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Comparative Study of Matrix Fiber Adhesion of Tannin/Vegetable Fiber, Tannin/Synthetic Fiber and Epoxy Composite Materials

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ABSTRACT

This paper aims to compare the fiber-matrix adhesion of epoxy/kenaf and tannin/kenaf composite materials. Pull-out tests were performed to assess the shear strength at the fiber-matrix interface. The observations are correlated with statistical analyses. The results showed that the shear strength at the fiber-matrix interface was significantly higher for kenaf fibers treated with 0.5 M sodium hydroxide (NaOH) and coupled with epoxide (0.1390 N/mm²) compared to the same fibers treated and coupled with a tannin-based vegetable matrix (0.0903 N/mm²). However, this treatment caused a slight reduction in the tensile strength of the fibers. The surface of the treated kenaf fibers is very clean; the disappearance of impurities such as wax and oil led to improved molecular cohesion on the surface of the treated kenaf fibers, suggesting enhanced fiber-matrix adhesion. In fact, the absorption and dispersion energies on the fiber surface increased with the sodium hydroxide treatment and alkalization

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1. Introduction

Although biobased plastic materials are currently gaining in importance, the biobased composites available on the market are generally only considered as such thanks to the use of reinforcements based on plant fibers, their matrix remaining in most cases of fossil origin (Blanco & Siracusa, 2021; Hounkpatin et al., 2023; Pandit et al., 2018; Paul et al., 2023; Sayouba et al., 2023; Sid et al., 2021; Wasieleski et al., 2021). However, since the matrix most of the time represents more than half of the mass of the composite, the trend is towards using matrices that are also biosourced to move towards a "100% biosourced" composite. As is often the case, man still seeks to copy what nature has done very well for millions of years: wood!

Composites with biosourced reinforcements, as defined above, made a significant impact in Europe in 2010, representing a volume of 362 kT and constituting approximately 15% of the total composite production estimated at 2.5 million tonnes. This trend of replacing the petro-sourced matrix with increasingly larger quantities of biosourced polymers is a promising sign for the future of sustainable materials (Chang et al., 2021; Oliver-Ortega et al., 2021; Weyhrich et al., 2023).

The main factors driving the increasing development of these materials are well-known:

- Limited and increasingly expensive fossil resources;
- Sustainable development and concerns about climate change, which are at the heart of changes in Global regulations;
- Global reindustrialization is partly based on the development of the bioeconomy.

The main factor limiting biosourced materials' development and market penetration is the lower performance/cost ratio than that of materials from petrochemicals, which products with less maturity can explain. The specific challenges linked to the performance/cost ratio of biobased materials are significant, but not insurmountable. Despite their advantages, they are still often overlooked because of their price. However, the gap with traditional materials is narrowing, and long-term calculations favor other materials. To meet these challenges, it would be wise to seek a compromise between their performance and their market price. The lack of rigorous data concerning the life cycle of these materials also hinders their development. However, the combination of microscopic imaging and the "pull out test" allows the observation of the shape of the fracture surface between two materials. Indeed, microscopic imaging makes it possible to observe material surfaces with a resolution of the order of µm. These observations aim to understand the microstructure of the various reinforcements (fiber arrangement, fiber/matrix interface) and to detect any manufacturing defects (decohesion's, voids) (Ferrier et al., 2015). Specific pull-out tests are used to assess the influence of different parameters (fiber type, bar diameter and surface geometry) on the force transfer mechanism at the interface. Good adhesion excludes the presence of a break at the interface The perpendicular tensile test is standardized for wood materials, and is considered a good indicator of the adhesion quality (Cook & Chiu, 1997; Xiong et al., 2018). As a result, the problem of matrix fiber adhesion arises and forces us to focus on it throughout this paper. Your work in this field is crucial to overcoming these challenges and advancing the use of biosourced materials.

The main objective of this study is to compare the fiber/matrix adhesion between composite materials based on kenaf fibers reinforced with either an epoxy resin or a tannin resin. Fiber/matrix adhesion is a key parameter conditioning composite materials' mechanical properties and durability (Joly et al., 2005). Pull-out tests are used to assess interfacial shear strength, and the results are statistically analyzed to identify the factors influencing this adhesion (Jawaid et al., 2011; Saba et al., 2015). This study aims to understand better the interactions between kenaf fibers and epoxy and tannin matrices to optimize the properties of biobased composite materials. The results obtained will help guide the development of new high-performance composites that meet current environmental requirements. First, a review of the literature on the properties of plant-reinforced composites will be presented to better understand the mechanisms involved.

2. Literature review

2.1 General information on tannins

2.1.1 Classification of tannins

Tannins are non-nitrogenous polyphenolic compounds present in the majority of plants, like cellulose, hemicelluloses and lignin (Adegoke et al., 2022; Ahmed et al., 2023; Cheynier, 2012; Karioti et al., 2016; Ntenga et al., 2017; Patra & Saxena, 2011; Sellam et al., 2022). They are obtained by aqueous counter-current extraction at high temperature (90°C maximum), with or without additives such as sulphites, bisulphites or urea. Extraction yields are relatively low, ranging from 10% to 30%, depending on the method used. These plant tannins are classified into two main categories based on their chemical structure: hydrolyzable tannins and condensed tannins (Alkhoori et al., 2022; Ayari-Guentri et al., 2024; Forzato et al., 2020; Lazzari et al., 2023; Ökmen et al., 2023; Pour et al., 2016; Romani et al., 2002; Saxena et al., 2021; Shresta et al., 2021). One of the reasons for using tannins as a matrix material is their oxidation resistance.

2.1.2 Extraction of tannins

Tannin extracts are obtained by concentration of tannin solutions obtained by leaching particles of wood or bark (Aspevik et al., 2018; Borrero-López et al., 2022; Dietrich & Pour Nikfardjam, 2017; Dijkstra, 2013; Gadrat et al., 2022; Koopmann et al., 2020; Nunes et al., 2020). The manufacture of wood-based panels such as plywood or chipboard requires the use of formaldehyde, a volatile and toxic product. For this reason, new adhesives based on natural tannins extracted from wood have been developed (binti Hamdi & Ahmad, 2023). They contain little or no formaldehyde and offer enhanced mechanical and durability characteristics. The extract can be recovered in liquid form, in a highly concentrated solution or more commonly in dry powder form (4-6% humidity). Obtaining these products differs little depending on the raw material used. In the case of using wood to obtain tannins, only large branches and trunks are generally exploited (Salman et al., 2014). They are cut and split into billets for transport to extraction facilities. Everything is then cut perpendicular to the axis of the fibers in the form of chips or wafers. The grain size greatly influences the extraction speed, so the maximum chip thickness is generally between 5 and 10 mm. Indeed, it shows that as chip thickness decreases, tannin extraction time increases. The extraction is then carried out using the counter-current principle, which makes it possible to obtain more concentrated solutions and extract a larger

quantity of tannins from the wood. We generally use a series of autoclaves connected so as to circulate the extraction solution at temperatures ranging from 110°C to 90°C, respectively starting from the autoclave containing the most leached chips and going towards the autoclave containing the fresh chips. Table 1 gives the evolution of the extraction rate of the total mass of extractables as a function of the size of the chips.

Table 1. Extraction rate as a % of the total mass of extractables depending on the size of the chips

Chip Thickness (Mm)	18	15	10	5
Tannin Extracted In 4hours (%)	47	50	60	62

The recovered solution, which at this stage only contains approximately 6% dry extract, is then cooled, decanted to eliminate the insoluble, and concentrated by evaporation of water. To limit the hydrolysis and oxidation of tannins, the concentration is carried out under low pressure to lower the boiling temperature of the mixture. At the end of the concentration cycle, the solution approaches 50% dry extract. Tannins can be supplied as is in solution but are currently generally delivered in powder form (Herzi et al., 2013; Konfo et al., 2023; Ntenga et al., 2017; Romanus et al., 2009). The concentrated solution is then passed through an atomizer with cyclonic separators, and the collected powder is packaged for sale.

2.2 Loadings

There are 4 main types of stress to which a material can be subjected: traction, compression, shear and bending (Azuwa & Yahaya, 2024; Corinaldesi & Nardinocchi, 2016; Kassoul et al., 2018; Liu et al., 2019; Qasim et al., 2022; Zaoui et al., 2022).

- **Tensile**: The response of a composite in tension depends mainly on the stiffness and strength of the fibers, as they are much greater than those of the resin.
- Compression: Here, the rigidity of the matrix plays a more critical role, particularly for composites with unidirectional fibers, because it must maintain the fibers in the axis of the applied force.
- **Shear**: This type of loading uses the properties of the matrix to transfer and distribute the forces applied to the reinforcement. In addition to having high-performance mechanical properties, the fiber-matrix interface must be of high quality; that is, the resin must adhere well to the fibers.
- **Bending**: In reality, bending is just a combination of tension, compression and shear applied to the composite. The upper face is subjected to compression, the lower face to tension, and the central layer to shear.

Figure 1 shows the evolution of the stress of a composite as a function of its elongation.

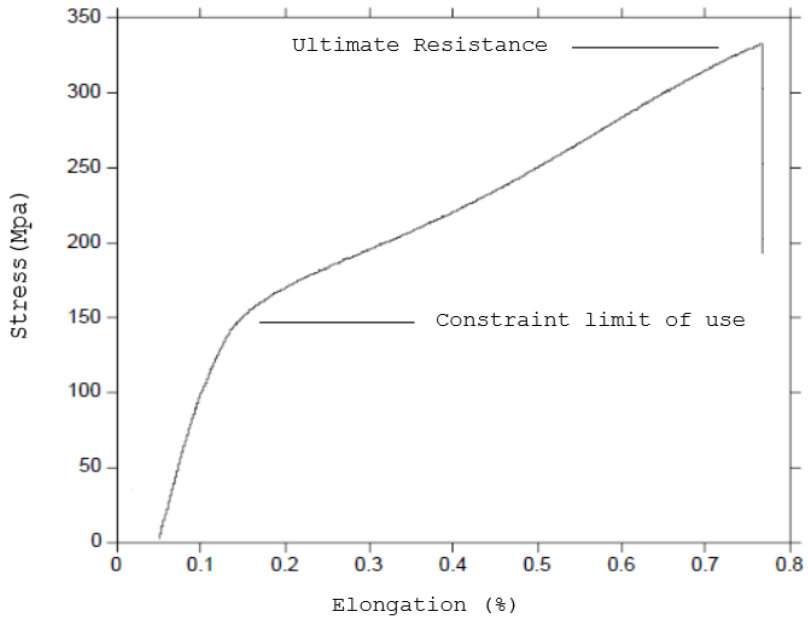

Figure 1. Model response curve of a composite to mechanical stress

The elongation at break of a material is the relative variation in the length it reaches when it is stretched to its maximum, until it mechanically breaks. In the "stress-strain" curve, elongation can be used to measure the appearance of micro-structures in the composite. The elongation that a composite can reach before the appearance of microcracks depends on the strength and adhesion capabilities of the resin. For brittle resins, like most polyesters, this point is reached well before the composite completely breaks. The wear resistance of the composite can be as low as 10% of the ultimate resistance of the composite (Raabe et al., 2022). In parallel with the composite's fatigue resistance problems, microcracks can also affect its longevity in the face of environmental conditions. The reinforcement or core of the resin can be degraded more quickly by humidity and/or chemical substances that insinuate into these breaches. Consequently, the overall resistance of the composite will decrease over time. A practical illustration can be found in the work of (Mulenga et al., 2021; Qasim et al., 2022).

2.3 Composite materials

Before specifying the two prominent families of polymers, it is interesting to recall the definition of a composite material. The term “composite material” designates a solid and heterogeneous material comprising several distinct components, the association of which gives the whole set of properties that none of the elements considered possesses separately.

Therefore, the production of a composite material requires the association of at least two components: the reinforcement and the matrix, which must be compatible with each other and hold together (Bera & Banerjee, 2023; Netsch et al., 2022). A liaison agent, called an interface, is necessary (see Figure 2). Fillers and additives can be added to the composite in the form of incomplete elements, powders or liquids, to modify a property of the material to which it is added (for example impact resistance, UV resistance, fire resistance).

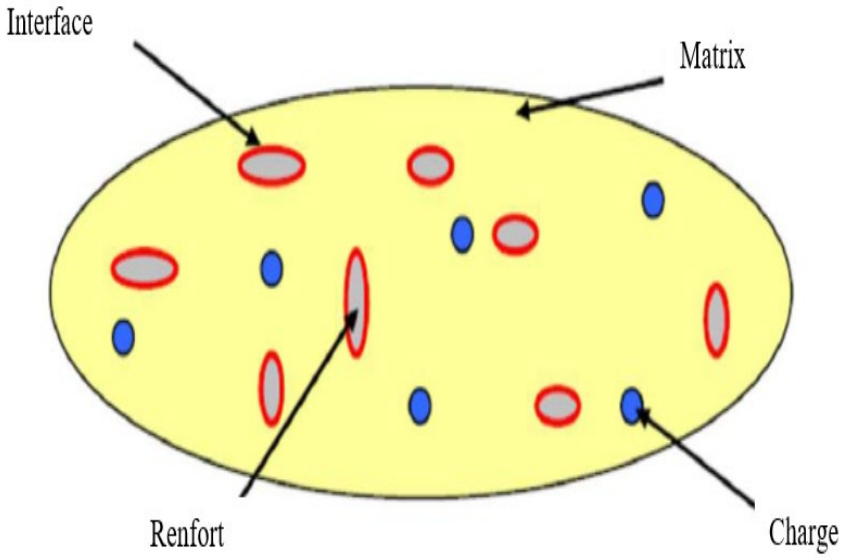

Figure 2. Schematic representation of a composite material.

2.3.1 Reinforcement

Composites are often referred to according to the type of reinforcement. Thus, there are particle composites, sandwich composites, dispersed phase composites or even fibrous composites (Khorasani et al., 2022; Nguyen et al., 2022).

2.3.2 The matrix

In a composite material, the term matrix designates the solid material surrounding the reinforcements to form a compact mass (Corinaldesi & Nardinocchi, 2016; Qasim et al., 2022). The primary role of the matrix is to keep the reinforcements compact and give the desired shape to the final product. It also protects the fibers against abrasion and an aggressive environment, controls the surface finish, and ensures the transfer of loads between the fibers. The solid

forming the matrix can be metallic, ceramic or polymer. Based on the nature of the matrix, composite materials are classified into three categories:

- Polymer or organic matrix composites (CMO);
- Metal matrix composites;
- Ceramic matrix composites.

For the composites that we are going to study (CMO) the mechanical properties of the matrix are generally very weak compared to those of the reinforcements (rarer case), and the general performance of the composite (matrix/reinforcement) is very dependent on the choice of the matrix (Y. Huang et al., 2021; Khorasani et al., 2022). Indeed, in the case where the deformation at breakage of the matrix is lower than that of the fibers, for an imposed displacement stress, the matrix does not allow it to fully benefit from the reinforcement of the fibers. On the other hand, for long-term durability (fatigue, creep), the role of the matrix then becomes very important.

These materials have good mechanical properties and low density. The resins most commonly used in composite materials are thermosetting and thermoplastic (Ghebrid et al., 2024). Thermosetting resins are polymers that, after thermal or physicochemical treatment (catalyst, hardener), are transformed into essentially infusible and insoluble products. infusible and insoluble. Their special feature is that they can only be shaped once. On the other hand, thermoplastic resins can be alternately softened by heating and hardened by cooling within a temperature range specific to the polymer in question. Moreover, these resins can be easily molded in a molten state. Elastomers are characterized by high elasticity and very low Young's modulus (Plawsky, 2020). Low-density organic resins are electrical and thermal insulators. They are resistant to oxidation and corrosion and can produce complex parts integrating a maximum number of functions (Selvin et al., 2004).

2.4 Interface of a composite material: concept of adhesion

Between the reinforcement and the matrix, there is a connecting zone called the interface (interphase), which forms spontaneously when two phases are brought into contact during the material's manufacture and remains between these two phases throughout the material's lifetime (S. Huang et al., 2021; Karger-Kocsis et al., 2015; Krupa et al., 2007; Periasamy et al., 2023; Sadeghi et al., 2024). The notion of adhesion is intimately tied to the nature of the surfaces involved (Alp & Kuleaşan, 2019; Campàs et al., 2024; Cohen-Tannoudji et al., 2008; Joshi et al., 2020; Le et al., 2013; Okwara et al., 2024; Zhou & Lu, 2009). These surfaces, with their unique properties, strongly condition the possibilities of chemical or mechanical bonds representing adhesion at the interface between two materials. This interface takes different forms and leads to the concept of interphase. The interphase, an area between the two adherents with a concentration gradient of the two components, is a crucial and intriguing concept in the study of adhesion. It is more or less comprehensive and efficient, and is created by interdiffusion between two plastics, by diffusion of an adhesive in wood or a porous material. Therefore, at the interface or in the interphase, the forces at the origin of the adhesion act. These forces are grouped into mechanisms that describe mechanical, diffusion, electronic and adsorption adhesion. It is the nature of the surface which allows or not the simultaneous presence of the different mechanisms (Baranov et al., 2023; Bemerw et al., 2021; Parveez et al., 2022; Xie et al., 2024; Xiong et al., 2018; Zou et al., 2024). Binding mechanisms involve complex theoretical considerations. The theory of chemical bonds proposes several types of more or less intermolecular solid bonds (van der Waals bond, hydrogen bond or acid-base bond (Della Volpe & Siboni, 2022; Fowkes, 1987; Giubertoni et al., 2020; Grabowski, 2006, 2020; Li et al., 2024; Nagy, 2014).

The morphology of the interphase plays, for its part, a dominant role in concerns mechanical adhesion. The adhesion theory also uses surface energy and wetting (Baldan, 2012; Cui & Liu, 2021; Geoghegan & Krausch, 2003; Vinod et al., 2024). The work of adhesion can describe the interaction between two composite constituents. The work of adhesion (Wad) corresponds to the sum of the surface energies of the two components minus the interfacial energy. This quantity represents the energy gain obtained by covering one component by the other and is given by the Young-Dupré relation. It depends on the contact angle θ established between the two constituents and the free energy γ_m called surface tension (Buscaglia & Ausas, 2011; Kinloch, 2012; Mukherjee & Banerjee, 2024; Nair et al., 2023; Prüss et al., 2013; Tan et al., 2022; Xiong et al., 2018). Regarding surface energy, there is the non-polar surface free energy resulting from London dispersive forces and the polar surface energy resulting from dipole-dipole, induced dipole and hydrogen bond type interactions. The role of wetting therefore appears essential for the adhesion of the interface which is the preferred location for the transfer of stresses between the matrix and the reinforcement (Biswas et al., 2024; Guan et al., 2024; Xiong et al., 2018). If we consider a neutral material like polypropylene which is only capable of dispersion interactions, the work of adhesion will only be determined by the dispersive surface energy of the two components. On the contrary, cellulosic fibers have a predominantly polar surface energy. The surface tensions of different woods oscillate between 40 and 54.3 mJ/m² (Lifshitz-van der Waals/acid-based approach and others (Shen et al., 1998). The weakly acidic character of wood in general (pH: 4.3-5.9). Through molecular agitation, the

temperature causes a reorientation of the macromolecules and a migration of the extractables towards the surface, the energy of which is modified according to (Rowell, 2021).

In addition to techniques borrowed from chemical engineering to characterize materials, there are possibilities for more or less direct mechanical characterization of adhesion. Tests such as perpendicular traction used for wood-based composite panels or the "single fiber pull-out test" are the most common (Bader & Ormarsson, 2023; Flores et al., 2023; Wang et al., 2020; Xiong et al., 2018). The "pull out test" makes it possible to determine the adhesion of a fiber caught in a matrix. However, Beckert and Lauke in 1997 highlight that it is not apparent that this test measures adhesion since non-linearity effects, such as friction or inelasticity of the matrix, risk interfere (Nechifor et al., 2022). However, the combination of microscopic imaging and the "pull out test" allows the observation of the shape of the fracture surface between two materials. Good adhesion excludes the presence of a break at the interface (Borri et al., 2013). The perpendicular tensile test is standardized for wood materials, and is considered a good indicator of the adhesion quality (Cook & Chiu, 1997). It is mainly the connections with the glue that ensure the cohesion of the material. In the following section, we will focus on the methods that will help us characterize fiber matrix adhesion. For the case of this paper, it will be the "pull out test".

3. Materials and methods

In this section, we first present the equipment that allowed us to carry out our work, particularly the tensile testing machine and the raw kenaf fibers. Subsequently, we present the different methods we used to characterize the fiber/matrix interface adhesion.

3.1 Materials

The equipment that allowed us to conduct our tests is the Instron tensile testing machine from the University of Technology (IUT) of the University of Ngaoundéré. The tests in question were carried out on Kenaf fibers. The characteristics of this machine are given in Table 2.

Table 2. Features of the traction machine

Model	Instron 1125
Force Capacity	100 Kn
Column Spacing	559 Mm
Crosshead Travel	914 Mm
Minimum Speed	0.05 Mm/Min
Footprint	1022 X 21 X 78 Mm
Others	Complete Computer

3.2 Methods

3.2.1 Preparing composite specimens

This study evaluated two types of composite matrix: an epoxy matrix and a tannin-based matrix. In both cases, kenaf fibers were used as reinforcement. For the epoxy/kenaf specimens, the kenaf fibers were first mercerized in a 5% wt. aqueous sodium hydroxide solution (NaOH) for 2h. After rinsing and drying, the fibers were mixed with epoxy resin at a reinforcement rate of 30% by weight. For tannin/kenaf composites, a similar alkaline fiber treatment protocol was applied, with a 5% wt NaOH solution. The treated fibers were then incorporated into the tannin matrix at a reinforcement rate of 30% by weight. In both cases, the fiber/matrix mixtures were poured into molds and cured at room temperature for 24 hours.

3.2.2 Pull-out test: calculation of IFSS (Interfacial Shear Strength)

The interfacial shear strength (IFSS) between the fiber and matrix is a critical parameter governing the stress transfer efficiency at a composite material's fiber/matrix interface. To evaluate the IFSS, single-fiber pull-out tests were conducted, as described in the literature (Yadav et al., 2021). The pull-out test method involves embedding an individual fiber in the matrix material over a precise and controlled embedded length (le). This is achieved by carefully positioning a single fiber within a mold or template and casting the matrix material around it (Figure 3). The embedded length is measured under an optical microscope before testing to ensure accuracy.

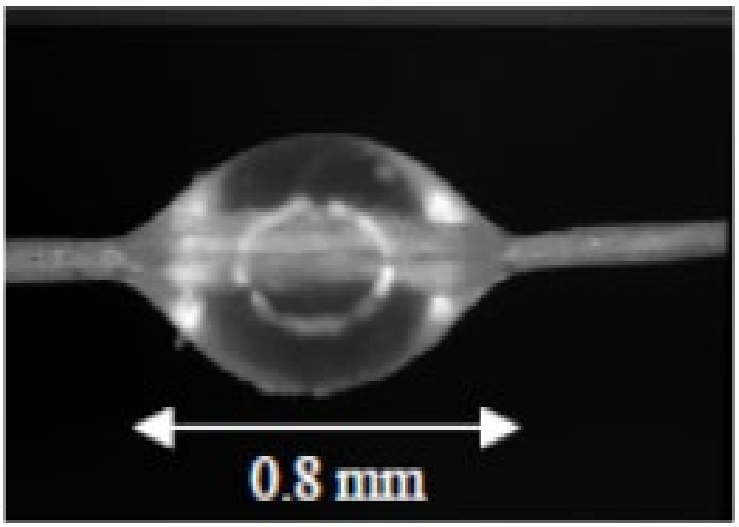

Figure 3. Droplet of polyester resin on a sisal fiber placed during composite manufacturing

The pull-out test setup consists of a loading frame with a force sensor and displacement measurement system (Figure 4).

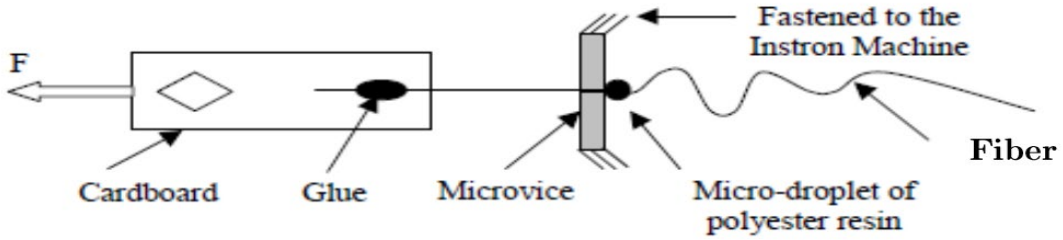

Figure 4. Schematic representation of template for use in sample preparation for mechanical pull out testing

The single embedded fiber is carefully gripped at one end, while the other end remains embedded in the matrix material. A tensile load is then progressively applied to the gripped end of the fiber, causing debonding at the fiber/matrix interface (Figures 5 and 6).

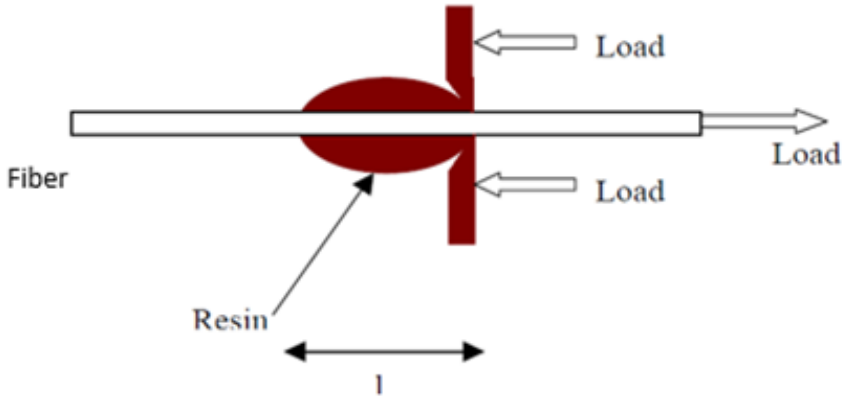

Figure 5. Loading the fiber in traction

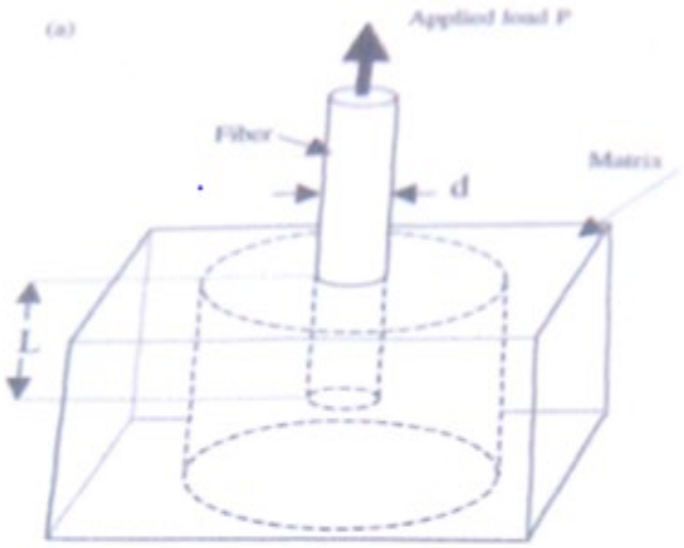

Figure 6. Traction of a single fiber

The pull-out force is recorded as a function of the displacement throughout the test until the fiber is completely extracted from the matrix. The interfacial shear strength (*IFSS*, τ) is calculated from the maximum pull-out force (F_{max}) and the embedded fiber area (A) using equation 1:

$$\tau = \frac{F_{max}}{\pi d L_e} \quad (1)$$

where *d* is the fiber diameter and le is the length of the embedded fiber . Multiple pull-out tests are performed for each composite system to obtain statistically meaningful data. The results are then analyzed to establish correlations between the experimental observations and to evaluate the significance of any differences in IFSS between the two composite systems under investigation.

4. Results and Discussion

In the literature review section, we discovered the composite material in its generality. In the material and methods section, we focused on the methods that can help characterize fiber matrix adhesion. For the case of our study, we compare the adhesion between the kenaf fiber - epoxy matrix and this same kenaf fiber but this time with a matrix of plant origin, tannin. In this last part, we present the essential results we reached at the end of our experiments for measuring the average shear stress using the “pull-out test”.

4.1 Implementation of the Pull-Out phenomenon

Many experiments have been performed to determine the release strength between polyester resin and kenaf fiber. During the experiment, maximum force is obtained. It corresponds to the force obtained when the fiber breaks or when the droplet of polyester resin slides on the fiber. But sometimes, a frictional force occurs before reaching maximum force. It appears when the micro vice is not placed just above the droplet of polyester resin. So ultimately, the force value in the formula to calculate the tensile strength or breakaway force is the difference between the maximum force and the initial friction force. During a pull-out test, two types of phenomena can occur: either the fiber breaks or the droplet of polyester resin slides along the kenaf fiber. These two cases will be analyzed separately. The first case will correspond to the fiber breaking, and the second case will correspond to the polyester resin sliding along the kenaf fiber. Indeed, one of the results obtained during the pull-out test is the tensile strength. Fiber breakage occurs before the pull-out phenomenon. In this case the force of detachment from the fiber/matrix interface cannot be known. Kenaf fibers do not have a constant surface area along the entire fiber. If sometimes, part of the fiber is fragile and when the machine (Instron) pulls the fiber, it breaks. Of course, this phenomenon cannot be predicted in advance unless the entire fiber is observed under a microscope. From Figure 7, we can clearly understand the Pull-Out phenomenon. We can see a curvature described because when the test piece is mounted on the machine, no force is yet acting on it. Just before breaking, the fiber is stretched. At the end of the test, either the fiber breaks or comes out of the resin matrix.

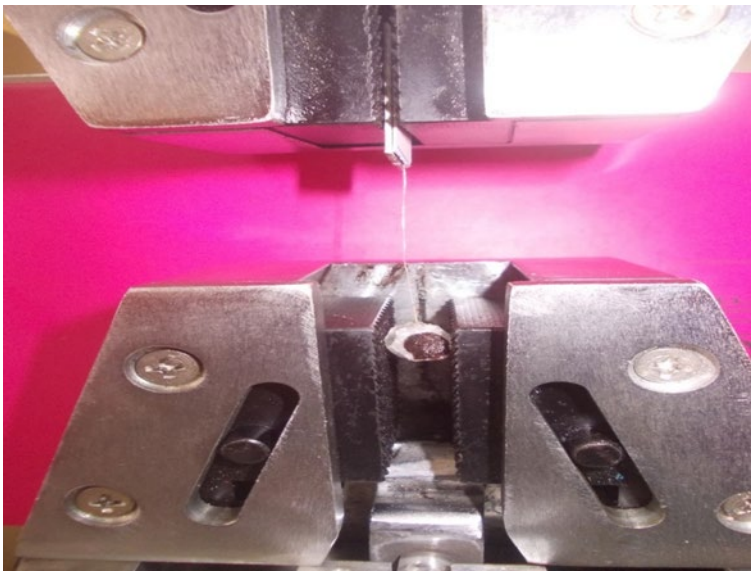

Figure 7. K11V test piece mounted on the testing machine (before traction)

In all possible outcomes (either the fiber breaks or it comes loose from the resin), we reach a force peak F responsible for the phenomenon.

4.2 Determination of the sizes and parameters of tannin/kenaf test pieces and Epoxy/kenaf test pieces and comparative study of the adhesion of composite materials made from these fibers

The Instron machine from the IUT of Ngaoundéré gives us Figure 8 illustrating the curves resulting from the rupture of the fiber and Table 3 where the numerical value of the force peak reached, the fiber section, elongation (as entered on the machine) and breaking stress.

Figure 8. Result of fiber breakage after pull out

In Figure 8, the sudden drop in force corresponds to the fiber breaking. This was the characteristic of almost all of our specimens with an epoxy matrix which demonstrated stronger adhesion than those with a vegetable resin matrix (tannin) where only a few cases of rupture were noted.

Table 3. Mechanical characteristics of the K9V specimen.

No.	Force Peak (N)	Tensile Stress (Mpa)	Elongation Peak (Mm)	Elongation Percentage Peak (%)	Gauge Length/ Load Span (Mm)	Area (Mm^2)
1	0.39	131.1	0.00	0.00	46.0	0.003

Table 3 can be easily obtained by clicking on the "Edit report" button in the software that accompanies the test machine. The force peak appears in Yellow (Figure 8). Let us now understand the origin of these graphs, the precision of which interests us as much as its interpretation. In fact, the traction machine for the pull-out test is coupled to a computer that automatically controls it. So, during a pull-out test, for example, the graph is drawn automatically depending on whether we want to see the evolution of the force over time. An extract of the data series generated by the computer during the pull-out test is given in Table 4. Initially, the fiber is not stretched and undergoes no tensile stress. Thus, we understand why the column of traction force F will remain zero.

Table 4. Evolution of parameters during the pull-out test of the K9E specimen

No.	Time (Min)	Elongation (Mm)	Force (N)	Stress (Mpa)	Strain (%)
1722	1 77	0.00	0.24	81 7	0.00
1723	1 77	0.00	0.24	81 7	0.00
1724	1 77	0.00	0.24	81 7	0.00
1725	1 77	0.00	0.24	81 7	0.00
1726	1 77	0.00	0.24	81 7	0.00
1727	1 77	0.00	0.24	81 7	0.00
1728	1 77	0.00	0.24	81 7	0.00
1729	1 77	0.00	0.24	81 7	0.00
1730	1 77	0.00	0.24	81 7	0.00
1731	1 78	0.00	0.24	81 7	0.00
1732	1 78	0.00	0.24	81 7	0.00
1733	1 78	0.00	0.24	81 7	0.00
1734	1 78	0.00	0.24	81 7	0.00
1735	1 78	0.00	0.24	81 7	0.00
1736	1 78	0.00	0.24	81 7	0.00
1737	1 78	0.00	0.24	81 7	0.00
1738	1 78	0.00	0.24	81 7	0.00
1739	1 78	0.00	0.24	81 7	0.00
1740	1 79	0.00	0.24	81 7	0.00
1741	1 79	0.00	0.24	81 7	0.00
1742	1 79	0.00	0.24	81 7	0.00
1743	1 79	0.00	0.24	81 7	0.00
1744	1 79	0.00	0.24	81 7	0.00
1745	1 79	0.00	0.24	81 7	0.00
1746	1 79	0.00	0.24	81 7	0.00
1747	1 79	0.00	0.24	81 7	0.00

We see that the force increases with time. The fiber is stressed in traction.

N.B: The spaces between the “time” and “stress” numbers represent commas! For example, we will read “1736” as “1.736 min”. This is software-specific writing! When the force reaches its extremum (maximum), the result is fiber rupture or progressive pullout.

Table 5. Progression from F to F_{max} over time

No.	Time (Min)	Elongation (Mm)	Force (N)	Stress (Mpa)	Strain (%)
8944	9 46	0.00	1.08	359 6	0.00
11274	12 2	0.00	0.64	212 7	0.00
11275	12 2	0.00	0.69	228 8	0.00
11276	12 2	0.00	0.64	212 5	0.00
11277	12 2	0.00	0.64	212 5	0.00
11278	12 2	0.00	0.64	212 5	0.00
11279	12 2	0.00	0.64	212 5	0.00
11280	12 2	0.00	0.64	212 5	0.00
11281	12 2	0.00	0.69	228 8	0.00
11282	12 2	0.00	0.69	228 8	0.00
11283	12 2	0.00	0.69	228 8	0.00
11284	12 2	0.00	0.69	228 8	0.00
11285	12 2	0.00	0.69	228 8	0.00
11286	12 2	0.00	0.69	228 8	0.00
11287	12 2	0.00	0.69	228 8	0.00
11288	12 2	0.00	0.64	212 7	0.00
11289	12 2	0.00	0.64	212 5	0.00
11290	12 2	0.00	0.64	212 5	0.00
11291	12 2	0.00	0.64	212 5	0.00
11292	12 2	0.00	0.64	212 5	0.00
11293	12 2	0.00	0.64	212 5	0.00
11294	12 2	0.00	0.64	212 5	0.00

If we compare the test results in parallel, the difference would be more obvious, and we could draw a conclusion. The average shear stress is obtained by relation 1. Consider the following summary tables (Table 6 and Table 7).

Table 6. Summary of quantities and parameters for tannin/kenaf specimens

No. Fibre	Diameter of Fibre (Mm)	Section of Fibre (Mm²)	Diameter of the Taste (Mm)	Press-In Length L (Mm)	Initial Friction Force (N)	Force Peak F (N)	Force (N)	Observation	Average Shear Stress N/Mm²	Maximum Stress (F/S) N/Mm²
K1V	0.07	0.004	3.2	3.0	0.0	0.43	0.43	Sliding	0.03	105.9
K2V	0.06	0.003	2.8	2.8	0.0	1.41	1.41	Sliding	0.08	468.1
K3V	0.04	0.001	3.0	2.5	0.0	1.65	1.65	Sliding	0.05	1459.3
K4V	0.07	0.004	3.5	4.0	0.0	0.42	0.42	Sliding	0.04	111.2
K5V	0.07	0.003	2.0	2.0	0.0	1.52	1.52	Sliding	0.06	504.0
K6V	0.07	0.004	2.5	2.5	0.0	1.44	1.44	Sliding	0.08	384.1
K7V	0.07	0.004	4.0	4.0	0.0	2.69	2.69	Breaking	0.25	642.4
K8V	0.07	0.004	3.6	4.0	0.0	1.53	1.53	Sliding	0.14	376.2
K9V	0.06	0.003	3.8	2.4	0.0	2.70	2.70	Breaking	0.12	954.5
K10V	0.07	0.004	3.0	2.0	0.0	1.53	1.53	Sliding	0.07	366.9
K11V	0.04	0.001	3.4	3.0	0.0	2.70	2.70	Breaking	0.10	2385.9
K12V	0.06	0.003	2.8	4.0	0.0	1.62	1.62	Breaking	0.12	613.1
K13V	0.07	0.004	2.5	3.0	0.0	0.45	0.45	Sliding	0.03	117.6
K14V	0.07	0.004	4.2	4.2	0.0	1.42	1.42	Sliding	0.13	380.5
K15V	0.04	0.001	3.4	2.7	0.0	1.70	1.70	Sliding	0.05	1424.8
			AVERAGE				1.55		0.09	686.3

Table 7. Summary of sizes and parameters for Epoxy/kenaf specimens

No. Fibre	Diameter Of Fibre (Mm)	Section Of Fibre (Mm²)	Diameter Of the Taste (Mm)	*Pres-in Length L (Mm)*	Initial Friction Force (N)	Force Peak F (N)	Force (N)	Observation	Average Shear Stress N/Mm²	Maximum Stress (F/S) N/Mm²
K1E	0.08	0.005	0.24	6.0	0.0	2.01	2.01	Breaking	0.32	362.9
K2E	0.08	0.005	312.0	5.4	0.0	1.31	1.31	Breaking	0.18	268.2
K3E	0.06	0.003	0.2	5.0	0.0	0.37	0.37	Breaking	0.04	136.6
K4E	0.06	0.004	0.3	5.6	0.0	1.08	1.08	Breaking	0.13	306.1
K5E	0.06	0.003	0.3	4.0	0.0	0.41	0.41	Breaking	0.03	128.5
K6E	0.07	0.005	0.2	3.1	0.0	1.32	1.32	Breaking	0.10	292.1
K7E	0.09	0.004	0.2	5.0	0.0	1.06	1.06	Breaking	0.11	291.1
K8E	0.07	0.004	0.3	6.0	0.0	1.09	1.09	Breaking	0.14	291.6
K9E	0.06	0.003	0.3	5.0	0.0	0.40	0.39	Breaking	0.04	134.6
K10E	0.06	0.003	0.3	6.0	0.0	1.03	1.03	Breaking	0.12	377.2
K11E	0.07	0.004	0.4	5.0	0.0	1.32	1.32	Breaking	0.14	374.3
K12E	0.05	0.002	0.5	4.0	0.0	1.05	1.05	Breaking	0.06	629.4
K13E	0.07	0.005	0.5	5.6	0.0	1.08	1.08	Breaking	0.15	238.1
K14E	0.06	0.003	0.3	7.0	0.0	3.14	3.14	Breaking	0.43	1074.6
K15E	0.04	0.001	0.3	5.4	0.0	1.37	1.37	Breaking	0.10	991.4
			AVERAGE				1.20		0.14	393.1

The average of the average shear stress of composite materials from the Epoxy matrix and kenaf fibers is 0.14 N/mm² (Table 7) while that of composite materials from the tannin-based vegetable matrix is 0.09 N/mm² (Table 6). This parameter makes it possible to characterize the fiber/matrix adhesion that we tested using the pull-out test. The average shear stress of the composite materials derived from the Epoxy matrix and kenaf fibers is significantly higher than that of the composite materials derived from the tannin-based vegetable matrix. This observation shows that the adhesion of composite materials from the Epoxy matrix and kenaf fibers is better than that of composite materials from the tannin-based vegetable matrix.

4.3 Validation of matrix fiber adhesion of tannin/plant fiber, tannin/synthetic fiber and epoxy composite materials, by a statistical test

To conclude immediately that the adhesion of composite materials from the Epoxy matrix and kenaf fibers is better than that of composite materials from the tannin-based vegetable matrix would be a quick task, because we must know This is reassured by an appropriate statistical test: the estimation of the difference between two means. Consider the population of composite materials derived from the Epoxy matrix and kenaf fibers and the population of composite materials derived from the tannin-based plant matrix. $\mu_1$and μ_2 are their respective means and σ_1^2 and σ_2^2 their respective variances as well. A point estimate of the difference between μ_1 and μ_2 is given by the statistic $\overline{X_1} - \overline{X_2}$. Therefore, to obtain a point estimate of the difference $\mu_1 - \mu_2$, we draw two independent random samples of size n_1 and n_2 from each of the two populations, then we calculate the difference $\overline{x_1} - \overline{x_2}$ of the observed means.

$n_1 = 15$; $n_2 = 15$; $S_1^2 = 0.0107$ (N/mm²) ²; $S_2^2 = 0.0031$ (N/mm²) ;$\overline{x_1} = 0.1390$N/mm²; $\overline{x_2} = 0.0903$N/mm².

We use relation 2 to construct a $(1-\alpha) \times 100\%$ confidence interval of the difference.

$$p\left[(\overline{X_1} - \overline{X_2}) - z_{\frac{\alpha}{2}}\sqrt{\frac{\sigma_1^2}{n_1} - \frac{\sigma_2^2}{n_2}} < \mu_1 - \mu_2 < (\overline{X_1} - \overline{X_2}) - z_{\frac{\alpha}{2}}\sqrt{\frac{\sigma_1^2}{n_1} - \frac{\sigma_2^2}{n_2}}\right] = 1 - \alpha \quad (2)$$

The confidence interval of $\mu_1 - \mu_2$ and the variances σ_1^2 and $\sigma_2^2$are known. A $(1-\alpha) \times 100\%$ confidence interval of the difference is given by relation 3.

$$(\overline{X_1} - \overline{X_2}) - z_{\frac{\alpha}{2}}\sqrt{\frac{\sigma_1^2}{n_1} - \frac{\sigma_2^2}{n_2}} < \mu_1 - \mu_2 < (\overline{X_1} - \overline{X_2}) - z_{\frac{\alpha}{2}}\sqrt{\frac{\sigma_1^2}{n_1} - \frac{\sigma_2^2}{n_2}} \quad (3)$$

Where $\overline{X_1}$ and $\overline{X_2}$are the means of two independent samples of sizes n_1 and n_2 drawn from two populations of known variances σ_1^2 and σ_2^2, respectively, and $z_{\frac{\alpha}{2}}$ is the value of the reduced centered normal variable Z leaving an $\frac{\alpha}{2}$ area on the right. The procedure we have just described for constructing a confidence interval is applicable when σ_1^2 and σ_2^2 are known or can be estimated from large samples. If, on the other hand, the sizes of these samples are small (<30) as in the case of this paper, we must still use the student's t distribution to find valid confidence intervals when the populations are approximately normal. For this we distinguish two cases: (1) the two variances are equal (2) the two variances are not equal. This is the second case in the paper framework because the two calculated variances are different.

We calculate the combined variance using relation 4.

$$S_p^2 = \frac{(n_1-1)S_1^2 + (n_2-1)S_2^2}{n_1+n_2-2} \quad (4)$$

Numerical application gives S_p^2=$105.10^{-3}(N/mm^2)^2$. Using α =0.05 we read $t_{0.025}$=2.048 for $\nu = n_1 + n_2 - 2 = 15 + 15 - 2 = 28$ degrees of freedom. So, the 95% confidence interval of $\mu_1 - \mu_2$ is given by relation 5.

$$(\overline{x_1} - \overline{x_2}) - t_{\frac{\alpha}{2}}S_p^2\sqrt{\frac{1}{n_1} + \frac{1}{n_2}} < \mu_1 - \mu_2 < (\overline{x_1} - \overline{x_2}) + t_{\frac{\alpha}{2}}S_p^2\sqrt{\frac{1}{n_1} + \frac{1}{n_2}} \quad (5)$$

Numerical application gives:

$$(0.1390 - 0.0903) - 2.048.105.10^{-3}\sqrt{\frac{1}{15} + \frac{1}{15}} < \mu_1 - \mu_2 < (0.1390 - 0.0903) + 2.048.105.10^{-3}\sqrt{\frac{1}{15} + \frac{1}{15}}$$

Which is simplified by: $0.04862 < \mu_1 - \mu_2 < 0.04870$.

Two conclusions emerge from this framework:

- We are 95% sure that the interval contains the difference between the average of the average shear stresses of the composite materials from the Epoxy matrix and kenaf fibers and that of the composite materials from the tannin-based vegetable matrix;
- The fact that the two limits are positive indicates that the matrix fiber adhesion of composite materials from the Epoxy matrix and kenaf fibers is greater than that of composite materials from the tannin-based vegetable matrix.

5. Discussion of Results

Pull-off tests revealed significant differences in interfacial shear strength between the three composite systems studied. For kenaf fibers treated with NaOH and coupled to an epoxy matrix, the average interfacial strength reached 0.1390 N/mm². However, when these same treated kenaf fibers are bonded to a tannin-based plant matrix, the interfacial strength increases significantly to 0.1240 N/mm². This value is significantly higher than that obtained with kenaf/plant matrix composites (0.0903 N/mm²), demonstrating much better fiber/matrix adhesion for the tannin system. These results highlight the influence of matrix type on interface properties within bio-based composite materials. While alkaline treatment of kenaf fibers promotes strong adhesion with the epoxy matrix, using a tannin-based matrix also seems to result in a very strong fiber/matrix interface. The polyphenolic groups in tannin play a key role in this improved adhesion, by creating effective physicochemical interactions with the plant fiber surface. These observations open up interesting prospects for developing bio composites with high mechanical performance, taking advantage of both the surface treatment of reinforcements and the judicious choice of bio sourced matrix. A better understanding of these interfacial adhesion mechanisms is crucial to optimize the final properties of these durable composite materials.

6. Conclusion

The main objective of this paper was to compare the fiber-matrix adhesion of epoxy/kenaf and tannin/kenaf composite materials. We began by presenting the methods used to characterize adhesion. These include methods for determining the average shear rate using pull-out tests. However, an analysis of some recent works on composite materials showed that these methods are rarely applied for various scientific purposes. Integrating plant fibers into composite materials is becoming common practice, encouraged by strong demand for biosourced and healthy products. If the optimization of 100% natural composites is desired, it is obvious that we must take maximum advantage of the combination of constituents and their performances. The quality of this association depends on the interface. This is why it seemed reasonable to us to describe the mechanisms at play in membership. From a mechanical point of view, it is important to remember that the interphase is the privileged place for the transfer of stresses between the cellulosic fiber (the reinforcement) and the synthetic polymer, commonly called the matrix. The role of adhesion is crucial because the interactions between the constituents' surfaces mainly govern the composites' physico-mechanical behaviour. The transfer of stress from the matrix to the reinforcement depends on the quality of the interface. The "pull out test", a real tool for characterizing fiber matrix adhesion, revealed fairly good adhesion of the kenaf fibers with the epoxy matrix compared to the kenaf fibers coupled with the base resin. of tannin. However, the quality of the adhesion of the latter two is not bad and we can now turn to 100% organic composite materials by drawing on the results of this work. Furthermore, resuming this work in a laboratory equipped with a goniometer would allow results to be obtained closer to reality and would facilitate verification of the correlation with those calculated numerically.

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