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Assessment of Durability and Aging Resilience in a Novel Structural Building Adhesive: An Examination of the Time-Temperature Equivalence Principle

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ABSTRACT

In the pursuit of characterizing the mechanical properties of an innovative structural adhesive for building applications, Dynamic Mechanical Analysis (DMA) testing, encompassing a multitude of temperature and frequency conditions, was utilized. Exceptional resilience was exhibited by this adhesive under rigorous long-term performance tests aimed at accelerated aging. Post a thirty-day aging period, consistent properties of the epoxy adhesive were discerned across varying humidity conditions, namely 85%, 75%, and 65% relative humidity. Such constancy elucidates the adhesive's impressive resistance to damp-heat aging, thereby asserting its aptitude for sustained applications. Utilization of the time-temperature equivalence principle was pivotal in the acceleration of the long-term performance characterization, culminating in a generalized curve for specific aging durations and environments. This methodology not only diminished testing timeframes but also provided further validation for its efficiency. The preferential use of this multiple equivalence principle is thus recommended to expedite the mechanical property characterization of the adhesive post-thermal aging. Selection of the test temperature as the reference temperature generated a refined generalized curve inclusive of extensive frequency alterations, suggesting a streamlined approach for studying the adhesive's long-term performance. Consequently, the findings from this exploration offer a dependable benchmark for evaluating the durability and reliability of structural adhesives utilized in building applications.

1. INTRODUCTION

The escalating importance of structural adhesives in construction activities has been recognized, with applications ranging from bonding and reinforcement, to the enhancement of transportation infrastructure, water conservancy, and hydropower engineering structure fortification. Their use has also been observed in the refurbishment of existing edifices. The nonlinear mechanical response of polymer materials, principally the viscoelastic nonlinearity, arises from factors such as excessive strain or extended duration. As polymers are viscoelastic substances, their mechanical properties are largely influenced by four main parameters: force, deformation, temperature, and time. Therefore, the time-dependent viscoelastic behavior of polymers renders short-term experiments a viable tool for predicting long-term mechanical properties [1-4].

Over several decades, extensive studies have confirmed that the creep and stress relaxation curves of a material during extended mechanical testing exhibit similar shapes at different time points throughout the physical aging process of polymers [5, 6]. These curves can be transposed along the time axis under varying circumstances to construct a master curve at a specific time point, thereby accelerating the process of characterizing the mechanical properties of materials [5, 6]. Through examining relaxation phenomena in molecular chain segments, it has been observed that mechanical relaxation can occur at both high temperatures over short periods and low temperatures over extended durations. The consistency of the effect of increased temperature and extended observation time on viscoelastic behavior exemplifies the time-temperature equivalence principle [7-9].

Given that many structural elements are designed to last several decades, their primary load-bearing components such as beams and columns should ideally possess similar lifespans. When structural adhesives are utilized in these parts, they are required to fulfill equivalent usage expectations. Consequently, the long-term performance of structural adhesives under service conditions has attracted significant research interest in recent decades [10-14]. Detailed stipulations exist in many relevant specifications for testing the longevity of engineering structural adhesives, such as the "Code for Design of Concrete Structures Reinforced with FRP" (GB50367-2013) and the "Technical Specification for Safety Identification of Engineering Structure Reinforcement Materials" (GB50728-2011). However, these technical specifications require extensive experimental periods for long-term performance testing, often spanning several months or even years. Despite this, the experimental duration for artificial aging of structural adhesives in the laboratory is comparatively shorter than the service cycle of building structures, which can span several decades or even centuries. As such, there is a necessity for faster characterization of adhesive properties over extended periods to adequately assess their long-term mechanical performance under service conditions [15-19].

A significant body of literature has explored the application of artificial accelerated characterization techniques to examine the long-term mechanical properties of epoxy resin adhesives [20-25]. These methods primarily involve exposing specimens to increased temperatures, stress, and physical aging. Modern research typically involves subjecting structural adhesives to experiments under varying temperature, stress levels, and aging conditions. Subsequently, principles such as the time-temperature equivalence and time-stress equivalence are utilized to anticipate their long-term properties. These artificially accelerated characterization methodologies, within a constrained experimental period and under specific conditions, have been found effective in aiding researchers to obtain a broader time range of data, thereby enabling a more comprehensive understanding of the properties of structural adhesives [25-30].

In many studies examining the creep behavior of epoxy resin adhesives, artificial acceleration methods have been utilized to extend the time span of creep behavior. However, these durations often fail to meet the experimental time mandated by relevant technical specifications. Therefore, it has been deemed necessary to employ artificial accelerated characterization of experimental data multiple times using principles such as time-temperature equivalence, time-humidity equivalence, and time-stress equivalence [25-30].

A recent study conducted a 30-day aging test on a newly developed structural adhesive for building applications under varying humidity (65%, 75%, 85%, and 95%) and temperature conditions (80℃, 100℃, 120℃, and 140℃). Following the aging process, DMA was performed on the samples. The current study examines the accelerated characterization of the long-term properties of structural adhesives at 85%, 75%, and 65% relative humidity based on the aforementioned research. It further discusses the validity of selecting different reference temperatures when employing the time-temperature equivalence principle for the artificial accelerated characterization of this structural adhesive [31-33].

2. MATERIALS AND METHODS

2.1 Material and sample preparation

A formulation comprising industrial-grade epoxy resin (grade E51) and phenolic resin (grade F51) served as the basis for this study. An additional component, Qishi BE toughening agent, along with m-phenylenedimethylamine as a curing agent, completed the mixture. The proportion of E51 to F51 was maintained at a 1:1 ratio, followed by the incorporation of 15g of Qishi BE per 100g of resin composite. The preparation of the adhesive involved a two-hour mechanical stirring process under heat, after which 18g of m-phenylenedimethylamine was introduced. Subsequent stirring for ten minutes was conducted, followed by degassing in a vacuum chamber for fifteen minutes to remove entrapped air. The resin mixture was then poured into a designated mold, where it was allowed to cure at room temperature over a seven-day period, resulting in samples of dimensions 25mm×5mm×2.5mm, as shown in Figure 1.

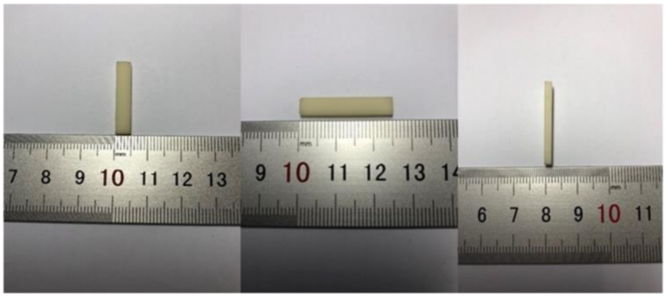

Figure 1. Sample of epoxy resin adhesive

2.2 Instrumentation and test procedure

The evaluation process incorporated several specialized devices, including the Constant Temperature and Humidity Aging Test Chamber (Huaxia Technology Co., Ltd.), the 401A Thermal Aging Test Chamber (Qidong Double Edge Test Equipment Co., Ltd.), and the DMA8000 Rheometer.

The Constant Temperature and Humidity Aging Test Chamber was used to induce controlled aging processes at pre-set relative humidity levels of 65%, 75%, 85%, and 95%, and a maintained temperature of 70℃. Concurrently, the Thermal Aging Test Chamber allowed for the regulation of thermal aging processes at temperatures of 80℃, 100℃, 120℃, and 140℃. Post a thirty-day aging period, batches were removed every five days for subsequent dynamic mechanical thermal analysis (DMTA) experiments.

A temperature scan was conducted on unaged samples and samples exposed to varied humidity levels using the DMA8000 Rheometer. For each condition, three samples were analyzed and the results averaged. The scanning process implemented the single cantilever beam mode in a temperature range from room temperature to 150℃, with a heating rate of 2℃/min and a strain level of 0.1%.

A frequency scan was performed on three additional samples employing the same single cantilever beam mode. Frequencies from 0.01Hz to 100Hz were applied at distinct temperature intervals of 40℃, 50℃, 60℃, 70℃, 80℃, 90℃, and 100℃, under a consistent strain level of 0.1%. The precision of these tests allowed for detailed insights into the long-term performance of the adhesive under a variety of conditions.

3. RESULTS AND DISCUSSION

3.1 Time-temperature equivalence principle for adhesive characterization post wet thermal aging

The utility of the time-temperature equivalence principle for predicting long-term mechanical properties of adhesives based on short-term experimental data is well-established [34-36]. The successful implementation of this principle enables detailed insights into the properties of polymer materials under constrained experimental conditions and time frames.

Previous investigations have generated generalized curves for the subject structural adhesive across different aging durations under conditions of 95% relative humidity [31]. The same methodology was applied to evaluate adhesive performance under varied aging durations at relative humidity levels of 85%, 75%, and 65%. A reference temperature of 70℃ was employed for each humidity condition and aging duration. Thereafter, experimental curves at different temperature levels were aligned with the reference curve. The translation of these results produced generalized curves for different aging durations under three humidity conditions (Figures 2-4).

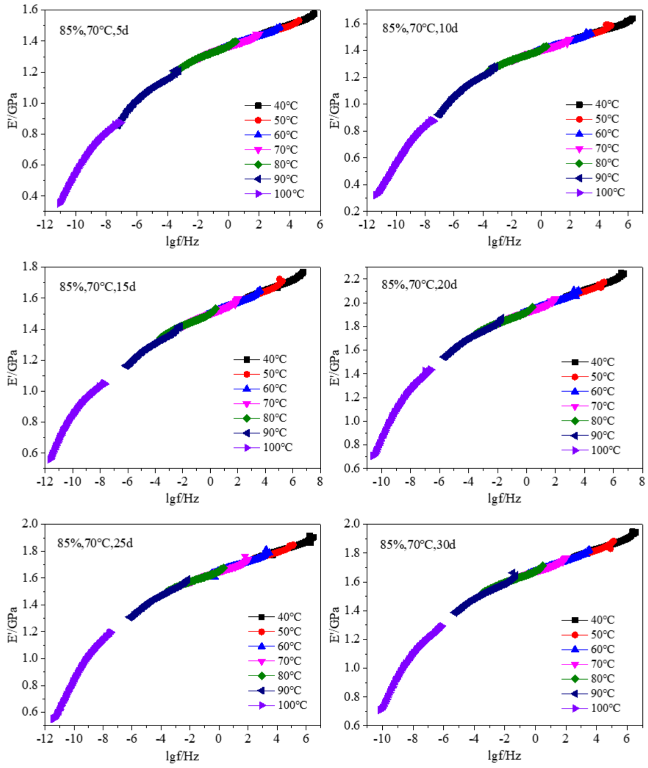

Figure 2. Generalized curves *E*'-lg*f* at a relative humidity of 85% with different aging days

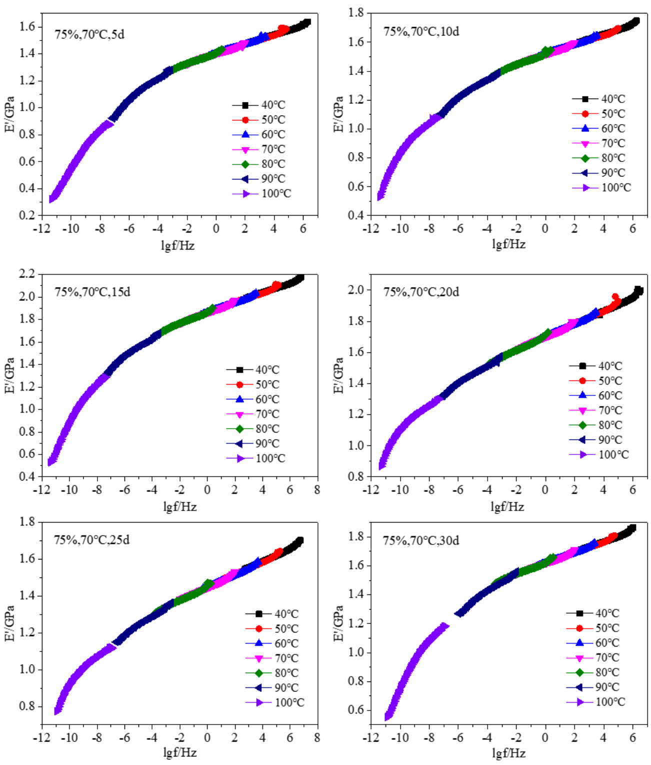

Figure 3. Generalized curves *E*'-lg*f* at a relative humidity of 75% with different aging days

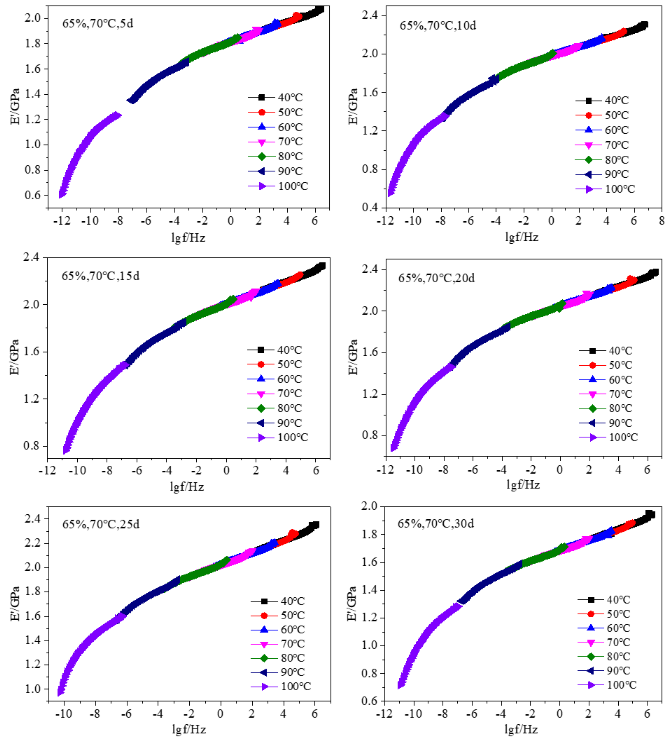

Figure 4. Generalized curves *E*'-lg*f* at a relative humidity of 65% with different aging days

For a comprehensive comparison and analysis, Figure 5 presents the generalized curves for different aging durations under four humidity conditions. As revealed in Figure 5, the energy storage modulus of the adhesive, post-aging, surpasses that of the non-aged curve at the identical test frequency. Furthermore, an increase in the test frequency at equivalent aging durations corresponds to an elevation in the energy storage modulus.

Interestingly, under an aging condition of 95% relative humidity, the curve from no aging to 10 days of aging ascends with the increase in aging time, while the curve from 10 to 30 days descends with increasing aging duration (Figure 5(a)). This phenomenon suggests a dominant role of adhesive post-curing during the initial 10 days, followed by a completion of post-curing and commencement of wet thermal aging after 10 days, thereby causing reduced swelling and plasticizing effects due to water molecules on the adhesive.

Similarly, at 85% relative humidity, an initial increase is observed in the aging curve up to 20 days, followed by a decrease upon further aging to 30 days (Figure 5(b)). This infers that post-curing plays a dominant role during the first 20 days, post which the wet heat aging dominates, leading to a decrease in adhesive performance.

In conditions with relative humidity levels of 75% and 65%, the curve from no aging to 10 days of aging increases with aging time, while the curve from 10 to 25 days decreases (Figures 5(c) and (d)). This indicates a predominant role of curing up to the first 25 days, beyond which the effects of water molecules on the adhesive become apparent. This further signifies the more pronounced and rapid water damage to adhesives in high humidity environments (95% and 85%) compared to lower humidity environments (75% and 65%).

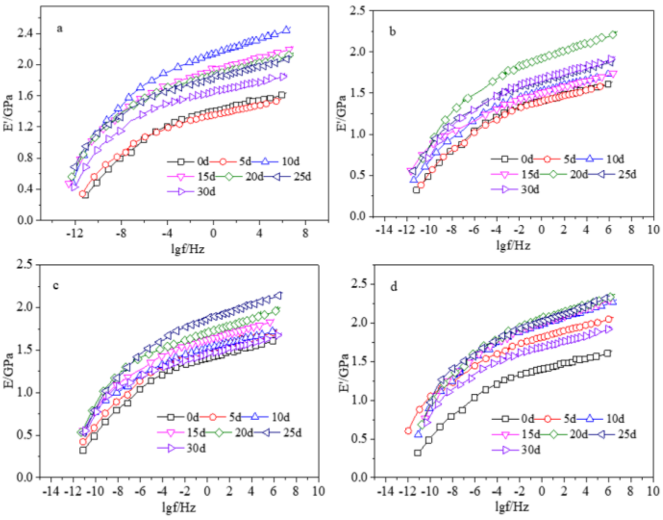

Figure 5. Generalized curves E'-lg$f$ at different relative humidity levels with different aging days (a-95%, b-85%, c-75%, d-65%)

Through the application of the time-temperature equivalence principle, the frequency range of the generalized curve from the initial experimental frequency spectrum is extended from four orders of magnitude to 20 orders of magnitude across varying humidity conditions and aging days. However, testing across a frequency range of 20 orders of magnitude is unfeasible in actual experimental setups, which underscores the value of the time-temperature equivalence principle in enhancing experimental efficiency and reducing the time required for experiments.

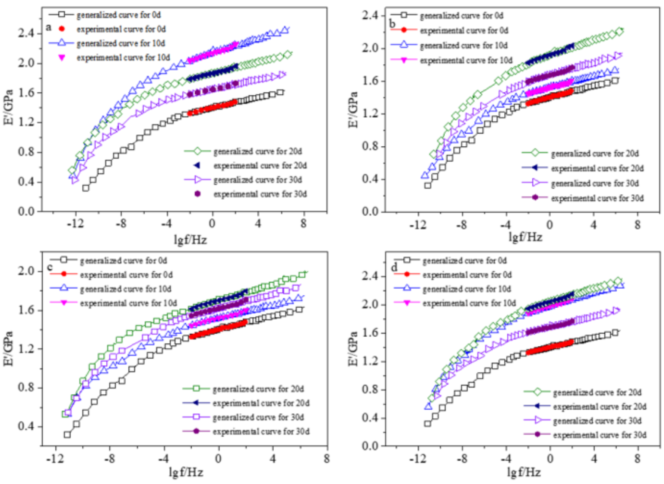

Figure 6. Comparison between generalized curves and experimental curves E'-lg$f$ at different relative humidity levels with different aging days (a-95%, b-85%, c-75%, d-65%)

The validation of the time-temperature equivalence principle was carried out through a comparison between the generalized curve and the experimental data (Figure 6). To ensure clarity, four curves at varying distances (0d, 10d, 20d, 30d) were chosen for comparison. The observed congruence between the generalized curve and experimental data provides evidence for the reliability and effectiveness of the time-temperature equivalence principle in this context.

3.2 Time-temperature equivalence principle for adhesive characterization post thermal aging

In previous research, the mechanical acceleration characterization of the frequency spectrum curve of epoxy resin adhesive after thermal aging was carried out using the time-temperature equivalence principle and time-aging time equivalence principle [33]. The resulting generalized aging curve was obtained for 20 days under an aging temperature of 100℃ and a test temperature of 100℃. The rheological and mechanical behavior characterization results from the adhesive after two accelerated tests were found to be in good agreement with experimental findings. However, it is essential to note that the frequency scan test temperature varied from 40℃ to 100℃ at each of the four selected temperature levels in the thermal aging experiment. Therefore, when employing the time-temperature equivalence principle to accelerate the characterization of adhesive mechanical properties after thermal aging, two temperatures were involved - one being the thermal aging temperature, and the other was the frequency sweep measurement temperature. In previous studies, the time-temperature equivalence principle was used for accelerated characterization, with the test temperature acting as the reference point. This section will explore the accelerated characterization of structural adhesives following thermal aging, using the aging temperature as the benchmark.

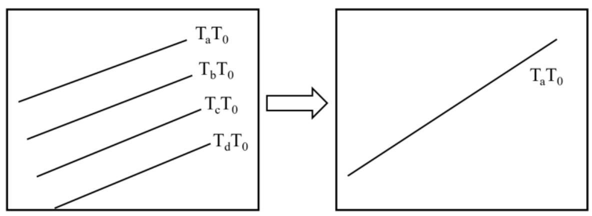

Figure 7. The diagram based on the aging temperature as reference temperature

Figure 7 demonstrates the aging temperature (a, b, c, d...) and the test temperature. The reference temperature is chosen as the aging temperature to obtain a generalized curve through translation. In line with the experiment conducted in this paper, Figure 8 presents the curve at a test temperature of 100℃ under four different aging temperatures (80℃, 100℃, 120℃, and 140℃) and varying aging durations (5-30 days). The time-temperature equivalence principle is employed to process Figure 8. To enable comparison with the above analysis, experimental data at a test temperature of 100℃ is selected as an example for analysis. Specifically, when T=100℃, the aging temperature of 100℃ is chosen as the reference temperature (Tref=100℃), and the curves corresponding to other aging temperatures (80℃, 120℃, and 140℃) are shifted onto this reference curve. When the aging temperature is 100℃ for various durations, the generalized curve of the test temperature remains at 100℃, as shown in Figure 9. It can be seen that after translation, the curve is not as smooth as that displayed in previous research by anther method, and there is a significant deviation in the curve aged at 140℃.

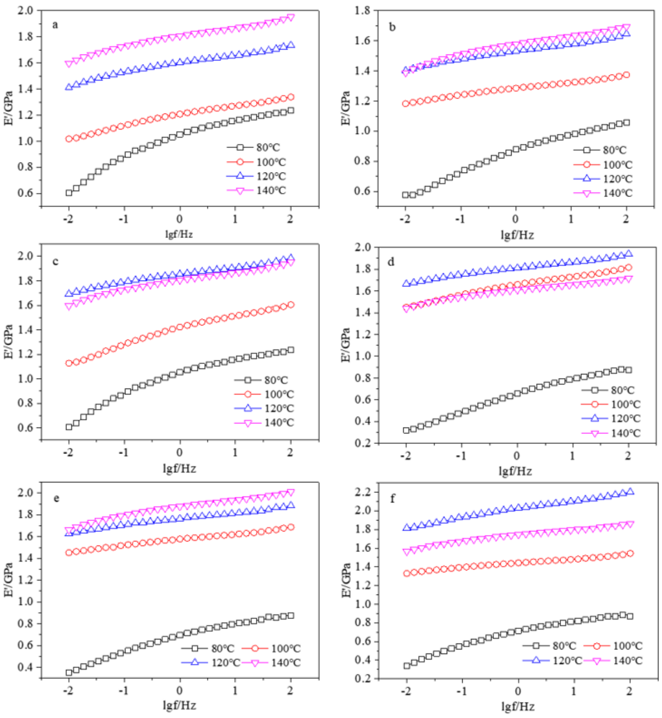

Figure 8. Curves E'-lg$f$ under 100°C with different aging temperature levels for different aging days (a-5 aging days, b-10 aging days, c-15 aging days, d-20 aging days, e-25 aging days, f-30 aging days)

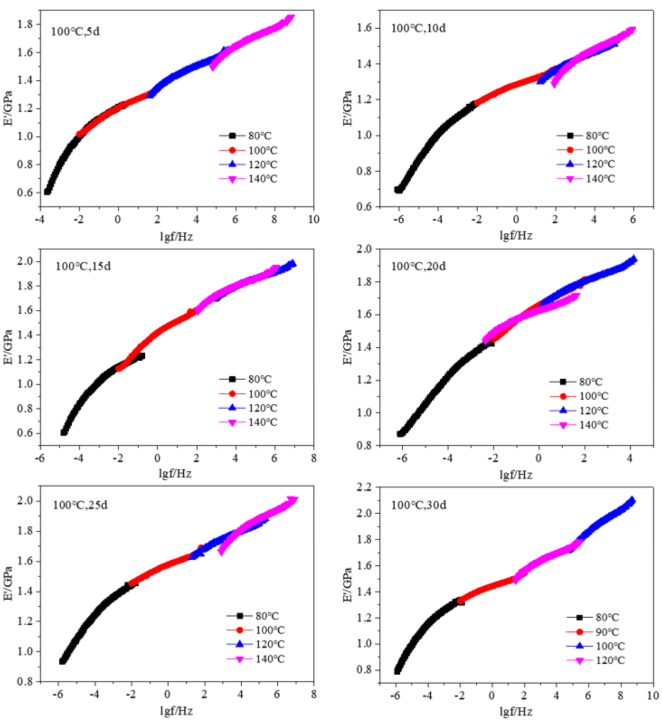

Figure 9. The generalized curves E'-lg$f$ with different aging days under testing temperature of 100°C (references temperature as aging temperature 100°C)

Moreover, Figure 9 was further processed concerning the time-aging time, and an aging duration of 20 days was chosen as a reference point. The results reveal that it is not possible to connect these curves into a smooth pattern. This is primarily due to the fact that the time-temperature equivalence principle is based on the theory of free volume. When an epoxy structural adhesive undergoes aging at a specific temperature, the movement of internal molecular chain segments reaches a state that matches that temperature. Consequently, mechanical properties are tested at a different temperature than that used for aging. In practice, the performance displayed during testing should reflect the conditions during prior aging. Therefore, when accelerating the rheological mechanical properties of epoxy resin adhesives after thermal aging and applying the time-temperature equivalence principle, if there is inconsistency between the aging temperature and test temperature, it is necessary to select a reference temperature that matches both temperatures for equivalent treatment of curves under different test temperatures.

4. CONCLUSION

(1) In this study, dynamic mechanical analysis (DMA) tests were conducted on a new structural adhesive at various temperatures and frequencies. The mechanical properties were characterized after wet and heat aging through long-term performance acceleration testing. The results show that the properties of the epoxy structural adhesive remain largely unchanged, even after 30 days of aging under three different humidity conditions (relative humidity 85%, 75%, and 65%). This demonstrates the adhesive's excellent resistance to moisture and heat aging.

(2) By employing the principles of time-temperature equivalence, time-aging time equivalence, and time-humidity equivalence, we were able to obtain a generalized curve depicting the frequency change of the epoxy structural adhesive under varying relative humidity levels (85%, 75%, 65%) at a temperature of 70°C for a period of 20 days. This approach effectively reduced the duration required for experimentation while providing an expedited method for characterizing the long-term mechanical properties of other polymers. This technique enables the acquisition of a generalized curve representing specific aging times of polymers under particular humidity and temperature conditions.

(3) For accelerating the mechanical properties of adhesives after thermal aging, the continuous application of the equivalent principle for accelerated characterization is recommended. However, when implementing the time-temperature equivalent principle first, if the aging temperature is inconsistent with the test temperature, selecting the aging temperature as the reference may not lead to successful second-time acceleration, and the curves may not fit well. In contrast, selecting the test temperature as the reference temperature can result in a second acceleration, yielding a smoother generalized curve with broader frequency changes.

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