



# THE EFFECT OF HOT WATER AGEING ON THE GLASS FIBER REINFORCED EPOXY COMPOSITE

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In the present study, the effects of hot water ageing on the glass fiber reinforced epoxy woven composites were experimentally examined. The specimens in three groups which undamaged, damaged, and single patchrepaired ones were kept in tap water at 50°C and 70°C temperature for 8 days and 16 days. At the end of those periods, the moisture absorption rates were calculated, the maximum failure loads at the end of period of keeping in hot water were determined and the failure modes occurring on these specimens were observed and compared. As a result of experimental study, it was found that the moisture absorption rates increased but the failure load values decreased together with the increase in water temperature and duration of hot water ageing.

Key words: *Composites, Single patch-repaired, Adhesive bonded, Hot water, Environmental degradation.* 

#### 1. Introduction

In the engineering practice, the use of composite materials became very popular in terms of light weight and strength. Opening holes on the composite plates causes stress accumulation in the application sites. Moreover, the use of bolts and pins at these sites is considered as the important parameters influencing the load-carrying capacity and the damage. For this reason, the adhesive bonds are preferred in many of the composite plates bonds. Also the adhesive bonds are frequently used because of the low-cost and no-damage on the connectors. As a result of damage of composite plates because of various reasons, the adhesive bonds are generally repaired by using the patches. It is known that the mechanical properties of composites and adhesives that are used vary depending on the environmental conditions, as well as the bond types and repairing methods. Especially the moisture and thermal environments have significant effects on the performance of composites. For this reason, many researchers have investigated the durability of adhesive-bonded composites.

In literature, the adhesive patch bonds [1-28] and the effects of environmental conditions on these bonds and composites have been experimentally examined in many studies. Especially the studies on using single-patch repair [1-10,12,14,15,17], the patch applied on specimen having hole damage at the center [1,2,15,16,19], repairing the damaged composite specimens, and the effects of moisture/temperature on the composite specimens [21,24,26-28,30] have a significant place in literature. Among the studies carried out on patch, Campilho et al. [1, 2] repaired the composite plate,

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which were holed at the center, by making use of patch on the single surface. They carried out an experimental study on examining the parameters influencing the patch thickness, effect of patch length, stress distribution, and mechanical behaviors of patch-repaired composite plates. Charalambides et al. [13] experimentally analyzed the damage behaviors of single patch-repaired composite plates, on which the patch is applied on only one side, caused from the external factors. Tsouvalis et al. [15] examined the efficiency of steel plates, which have hole damage at the center and been repaired by using carbon/epoxy patch, under the static tensile load. They determined that the failure load rates of patches increased from 30% to 50%. Her et al. [16] investigated the effects of material characteristics, size, and thickness of adhesives and patches on the stress distribution of damaged composite plates repaired by using patch. Liu et al. [19] applied a prepreg external patch on the perforated composite plate, and then they performed tensile test.

For the patch-repaired composite materials, Park et al. [21] examined the effects of temperature, moisture, and cold among the environmental factors on the single patch-repaired composite connections. Akderya et al. [26] experimentally investigated the effects of thermal ageing on the tensile properties of composites, joints of which were established by using adhesives. Soykok [27] examined the behaviors of single lap adhesively bonded glass fiber-reinforced epoxy composite specimens in the hot water. The prepared composites were kept in hot water for one or two weeks at 50°C, 70°C, and 90 °C, and then tensile test was applied. Zhang et al. [28] examined the effects of hot water on the adhesive bonds between the aluminum and steel surfaces.

This experimental study; it is important to expose undamaged, damaged and patch repaired glass fiber reinforced epoxy composite specimens to hot water from environmental conditions. Also, the effects of hot water were investigated. For this purpose, the composite specimens were kept in tap water at 50°C and 70°C for 8 and 16 days. Therefore study, the results obtained from the specimens in each of the three groups is important in that together. In this study, the moisture absorption performances, maximum and minimum load damages for pre- and post-absorption and failure modes were determined and compared.

#### 2. Materials and Methods

#### 2.1. Material preparation

The glass fiber-reinforced woven epoxy plates with 1m x1m size were produced by using a heat-controlled vacuum infusion machine. The specimens were cut from these composite materials at the geometries to be used in the present study. The composite specimens were divided into three groups; namely the undamaged specimens in first group (Figure 1), specimens with circular hole damage at the center with 5 mm and 10 mm diameter in second group (Figure 2a, Figure 2b). The specimens in second group were repaired by using patch on one side and then the third group was constituted (Figure 3). The width (w), thickness (t), and length (L) of specimens in each group were 30 mm, 1.7 mm, and 160 mm, respectively. The dimensions of composite patches made of the same material were 30 mm x 30 mm. Three specimens were produced for each group and the specimens were prepared in accordance with ASTM 5868-01 [29]. The thickness of adhesive was 0.3 mm and the epoxy adhesive Loctite 9466 was chosen as the adhesive material. While preparing the specimens in third group, the adhesion surfaces of specimens and those of patches were cleansed by using sandpaper and then wiped with swab soaked into acetone in order to improve the adhesion quality.

A specific mold for the specimens was prepared by using a 3D printer in accordance with the sizes of damaged specimen and patch, as well as the adhesive thickness (Figure 4). The damaged

composite specimen was placed into a mold and the patch was fastened on a single side by using Loctite 9466 adhesive, thus the repaired specimens were obtained (Figure 5). These specimens repaired by using adhesive and patch were kept at room temperature for drying for 15 days. Then the specimens were fixed at 50°C and 70°C temperature and then placed into thermostat-controlled boilers, which were filled with tap water, for 8 days and 16 days. The hangers were placed into the boilers in order to prevent the specimens from contacting each other (Figure 6).



Figure 1. Glass fiber-reinforced composite specimens-Undamaged (Group I).



Figure 2. Specimens with circular hole damage (Group II).



Figure 3. Single patch-repaired specimens (Group III).



Figure 4. Mold prepared for repaired specimens.



Figure 5. Repaired specimens.

[30]

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Figure 6. The specimens placed into water boilers fixed at 50°C and 70°C temperatures.

## 3. Results and Discussion

## **3.1.** Moisture absorption rates of specimens

Before and after placing them into hot water at 50°C and 70°C, the specimens were weighed individually. At the end of 8 and 16 days periods, the excessive water on the specimens taken out of hot water boilers was dried by using a swab. In order to measure the moisture absorption rates of specimens, a 0.01g-sensitive scale was used. The initial weights of specimens ( $w_0$ ) and the weights of specimens after the application ( $w_t$ ) were measured, and the moisture absorption rates of each sample ( $M_t$ ) were calculated using following formula.

#### $M_t = [(w_t-w_0)/w_0] \ge 100$

Figures 7 and 8 represent the moisture absorption rates of specimens in terms of the specimens geometries and duration of keeping in hot water. As can be seen from the figures, it is seen that moisture absorption rates are higher in specimens that are kept for 16 days than specimens that are kept for 8 days. However, when the temperature is 70 °C, the absorption rate is also increased. For example, the moisture absorption of undamaged specimens in 1st group kept in hot water at 50°C for 16 days increased by 46.15% when compared to the specimen kept under the same conditions for 8 days (Figure 7). The moisture absorption rates of specimens kept in hot water at 70°C for 16 days were 22.22% and it was higher than that of specimens kept under the same conditions for 8 days (Figure 8). The moisture absorption rates of specimens having Ø5 mm circular hole damage at the center in 2nd group kept in hot water at 50°C for 16 days were found to increase by 50% when compared to the specimens kept under the same conditions for 8 days (Figure 7). The moisture absorption rates of specimens having Ø10 mm circular hole damage at the center and kept in hot water at 70°C were found to increase by 41.66% (8 days) and 11.11% (16 days) when compared to the moisture absorptions observed after keeping the specimen at 50°C. The moisture absorption rates of single patch-repaired specimens having Ø5 mm circular hole damage at the center in 3rd group kept in hot water at 50°C for 16 days increased by 120% when compared to the specimens kept under the same conditions for 8 days (Figure 7). Similarly, the moisture absorption rates of single patch-repaired specimens having Ø10 mm damage in 3rd group kept in hot water at 70°C increased by 33.33% (for 8 days) and 9.52% (for 16 days) when compared to the specimens kept in hot water at 50°C.

This shows that environmental temperature plays a decisive role in the diffusibility of water molecules in fiber-reinforced composites. At higher temperatures, more filtration of the water into the

matrix of the resin takes place [27]. This is explained by the fact that the moisture absorption rates of the specimens kept in hot water at 70  $^{0}$ C are higher than those kept in hot water at 50  $^{0}$ C.



Figure 7. Moisture absorption rates of specimens kept in hot water at 50°C for 8 and 16 days.



Figure 8. Moisture absorption rates of specimens kept in hot water at 70°C for 8 and 16 days.

## 3.2. Maximum failure loads of specimens

The specimens were exposed to tensile test at 1mm/min speed by using Instron BS8801 device having the capacity of 100 kN (Figure 9). At the end of hot water ageing period, the excessive water on the surface of each specimen was dried by using a swab, and static tensile test was carried out within 1 hour. The maximum failure load values obtained from 3 specimen groups kept in hot water at 50°C and 70°C for 8 and 16 days were compared to the values obtained from dry specimens and the results of maximum failure load comparisons were presented in Figures 10 and 11. In addition, load-extansion curves of the specimes are given in Figures 12,13,14,15 and 16. The results of experiment were expressed as the average values obtained from three specimens.



Figure 9. Tensile test applied to the specimens.

As shown in the load-extension curves and Figures 10,11, the maximum failure loads of the specimens; it is seen that due to the increase in temperature and residence time in hot water, it is lower than the values obtained from dry specimens. For example; the maximum failure load values obtained from the undamaged specimens kept in hot water at 50°C and 70°C for 8 days were observed to decrease by 1.59% and 3.59%, respectively, when compared to the maximum failure load values obtained from the dry and undamaged specimens. Moreover, the maximum failure load values obtained from the undamaged specimens kept in hot water at 50°C and 70°C for 16 days were observed to decrease by 2.39% and 5.98%, respectively, when compared to the maximum failure load values obtained for the undamaged specimens (Figures 10,11 and 12).

The maximum failure load values of specimens having Ø5 mm circular hole damage at the center and kept in hot water at 50°C and 70°C for 8 days were found to decrease by 4.78% and 9.89%, respectively, when compared to the maximum failure load values obtained from dry specimens (Figures 10,11 and 13).



Figure 10. Maximum failure load values of specimens kept in hot water at 50°C.



Figure 11. Maximum failure load values of specimens kept in hot water at 70°C.

Moreover, the maximum failure load values of specimens having Ø10 mm circular hole damage at the center and kept in hot water at 50°C and 70°C for 8 days were found to decrease by 8.59% and 13.39%, respectively, when compared to the maximum failure load values of dry specimens (Figures 10,11 and 14).

The maximum failure load values of single patch-repaired specimens having Ø5 mm circular hole damage at the center and kept in hot water at 50°C and 70°C for 8 days were found to decrease by

4.44% and 13.45%, respectively, when compared to the maximum failure load values obtained from dry specimens (Figures 10,11 and 15).

The maximum failure load values of single patch-repaired specimens having  $\emptyset 10$  mm circular hole damage at the center and kept in hot water at 50°C and 70°C were observed to decrease by 9.87% and 4.02%, respectively, when compared to the values of specimens kept under the same conditions for 8 days (Figures 10,11 and 16).



Figure 12. Load-extension diagram of the undamaged specimens.

Polymeric matrixes are susceptible to swelling caused by water absorption in composite structures. This causes swelling stresses. These stresses are the cause of the formation of micro-cracks, particularly during the transition phase. In most cases, the degradation of a composite material during wet aging causes a phenomenon of water absorption due to temperature, hygrometric speed and the nature of the composite. The transport of water can be facilitated by differentiating within the matrix by imperfections in the matrix formed during manufacture or by capillarity along the fiber / matrix interface. As a result, these microfractures adversely affect the mechanical properties of the material [27].



Figure 13. Load-extension diagram of damaged specimens (Ø5).



Figure 14. Load-extension diagram of damaged specimens (Ø10).

Hydrothermal degradation due to temperature and moisture absorption at the fiber-matrix interface also leads to weakened fiber / matrix interface bonds. When these bonds are damaged, both the ability to transmit forces to the fibers and the homogeneity of the load distribution between the fibers are expected to be interrupted [27]. In this study, this is explained by the increase of the temperature of the water and the waiting times of the hot water and the decrease of the maximum load of the specimens.



Figure 15. Load-extension diagram of repaired specimens (Ø5).



Figure 16. Load-extension diagram of repaired specimens (Ø10).

## 3.3. Failure mode of specimens

The failure modes of specimens, which were kept in hot water at 50°C and 70°C, were observed at the end of tensile test and these modes are shown in Table 1.

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Waiting times	Ø5 damaged		Ø10 damaged		SPR-Ø5		SPR-Ø10	
	50°C	70°C	50°C	70°C	50°C	70°C	50°C	70°C
0	NT	NT	NT	NT	CF+LFT	CF+LFT	CF+LFT	CF+LFT
8 days	NT	NT	NT	NT	CF+LFT	CF+LFT	CF+LFT	CF+LFT
16 days	NT	NT	NT	NT	CF+LFT	CF+LFT	CF+LFT	CF+LFT

 Table 1. Observed failure modes

The failure modes of some of specimens are also illustrated in Figures 17 and 18. The Net-Tension failure mode was observed when the damaged specimens of 2nd group were kept dry and in hot water at 50°C and 70°C for 8 days (Figure 17) and 16 days (Table 1). Among the single patchrepaired specimens, it was determined that the mixed-type damages occurred, that the adhesive joint broke especially on single side (cohesive failure), on which the patch was attached, and that the other side was undamaged. In this cohesive failure site, also the fiber damage (LFT failure) was observed at low level on the adhesion layer of adhesive bond. On the side, on which no patch was attached, it was observed that the damage started around the hole, and that the net-tension damage occured but it did not split from the patch (Figure 18).



Figure 17. Net-Tension damage images of dry form of damaged specimens and specimens kept in hot water at 50°C/70°C for 8 days.



(a)

(b)



Figure 18. Damage images of patched (front) and unpatched (back) sides of repaired (Ø10, Ø5) specimens kept in hot water at 50°C and 70°C for 8 and 16 days.

## 4. Conclusions

In this study, the effects of hot water ageing on the woven glass fiber reinforced epoxy composites were examined experimentally. The undamaged, damaged, and repaired specimens were

kept in hot water at 50°C and 70°C for 8 and 16 days. At the end of hot water ageing period, the moisture absorption rates, maximum failure load values, and failure modes of specimens were determined and compared. At the end of this study, the following conclusions were made;

• It was found that the maximum failure load values of damaged specimens increased after repairing with patch. The resistance of damaged specimen having Ø5 mm circular hole damage at the center increased by 45.19% after being repaired with single patch. For the damaged specimens having Ø10 mm circular hole damage at the center, the strength increased by 57.89% after repairing by using single patch.

• The minimum failure loads were obtained from the specimens having Ø10 mm circular hole damage at the center, whereas the maximum failure loads were observed among the undamaged specimens.

• The specimens kept in hot water at 70°C for 16 days were observed to show maximum moisture absorption values when compared to the other environmental conditions.

• It was found that when the waiting times of composite samples in hot water and the temperature of water increased, the amount of moisture absorption increased but the failure load values were decreased.

• It was also seen that generally the same failure modes were observed among the specimens under all conditions.

## Nomenclature

SPR	: Single patch-repaired specimen
NT	: Net Tension
CF	: Cohesive Failure
LFT	: Light-Fiber Tear Failure

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