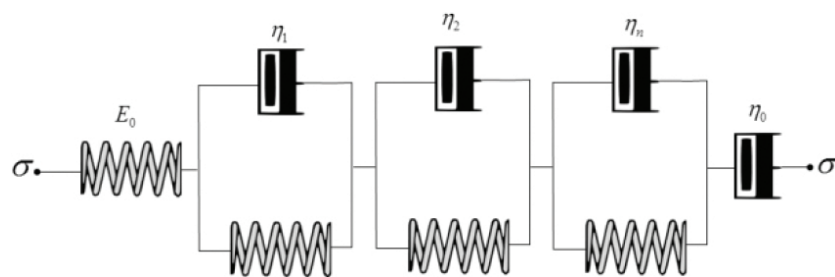


Figure 8. Multiple models for adjusting creep curves.

Fundamental viscoelastic properties such as creep compliance module or relaxation can be found by solving the differential equation that represents including for the appropriate load if necessary. For example, the compliance fluence can be determined using the conditions for a creep test and would be represented by:

$$D_{(t)} = \frac{1}{E_1} + \frac{t}{\eta_1} + \sum_{i=2}^n \frac{1}{E_i} \left[1 - e^{-\frac{t}{\tau_i}} \right] \tau_i = \frac{\eta_i}{E_i} \varepsilon_{(t)} = D_{(t)} \sigma \quad (11)$$

The determination of initial conditions is achieved by inspection of the physical model. Since the input force is constant for the creep test, the change in stress is zero. The solutions of differential equations for relaxation conditions, constant deformation, or changes in stress and other conditions can be obtained similarly.

3.4. Time-dependent deformation behavior of biocomposite

A way to study the effect of a reinforcement or filler natural fibers to manufacture biocomposites is to show the interdependence of the stress, strain, and time through curve creep and recovery creep performed in short tests using the DMA technique dynamic mechanical analysis. To perform the creep tests and recovery (creep and creep recovery), it is necessary to set a DMA with the force of flow required to maintain the constant effort during testing of

creep at a given time, and rezero when it reaches the estimated time then deformation is recorded by another equal time. For example, for a biocomposite made of polyethylene aluminum fiber fique, we can observe the response to creep and creep recovery in **Figure 9**, a maximum stress of 1.2 MPa was applied in a bending test at three points at 25°C, 1.2 MPa effort is an attempt that was obtained after identifying the linear viscoelastic region, a biocomposite test strain sweeps at 25°C in a DMA (RSAIII).

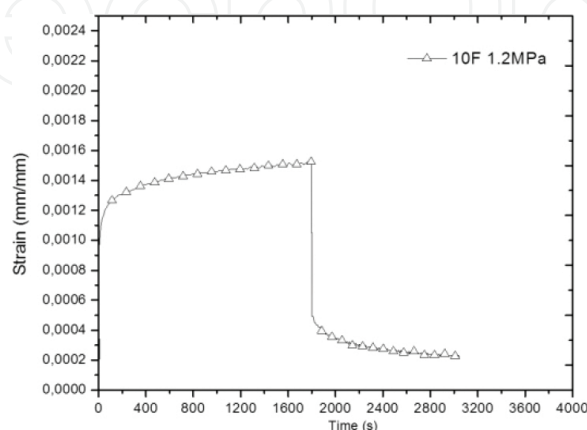


Figure 9. Creep curves and creep recovery for biocomposite LDPE-Al-Fique 10% reinforcement of natural fibers.

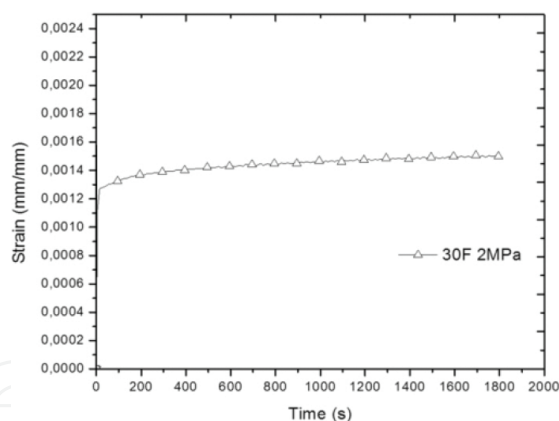


Figure 10. Creep curves for biocomposite LDPE-Al-Fique 30%.

As presented schematically in **Figure 6**. For an adjustment with a four-parameter interpretation is the same, it is important to note that the effect of reinforcement on a biocomposite can be studied by this method, especially when amending fiber volume, nanoreinforcements, fillers, filler, or some surface modification is made to the fibers or fillers in order to improve performance micromechanical, and default creep performance is used. In **Figure 10**, one can see a behavior of an LDPE manufactured biocomposite-Al-Fique 30% fiber volume. Being possible to observe the effect of increased volume is positive with respect to creep, decreasing the speed of decoration and to increase enforcement effort in the linear viscoelastic region by nearly 60%.

4. Strain rate of biocomposites

To calculate the strain rate of the biocomposites subjected to creep tests, one can be approximated using the four-parameter model represented by Eq. (6), or model parameters n represented by Eq. (11). By making adjustments mathematical models, is provided mainly for comparing quantitatively the effect of strain rate, and further study of the performance of the incorporation of fibers, surface treatment agents couplings for fibers or polymers, fillers, nanoreinforcements, and fillers to manufacture biocomposites, controlled conditions of temperature and constant effort. In **Figure 11**, one can observe an experimental curve creep, and the respective model adjustment four parameters and the model of n parameters, it is emphasized that in the two curves creep models over four parameters. It is better than the four-parameter fit, which facilitates the study of the incorporation of fillers, reinforcements, nanoreinforcements for biocomposites.

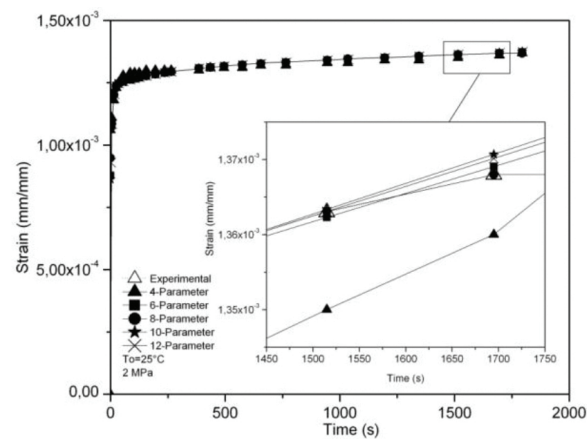


Figure 11. Adjustment of an experimental curve of a biocomposite creep, using the four-parameter model and model parameters n .

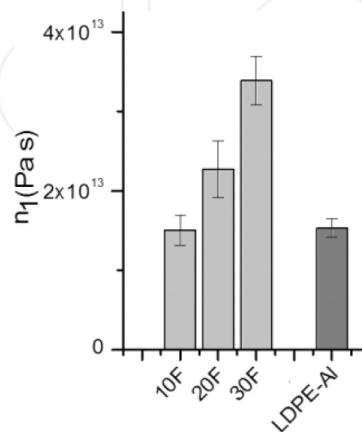


Figure 12. n_1 parameter for 10, 20, and 30% biocomposites reinforced with natural fiber sisal, and unreinforced LDPE-Al.

In **Figure 12**, we can see the response of viscous parameter n_1 , which allows the calculation of the strain rate for creep tests; this exercise was carried out, varying the volume of natural fibers fique biocomposite, corroborating the possibility of deepening the study of the effect of reinforcements or fillers to biocomposites.

Figure 13 shows the setting of the four-parameter model, two biocomposites, with different volumes of natural fiber reinforcement sisal. It is possible to corroborate and validate the model.

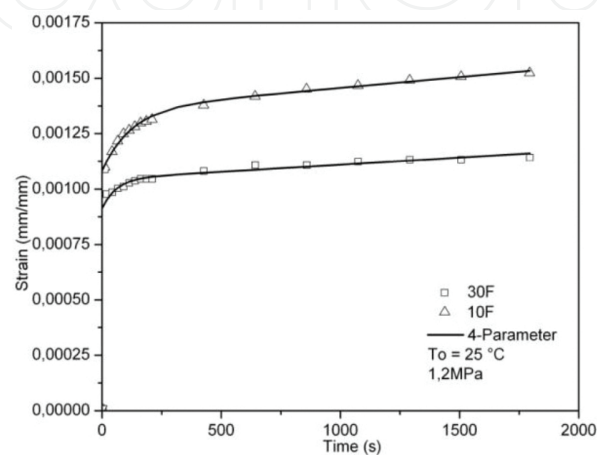


Figure 13. Setting the four -parameter model to LDPE-Al-Fique 10 and 30% incorporation biocomposites of fibers.

Table 7 shows the adjustment of the different parameters for a model of six parameters, including error, which is the estimate of least squares adjustment parameters can be obtained using the weighted sum of squares (weighted sum of squares, WSS), using Eq. (12):

Parameter	10%	30%
E1 (Pa)	1.10E + 09	1.50E + 09
E2 (Pa)	4.79E + 09	1.53E + 10
E3 (Pa)	7.91E + 09	8.39E + 07
N1 (Pa.s)	1.49E + 13	3.12E + 13
N2 (Pa.s)	6.48E + 11	9.32E + 11
N3 (Pa.s)	7.83E + 13	3.33E + 15
WSS	9.23E-08	6.43E-08

Table 7. Parameters of creep tests obtained by adjustment to a model of six parameter biocomposites LDPE-Al-Fique 10 and 30% incorporation biocomposites of fibers.

$$WSS = \sum_{i=1}^n w_i \left[\varepsilon(t_i) - \hat{\varepsilon}(t_i) \right]^2 \tag{12}$$

where $\varepsilon(t_i)$ represents the observed at time t_i experimental deformation (t_i), ε is estimated by the model, and w_i the difference between two samples of time deformation. A smaller value indicates a better fit WSS model to experimental data.

In **Figure 14**, we can see micrographs obtained by electronic scanning microscopy of an LDPE-Al-Fique biocomposite, where you can see that the fiber has a hydrophobic property, not adhere completely to the polymer, while aluminum has some adhesion but the manufacturing process of compression molding favors the adhesion between the faces, which can be reflected in a decrease of mechanical and viscoelastic performance of the biocomposites.

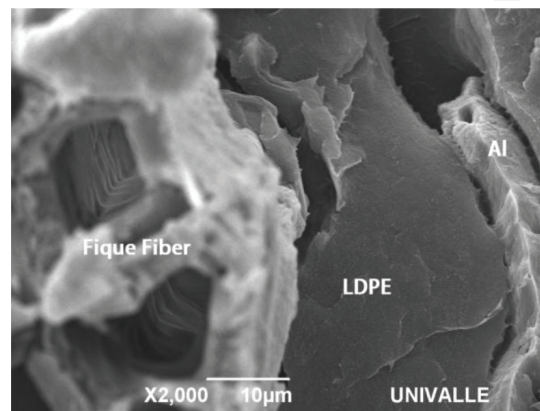


Figure 14. SEM micrographs of LDPE-Al-Fique biocomposite.

Currently, it is known that these defects can be corrected by surface treatments in the fibers, primarily using coupling agents or modifications to the polymers to achieve a greater adhesion and micromechanical relationship to reduce the strain rate.

5. Conclusions

Natural fibers as reinforcement of nonbiodegradable and biodegradable polymers have been used for decades for the development of various products, especially in the automotive, construction, and packaging industry. The importance of studying the viscoelastic behavior of biocomposites lies in understanding the impact of natural fibers or fillers on any scale, in the matrices that you want to use for different applications. Now, it is known that the structural rigor for automotive, construction, and/or packaging parts lies mainly in the relationship of the shape of the products with the stresses to which these materials are subjected, temperature changes, environmental and even other physicochemical factors that could alter the performance of the biocomposite in time. The articulation of theoretical models and experimental results using the technique of dynamic mechanical analysis (DMA) to predict the behavior over time, and the effect of filler or reinforcement, nanoreinforcements, micromechanics, surface treatments to the fibers, or modifications to polymer matrices facil-

itate a better understanding of its operation and its use at an industrial level for various applications. In South America, the natural fibers have recently become an attractive reinforcement or filler for researchers, engineers, and scientists as an alternative to develop biocomposites. Because of its low cost, sometimes good mechanical properties and good specific resistance generate a high environmental impact and additionally, it is biodegradable. It has been found that the use of natural fibers such as fique of South American Andean region can be used as reinforcement in composite materials and generate a lot of possibilities for industrial applications. It was found that the addition of fibers in different polymer matrices leads to produce new natural compounds with good physical properties for use in different sectors. The study revealed that biocomposites inherited effects relaxations attributed to transitions suffered by the polymer matrices used, whereby the relationship of fiber-matrix-filled showed evidence that the biocomposites always have a positive effect creep (creep), however, and while being exposed to constant loads over time and while working above the glass transition temperature, the biocomposite will behave predominantly as a super-cold fluid, and not likely as an elastic solid.

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