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# Predicting the Relaxation Modulus for the Study of the Delayed Behaviour of Kenaf Fibres in Stress Relaxation

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**Abstract**
*Plant fibres (PFs) are preferred reinforcements of bio-composites. Knowledge of their lifespan requires a study of their viscoelastic behaviour. In this paper, a stress relaxation analysis of kenaf fibres was performed at a constant rate of deformation at room temperature. A method for extracting the relaxation modulus in the deferred zone was proposed. This method was compared, using simulation, with the Zapas-Phillips method and experimental data via three predictive models: the stretched exponential function or KWW, the inverse power law of Nutting and the prony series. The results indicate that the relaxation modulus obtained by the method proposed is in good agreement with the experimental modulus. In addition, the estimated error is of the same order of magnitude as in the case of the Zapas-Phillips method. The parameters estimated from the KWW function (β = 0.4) and prony series model showed an important contribution in the study of the delayed response of kenaf fibres. These results can have a significant impact on the use of kenaf fibres in midterm and long-term loading applications.*

**Key words:** *kenaf fibres, relaxation test, relaxation modulus, predictive model, delayed behavior.*



## Introduction

Natural fibres in general and PFs in particular are increasingly used in several fields of engineering applications because of their interesting properties. These fibres are biodegradable and environmentally friendly. For an optimal use of PFs in structures, knowledge of their behaviour in service is essential. Many applications require that fibres be permanently loaded in the long run [1]. However, it is difficult or impossible to carry out long-term tests [2]. Thus, limited laboratory tests such as the creep and relaxation are used to predict their behaviour in service by extrapolation. A significant number of works have already been conducted to characterise and model the elastic behaviour of PFs [3-7]. Yet, only few studies have been devoted to the viscoelastic behaviour of PFs.

The linear viscoelasticity of a material in stress relaxation depends largely on the relaxation modulus. The modulus of relaxation in stress can be determined by the progressive application of deformation [8] or by instantaneous deformation [9]. In addition, approaches for the characterisation of material stress relaxation behaviours are reported [10-15]. The stress relaxation behaviour of reinforced composites of plant fibres are also reported [16, 17]. These authors concluded that the introduction of short fibres for the reinforcement of composites increases the rate of stress relaxation.

Different methods of extracting the modulus from relaxation experiments have also been proposed in the literature. Kelchner and Aklonis [18], Zapas and Phillips [8, 19], Smith, and Lee and Knauss [19-21] proposed more or less accurate and simple methods based on the simulated response of virtual polymeric materials, rather than real experimental data.

Modelling the viscoelastic properties of PFs is necessary to accurately predict the long-term performance of PF composites. Predictive models are generally used to model the delayed behaviour of natural fibres in creep and relaxation experiments. However, in the literature, several studies have focussed on modelling the viscoelastic behaviour of materials. Cisse [22] highlighted the viscoelastic nature of flax fibre after a creep test. He used analogic rheological models to predict the viscoelastic behaviour of flax fibre. Bourmaud et *al*. [23] studied the viscoelastic behaviour of flax fibre nanoindentation footprint recovery. The authors used Maxwell's two branch and three branch models to describe the viscoelastic behaviour of flax fibres. In addition, they also used the Kolrausch-Williams-Watts (KWW) function to replicate the viscoelastic behaviour of flax fibre. Sasaki [24] used mechanical models based on the function of KWW to predict viscoelastic behaviour. He also presented several other methods to study the viscoelastic properties of biological materials. Saiful et *al*. [25] proposed linear viscoelastic models based on the prony series. Chen [26] proposed a method of determining the coefficients of viscoelastic modulus materials using prony series representation from the rate dependent data. Goh et *al*. [27] proposed a method based on the finite time increment formulation of the convolution integral, and is applicable for materials which exhibit separable strain and time variables. The authors applied this method to determine the constitutive constants of a non-linear viscoelastic material. The selected time-dependent function is based on the prony series. Recently, Xu and Engquist [28] proposed a mathematical model based on the sigmoidal function to predict and model the linear viscoelasticity relaxation modulus of biological materials. The stress relaxation behaviour of nanocomposites was reported in [29]. The KWW and Maxwell-Weichert models were used to predict the relaxation phenomenon observed. However, the authors conclude that the models fit the experimental points well.

However, these authors used various classical functions for predicting the parameters and adjusting the experimental curve. In addition, knowledge of the mechanical properties of the constituents taken separately makes it possible to optimise the bio-composite.

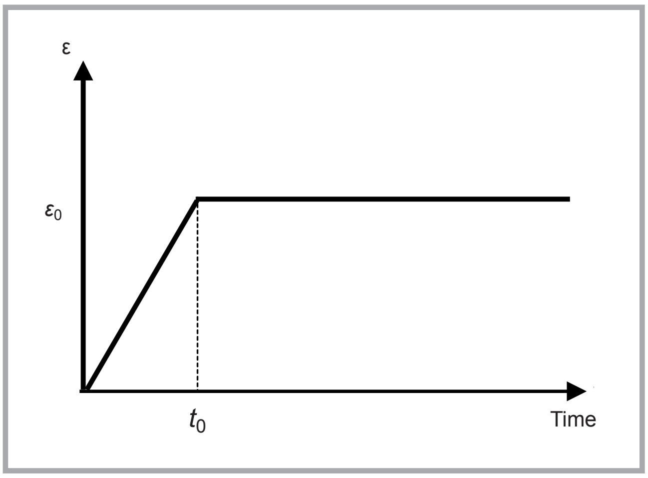


***Figure 1.*** *Ramp of strain.*

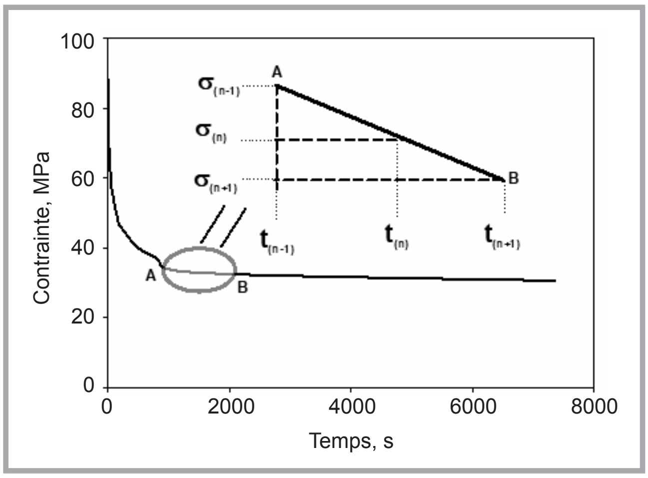


***Figure 2.*** *Illustration of the discretisation method's relaxation curve.*

In this work, an extraction method for the relaxation modulus of stress that bypasses the integral equation solution will be introduced. The method applied is based on the discretisation of the experimental curve in the delayed field. The current paper is directed towards replacing the classical functions in the integral equation to compare the experimental data with the values calculated by the Zappas-Phillips and discretisation methods for the experimental curve.

## Material and methods

**Stress relaxation test**

The relaxation test consists of applying a quasi-instantaneous constant deformation to the fibre, and monitoring the response (stress) over time. Twenty kenaf fibres with a $60 \pm 1.8$ µm mean diameter were prepared and tested on a LIYI 1066A type universal tensile testing machine. The LIYI machine is equipped with a 50 N load sensor of 0.05% accuracy and controlled by TM2101 software. The tests were conducted at a room temperature of $24 \pm 1$ °C and relative humidity of $50 \pm 1.5$%.

An almost instantaneous strain of the order of 0.25% was applied with a crosshead speed of 1 mm/min$^{-1}$, followed by a hold time of 7200 s. Fibre preparation was carried out according to the ASTM D3822 standard.

**Linear viscoelasticity analysis method**

In theory, when the strain is instantaneously applied, the relaxation modulus can be determined as:

$$E(t) = \frac{\sigma(t)}{\varepsilon_0} \qquad (1)$$

Where, $\sigma(t)$ is the time dependent stress response, and $\varepsilon_0$ is the strain amplitude. However, in practice the application of an instantaneous strain is not possible. In a stress relaxation test, with time $0 \leq t \leq t_0$, the strain is applied at a constant rate. Where, $t_0$ is the time for the stress to reach its maximum value. Subsequently, a constant deformation level is maintained at time $t \geq t_0$ (***Figure 1***). Generally, a discrepancy is observed between the responses to a constant strain and the ideal response to an instantaneous strain.

Therefore, ***Equation (1)*** is not applicable for calculation of the relaxation modulus. According to [8], the integral Boltzmann equation may be appropriate. In the stress relaxation of a linear viscoelastic material under uniaxial loading, the Boltzmann ***Equation (2)*** can be given as [9, 30]:

$$\sigma(t) = \int_0^t \mathrm{E}(t-\tau)\frac{d\varepsilon(\tau)}{dt}d\tau \qquad (2)$$

Where, σ is the stress, t the time, E the relaxation modulus, and $\frac{d\varepsilon(\tau)}{dt} = \dot{\varepsilon}$ the strain rate.

The strain in the relaxation experiment (***Figure 1)*** makes it possible to define the following relation [19]:

$$\varepsilon(t) = \begin{cases} \dot{\varepsilon}_0 t & t \prec t_0 \\ \varepsilon_0 & t \geq t_0 \end{cases} \qquad (3)$$

Combining ***Equations (3)*** and ***(2)***, gives:

$$\sigma(t) = \begin{cases} \dot{\varepsilon}_0 \int_0^t E(t-\tau)d\tau & t \prec t_0 \\ \dot{\varepsilon}_0 \int_0^{t_0} E(t-\tau)d\tau & t \geq t_0 \end{cases} \qquad (4)$$

**Methods of extraction of the relaxation modulus**

***Zapas and Phillips method***

Zapas-Phillips [8] and recently Sorvari-Malinen [19] proposed a method for determining the relaxation modulus of viscoelastic materials. The method of Zapas and Phillips was to integrate ***Equation (4)*** using the midpoint rule for $t \geq t_0$. Thus, the relaxation modulus is estimated following ***Equation (5)*** below:

$$E(t - t_0/2) = \frac{\sigma(t)}{\varepsilon_0} \quad t \geq t_0 \qquad (5)$$

Or

$$E(t) = \frac{\sigma(t + t/2)}{\varepsilon_0} \quad t \geq t_0/2 \qquad (6)$$

***Sorvari and Malinen method***

Sorvari and Malinen [19] derived ***Equation (4)*** as a function of time for $t \geq t_0$ and finally integrated it using the two-point trapezoidal rule to estimate the relaxation modulus using ***Equation (7)*** or ***Equation (8)*** below:

$$E(t - t_0) = \frac{\sigma(t)}{\varepsilon_0} - \frac{\dot{\sigma}(t)}{2\dot{\varepsilon}_0} \quad t \geq t_0 \qquad (7)$$

Or

$$E(t) = \frac{\sigma(t + t_0)}{\varepsilon_0} - \frac{\dot{\sigma}(t + t_0)}{2\dot{\varepsilon}_0} \quad t \geq 0 \qquad (8)$$

Where $\dot{\varepsilon}_0 = \frac{\varepsilon_0}{t_0}$

For the stress rate, the following formula has been defined:

$$\dot{\sigma}(t) = \frac{\sigma(t+h) - \sigma(t-h)}{2h} \qquad (9)$$

Where, $h$ is the length of the time step, taking arbitrary small values.

***Discretisation method***

An infinite curve can be considered an infinite succession of segments. Accordingly, the curve of ***Figure 2***, obtained during the stress relaxation test, is discretised here using a simple method that bypasses the integral equation used in viscoelasticity theory. Using a square triangle obtained from a segment as illustrated in ***Figure 2***, we can obtain the relationship between the stress $\sigma(t)$ and the corresponding times $t_n$. as below:

$$\frac{\sigma_{(n-1)}(t_{n-1})-\sigma_{n+1}(t_{n+1})}{t_{n+1}-t_{n-1}}= = \frac{\sigma_{(n-1)}(t_{n-1})-\sigma_{n}(t_{n})}{t_{n}-t_{n-1}} \quad (10)$$

$$t_n = t_{n-1} + \frac{t_{n+1}-t_{n-1}}{2} = \frac{t_{n-1}+t_{n+1}}{2} \quad (11)$$

Combining ***Equations (10)*** and ***(11)*** gives:

$$\sigma_n(t_n) = \frac{\sigma_{(n-1)}(t_{n-1})+\sigma_{n+1}(t_{n+1})}{2}, \quad t_n \in \left[t_{n-1}\ t_{n+1}\right] \quad (12)$$

Where, the abscissa $t_n$ is supposed to be the midpoint of the time interval of ***Equation (12)***. Thus, the relaxation modulus at time $t_n$ is given by:

$$E_n(t_n) = \frac{\sigma_{(n-1)}(t_{n-1})+\sigma_{n+1}(t_{n+1})}{2\varepsilon_0}, \quad t_n > t_0 \quad (13)$$

$n \in [1, k]$,

Where, $k$ is the total number of experimental data obtained.

The discrepancy between the experiment and numerical calculations is given by [19]:

$$erreur = \left\| \frac{E_{\exp}-E_{num}}{E_{\exp}} \right\| \quad (14)$$

Where, $E_{exp}$ is the experimental relaxation modulus, and $E_{num}$ is the relaxation modulus computed using ***Equation (13)*** of the discretisation method.

**Viscoelasticity theory modelling**

To describe the delayed behaviour of kenaf fibre in stress relaxation testing from ***Equation (4)***, ***Equation (2)*** is rewritten as:

$$\sigma(t) = \dot{\varepsilon}_0 \int_0^t E(t-\tau)d\tau = \dot{\varepsilon}_0 \int_0^t E(x)dx \quad (15)$$

However, three classical models are generally used to represent the relaxation function: the prony series [28-29], the power law [9, 33], and the stretched exponential law [8, 34, 35].

$$E(t) = E_e + \sum_{i=1}^{n} E_i \exp^{-t/\tau_i} \quad (16)$$

$E_e$ is an equilibrium modulus, $E_i$ the elastic component, and $\tau_i$ the associated relaxation times.

The stretched exponential function, or the Kohlrausch Williams Watts (KWW) [8, 35] ***Equation (17)***, to represent the relaxation function is defined as:

$$E(t) = E_0 \exp(-(t/\tau)^{\beta}), \quad 0 \prec \beta \prec 1; \tau \succ 0 \quad (17)$$

Or

$$E(t) = E_0 \exp(-(t/\tau)^{\beta}) + E_e \quad (18)$$

$E_0$ and $E_e$ are the instantaneous and equilibrium modulus, respectively. $\beta$ is a characteristic coefficient that depends on the viscoelastic behaviour of different materials. $\beta = 0$ describes the elastic behaviour and $\beta = 1$ the viscous behaviour.

The empirical power law was proposed by Nutting [33] to adjust experimental creep data. In addition to the inverse relationship between the creep modulus and relaxation modulus, this law can be put in the following form [33]:

$$E(t) = K_0.t^{-m} \quad (19)$$

Where $K_0$ and m are parameters of the model m = 0, and would represent the elastic behaviour of the material, and m = 1 would correspond to the viscous behaviour. The behaviour of viscoelastic material is between these two values. The power law has the advantage of being simple to use, with only two parameters materials ($K_0$ and $m$).

Substituting ***Equations (16)***, ***(18)*** or ***(19)*** in ***Equation (15)***, after integration and derivation of $\frac{d\sigma(t)}{d\varepsilon(t)} = \frac{d\sigma(t)}{dt}\frac{dt}{d\varepsilon(t)} = E(t)$ [36] and rearranging it, we obtain:

$$E(t)_p = \frac{1}{\dot{\varepsilon}_0}\left[E_e + \sum_{i=1}^{n} E_i \exp^{-t/\tau_i}\right] \quad (20)$$

Or

$$E(t)_p = \frac{t_0}{\varepsilon_0}\left[E_e + \sum_{i=1}^{n} E_i \exp(-t/\tau_i)\right] \quad (21)$$

$$E(t)_{KWW} = \frac{1}{\dot{\varepsilon}_0}\left[E_e + E_0 \exp^{-\left(\frac{t}{\tau}\right)^{\beta}}\right] \quad (22)$$

Or

$$E(t)_{KWW} = \frac{t_0}{\varepsilon_0}\left[E_e + E_0 \exp^{-\left(t/\tau\right)^{\beta}}\right] \quad (23)$$

$$E(t)_{lp} = \frac{K_0}{\dot{\varepsilon}_0} t^{-m} \quad (24)$$

Or

$$E(t)_{lp} = \frac{t_0 K_0 t^{-m}}{\varepsilon_0} \quad (25)$$

This modelling approach allows to predict the material parameters more accurately. The various parameters of the models ***Equations (20)-(25)*** are estimated by fitting the experimental data using the "nlinfit" function in matlab, based on the Levenberg-Marquardt optimisation algorithm.

## Results and discussion

**Stress relaxation test of kenaf fibres**

***Figure 3*** below shows the stress relaxation test result of kenaf fibre. The strain applied (0.25%) is chosen in the elastic domain, in the area where the tensile curve of the kenaf fibre exhibits non-linearity [37]. This value of the strain is selected for better appreciation of the relaxation phenomenon. Results show a significant decrease in stress under constant strain over time, indicating the viscoelastic behaviour of the fibre.

The stress time curve of ***Figure 3*** allowed to identify the value of instantaneous stress for the application of the strain rate. Under constant strain, the evolving stress observed over time is termed deferred (viscoelastic solution). In this so-called deferred range, two zones can be distinguished: The first zone of relaxation is characterised by a rapid descent of the stress until its equilibrium state, followed by the second linear zone, characterised by the stability of the stress over time. After 7200 s, corresponding to the relaxation holding time, a decrease in stress is observed in the interval 53.34 -146.98 MPa.

**Stress relaxation behaviour of kenaf fibres**

To study the stress relaxation behaviour of the fibre, the instantaneous stress is subtracted from the deferred response. Only data obtained during the period of constant strain are used to determine the properties of the material. The results obtained are shown in ***Figure 4*** in terms of normalised deferred stress $\frac{\sigma(t)}{\sigma_0}$ versus the logarithm of time. Where, $\sigma_0$ is

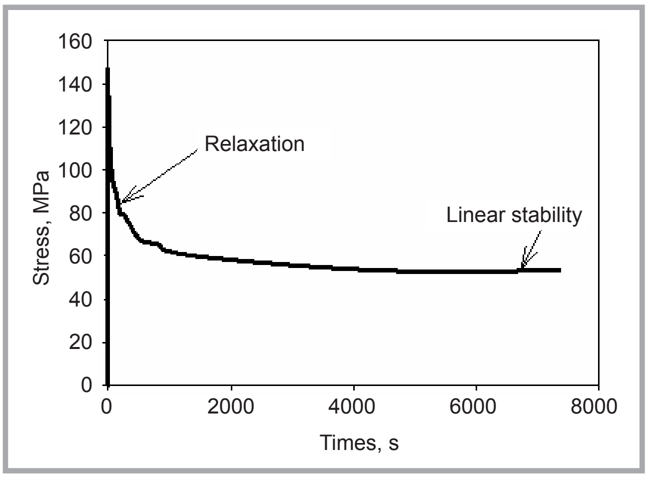


*Figure 3. Illustration of the relaxation stress curve versus time of kenaf fibre.*

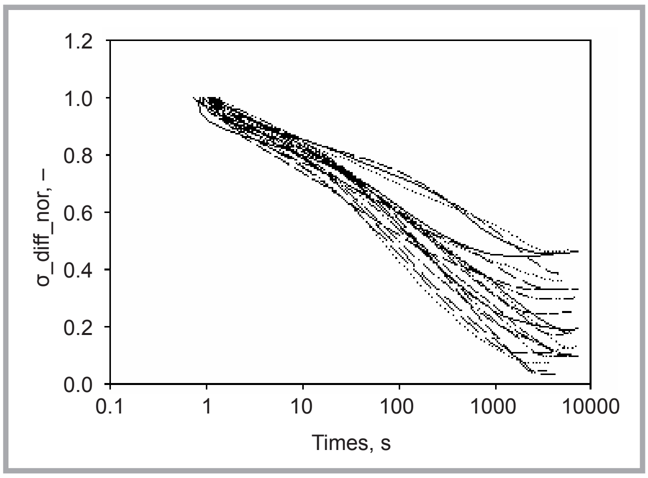


*Figure 4. Normalised deferred stress curves as a function of the logarithm of time.*

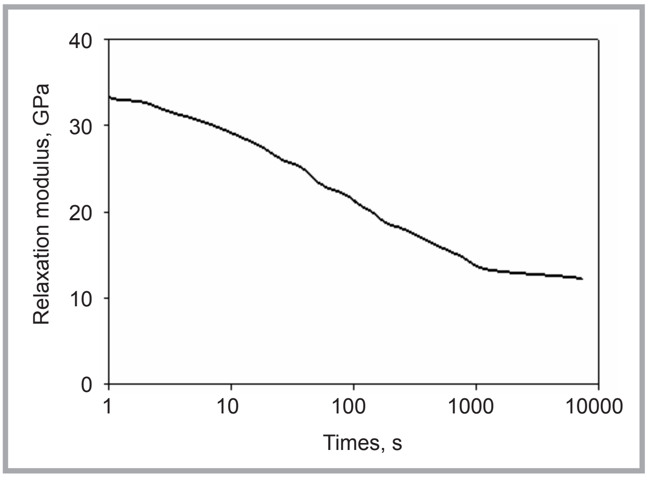


*Figure 5. Master curve of kenaf fibres of the response of the relaxation modulus as a function of the logarithm of time.*

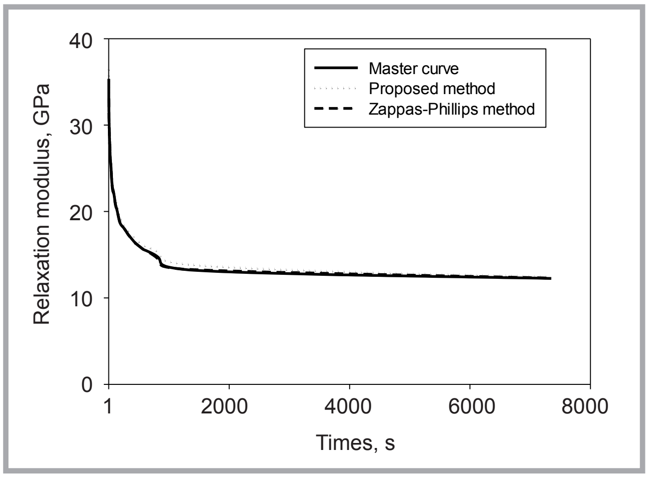


*Figure 6. Comparison between the experimental master curve and calculated data.*

the instantaneous stress, and σ($t$) is the deferred stress (during the relaxation). The normalised deferred stress relaxation curves plotted against the logarithm of time all have the same shape.

Given the similarity of the different curves, computation of the average is performed on the curves resulting from the stress relaxation test of kenaf fibre. This allows to build a relaxation "master curve" at room temperature, shown in ***Figure 5***.

Plant fibres have polymeric constituents (cellulose, hemicellulose and lignin). The curve of ***Figure 5*** presents a plateau at short times corresponding to the instantaneous modulus $E_0$ = 35.37 GPa. This plateau would correspond to the molecular mobility of the cellulose chains. Then it decreases exponentially to reach an equilibrium, as defined by the relaxation modulus $E_r$ = 12.255 GPa, which would correspond to the total equilibrium of cellulose molecules. The part that decreases exponentially could be attributed to the branching of long chains of cellulose molecules. In addition, the master curve of the relaxation modulus makes it possible to obtain an equilibrium modulus greater than zero. This would show the influence of pure elastic behaviour on kenaf fibre behaviour.

## Comparison between the master curve and calculated relaxation modulus

The analytical results obtained are compared with the experimental relaxation master curve. This is plotted in ***Figure 6***. A good correlation is observed between the experimental master curve of the relaxation modulus and curves resulting from the extraction methods of the relaxation modulus. The results of the error calculation indicate that the mean relative error affecting the discretisation method with respect to the experimental points is around 2.39%, while that of the Zapas-Phillips method is 0.54%.

## Modelling the delayed behaviour of kenaf fibre

***Figures 7, 9*** and ***11*** illustrate the relaxation modulus curves as a function of the logarithm of time. These curves show a comparison between experimental data of the relaxation modulus and the values of the relaxation modulus extracted by the Zapas-Phillips method and the method proposed herein. The Zapas-Phillips method is used here because of the ease of implementation and the quality of accuracy. Three predictive models (the KWW function, Prony series and the Nutting inverse power law) are used to adjust the master curve in this work. Simulations were performed under the same experimental conditions with a final strain of the order of 0.25%, for a time during which the stress reaches its maximum value $t_0$ = 0.9 s, using a crosshead

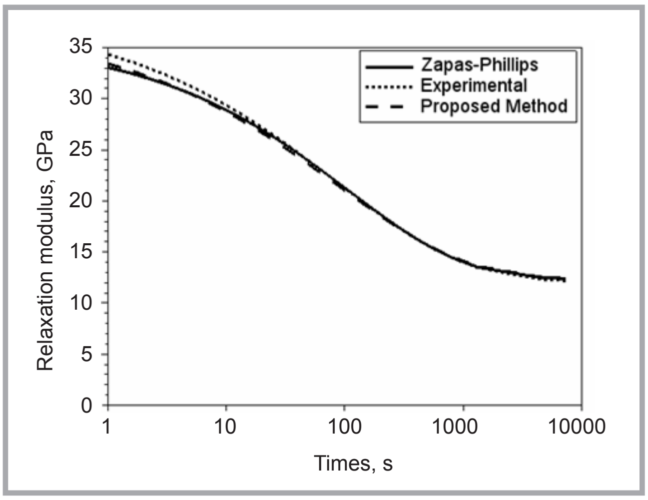


*Figure 7.* Simulation between the experimental data, Zapas-Phillips method and proposed method using the KWW.

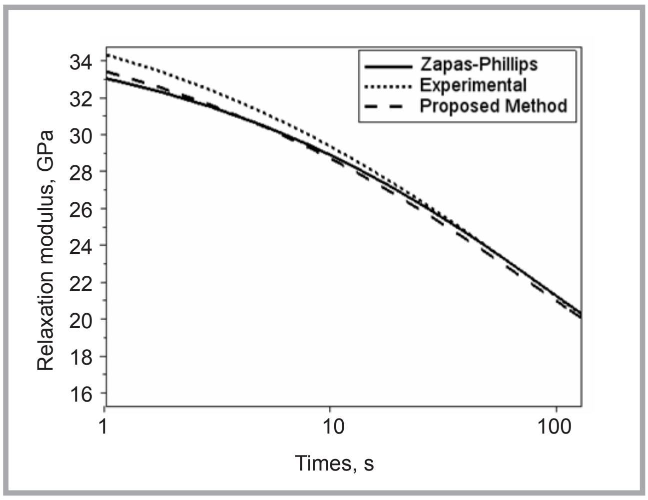


*Figure 8.* Simulation between the experimental data, Zapas-Phillips method and proposed method using the KWW at t ≤ 100 s.

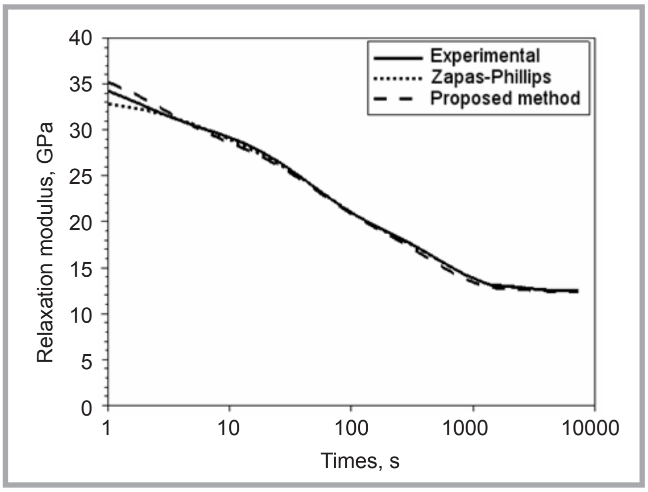


*Figure 9.* Comparison of proposed method with experimental data and Zapas-Phillips method using the prony series.

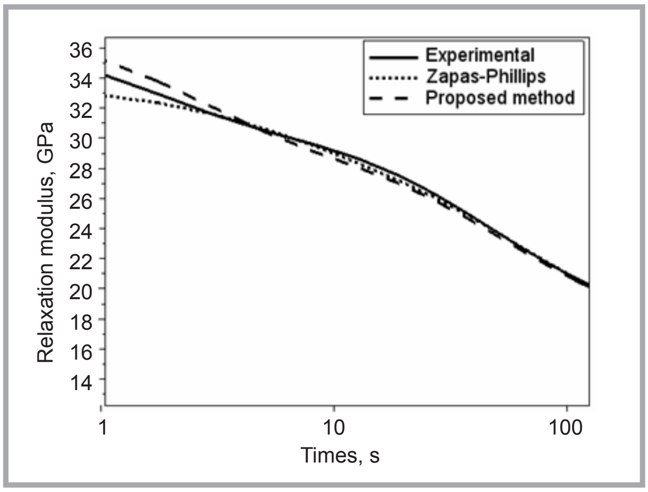


*Figure 10.* Comparison of proposed method with experimental data and Zapas-Phillips method using the prony series at t ≤100 s.

speed of 1 mm.min$^{-1}$. In line with the different curves, predictive models allow a better smoothing of the master curve. ***Tables 1, 2*** and ***3*** summarise the values of estimated parameters of the predictive models. These are yielded from the non-linear regression based on the Levenberg-Marquardt algorithm for different values of the relaxation modulus.

The average values of $\beta$ = 0.40 and 0.44 respectively obtained by the method proposed and by the Zapas-Phillips method are close to that obtained by experiment. These $\beta$ values are significantly higher than those of dental polymers ($\beta$ = 0.33) at 37 °C [33]. In addition, these high $\beta$ values are noticeable, and the average relaxation times are high ($\tau$ = 101.7, 107.07 and 99.8 s), indicating a significant contribution of viscosity. Although such a model is accurate to within 2.73%, it

***Table 1.*** *Estimated KWW function parameters.*

| KWW parameters | $E_0$ | $\tau$ | $\beta$ | $E_e$ |
|---|---|---|---|---|
| Experimental | 26.32 | 101.7 | 0.4 | 12.12 |
| Zapas-Phillips | 23.51 | 107.078 | 0.44 | 12.35 |
| Proposed method | 24.94 | 99.8 | 0.4 | 12.32 |

***Table 2.*** *Estimated model parameters of the Prony Series.*

| Prony Series Parameter s (n = 3) | $E_1$ | $E_2$ | $E_3$ | $E_e$ | $\tau_1$ | $\tau_2$ | $\tau_3$ |
|---|---|---|---|---|---|---|---|
| Experimental | 5.69 | 9.59 | 9.36 | 12.49 | 1.606 | 40.29 | 507.8 |
| Zapas-Phillips | 3.297 | 8.723 | 9.06 | 12.52 | 5.31 | 46.87 | 510.26 |
| Proposed method | 7.33 | 8.36 | 9.76 | 12.421 | 2.47 | 40.16 | 425.63 |

***Table 3.*** *Estimated model parameters of the Nutting Inverse Power Law.*

| | $K_0$ | n |
|---|---|---|
| Experimental | 36.96 | 0.124 |
| Zapas-Phillips | 35.23 | 0.118 |
| Proposed method | 34.87 | 0.116 |

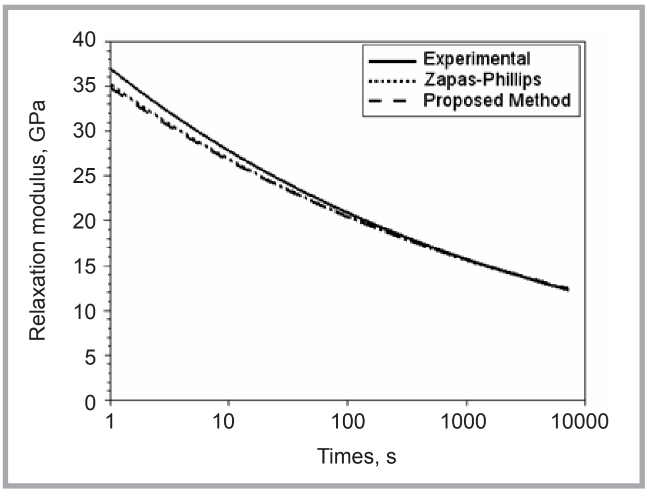


***Figure 11.*** *Comparison of proposed method with experimental data and Zapas-Phillips method using the power law.*

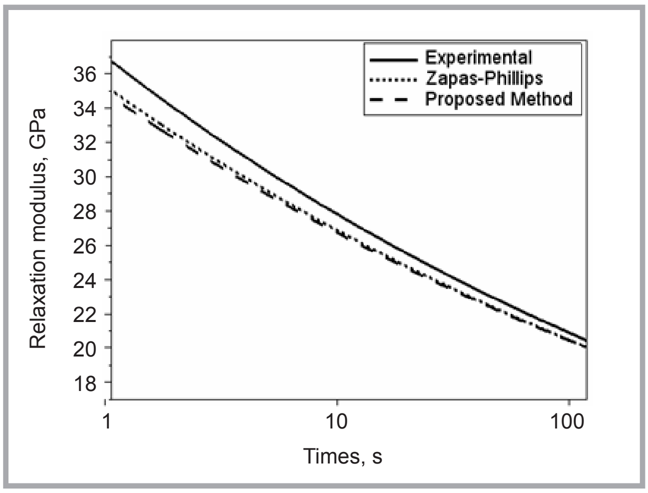


***Figure 12.*** *Comparison of proposed method with experimental data and Zapas-Phillips method using the power law at t ≤ 100 s.*

does not provide accurate predictions at short times.

However, the Nutting inverse power law applied to the different methods of extraction of the modulus presents a quite low exponent value of $m$ = 0.12. This value, close to that of an elastic solid ($m$ = 0), would indicate a less important contribution of the viscoelasticity of kenaf fibre to stress relaxation. The values of coefficient $K_0$ are of the same order of magnitude as the value of the short-term modulus. Although this law allows to accurately represent the master curve, it remains insensitive to the response during strain under stress. In addition, it is obvious that the equation is useful only at times greater than 100 s.

However, the model based on the prony series (***Figure 9)*** shows the fitting between experimental data and the different methods. For all three branches, the model shows increasing relaxation times (***Table 2***). This also implies the evolution of the viscosity modulus, as defined by $\eta_i = \tau_i \times E_i$, from the first branch to the third. This would also justify a significant part of the viscoelastic behaviour of kenaf fibre.

Nevertheless, for all the curves obtained by the predictive models, the factor of ten rule holds quite well.

As suggested by Flory and McKenna [8], errors seem to be negligible for times t ≥ 100 s. In addition, simulations, as plotted in ***Figures 8, 10*** and ***12,*** allow to visualise not only the difference between experimental data and the method proposed for t ≤ 100 s, but also a good agreement at times t ≥ 100 s. The results show a good correlation of the method proposed with long-term experimental data. While at short times, a slight dispersion is observed.

## Conclusions

In this work, the delayed behaviour of kenaf fibres was investigated. To this end, stress relaxation experiments were carried out. The average values of the instantaneous modulus ($E_0$) and of the relaxed modulus ($E_r$) are equal to 35.37 GPa and 12.25 GPa, respectively. Experimental normalised deferred stress-time curves were plotted to represent the stress relaxation behaviour of kenaf fibres. A mathematical method for extracting the relaxation modulus from relaxation experimental data was proposed. The said method was compared to the Zapas-Phillips method and three other predictive models by means of the prony series and power law. As reported above, results for the relaxation modulus show good agreement between the experimental data and the different theoretical methods in the delayed range. Material parameters estimated ($\beta$ = 0.4) from the KWW function and prony series ($\tau$ = 507.8 s) are particularly important to be accounted for in studying the delayed behaviour of plant fibres. Eventually, an error estimate shows that the errors obtained with the method proposed are merely the same as with the Zapas-Phillips method. The method proposed could serve for the extraction of the relaxation modulus over the entire deferred area of a viscoelastic material.


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## Conflicts of interest

*The authors declare that there is no conflict of interest.*

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