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An investigation of the effect of relative humidity on viscoelastic properties of flax fiber reinforced polymer by fractional-order viscoelastic model

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ABSTRACT

As an environmental-friendly material with eligible mechanical properties, FFRP (Flax Fiber Reinforced Polymer) composites are being widely studied and used in the construction industry. This paper presents an investigation of the effect of environmental humidity on the viscoelastic properties of FFRP. Frequency sweep tests are conducted, and results are re-organized by the Time-Temperature Superposition Principle (TTSP). The Huet-Sayegh viscoelastic model is introduced to describe the relationship between viscoelastic properties and loading frequency. The fractional derivative is applied to the model for more accurate results. It is found both storage modulus and loss modulus decrease with the increase of relative humidity. However, the loss factor does not have a monotonical correlation with the relative humidity of the environment. In general, it can be concluded both the capacity of FFRP to store and dissipate the energy decreases over the hygroscopicity absorption in the air, but there is no specific relationship between the amount of their reduction.

1. Introduction

Fiber Reinforced Polymer (FRP) materials are composites made of polymer matrix and fiber. It has been widely used in practical engineering due to its advantages of high strength, easy manufacturing, fatigue resistance, corrosion resistance, and can reduce the self-weight of the structure significantly. However, the fiber currently used is mainly carbon fiber and glass fiber which are high energy-consuming and non-renewable products. Due to the concern for the environment and demand for environmentally friendly construction materials, natural fibers made from plants are developed rapidly in recent years. Flax fiber is one of the materials that could be the potential substitute for traditional fiber to be used in construction industries. Flax fiber reinforced polymer (FFRP) has been employed in several engineering projects as the main material such as being used to wrap concrete columns [1,2] and the concrete beam [3] as a strengthen and working in a sandwich panel outside the core foam [4–6]. In 2016, the world's first pedestrian bridge fully made of flax fiber reinforced polymer (FFRP) has been built in Eindhoven, the Netherlands [7]. This engineering practice indicates that FFRP shows great potential for constructing load-bearing structures. However, its mechanical properties are sensitive to environmental factors, especially moisture which needs to be further investigated.

FFRP is susceptible to environmental moisture due to the porous structure of flax fibers [8]. Thuault et al. have found the tensile strength of flax fiber decreases a lot when the relative humidity is over 68% [9]. Burges et al. reported the strength of the flax-epoxy laminates is not decreased by the high relative humidity environment, but the tensile modulus decreased severely [10]. Kollia et al. tested flax fiber/bio-based resin composites after hydrothermal aging and found both the strength and the stiffness would not recover after a significant decrease [11]. Wang et al. introduced the chemical treatment on the flax fiber and found the treatment can help to prevent the flax fiber from absorbing moisture [12]. Viscoelastic properties of the material are also found to be correlated with environmental humidity. Li et al. [13]studied the flax fiber composites beams manufactured at different relative humidity levels by impact hammer testing. The results indicated the damping ratio of the beam increases with the relative humidity. Prabhakaran et al. [14] observed there is a 51.03% higher vibration damping ratio of flax fiber reinforced composites than that of the glass fiber reinforced composites, which is caused mainly by the viscoelasticity of flax fiber and energy dissipation of fiber-matrix interface [15]. It has been proven that the FFRP is sensitive to moisture, but the knowledge about the influence of environmental humidity on the viscoelastic properties of FFRP is still limited.

The viscoelastic behavior should be properly described to study and compare the viscoelastic properties of FFRP in different humidity en-

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vironments. The calculation model is a powerful tool to study the viscoelastic behavior of the FRP. In the calculation model proposed by Cardoso et al. [16]. The glass fiber is taken as elastic and represented by the Hook spring, the viscoelastic resin, as well as the fiber-matrix interface, which are calculated by a combination of spring and Newton dashpot. However, the flax fiber is viscoelastic rather than elastic, therefore a more accurate constitutive model is needed to investigate the viscoelastic behavior of the FFRP. The four-element Burgers model is one of the most popular constitutive models for the viscoelastic behavior of the natural fiber reinforced polymer composites [17,18]. The model is a combination of springs and dashpots, arranged as a combination of the separate component in series (Fig. 1(a)). However, the Burgers model is not ideal for the viscoelastic analysis under high frequencies due to the lack of the parameter for time. In addition, there is no temperature-related parameter in the equation that can reflect the temperature change. By introducing two variable dashpots, the Huet-Sayegh model [19] has been proven valid over a wide range of frequencies [20]. The two variable dashpots are seen as a rheological element between the linear spring ($a = 0$) and the linear dashpot ($a = 1$) and are defined as:

$$\sigma\left\{e^{i\omega t}\right\} = \eta\tau^{\alpha-1}\Omega^{\alpha}\left[\varepsilon\left\{e^{i\omega t}\right\}\right] = \frac{\eta}{\tau}(i\omega\tau)^{\alpha}\varepsilon\left\{e^{i\omega t}\right\} \tag{1}$$

where i the complex number defined by $i^2 = -1$, ω is the frequency (rad/s), η is the viscosity dimension (Pa·s), and τ is the time constant.

A general Huet-Sayegh model can be defined as:

$$E(\omega) = E_o + \frac{E_\infty - E_o}{1 + \delta_1(i\omega\tau_1)^{-\alpha} + \delta_2(i\omega\tau_2)^{-\beta}} \tag{2}$$

where E_0 is the complex modulus when ω is approaching zero, E_∞ is the complex modulus when ω is approaching infinity, and δ_1 and δ_2 are model parameters.

However, the Huet-Sayegh model has not been widely used until now due to the parameters in the equation are hard to be determined. It helps to overcome the disadvantage of using too many parameters in the integer-order differential model [21] by employing fractional derivatives in the viscoelastic model. Bagely et al. [22–24] found that some special properties of the viscoelastic material can be better described by the mechanical model modified with fractional order calculus. Koeller [25] first introduced the fractional exponent in the classical viscoelastic model and found it helps to get a good fitting result when the mechanical experiment results are hard to be fitted by integer order viscoelastic models. Xu et al. [26,27] proposed an equivalent higher-order fractional derivative model which considers the effects of temperature and frequency simultaneously and found the model is efficient for studying the viscoelastic behavior of the rubber damper. Wang et al. [28] applied the fractional derivative model to study the FFRP after water aging and found the modeling results agreed well with the experiment. The viscoelastic behavior of FFRP can be conveniently described with fewer parameters by selecting a proper material constitutive model and applying the fractional-order derivative method to the model.

To integrate the fractional calculus into the Huet-Sayegh model, the Riemann-Liouville fractional derivative is applied as [29]:

$$D^{a}f(t) = \frac{1}{\Gamma(1-a)_{dt}}\int_0^{\tau}\frac{f(\tau)}{(t-\tau)^{\alpha}}d\tau \tag{3}$$

where D^α is the order differential operator, $0 < \alpha < 1$. The Huet-Sayegh modified with Fractional order differential is:

$$\sigma(t) = \left(E_0 + \frac{1}{\frac{1}{E_1} + \frac{1}{E\tau^{\alpha}D^{\alpha}} + \frac{1}{E_2\tau_{\alpha}^{\beta}D^{\beta}}}\right)\varepsilon(t) \tag{4}$$

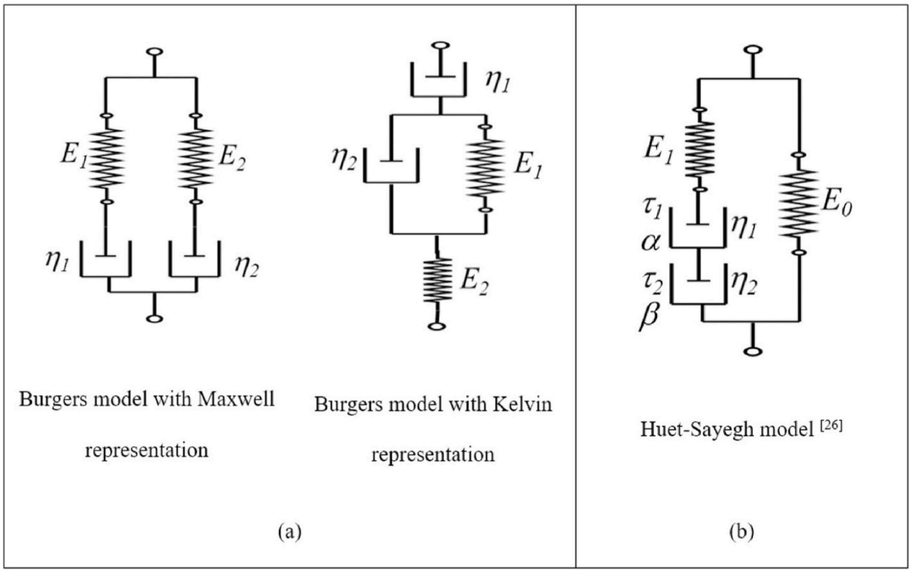
while set $E_1 = E_2 = E$, $\tau_1 = \tau_2$, the storage modulus and loss modulus can be obtained as:

$$E'(\omega) = E_0 + \frac{EAB\left(AB + A\cos\frac{\beta\pi}{2} + B\cos\frac{\alpha\pi}{2}\right)}{A^2B^2 + A^2 + B^2 + 2AB\cos\frac{(\alpha-\beta)}{2} + AB^2\cos\frac{\alpha\pi}{2} + 2A^2B\cos\frac{\beta\pi}{2}} \tag{5}$$

$$E''(\omega) = \frac{EAB\left(A\sin\frac{\beta\pi}{2} + B\sin\frac{\alpha\pi}{2}\right)}{A^2B^2 + A^2 + B^2 + 2AB\cos\frac{(\alpha-\beta)}{2} + 2AB^2\cos\frac{\alpha\pi}{2} + 2A^2B\cos\frac{\beta\pi}{2}} \tag{6}$$

where $A = \tau^\alpha\omega^\alpha$, $B = \tau^\beta\omega^\beta$.

This study aims to provide an investigation of the viscoelastic properties of FFRP in different humidity environments to ensure its safety during service and provide design parameters as a reference. The dynamic mechanic analysis experiment was conducted on the FFRP samples conditioned in different humidity environments and the fractional order differential Huet-Sayegh model is employed to describe the viscoelastic properties of the FFRP with different relative humidity. Finally, the change of viscoelastic properties under the loading frequency range from 10^{-10} Hz to 10^{10} Hz in different environments is



Burgers model with Maxwell representation

Burgers model with Kelvin representation

Huet-Sayegh model [26]

(a)

(b)

Fig. 1. The schematic diagram of Burgers model (a) and Huet-Saygh model (b).

presented and compared.

2. Material and methods

FFRP samples were made of flax fiber and unsaturated polyester with a 40% fiber volume fraction. The sample size is 40 mm × 5 mm × 0.6 mm as shown in Fig. 2. Firstly, FFRP plates were manufactured by the vacuum infusion method. The vacuum pressure of the infusion system was kept at -1 Bar during the injection process. A post-curing process at 120°C was carried out following the 6-h initial curing process. The fully cured FFRP plates were cut into samples by a diamond saw. Then these samples were dried in a vacuum oven at 60°C until they got a constant weight before, they were placed in environments with different relative humidity (RH) as listed in Table 1. The weight of samples was periodically measured to assess moisture absorption.

Dynamic frequency sweep tests were performed with tension clamps on DMA Q800 dynamic mechanical analyzer. The temperature range is from 20°C to 200°C with an interval of 20°C. The loading frequency was set from 0.01 Hz to 100 Hz. Storage modulus *E'*, loss modulus *E''*, and loss factor *tanδ* at different stress frequencies were recorded under each testing temperature step. The dynamic amplitude during the test was set as15 μm. All experimental values were obtained by an average value of three samples.

3. Results and discussion

3.1. Moisture absorption results

The weight of moisture absorbed by samples over time fitted by the Fickian diffusion law [30] as follows:

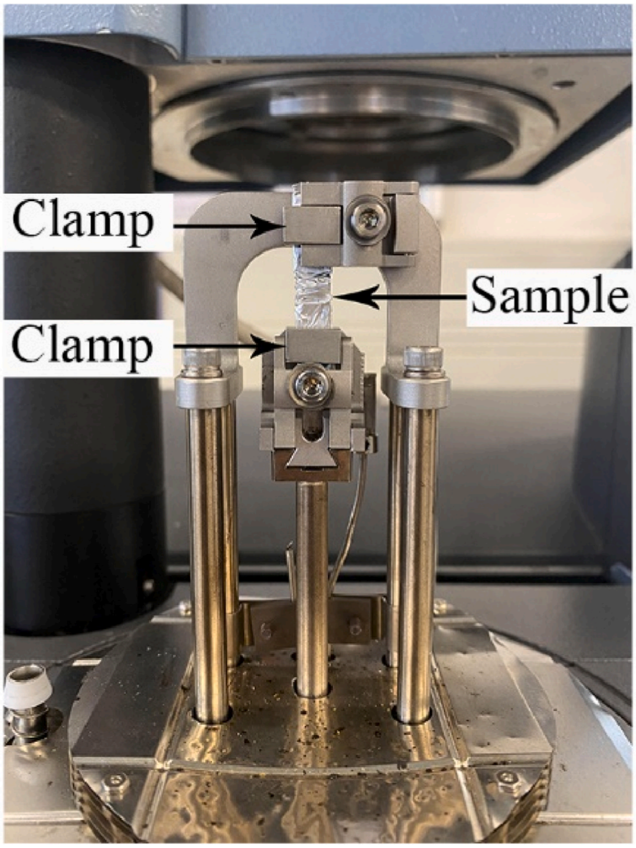


Fig. 2. The testing configuration on DMA Q800.

$$\frac{M_t}{M_m} = \left\{ \begin{array}{l} \frac{4}{h}\sqrt{\frac{Dt}{\pi}}, \frac{M_t}{M_m} < 0.6 \\ 1 - \exp\left[-7.3\left(D\frac{t}{h^2}\right)^{0.75}\right], \frac{M_t}{M_m} \geq 0.6\frac{M_t}{M_m} \end{array} \right\} \tag{7}$$

where t is time (h), h is the thickness of the sample (mm), M_t is the relative moisture uptake (g) at time t, M_m is the maximum moisture uptake (g), and D is the diffusion coefficient (mm^2/s). The fitting results with Eq. (7) indicate that all samples got moisture absorption saturated and diffusion coefficients corresponding to different relative humidity are listed in Table 1.

3.2. Dynamic mechanical analysis (DMA) results

During the frequency sweep test, storage modulus (*E'*) and loss modulus (*E''*) are recorded under each isothermal step. The storage modulus refers to the energy stored because of the elastic (reversible) deformation, and the loss modulus refers to the energy lost because of the viscous deformation (in inverse). The ratio of loss to storage modulus as $tan\ \delta(\omega) = E''(\omega)/E'(\omega)$ is the loss factor which reflects the viscous elastic ratio of materials. There is an inherent correlation between the storage modulus and the loss modulus because they are both dependent on the complex modulus and the phase angle of the material. Hence either of them can help to determine the parameters of the viscoelastic model as Eq. (5) and Eq. (6) to reflect the viscoelastic properties of the FFRP. The storage modulus measured is employed to determine the viscoelastic model as Eq. (5) for it is readily available in most experiment cases. Then other viscoelastic properties can be discussed based on the theoretical model. The glass transition temperature T_g of the sample has been tested before this study as around 130°C. The effect of rising temperature and frequency on decreasing storage modulus is diminishing when the temperature is over 120 °C. This phenomenon means the ability of the material to resist deformation is almost gone when the material got into the glass transition stage, and it will barely be influenced by the loading frequency when the temperature is high enough. The Time-Temperature Superposition Principle (TTSP) is applied to construct a master curve of the storage modulus. The change of storage modulus with frequency can be obtained as:

$$E'(\omega, T) = E'\left(a_T \omega_{ref}, T_{ref}\right) \tag{8}$$

where T_{ref} and ω_{ref} are the reference temperature and the reference frequency respectively, T and ω are the testing temperature and the testing frequency respectively, and α_T is the shifting factor. Two methods can be used to calculate the shifting factor, one is the Williams–Landel–Ferry (WLF) equation [31] as Eq. (9) and the other one is the Arrhenius equation [18] as Eq. (10):

$$\lg a_T = \frac{C_1\left(T - T_{ref}\right)}{C_2 + \left(T - T_{ref}\right)} \tag{9}$$

$$\ln a_T = -\frac{E_a}{R}\left(\frac{1}{T} - \frac{1}{T_{ref}}\right) \tag{10}$$

where C_1 and C_2 are constant parameters, E_a is the active energy of the material, and R is the universal gas constant. The WLF has a limitation that T and T_{ref} must be below or above T_g together [1]. The Arrhenius equation is employed to estimate shifting factors in this study. The testing results in Fig. 3 can be shifted to construct the master curve of storage modulus which can cover a wide range of frequencies as shown in Fig. 4. Then the master curves are fitted by the Huet-Sayegh Model with fractional order following Eq. (3)-Eq. (6) and the fitted results are listed in Table S1. Then the relationship between loss modulus and loading frequency can be generated with Eq. (5) and Eq. (6).

The viscoelastic properties of FFRP with loading frequency in

Table 1
The diffusion coefficient and saturated moisture content.

Code	Relative humidity (%)	Ambient Temperature (°C)	Method to provide the environment	Saturated moisture content (%)	Diffusion coefficient (mm^2/h)
Dry	–	60	Vacuum oven	–	–
11% RH	11	23	LiCl solution	0.872	0.95×10^{-3}
33% RH	33	23	$MgCl_2$ solution	1.468	1.47×10^{-3}
50% RH	50	23	Climate chamber	2.037	1.60×10^{-3}
75% RH	75	23	NaCl solution	3.412	1.95×10^{-3}
97% RH	97	23	K_2So_4 solution	6.947	2.47×10^{-3}

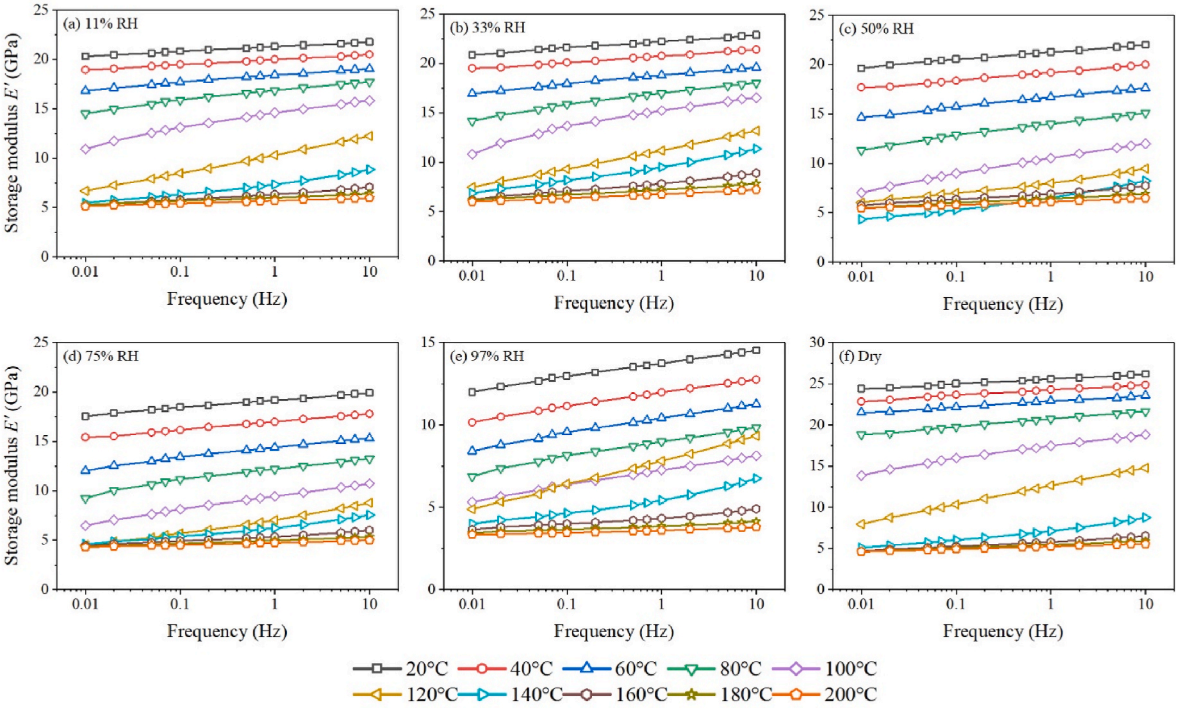


Fig. 3. Relationship between storage modulus and frequency under different testing temperature.

different humidity environments are shown in Fig. 5 (a)~(c), and the result revealed that the sample in all environments shows a typical viscoelastic behavior which is the storage modulus *E'* increases monotonically with the loading frequency while the loss modulus *E''* increases to the peak and then begin to drop with the increase of frequency. Under the same loading frequency, *E'* is found to decrease with the increase of relative humidity and the reduction is more obvious when RH is over 50% (Fig. 5(a)). This is consistent with the results on the tensile modulus of similar material tested in static tensile tests [32]. The 3D colormap (Fig. S1) confirms that the influence of RH and loading frequency on the *E'* is monotonic. What's more, it is surprisingly found that the relationship between *E'* and RH is almost linear under different loading frequencies (Fig. S2). However, further research is needed to provide more details. The changing rate of storage modulus in Fig. 5 (a) also indicates that the sample is in the viscoelastic solid state when the frequency is between 10^{-4} Hz and 10^4 Hz. In this frequency range, the loss modulus is negatively related to the relative humidity, the loss modulus of the sample in the dry state is almost two times more than that in 97% RH. This means the influence of environmental humidity on the viscoelastic properties of FFRP should be specially considered during the design of the FFRP structure aimed at energy dissipation function. Though the magnitude of the peak loss modulus increases with the relative humidity, the frequency corresponding to the peak value seems not seriously influenced by the relative humidity. These findings can be interpreted based on polymer chain movements and fiber-matrix interactions which is the storage modulus of FFRP is mainly dependent on flax fiber and the loss modulus is determined by the chain movement and weakened flax-matrix interface. However, these changes are not synchronized which results in the change of loss factors *tanδ* with humidity not following a specific pattern. Taken together, the results indicated that both the storage modulus and loss modulus of FFRP increase monotonically with the relative humidity when it is working in the viscoelastic solid state, but the loss factor is not changing uniformly with the relative humidity.

4. Conclusion

This work presents a study on the effect of environmental humidity on the viscoelastic properties of the FFRP. Time-temperature superposition principle is employed to organize the frequency sweep test results to get mechanical properties in a wider range of stress frequencies. The modified Huet-Sayegh model with a fractional differential order is proved to describe the viscoelastic behavior of FFRP effectively. The storage modulus of FFRP is found to decrease with the humidity of the environment under the same loading frequency. The loss modulus is found to decrease with the humidity when the material is in a viscoelastic solid state (with frequency from 10^{-4} to10^4 Hz). However, there is no monotonic relationship can be found between loss factor and humidity due to the complex mechanism of viscoelastic properties. The results presented in this work validate the effectiveness of the Huet-Sayegh model with the fractional differential and improve the understanding of the viscoelasticity of FFRP. However, only the FFRP samples

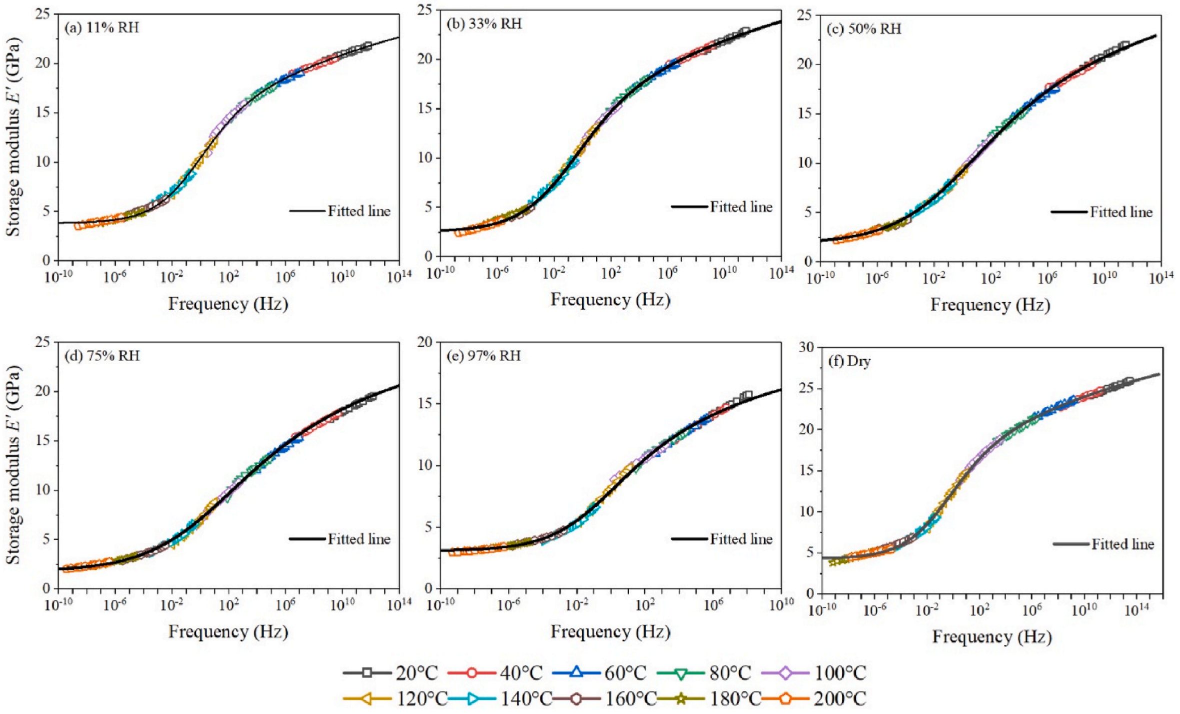


Fig. 4. Superposition results of storage modulus at a reference temperature of 120 °C and the curves fitted by the Huet-Sayegh model.

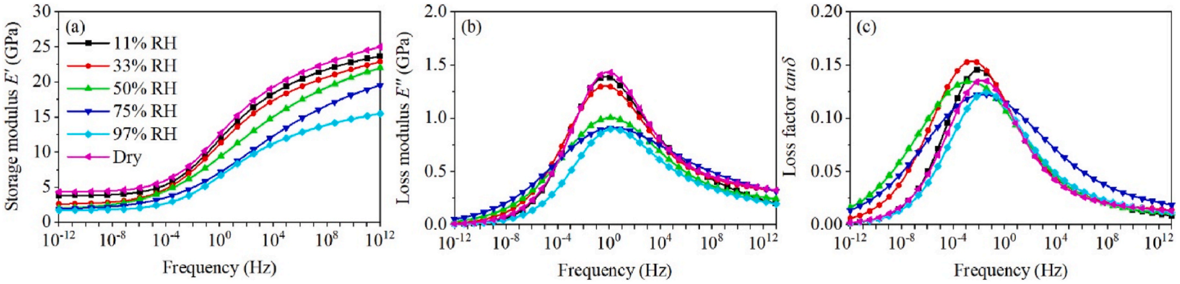


Fig. 5. A comparison of viscoelastic properties in different relative humidity under a reference temperature of 120°C.

with 40% fiber volume fraction in five relative humidity environments are studied. More situations can be tested for the validity of the proposed conclusion.

CRediT authorship contribution statement

Bowen Xu: Conceptualization, Methodology, Experiment, Data curation, Writing – original draft. **Rijk Blok:** Writing – review & editing, Supervision. **Patrick Teuffel:** Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.coco.2022.101406.

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