



## Effect of hydrothermal aging behavior on surface treated Kevlar fiber laminated composites

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**Abstract:** The water sorption characteristics of Kevlar fiber–reinforced epoxy composites were studied by immersion in water at 80 °C. The hydrothermal aging process was conducted on treated and untreated Kevlar/epoxy composites; also, the composite was evaluated by the three-point bending test. The phosphoric acid (PA) pretreated with Epichlorohydrin (ECH) was used for the surface modification of Kevlar. In the case of chemically modified fiber composites, water uptake was found to be dependent on the chemical treatment done on the fiber surface. The lowest water uptake was observed for composites treated with PA with ECH. The effect of thermal aging on the flexural strength of the treated Kevlar composite was 20.42% higher than the untreated composite. Consequently, the flexural modulus was 13.9% higher than the untreated Kevlar composite. Moreover, the water diffusion coefficient of treated composite is 30.19% higher than untreated composite. It was concluded that fiber/matrix degradation time at the interface region was increased in the case of the treated composite due to the increase of interfacial polar bonding in the Kevlar composites. The surface morphology (SEM) and XRD analysis were used to validate the experimental results.

**Keywords:** Kevlar fiber, flexural strength, hydrothermal, surface treatment

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## 1. Introduction

Kevlar is a fiber that is synthetic and organic in fragrant polyamide own family. Kevlar has a specific residence and wonderful chemical composition of aromatic polyamides (aramids). It has a unique mixture of excessive energy, high modulus, sturdiness, and thermal stability (Marshall, 1985). However, the mechanical properties of Kevlar fiber composites were decreased at high-temperature environmental conditions. Moreover, the Kevlar fiber has more strength degradation compared with glass fiber composite in the wet medium state (Menail et al., 2009). Further, the authors were presented surface chemical modification was the prominent role to decrease the water uptake in the composites; also, the diffusion coefficient of untreated banana/glass hybrid composite was higher compared with treated composites (Pothan & Thomas, 2004). Further, the authors stated that the diffusion coefficient and equilibrium water content were increased in Kevlar composites by about 5 to 10 times compared with glass/graphite composites; also, the permeable effect of Kevlar fiber was attributed to increasing the water absorption level in the Kevlar composites (Gopalan et al., 1986). The researchers have investigated several methods for moisture absorption characteristics and their effect on the mechanical properties of a composite material (Shen & Springer, 1976; Gopalan et al., 1989; Xian et al., 2012). The researchers have discussed the effect of the aging process on hybrid (Glass / Kevlar) / polyester composites. Further, the damage mechanism (Kevlar fiber fraying and micro buckling) was promoted after the aging of hybrid composites, which was attributed to reducing the strength of composites (Felipe et al., 2019; Demuts, & Shyprykevich, 1984). Furthermore, the damage initially occurred at the resin-rich boundaries; also, the hydrothermal residual stresses lead to change from tensile to compressive at the resin region. In contrast, uniform distribution of fiber was attributed to changing the residual stresses from compressive to tensile at the fiber/matrix interface (Wood & Bradley, 1997). Further, the researchers have indicated that flexural, interlaminar, and compressive strength of glass fiber/epoxy composite were severely damaged in saltwater compared with distilled water immersion for the same temperature and duration (Rege & Lakkad, 1983). The water immersion was attributed to affecting the microstructural integrity of composite, which lead to internal defects. Moreover, the interfacial crack and fiber/matrix debonding occurred due to poor interfacial adhesion in composites (Imielińska & Guillaumat, 2004). The authors were addressed the interfacial adhesion could be enhanced by surface modification techniques; also, fiber/matrix debonding was improved through surface modification of Kevlar fabric using various chemical treatments (Ramasamy et al., 2019). The

focus on the present investigation is to find the results on mechanical properties under the controlled environmental condition at 80 °C constant temperature with chemical treatment and without chemical treatment on Kevlar/epoxy composites.

The novel contribution of the present study is to establish whether any thermally induced deterioration occurs in Kevlar-29 fibers after the surface modification process of fiber composites. The effects of water exposure on Kevlar/epoxy composites, moisture absorption, and its effect on flexural strength have been studied. The novel contribution in this research for better understanding of the pre-treatment approach with chemical treatments on Kevlar fiber under a controlled temperature of 80 °C to reduce the moisture absorption and to increase the durability of composites.

## 2. Materials and methods

### 2.1. Materials

Kevlar 29 was used as the Kevlar fiber for fabricating the composite specimen with the epoxy matrix. Epoxy resin (Araldite LY 556) matrix having outstanding properties has been used. And work Hardener (Araldite) HY 951 was used. Phosphoric acid (PA), KOH, and Epichlorohydrin (ECH) purchased through sugar biological and chemicals, Chennai, India.

### 2.2. Fiber treatment

The Kevlar fiber was treated by ECH with PA pre-treated composite. The fiber was immersed in acetone for four hours to remove the impurities and dust from the Kevlar fiber. The Kevlar fabric was soaked in one percent KOH solution at room temperature for 2 hours, subsequently washed with purred water and dried for 48 hours at room temperature. Then fiber was treated by 15 weight % phosphoric acid, then cleaned and dried. The PA treated fiber was grafted by ECH for 1 hour at 70 °C, and then grafted fiber was washed and dried in a vacuum oven for improving debonding strength of Kevlar/epoxy.

### 2.3. Hydrothermal ageing treatments

The hydrothermal aging process was used for treated and untreated Kevlar/epoxy composites. The moisture was absorbed during the aging process of composite; also, water absorption percentage ( $M_t$ ) was calculated by using the Equation (1) (Zhang & Mi, 2019).

$$M_t = \frac{w_t - w_o}{w_o} \times 100\% \quad (1)$$

$$D = \frac{\pi h^2 S^2}{16M^2} \quad (2)$$

Where,

$W_t$ -water absorption at the period (Kg)

$W_0$ -initial water absorption (Kg)

H-thickness of specimen (3.2 mm)

S-modulus of water absorption ( $1/\sqrt{s}$ )

M-equilibrium moisture content (%)

The construction of hydrothermal aging equipment for aging the samples up to 66 days (9 weeks) at 80 °C (Menail et al., 2009). The samples were taken for flexural testing every three weeks. For each week three samples were used for evaluating the samples of treated (names as T3, T6, and T9) and untreated (named as NT3, NT6, and NT9). Consequently, the weight of water absorption was observed after cleaned samples on all days by using 0.1g electronic weight measurement. The components used for this equipment are W 1209 temperature relay, immersion heater, and temperature sensor. The temperature relay module can intelligently monitor the power of most types of electrical devices based on the temperature sensed by the high precision NTC temperature sensor. Immersion heaters have a metal tube that holds a temperature sensor. Unlike a gas-heated tank with burners underneath it, immersion heaters heat water directly from inside the tank. A strong electric current is passed through the part that heats the surrounding water. AC adapters are used for electrical devices that need power. The immersion heater is immersed in a bucket filled with water for the hydrothermal aging process.

To maintain a constant temperature inside the bucket it is surrounded by insulation material such as sand. To maintain the temperature of the heater the relay is used. The tempera-

ture sensor connected to the relay is dipped into the water. When the water reaches a certain temperature (80 °C) the relay automatically cuts the power supply to the immersion heater, so the temperature is maintained in the setup was shown in Figure 1a. Moreover, the water diffusion coefficient (D) was determined from Equation (2).

#### 2.4. Experimental characterization

The three-point bending flexural test provides values for the modulus of elasticity in bending, flexural stress, flexural modulus, and the reduction of flexural stress response of the material. This test was performed on a universal testing machine in 100kN load carrying capacity Tinius Olsen universal testing machine (UTM) with a three-point bend fixture which is shown in Figure 1b. The sample was prepared as per ASTM D 790-03 and three samples were used for each group of testing. The flexural test has been done treated (T) and untreated (NT) Kevlar specimen without hydrothermal aging.

Subsequently, the hydrothermal aging samples were evaluated for the performance behavior of Kevlar composites. The crystallinity index of surface treated aramid fiber and untreated aramid fiber were determined by using X-ray diffraction (XRD). The step size adopted for this study was  $0.02^\circ$  ( $2\theta$ ) in the angular range of  $5^\circ$ - $100^\circ$ . The sealed-tube of Cu-K $\alpha$  radiation was operated at 40 kV and 50 mA at a wavelength of

$1.54 \text{ \AA}$  for calculating crystallinity index by Segal peak height method. Further, fracture surface morphology of treated and untreated Kevlar fiber composites was observed using in a scanning electron microscope (model- SNE-3200M, SEC).

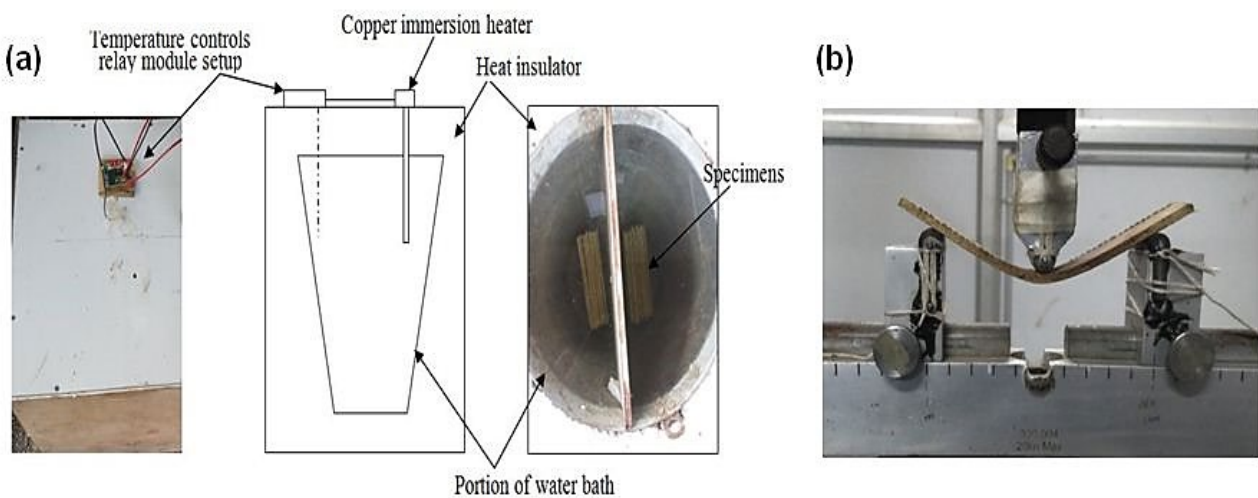


Figure 1. a) Hydrothermal setup and b) flexural testing.

### 3. Results and discussion

#### 3.1. XRD analysis

The XRD spectra of treated and untreated Kevlar fiber was shown in Figure 2. It was reported that there was no additional peak in the treated fiber, indicating that the fiber is unaffected by the Kevlar fiber surface chemical treatment. Moreover, the untreated fiber has more crystallinity materials compared with treated Kevlar fiber (Zhao, 2013). Hence, the surface treatment on Kevlar fiber was attributed to decreased crystallinity index value by about 21.09% compared with untreated Kevlar fiber.

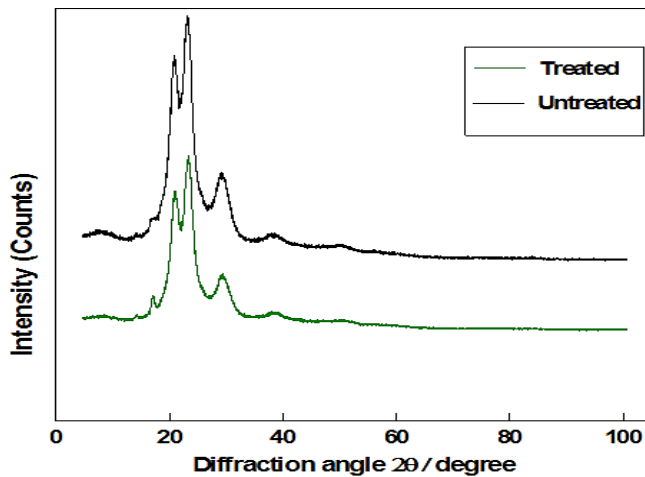


Figure 2. XRD analysis of untreated and treated Kevlar fiber.

#### 3.2. Effects of water absorption on Kevlar composites

The water absorption of surface treated and untreated Kevlar/epoxy composite on the hydrothermal aging process was shown in Figure 3(a), where the x-axis is the square root of immersion time in hours and the y-axis is water absorption percentage (Imielińska & Guillaumat, 2004). The treated Kevlar composites absorbed less water with increasing immersion time. Consequently, the water absorbed was more in untreated composites. The water absorption attained maximum level in 32 days at 4.35% for untreated and 3.28% in 44 days for treated composites. This can be due to surface modification of Kevlar having more wettability with epoxy matrix lead to increase interphase strength of the composite. Moreover, the void and flaws were occurred less in treated composites (Tao et al., 2016) due to which water inhibits into the matrix very slowly. Figure 4 shows the fracture surface morphology of untreated and treated Kevlar fiber composites. It was indicated that voids and flaws were higher than untreated Kevlar/epoxy composites.

Furthermore, the diffusion coefficient was determined for treated (T) and untreated (NT), which was shown in Figure 3(b). The modulus of water absorption (S) was calculated from Figure 3(a). The D of treated and untreated composites was  $5.63 \times 10^{-12} \text{ m}^2/\text{s}$  and  $7.33 \times 10^{-12} \text{ m}^2/\text{s}$ , respectively. This could be concluded that D was less for treated Kevlar composites because polar functional groups were activated on the surface of treated composites (Ramasamy et al., 2019) lead to slow water ingress in treated composites.

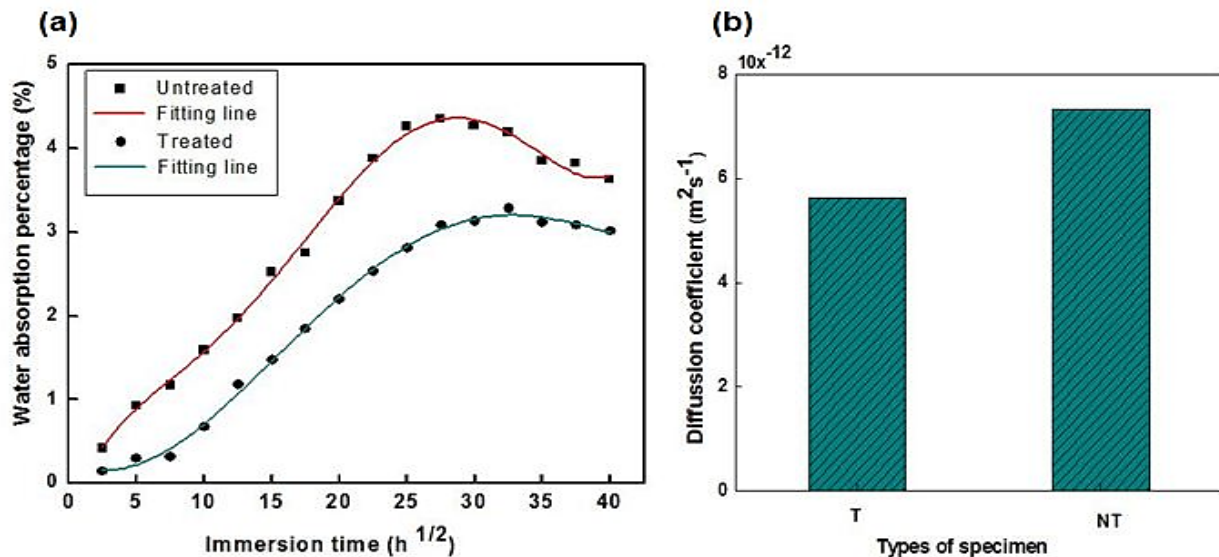


Figure 3. Kevlar/epoxy composite of a) water absorption and b) diffusion coefficient.

### 3.3. Properties of flexural

The mechanical property of composites depends on the interfacial strength between fiber and matrix. The load-displacement curves were shown in Figure 5. For the non-hydrothermal aging process, the load was attained 440N for treated and 400N for untreated. Consequently, the attained load was decreased after the hydrothermal aging process.

In particular, the flexural load was significantly reduced after three weeks of aging. Meanwhile, the flexural load was decreased after water absorption reached saturation level. The flexural strength of Kevlar composites as shown in Figure 6a. The flexural strength of treated composites was 277.5MPa, 202.7MPa, 185.8MPa, 182.1 MPa; also, the non treated were 249.5MPa, 169.5MPa, 145.4MPa, 144.9 MPa for non-hydrothermal aging, 3 weeks, 6 weeks, 9 weeks, respectively.

The flexural strength was reduced after the aging process due to the mechanism of capillary action between the fiber and matrix composite. The water ingress in the composite leads to plasticization and swelling of the matrix system.

Moreover, the untreated Kevlar fiber has poor bonding with the epoxy matrix; also, the voids may affect the flexural strength of composite (Li et al., 2013). Furthermore, the flexural modulus also drastically reduced in untreated Kevlar/epoxy composites as shown in Figure 6b.

The aging process in Kevlar composite might be reduced crystallinity of composite (Arrieta et al., 2011). The prolonged aging in composite was attributed to reducing the mechanical properties as well as load-carrying capability was decreased. This was concluded that treated composite could be attained the required performance.

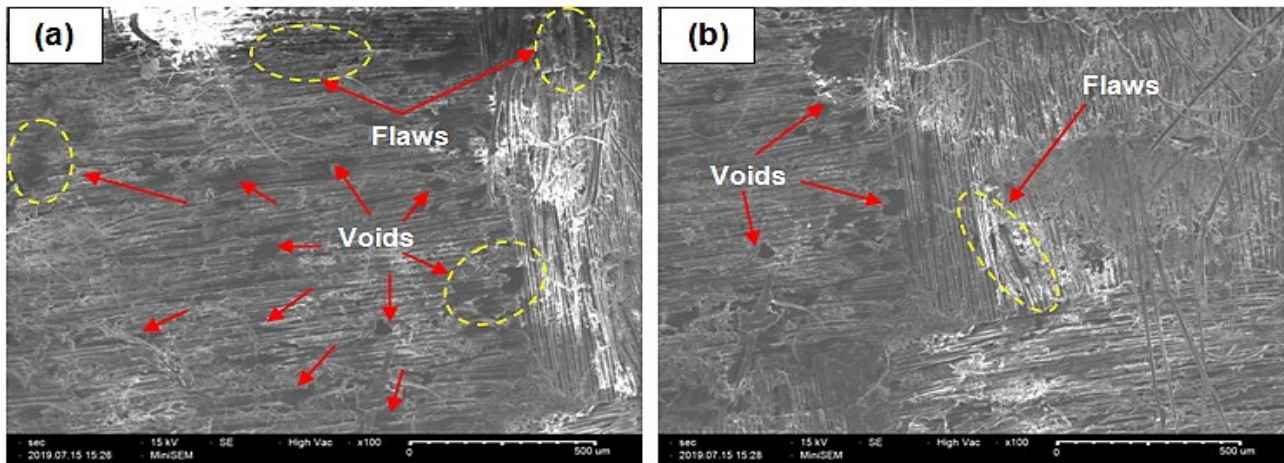


Figure 4. Fracture surface morphology of Kevlar/epoxy composites a) untreated and b) treated.

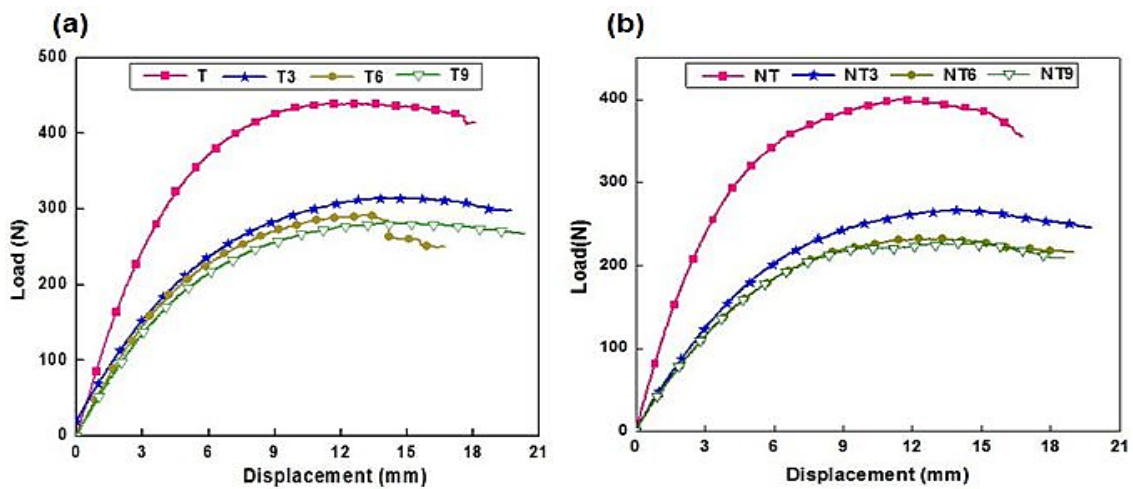


Figure 5. Load versus displacement of a) treated and b) untreated.

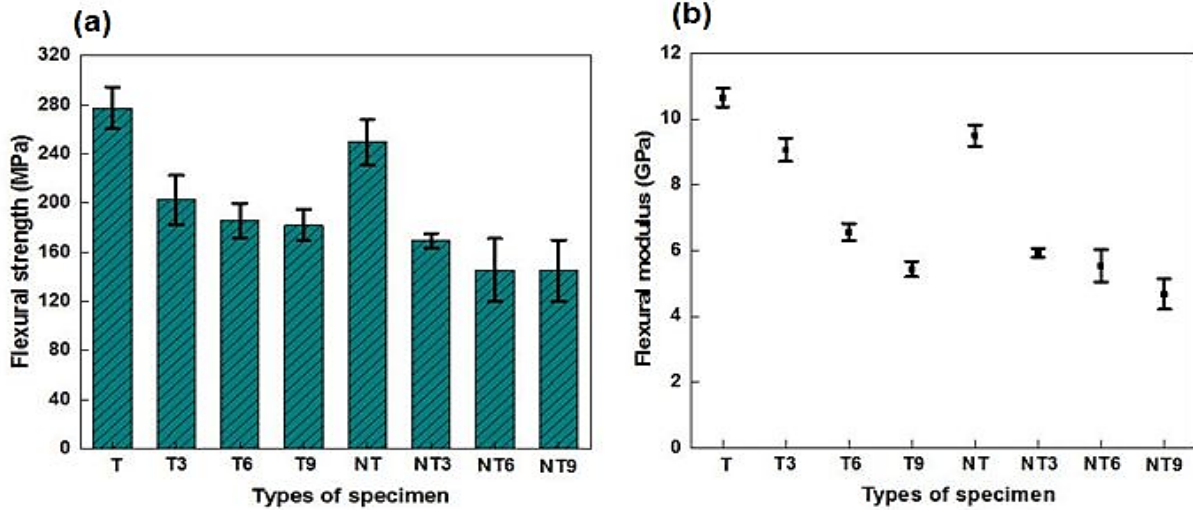


Figure 6. Kevlar/epoxy composites of a) flexural strength and b) flexural modulus.

### 3.4. Influence of aging in flexural

The hydrothermal aging process was the influencing factor for reducing of flexural behavior of composite. Figure 7 was concerned about the reduction of flexural strength in the percentage. After the hydrothermal aging untreated composites were 32.06%, 41.72%, and 41.94% for NT3, NT6, and NT9, respectively. The treated composite has process, the strength for treated composite comparison with non-hydrothermal aging composites were 26.9%, 33.03%, 34.38% for T3, T6, and T9, respectively. Consequently, the strength of improved interfacial characteristics lead to improve mechanical properties of composites

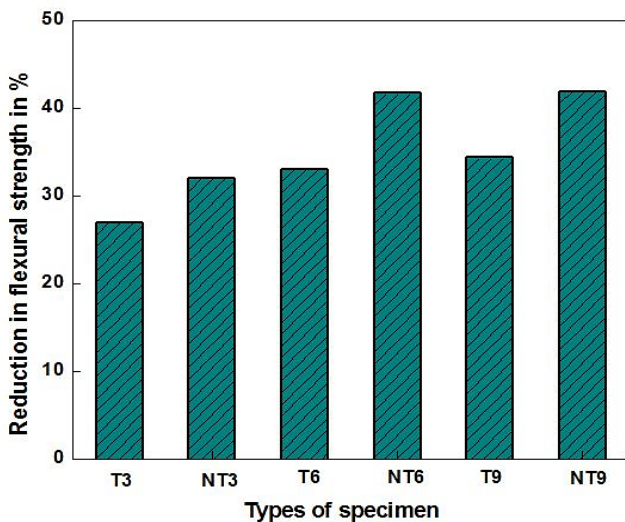


Figure 7. Reduction of the strength of hydrothermal aging composite.

### 4. Conclusion

The water absorption of treated and untreated Kevlar composite was discussed in this research. The results were concluded the followings:

- The water absorption results state that the absorption is less when the fiber is treated with epichlorohydrin compared to untreated fiber.
- During Hydrothermal aging, the attaining saturation state of absorption, there is a certain decrease in weight gain in composites. This was caused by the degradation of the fiber.
- The flexural strength of treated Kevlar composite was 10% higher than the untreated for the non-hydrothermal process as well as 16.3% higher than untreated for T3 aging process.
- The flexural modulus of treated Kevlar composite was 10.97 % higher than the untreated for non hydrothermal process as well as 34.65 % higher than untreated for T3 aging process.
- Thus, it was perceived that the treatment of fiber increases the performance of the composite at temperature and water immersion conditions.

## Conflict of interest

The author(s) declare no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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