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Long-Term Tensile Properties of Natural Fibre-Reinforced Polymer Composites: Comparison of Flax and Glass Fibres

Dimo Hristozov [a], Laura Wroblewski [b], Pedram Sadeghian [c]

[a] *Department of Civil and Resource Engineering, Dalhousie University, 1360 Barrington Street, Halifax, NS, B3H 4R2, Canada.*

[b] *School of Science, Engineering and Technology, Penn State Harrisburg University, 777 W. Harrisburg Pike, Middletown, Middletown, PA 17057, USA.*

[c] *Department of Civil and Resource Engineering, Dalhousie University, 1360 Barrington Street, Halifax, NS, B3H 4R2, Canada. Email: Pedram.Sadeghian@dal.ca (corresponding author)*

ABSTRACT: Despite of significant research on the mechanical properties of fibre-reinforced polymer composites made of natural fibres, long-term performance of the natural fibre composites against moisture and other environmental conditions is not well-known. In this paper, accelerated durability tests were conducted in order to investigate the long-term performance of flax fibre composites in comparison with glass fibre composites. A total of 490 tensile specimens were prepared and exposed to four different environmental conditions namely dry heat, water (distilled), salt water (3.5% by weight), and alkaline (pH=12.5) solutions. The temperature of the solutions was controlled at 20, 50, and 60 degrees of Celsius (°C). Tensile tests were performed after 21, 42, 83, and 125 days exposure to the environmental conditions. Overall, the tensile strength of both flax and glass composites significantly decreased with increasing temperature and exposure time, however initial elastic modulus of both materials was not affected. Flax composites showed a bilinear stress-strain behavior and the secondary slope of the curve become slightly softer after the environmental conditionings, although glass composites almost retained their linear behavior. In terms of tensile

strength retention percentage, flax composites not only were not worse than glass composites but also they had even slightly better strength retention percentage.

KEYWORDS: A. Fibres; B. Environmental degradation; B. Strength; D. Mechanical testing.

1. INTRODUCTION

Fibre-reinforced polymer composites (hereafter called composites) are unique materials that take advantages of both fibre and polymer materials to achieve higher performance in desired direction in comparison with traditional homogeneous materials such as steel. It has already been established that fibre reinforcements elevate the mechanical properties of the polymer materials. Classic fibre materials for the composites are usually made of non-renewable resources (i.e. synthetic fibres), and typically emit significant greenhouse gases during production and fabrication. At the end of their service life, they are incinerated, sent to landfills, or recycled to materials with less quality, usually by energy-intensive methods. Thus, there is a major need to (i) limit the use of non-renewable materials by using more renewable ones, (ii) reduce pollution and energy costs of material production, and (iii) use materials that have more potential after service life or demolition. Considering synthetic materials' lack of renewability, this research was aimed at fibres from renewable resources. Natural fibres extracted from plants (e.g. flax, hemp, jute, and etc.) are good examples of renewable materials and offer several economic, technical, and ecological advantages over synthetic fibres [1][2][3][4][5].

Composites made of natural fibres (hereafter called natural fibre composites) have been extensively researched for the past two decades [6][7][8][9][10][11][12]. Due to the relative large quantity, low cost of raw material, low density, high specific properties, and positive environmental profile; natural fibres have been considered as prospective substitutes to synthetic fibres, specifically glass fibres. Despite of positive characteristics, natural fibres have some negative characteristics. They

are highly hydrophilic and their mechanical and physical properties are strongly dependent on the climate, location, and weather; so it is difficult to predict their respective composite properties and failure mechanism [13][14][15][16][17]. In terms of long-term performance of natural fibre composites, there are drawbacks with respect to their durability against moisture and other environmental conditions [18][19][20][21][22][23].

In the literature, there are limited studies on the long-term mechanical performance of natural fibre composites. Michel and Billington [24] studied the performance of Poly-Hydroxybutyrate (PHB) films and PHB/hemp composites after accelerated exposure to ultraviolet (UV) radiation, moisture, and temperature change. The weathering was applied in 74 measured cycles, with the procedures including moisture lasting a total of 1998 hours, and 384 hours for the heat and UV only procedures. The specimens were tested in tension to evaluate effect of the weathering on the mechanical properties. Both film (without fibre) and composite specimens showed a reduction in tensile strength after weathering. The film specimens displayed 30% decrease in the presence of moisture, and a 42% decrease in heat and UV alone. The tensile strength of the composite specimens were observed to decrease 47% with moisture, but only 25% with solely UV and heat.

Recently, Yan et al. [25] implemented accelerated UV weathering to study the durability of flax composites for civil engineering applications as an alternative to steel reinforcement. In tension, the strength and initial elastic modulus were reduced by 29.9% and 34.9% after weathering; respectively. In flexure, the strength and initial elastic modulus were reduced by 10.0% and 10.2%; respectively. The effect of weathering on tension properties had a much greater effect. More recently, Mak et al. [26] investigated the short- and long-term performances of flax composites and compared its behavior to glass composites. The impact of manufacturing method, namely wet layup and vacuum bag molding, and number of layers on short-term mechanical properties was also examined. Long-

term performance was determined through environmental aging in salt water containing 3.5% salt content by weight, where flax composite coupons were subjected to 23, 40, and 55 °C water for up to a maximum of 365 days. All mechanical properties and degradation were assessed through tension tests. Results showed that using the vacuum bag process, flax composites showed a strength and elastic modulus 18 and 36% higher, respectively, than wet layup specimens. After 365 days of conditioning at 23, 40 and 55 °C, wet layup specimens showed a strength retention of 81, 73, and 69%, respectively.

Despite the recent progress towards the understanding of the long-term performance of natural fibre composites, there are many gaps in the field. First of all, it is crucial to compare the overall performance of both natural and synthetic fibre materials, as degradation problems might not be limited to natural fibres. Moreover, the effect of temperature, moisture, and aging chemicals must be studied intensively to identify the source of degradations. It should be noted that direct comparison between the long-term performances of natural and synthetic fibre composites cannot be made in a quantitative way, since the composites may contain different volumes of fibres resulting different resin retention ratios. Although, the data obtained can be generally compared towards the applications of each material system.

From the literature, it has been reported that the fibre volume fraction influences the amount of moisture absorption and may enhance or worsen the matrix properties [27]. Porous nature of natural fibres at microstructure level can create more moisture path into the matrix leading to higher drying shrinkage and some enhancements [28]. However, higher fibre volume composites have greater moisture absorption and more degradation in mechanical properties are expected [29][30][31][32]. Therefore, for a natural fibre composite to be used outdoor, fibre content should be limited to attain

its intended long-term strength [27]. It should be noted that the effect of fibre content is beyond the scope of the current experimental research.

In this research, accelerated durability tests were conducted in order to investigate the long-term mechanical performance of composites made of flax fibres exposed to a variety of environmental conditions. For comparison, similar tests were conducted on composites made of glass fibres. The specimens were exposed to four different environmental conditions, namely dry heat, water, salt water, and alkaline solutions. The temperature of the specimens was controlled at three levels and the mechanical tensile tests were conducted after four different exposure durations to the environmental conditions.

2. EXPERIMENTAL PROGRAM

The sections below present a detailed description of test matrix, material properties, specimen fabrication, environmental conditioning, and test setup and instrumentation.

2.1. Test Matrix

A total of 490 specimens (245 flax and 245 glass composites) were prepared and tested under uniaxial tensile loading. The test matrix is shown in Table 1. The test parameters are: fibre type (flax vs. glass), environmental aging condition (dry heat and immersion in water, salt water, and alkaline solutions), exposure duration (21, 42, 83, and 125 days), and temperature during exposure (20, 50, and 60 °C).

2.2. Material Properties

For flax composites, a 275 g/m^2 stitched unidirectional flax fabric was used. The fabric was made of flax fibres with the density of 1.5 g/cm^3, diameter of 20 µm, tensile strength of 500 MPa, elastic modulus of 50 GPa, and rupture strain of 2% reported by manufacturer. For glass composites, a 955

g/m^2 stitched unidirectional glass fabric was used. For both flax and glass composites, a vinyl ester resin catalyzed with 1.25% methyl ethyl ketone peroxide (MEKP) was used. The resin cured at room temperature for 24 hours and post-cured for 2 hours at 138°C was reported by manufacturer to have the tensile strength of 82 MPa, elastic modulus of 3.72 GPa, and rupture strain of 7.9%.

2.3. Specimen Fabrication

Initially, composite sheets (500×500 mm) were made of two layers of either the flax or the glass fabrics. Wet-layup method using a vinyl ester resin was implemented as shown in Fig. 1(a). The composite sheets were cured for 7 days at room temperature and then were cut into 25 mm wide and 165 mm long strips using a precise band saw. Then, the strip-shaped specimens were pre-conditioned for a period of 48 hours in a circulating-air oven at a temperature of 50 °C. The purpose of the pre-conditioning was to ensure all specimens have the same moisture content before the environmental aging process as well as to measure the weight of the specimen at dry condition. After weighing, the specimens were ready to be aged in different environmental conditions. Based on the weight of the fabrics and composites, the fibre ratio (by weight) of the flax and glass specimens was calculated to the average of 0.28 and 0.58 with the standard deviation of 0.03 and 0.04, respectively. This implies a high resin ratio for the flax composites, which is compatible with the authors' observations regarding high resin absorption of flax fabrics during the composite fabrication.

2.4. Environmental Conditioning

The test specimens were immersed into three different solutions, namely water (W, distilled), salt water (SW, 3.5% salt by weight), and alkaline (AL, pH=12.5) solutions as shown in Fig. 1(b). In addition, control specimens were kept in an air dry (dry heat) condition for comparison. The

temperature of the specimens was controlled at three levels, namely 20 °C (room temperature), 50 °C, and 60 °C. The mechanical tests were conducted at 21, 42, 83, and 125 days (i.e. 500, 1000, 2000, and 3000 hours) exposure into the environmental conditions. For each case, five identical specimens were prepared and tested. At the time intervals, the specimens were taken out of the solutions, their surfaces were made dried using paper towel and immediately the weight of the surface-dried specimens were obtained. The changes in weight of the specimens were recorded over the exposure periods to study the moisture absorption of the materials.

The temperature and pH level of the solutions were also controlled during the environmental conditioning. For room temperature cases, the specimens were kept in containers in a cabinet in the laboratory. The temperature of the solutions in room temperature was measured three times when the containers were opened at the time of the mechanical testing. As the laboratory was air conditioned, the temperature was almost constant with the average of 20.2 °C and the standard deviation of 0.2 °C, which is why the room temperature condition is referred as 20 °C. The specimens and solutions were kept in sealed four-liter containers to control evaporation and exposure for maintaining the chemical concentration of the solutions. For the two high temperatures of 50 and 60 °C, the containers were kept in separate ovens with accurate temperature control system. The average temperature of the solutions in the two ovens was 50.7 and 60.6 °C with the standard deviation of 0.4 and 0.8 °C, respectively.

The salt water solution was prepared using distilled water and 3.5% NaCL by weight to model typical ocean water. The level of salt water solution in containers was marked to ensure that salt concentration is constant. As the containers were sealed properly, no evaporation was observed, even at 60 °C. For the alkaline solution, 19.6 gram/liter (g/l) KOH, 2.4 g/l NaOH, and 2.0 g/l $Ca(OH)_2$ were added to distilled water. The pH of the fresh alkaline solution was 13.2. The alkaline solution

containing the specimens was kept in sealed container to prevent evaporation and interaction with atmospheric CO_2. At each time interval of taking the specimens out of the solutions, the pH level of the solutions was measured using a pH meter, three times per container. The average pH of the alkaline solution at 20 °C during the period of 125 days was 13.10 with the standard deviation of 0.13. The average pH of the alkaline solution at 50 °C during the same period of 125 days was 12.20 with the standard deviation of 0.20. The average pH of the alkaline solution at 60 °C during the same period was 11.88 with the standard deviation of 0.21. As observed, the solutions at higher temperature were more inclined to lose their alkalinity. Overall, the average pH level of the alkaline solution used in this study was 12.5 with the standard deviation of 0.6.

2.5. Test Setup and Instrumentation

After the end of each environmental conditioning, the specimens were grinded into dumbbell-shaped specimens to be tested in tension based on ASTM D638 [33] standard as shown in Fig. 1(c). Before the mechanical testing, the specimens were post-conditioned for a period of 48 hours in a circulating-air oven at a temperature of 50 °C based on ASTM D618 [34] to make sure all specimens have the same moisture content at the time of the mechanical testing. Then the post-conditioned specimens were removed from the oven and were cooled to the room temperature for a period of at least 5 hours prior to the mechanical testing. A universal testing machine with a 25 mm gauge length extensometer sensor was used to provide a stress-strain curve for each specimen as shown in Fig. 1(d). The tests were displacement controlled with the rate of 2 mm/min. Axial load and stroke and the elongation of the gauge length were recorded at the rate of 10 Hz using the data acquisition system of the testing machine up to the rupture of each specimen in tension. A sample set of flax specimens after the tensile test is shown in Fig. 1(d).

3. RESULTS AND DISCUSSIONS

The sections below present a detailed discussion on weight change, tensile strength, elastic modulus, and stress-strain behavior of the flax and glass specimens exposed to the accelerated environmental conditions.

3.1. Weight Gain

The weight gain of glass and flax composite specimens can be compared in order to understand the difference in water absorption. This can help to understand the difference in long-term mechanical properties of the two materials. When flax specimens were placed into the solutions for different durations, there was usually a weight gain due to absorption. Fig. 2 shows the variation in weight gains of flax and glass specimens at three temperatures of 20, 50, and 60 °C after 21, 42, 83, and 125 days immersing in the solutions. The weight gains were calculated based on oven dry weight and saturated surface dry weight of each specimen. The weight gains are the average of five identical specimens and the bars in the figure show a standard deviation below and above the average values. The weight gain is obtained as follows:

$$M_t = \frac{W_t - W_d}{W_d} \times 100 \tag{1}$$

where M_t is the percentage of weight gain at time t, W_d is the weight of the dry specimen at time t= 0 and W_t is the weight of the saturated surface dry specimen at time t. The effects of each weathering solution are discussed in the sections below.

3.1.1. Effect of Water Solution on Weight Gain

As shown in Fig. 2(a), the weight gain percent of flax specimens in water solution at 20 °C was 6.30±1.03% after 21 days. It should be mentioned that 6.03% is the average value of the five identical specimens and 1.03% is the standard deviation of the five values as its range from the average is shown with the bars in the figure. For the specimens taken out after 42 days the weight gain percent was 7.37±1.38%. After 83 days the weight gain percent was 6.86±1.30% and after 125 days the weight gain percent was 6.08±2.28%. With a small variation, the weight gain in the water solutions at 20 °C was almost constant throughout the different time intervals. The weight gain percent of flax in water solution at 50 °C was 11.66±1.36% after 21 days. For the specimens taken out after 42 days the weight gain percent was 12.01±1.65%. After 83 days the weight gain percent was 12.87±1.22% and after 125 days the weight gain percent was 14.12±1.98%. The weight gain in the solutions at 50 °C increased as the exposure time increased. The weight gain percent of flax specimens in water solution at 60 °C was 10.15±0.91% after 21 days. For the specimens taken out at the 42 day interval the weight gain percent was 11.89±2.45%. After 83 days the weight gain percent was 11.35±1.26% and after 125 days the weight gain percent was 11.31±1.15%. The weight gain in the solutions at 60 °C increased in between 21 days and 42 days although the rest was almost constant. At 20 °C the average was about 6.6% throughout the different exposure durations. At 50 °C the average was 12.6% and at 60 °C 11.2%. Although the gain is lower at 60 °C than 50 °C it is still much greater than at 20 °C. The loss of mass due to higher degradation at higher temperature could be the reason of a lower gain at 60 °C, which is compatible with the faded color of the specimens. In general, it can be concluded that increased temperature has a significant factor in weight gain.

As shown in Fig. 2(b), the weight gain percent of glass specimens in water solution at 20 °C was 1.88±0.05% after 21 days. For the specimens taken out at the 42 day interval the weight gain percent was 1.93±0.28%. After 83 days the weight gain percent was 2.17±0.46% and after 125 days

the weight gain percent was 2.07±0.51%. The weight gain percent of glass specimens in water solution at 50 °C was 3.42±0.47% after 21 days. For the specimens taken out at the 42 day interval the weight gain percent was 4.42±0.27%. After 83 days the weight gain percent was 4.06±0.48% and after 125 days the weight gain percent was 4.92±1.21%. The weight gain percent of glass in water solution at 60 °C was 2.44±0.25% after 21 days. For the specimens taken out after 42 days the weight gain percent was 3.21±0.27%. After 83 days the weight gain percent was 3.91±0.67% and after 125 days the weight gain percent was 4.11±1.47%. By comparing Figs. 2(a) and 2(b), it can be seen that the effect of temperature on the weight gain of both flax and glass specimens had the same trend, however the weight gain in glass specimens is much lower than flax specimens. For example after 125 days in water at 20, 50, and 60 °C; the weight gain percentages of flax specimens were 2.94, 2.57, and 2.75 times of the weight gain percentages of glass specimens, respectively. Weight gain is mainly due to water absorption of specimens, however at high temperatures the rate of weight gain in water is slightly reduced due to the degradation of materials. The figures show that the highest weight gain percentage happens at 50 °C, then slightly lower at 60 °C, and then the lowest at 20 °C.

3.1.2. Effect of Salt Water Solution on Weight Gain

As shown in Fig. 3(a), the weight gain of flax specimens in salt water solution at 20 °C was 4.84±0.26% after 21 days, 7.85±1.11% after 42 days, 6.00±0.59% after 83 days, and 5.11±1.05% after 125 days. The weight gain of the specimens at 50 °C was 6.95±1.34% after 21 days, 10.36±1.12% after 42 days, 8.25±1.22% after 83 days, and 9.60±1.02% after 125 days. At 60 °C, the weight gain was 7.7±1.93 after 21 days, 9.49±0.62% after 42 days, 8.40±0.14% after 83 days, and finally 9.87±0.58% after 125 days. The results show that flax specimens in salt water experienced slightly lower weight gain than flax specimens in water, across all temperatures. As shown in Fig.

3(b), the weight gain of glass specimens in salt water solution at 20 °C was 1.57±0.30% after 21 days, 3.04±0.33% after 42 days, 1.52±0.32% after 83 days, and 1.73±0.22% after 125 days. The weight gain of the glass specimens at 50 °C was 1.84±0.25% after 21 days, 2.72±0.21% after 42 days, 1.39±0.09% after 83 days, and 1.79±0.13% after 125 days. At 60 °C, the weight gain was 1.62±0.35 after 21 days, 1.96±0.27% after 42 days, 1.54±0.17% after 83 days, and finally 1.69±0.37% after 125 days. Under the environmental condition of salt water, glass specimens showed weight gains significantly less than the glass specimens in water. The figures show that the water absorption of both flax and glass specimens in salt water solution is not as severe as distilled water. For example, after 125 days in salt water at 20, 50, and 60 °C, the weight gains of flax specimens were 84, 68, and 87% of the same materials in water, respectively. For glass specimens, the ratios at 20, 50, and 60 °C were 83, 36, and 41%, respectively.

3.1.3. Effect of Alkaline Solution on Weight Gain

As shown in Fig. 4(a), the weight gain of flax specimens in alkaline solution at 20 °C was 8.64±0.81% after 21 days, 8.95±1.01% after 42 days, 9.16±0.49% after 83 days, and 8.45±0.54% after 125 days. The weight gain of the specimens at 50 °C was 10.96±0.82% after 21 days, 13.88±0.73% after 42 days, 6.80±0.79% after 83 days, and 6.73±0.52% after 125 days. At 60 °C, the weight gain was 21.92±6.36 after 21 days, 7.18±4.98% after 42 days, 6.69±2.64% after 83 days, and finally 5.75±1.20% after 125 days. In the specimens exposed to alkaline solution there was a combination of gaining weight due to water absorption and losing weight due to the solution eating away the material. As presented, the maximum weight gain recorded after 21 days due to water absorption and then the rate was reduced due to dissolving the material in the alkaline solution. As shown in Fig. 4(b), the

weight gain of glass specimens in alkaline solution at 20 °C was 2.54±0.65% after 21 days, 2.66±0.59% after 42 days, 2.91±1.56% after 83 days, and 2.93±1.04% after 125 days.

The weight gain of the glass specimens at 50 °C was 4.47±1.26% after 21 days, 3.45±1.02% after 42 days, 2.75±1.98% after 83 days, and 1.34±0.54% after 125 days. At 60 °C, the weight gain was 3.88±1.155 after 21 days, 3.16±0.21% after 42 days, 2.09±0.32% after 83 days, and finally 1.71±0.58% after 125 days. During the first two durations of 21 days and 42 days the weight gain showed similar results as the water. This is due to water absorption only. The 50 and 60 °C have a much larger absorption. After 83 and 125 days the alkaline solution had enough time to eat away from the material at the higher temperatures therefore the weight giant is lower than 20 °C. The figures show that the weight gain of both flax and glass specimens in the alkaline solution had a peak. After the peak, the rate of weight lost due to alkaline was larger than the weight gain due to absorption. For example, after 125 days in the alkaline solution at 20, 50, and 60 °C, the weight gains of flax specimens were 139, 48, and 51% of the same materials in the water, respectively. For glass specimens, the ratios at 20, 50, and 60 °C were 141, 27, and 42%, respectively.

3.2. Tensile Strength

After four different environmental conditionings (i.e. air day, water, salt water, and alkaline conditions) at three different temperatures (i.e. 20, 50, and 60 °C) with four different exposure times (i.e. 21, 42, 63, and 125 days), the tensile strength of both flax and glass specimens was obtained through uniaxial tensile test. It should be mentioned that all specimens were made of two layers of unidirectional fabric. The average actual thickness of flax and glass specimens before exposure to the environmental solutions was 2.11 and 2.33 mm with standard deviation of 0.28 and 0.12 mm, respectively. For consistency, the nominal thicknesses of 1.0 mm and 1.5 mm were uses for flax and

glass specimens, respectively. The nominal thicknesses selected based on the actual thickness measurements of dry fabrics. The sections below present the detailed discussion about the results.

3.2.1. Effect of Air Dry Condition on Tensile Strength

The purpose of the air dry conditioning was to study the effect of heat without any moisture as well as to capture any heat effect on the curing of the composites. Fig. 5(a) shows the tensile strength of flax specimens in air dry condition before and after the conditioning. The average tensile strength of five identical control flax specimens was 207.42 MPa with the standard deviation of 11.99 MPa (207.42±11.99MPa). As shown in Fig. 4(a), the tensile strength of flax specimens in air dry condition at 20 °C was 193.00±13.72 MPa at 21 days, 185.55±17.57 at 42 days, 200.27±14.63 MPa at 83 days, and 186.55±6.74 at 125 days. The tensile strength of the specimens at 50 °C was 200.02±9.65 MPa at 21 days, 170.60±17.41 MPa at 42 days, 167.59±15.47 MPa at 83 days, and 168.38±10.96 MPa at 125 days. At 60 °C, the tensile strength was 160.86±14.57 MPa at 21 days, 174.62±9.91 MPa at 42 days, 168.34±16.08 MPa at 83 days, and finally 165.59±17.89 MPa at 125 days. Fig. 5(b) presents the tensile strength retention percentage (i.e. the tensile strength of aged specimen over the tensile strength of control specimen) of flax specimens. The results show that the tensile strength of flax specimens decreased with increasing both heat and exposure time. For example, after 125 days in the air dry condition at 20, 50, and 60 °C, the tensile strength retention of flax specimens was 90, 81, and 80%. Overall, the average tensile strength retention of flax specimens after 21 to 125 days in air dry was 86%.

Fig. 5(c) shows the tensile strength of glass specimens, where the tensile strength of five identical control glass specimens was 865.35±41.88 MPa. As shown in the figure, the tensile strength of glass specimens in air dry condition at 20 °C was 716.14±69.39 MPa at 21 days, 802.26±39.13

MPa at 42 days, 732.24±42.25 MPa at 83 days, and 756.96±59.59 MPa at 125 days. The tensile strength of the specimens at 50 °C was 765.38±50.35 MPa at 21 days, 796.99±62.64 MPa at 42 days, 833.48±19.02 MPa at 63 days, and 792.50±52.86 MPa at 125 days. At 60 °C, the tensile strength was 734.13±76.42 MPa at 21 days, 789.37±57.28 MPa at 42 days, 772.84±27.03 MPa at 83 days, and finally 803.00±25.63 MPa at 125 days. Fig. 5(d) presents the tensile strength retention percentage of glass specimens. The results show that the tensile strength of glass specimens slightly decreased with increasing both heat and exposure time. For example, after 125 days in the air dry condition at 20, 50, and 60 °C, the tensile strength retention of flax specimens was 87, 92, and 93%. It means despite of flax specimens, the dry heat reduced the tensile strength of glass specimens in short term, however in long term the strength increased. The increase can be due to the curing of the polymer at higher temperatures. Overall, the average tensile strength retention of glass specimens after 21 to 125 days in air dry was 90%, that is slightly higher that the value of 86% for flax specimens.

3.2.2. Effect of Water Solution on Tensile Strength

As presented in the Fig. 6(a), the tensile strength of flax specimens aged in water at 20 °C was 173.12±20.21 MPa at 21 days, 190.14±10.58 MPa at 42 days, 170.96±16.48 MPa at 83 days, and 181.69±12.34 MPa at 125 days. The tensile strength of flax specimens aged in water at 50 °C was 147.68±10.48 MPa at 21 days, 155.14±14.46 MPa at 42 days, 147.97±13.53 MPa at 62 days, and 146.36±5.05 MPa at 125 days. At 60 °C, the tensile strength was 144.28±9.29 MPa at 21 days, 142.46±4.88 MPa at 42 days, 137.73±14.31 MPa at 83 days, and finally 141.35±5.76 MPa at 125 days. Fig. 6(b) shows the tensile strength retention percentage of the flax specimens aged in water. The results indicate that the tensile strength of the specimens decreased with increasing heat, however after an initial drop at 21 days, the strength stayed almost constant up to the end of aging process. For

example, at 20, 50, and 60 °C at 21 days, the tensile strength retention of flax specimens was 83, 71, and 70 %, respectively. At the same temperatures at 125 days, the tensile strength retention was 88, 71, and 68%. Overall, the average tensile strength retention of flax specimens aged in water was 75%, which was 13% less than the average value of 86% for the same materials in the air dry condition.

As shown in the Fig. 6(c), the tensile strength of glass specimens aged in water at 20 °C was 739.26±36.61 MPa at 21 days, 782.43±22.73 MPa at 42 days, 735.01±45.29 MPa at 83 days, and 771.47±61.55 MPa at 125 days. The tensile strength of the specimens aged in water at 50 °C was 718.23±45.87 MPa at 21 days, 653.04±53.65 MPa at 42 days, 573.12±23.92 MPa at 83 days, and 502.29±41.36 MPa at 125 days. At 60 °C, tensile strength was 550.04±43.62 MPa at 21 days, 485.63±24.62 MPa at 42 days, 437.97±11.18 MPa at 83 days, and finally 386.15±14.26 MPa at 125 days. Fig. 6(d) presents the tensile strength retention percentage of glass specimens. The results show that the tensile strength of glass specimens significantly decreased with increasing both heat and exposure time. For example, after 125 days aging in water at 20, 50, and 60 °C, the tensile strength retention of glass specimens was 89, 58, and 45%. Overall, the average tensile strength retention of glass specimens after 21 to 125 days aging in water was 71% that is slightly less that the value of 75% for flax specimens in the same condition. It means the degradation of flax specimens in water was not worse than glass specimens.

3.2.3. Effect of Salt Water Solution on Tensile Strength

As shown in the Fig. 7(a), the tensile strength of flax specimens aged in salt water at 20 °C was 173.74±12.58 MPa at 21 days, 172.34±12.77 MPa at 42 days, 171.73±9.39 MPa at 83 days, and 167.17±15.14 MPa at 125 days. The tensile strength at 50 °C was 134.44±8.93 MPa at 21 days, 151.27±12.56 MPa at 42 days, 146.28±6.44 MPa at 83 days, and 142.36±13.62 MPa at 125 days. At

60 °C, the tensile strength was 146.29±5.03 MPa at 21 days, 189.69±29.82 MPa at 42 days, 135.94±8.59 MPa at 83 days, and finally 135.08±7.12 MPa at 125 days. Fig. 7(b) shows the tensile strength retention percentage of the flax specimens aged in salt water. The results shows an initial drop at 21 days and then the strength stayed almost constant up to the end of aging process. The bumps at 42 days seems anomaly, as there is no explanation for that. At 20, 50, and 60 °C at 125 days, the tensile strength retention was 81, 69, and 65%. Overall, the average tensile strength retention of flax specimens aged in salt water (21 to 125 days) was 75%, which is equal to the results of the same materials in water. Thus, there is no significant difference between water and salt water for flax specimens.

As shown in the Fig. 7(c), the tensile strength of glass specimens aged in salt water at 20 °C was 739.70±61.35 MPa at 21 days, 747.79±47.07 MPa at 42 days, 701.08±61.60 MPa at 83 days, and 763.65±56.58 MPa at 125 days. The tensile strength at 50 °C was 728.57±60.89 MPa at 21 days, 688.08±24.88 MPa at 42 days, 584.33±37.47 MPa at 83 days, and 517.05±27.07 MPa at 125 days. At 60 °C, the tensile strength was 591.13±51.65 MPa at 21 days, 499.37±47.69 MPa at 42 days, 481.91±22.04 MPa at 83 days, and finally 470.57±35.89 MPa at 125 days. Fig. 7(d) presents the tensile strength retention percentage of glass specimens aged in salt water. The results show that the tensile strength of glass specimens significantly decreased with increasing both heat and exposure time. For example, after 125 days aging in salt water at 20, 50, and 60 °C, the tensile strength retention of glass specimens was 88, 60, and 54%. Overall, the average tensile strength retention of glass specimens after 21 to 125 days aging in salt water was 72% that is slightly less that the value of 75% for flax specimens in the same condition. It means the degradation of flax specimens in salt water was not worse than glass specimens. In addition, there is no significant difference between aging in water and aging in salt water.

3.2.4. Effect of Alkaline Solution on Tensile Strength

As shown in the Fig. 8(a), the tensile strength of flax specimens aged in the alkaline solution at 20 °C was 164.99±6.17 MPa at 21 days, 151.75±20.64 MPa at 42 days, 141.13±9.24 MPa at 83 days, and 154.11±12.61 MPa at 125 days. The tensile strength at 50 °C was 138.31±5.27 MPa at 21 days, 116.44±12.16 MPa at 42 days, 126.98±6.62 MPa at 83 days, and 122.00±10.71 MPa at 125 days. At 60 °C, the tensile strength was 120.09±10.60 MPa at 21 days, 116.10±12.45 MPa at 42 days, 128.36±11.90 MPa at 83 days, and finally 101.93±6.35 MPa at 125 days. Fig. 8(b) shows the tensile strength retention percentage of the flax specimens aged in the alkaline solution. As same as aging in water and salt water, the results shows an initial drop at 21 days and then the strength remained almost constant up to the end of aging process. After the maximum exposure time (i.e. 125 days) at 20, 50, and 60 °C, the tensile strength retention was 74, 59, and 49%; respectively. Overall, the average tensile strength retention of flax specimens aged in the alkaline solution (21 to 125 days) was 64%, which is 85% of the overall results of the same materials in the water and salt water solutions.

As shown in the Fig. 8(c), the tensile strength of glass specimens aged in the alkaline solution at 20 °C was 722.30±38.79 MPa at 21 days, 691.09±68.06 MPa at 42 days, 621.30±85.02 MPa at 83 days, and 589.99±104.40 MPa at 125 days. The tensile strength at 50 °C was 634.30±31.72 MPa at 21 days, 506.56±43.14 MPa at 42 days, 418.45±11.29 MPa at 83 days, and 374.92±32.92 MPa at 125 days. At 60 °C, the tensile strength was 494.41±32.76 MPa at 21 days, 363.08±22.52 MPa at 42 days, 284.48±28.18 MPa at 83 days, and finally 255.27±17.86 MPa at 125 days. Fig. 8(d) shows that the tensile strength of glass specimens significantly decreased with increasing both heat and exposure time. For example, after 125 days at 20, 50, and 60 °C, the tensile strength retention was 68, 43, and 29%. Overall, the average tensile strength retention of glass specimens after 21 to 125 days aging in

the alkaline solution was 0.57% that is slightly less that the value of 64% for flax specimens in the same condition. It means the degradation of flax specimens in the alkaline solution was not worse than glass specimens.

In general, the tensile strength of the specimens aged in the solutions were always lower than the control specimens. Although glass specimens were much stronger than flax specimens under all conditions, the percent reduction of glass specimens was always greater than flax specimens. With the harshest condition at 60 °C after 125 days in the alkaline solution, the flax specimens were approximately at one half the strength while the glass specimens were at one fourth of the original strength.

3.3. Elastic Modulus

The elastic modulus or Young's modulus of both flax and glass specimens were calculated based on the slope of the initial linear portion of the stress-strain curve of the specimens in tension. The initial linear part was identified by the TestWork software which was used for collecting data from the universal testing machine. The elastic modulus of control flax specimens was 21.94±1.29 GPa which is almost half that of control glass specimens with the value of 37.37±1.31GPa. It should be mentioned that the results were based on testing five identical tensile specimens with two layers of the fabrics and the total nominal thickness of 1.0 mm for flax specimens and 1.5 mm for glass specimens.

3.3.1. Effect of Air Dry Condition on Elastic Modulus

As shown in Fig. 9(a), the elastic modulus of flax specimens in the air dry condition at 20 °C was 20.31±1.50 GPa at 21 days, 24.44±1.82 GPa at 42 days, 22.60±2.47 GPa at 83 days, and 25.67±2.87 GPa at 125 days. The elastic modulus in the same condition at 50 °C was 22.39±1.50 GPa at 21 days,

22.31±2.06 GPa at 42 days, 28.03±3.51 GPa at 83 days, and 24.55±1.10 GPa at 125 days. At 60 °C, it was 22.11±1.89 GPa at 21 days, 21.64±0.69 GPa at 42 days, 24.02±2.20 GPa at 83 days, and finally 25.59±1.21 GPa at 125 days. The results show that the elastic modulus increased slightly as the age of the specimens increased. For example after 125 days at 20, 50, and 60 °C, the elastic modulus of flax specimens increased 17.01, 11.93, and 16.67% with respect to the control specimens, respectively. This can be explained by the post-curing of the resin over time and the increasing of the cross-links density which is reflected in an increase of stiffness of the composites.

As shown in Fig. 9(b), the elastic modulus of glass specimens in the air dry condition at 20 °C was 36.98±1.40 GPa at 21 days, 39.88±1.72 GPa at 42 days, 42.52±2.30 GPa at 83 days, and 41.49±3.65 GPa at 125 days. The elastic modulus in the same condition at 50 °C was 37.45±0.71 GPa at 21 days, 39.03±2.17 GPa at 42 days, 41.24±2.03 GPa at 83 days, and 40.21±3.22 GPa at 125 days. At 60 °C, it was 37.27±2.41 GPa at 21 days, 39.45±1.24 GPa at 42 days, 40.43±1.55 GPa at 83 days, and finally 40.06±1.14 GPa at 125 days. Similar to flax specimens, there is an increase of elastic modulus for glass specimens, however the rate of increase is lower. For example after 125 days at 20, 50, and 60 °C, the elastic modulus of glass specimens increased 11.03, 7.61, and 7.21%, respectively. The lower increase can be explained by lower contribution of the resin in the stiffness of the composite due to higher stiffness of glass fibres with respect to flax fibres. In addition, fibre content of the flax specimens are less than that of the glass specimens.

3.3.2. Effect of Water Solution on Elastic Modulus

As shown in Fig. 10(a), the elastic modulus of flax specimens aged in the water solution at 20 °C was 20.28±1.78 GPa at 21 days, 19.96±1.65 GPa at 42 days, 18.65±3.44 GPa at 83 days, and 18.32±1.52 GPa at 125 days. The elastic modulus of flax specimens aged in the same solution at 50 °C was

17.77±1.88 GPa at 21 days, 20.53±1.26 GPa at 42 days, 18.48±1.97 GPa at 83 days, and 17.18±0.90 GPa at 125 days. At 60 °C, the elastic modulus was 19.58±1.76 GPa at 21 days, 20.99±1.39 GPa at 42 days, 18.75±2.23 GPa at 83 days, and finally 16.17±1.78 GPa at 125 days. The results show that the elastic modulus of flax specimens decreased slightly with the increasing of exposure time to the water solution. For example, after 125 days at 20, 50, and 60 °C, the elastic modulus decreased to 83.53, 78.32, and 73.71% that of the control specimens, respectively.

As shown in Fig. 10(b), the elastic modulus of glass specimens aged in the water solution at 20 °C was 38.10±3.46 GPa at 21 days, 40.93±2.79 GPa at 42 days, 38.14±2.03 GPa at 83 days, and 39.72±1.73 GPa at 125 days. The elastic modulus of glass specimens aged in the same solution at 50 °C was 38.62±1.73 GPa at 21 days, 38.47±2.64 GPa at 42 days, 39.21±1.43 GPa at 83 days, and 41.71±3.09 GPa at 125 days. At 60 °C, the elastic modulus was 39.39±4.26 GPa at 21 days, 38.58±1.26 GPa at 42 days, 39.11±2.82 GPa at 83 days, and finally 36.04±2.99 GPa at 125 days. The results show that in the water solution the elastic modulus of flax specimens slightly dropped over the different exposure times while for glass specimens it stayed almost the same.

3.3.3. Effect of Salt Water Solution on Elastic Modulus

As shown in Fig. 11(a), the elastic modulus of flax specimens aged in the salt water solution at 20 °C was 19.72±3.01 GPa at 21 days, 21.51±1.64 GPa at 42 days, 20.61±2.64 GPa at 83 days, and 19.45±1.57 GPa at 125 days. The elastic modulus at 50 °C was 19.75±0.93 GPa at 21 days, 18.49±1.63 GPa at 42 days, 19.13±1.36 GPa at 83 days, and 18.26±2.36 GPa at 125 days. At 60 °C, the elastic modulus was 18.18±1.53 GPa at 21 days, 25.64±3.36 GPa at 42 days, 18.86±0.70 GPa at 83 days, and finally 16.14±1.51 GPa at 125 days. The results show that the elastic modulus of flax specimens decreased slightly after aging in the salt water solution. For example, after 125 days at 20,

50, and 60 °C, the elastic modulus decreased to 88.67, 83.22, and 73.56% that of the control specimens, respectively. The variation of the elastic modulus of flax specimens aged in the salt water is very close to the same specimens aged in the water solution.

As shown in Fig. 11(b), the elastic modulus of glass specimens aged in the salt water solution at 20 °C was 35.26±0.99 GPa at 21 days, 38.59±0.85 GPa at 42 days, 37.31±3.02 GPa at 83 days, and 39.93±1.41 GPa at 125 days. The elastic modulus of glass specimens aged in the same solution at 50 °C was 37.92±1.63 GPa at 21 days, 38.73±1.86 GPa at 42 days, 37.71±1.01 GPa at 83 days, and 39.31±1.10 GPa at 125 days. At 60 °C, the elastic modulus was 36.05±1.91 GPa at 21 days, 38.05±1.31 GPa at 42 days, 39.08±1.49 GPa at 83 days, and finally 40.06±2.56 GPa at 125 days. By comparing the result, it can be concluded that after aging in the salt water solution the elastic modulus slightly decreased slightly for flax specimens throughout the different temperatures, while for glass specimens the elastic modulus stayed almost constant.

3.3.4. Effect of Alkaline Solution on Elastic Modulus

As shown in Fig. 12(a), the elastic modulus of flax specimens aged in the alkaline solution at 20 °C was 21.40±3.24 GPa at 21 days, 19.64±1.35 GPa at 42 days, 20.26±1.38 GPa at 83 days, and 19.12±0.94 GPa at 125 days. The elastic modulus of flax specimens aged in the same solution at 50 °C was 20.64±1.26 GPa at 21 days, 16.90±1.03 GPa at 42 days, 18.70±1.44 GPa at 83 days, and 15.44±1.59 GPa at 125 days. At 60 °C, the elastic modulus flax specimens aged in the same solution was 20.07±1.70 GPa at 21 days, 19.54±2.19 GPa at 42 days, 23.25±3.39 GPa at 83 days, and finally 16.94±2.79 GPa at 125 days. As presented, the elastic modulus of flax specimens decreased after aging in the alkaline solution. For example, after 125 days at 20, 50, and 60 °C, the elastic modulus decreased to 87.17, 70.37, and 77.24% that of the control specimens, respectively. The variation of

the elastic modulus of flax specimens aged in the alkaline is very similar to the same specimens aged in the water and salt water solution.

As shown in Fig. 12(b), the elastic modulus of glass specimens aged in the alkaline solution at 20 °C was 39.36±3.42 GPa at 21 days, 39.20±2.78 GPa at 42 days, 40.31±1.43 GPa at 83 days, and 38.11±1.73 GPa at 125 days. The elastic modulus of glass specimens aged in the same solution at 50 °C was 36.48±2.36 GPa at 21 days, 38.24±1.45 GPa at 42 days, 37.06±0.84 GPa at 83 days, and 39.47±5.55 GPa at 125 days. Finally at 60 °C, the elastic modulus was 38.47±3.27 GPa at 21 days, 36.62±1.84 GPa at 21 days, 37.27±2.25 GPa at 21 days, and 35.61±4.24 GPa at 21 days. By comparing the results, it can be concluded that the elastic modulus of glass specimens aged in the alkaline solution was stayed constant similar to that of water and salt water, however the elastic modulus of flax specimens averagely decreased to 87% of the control specimens over the period of 21 to 125 day exposure to all three solutions.

3.4. Stress-Strain Behavior

3.4.1. Stress-Strain Behavior of Control Specimens

Figure 13 presents the stress-strain curves of the control flax and glass specimens tested under uniaxial tension. Five identical specimens were tested and presented for each material. The stress was calculated based on the load, average width of the gauge length, and nominal thickness of the specimen. The widths of the specimen was constant over the gauge length of 25 mm, however the width was measured three times, at the middle and two sections ±12.5 mm off the mid-section, then the average was calculated. The strain was calculated using the elongation of the gauge length measured by a 25 mm extensometer. As shown in Fig. 13(a), flax specimens have a bilinear stress-strain curve, where the slope changes with a transition zone at the strain range 0.002 to 0.003 mm/mm

(i.e. 0.2-0.3%). All five specimens have similar stress-strain curves. The straight dotted line shows the average elastic modulus of the five identical specimens terminated at the maximum tensile strength. The secondary slope of the flax specimens was calculated 14.40±0.32 GPa, while the initial slope was obtained 21.94±1.29 GPa. The secondary slope is almost two-third of the initial slope. The intersection of the average initial and secondary slops is located at the stress of 55 MPa and at the strain of 0.0025 mm/mm (i.e. 0.25%). The rupture strain of flax specimens was 1.31±0.11%. Fig. 13(b) shows that glass specimens have a linear behavior throughout most of the graph as expected. The rupture strain of glass specimens was 2.54±0.15%. For both flax and glass specimens, at the break there is a sudden rupture which shows that both materials are brittle. The rupture of glass specimens was noisier than flax specimens.

3.4.2. Effect of Aging on Stress-Strain Behavior of Flax Specimens

Figure 14 shows the stress-strain curves of flax specimens after 125 days immersion into the water, salt water, and alkaline solutions at 20, 50, and 60 °C. For each case, the result of five identical specimens are presented. Comparing the curves to the control specims in Fig. 13(a) indicates that similar to the control specimens, the aged flax specimens have also a bilinear stress-strain behavior. However, the length of the initial linear part is become shorter by increasing the temperature of the solutions. While both the initial and secondary slopes are soften, the secondary slope is become softer by increasing the temperature. For the water and salt water solutions, the stress-strain curves of flax specimens are almost similar at the same temperature, however the curves related to the alkaline solution tend to convert from a bilinear shape to a nonlinear shape as the temperature increases. This shows that the alkaline solution greatly influenced the material, more than the water and salt water solutions.

3.4.3. Effect of Aging on Stress-Strain Behavior of Glass Specimens

Figure 15 shows the stress-strain curves of glass specimens after 125 days immersion into the water, salt water, and alkaline solutions at 20, 50, and 60 °C. Similar to flax specimens, the result of five identical specimens are presented for each case. Comparing the curves to the control specims in Fig. 13(b) indicates that similar to the control specimens, the aged glass specimens have also a linear stress-strain behavior. Overall, the stress-strain curves of glass specimens are become shorter by increasing the temperature of the solutions. However, the specimens aged in the alkaline solution show a little nonlinearity before the failure. As expected, the figure shows that the alkaline solution greatly influenced glass specimensa, much more than the water and salt water solutions. Despite of flax specimens, the glass specimens almost maintaned their stiffness after exposure to the heat and environmental conditions.

4. CONCLUSION

In this study, a total of 490 fibre-reinforced polymer composite specimens were prepared and tested under uniaxial tensile loading. All specimens were made of two layers of unidirectional flax/glass fabrics and vinyl-ester resin as a matrix for the fibres. The specimens were immersed into different environmental conditions, namely water, salt water (3.5% salt by weight), and alkaline solutions (pH=12.5). In addition, control specimens were kept in an air dry condition for comparison. The temperature of the specimens was controlled at three levels, namely 20, 50, and 60 °C. The mechanical tests were conducted after 21, 42, 83, and 125 days (i.e. 500, 1000, 2000, and 3000 hours) exposure to the environmental conditions. After 125 days of immersing in water at 20, 50 and 60 °C, the tests showed a weight gain of 2.1, 4.9, and 4.1% for glass specimens; and

6.1, 14.1, and 11.3% for flax specimens; respectively. For the specimens immersed in water and salt water the weight gain increased as temperature increased, however for the specimens immersed in alkaline that was not the case. The weight gain was predominately lower in glass specimens than it was with flax specimens. Moreover, the tensile strength, elastic modulus, and rupture strain were studied to determine the degree of deterioration of the composites. After 125 days of conditioning at 20, 50 and 60 °C, flax specimens showed a strength retention of 88, 71, and 68% in water; 81, 69, and 65% in salt water; and 74, 59, and 49% in alkaline; respectively. At the same conditions, glass specimens showed a strength retention of 89, 58 and 45% in water; 88, 60, and 54% in salt water; and 68, 43, and 30% in alkaline; respectively. Despite of almost three times water absorption of flax specimens in comparison with glass specimens, flax specimens showed slightly higher strength retention with an average of 23%. The results show that the long-term mechanical behavior of composites made of flax fibres is not worse than composites made of glass fibres.

ACKNOWLEDGEMENTS

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Table 1. Test matrix

Exposure (days/hrs)	Air dry (AD)			Water (W)			Salt water (SW)			Alkaline (AL)		
	20 °C	50 °C	60 °C	20 °C	50 °C	60 °C	20 °C	50 °C	60 °C	20 °C	50 °C	60 °C
Control	5	-	-	-	-	-	-	-	-	-	-	-
21/500	5	5	5	5	5	5	5	5	5	5	5	5
42/1000	5	5	5	5	5	5	5	5	5	5	5	5
83/2000	5	5	5	5	5	5	5	5	5	5	5	5
125/3000	5	5	5	5	5	5	5	5	5	5	5	5
Total	245 flax and 245 glass specimens											
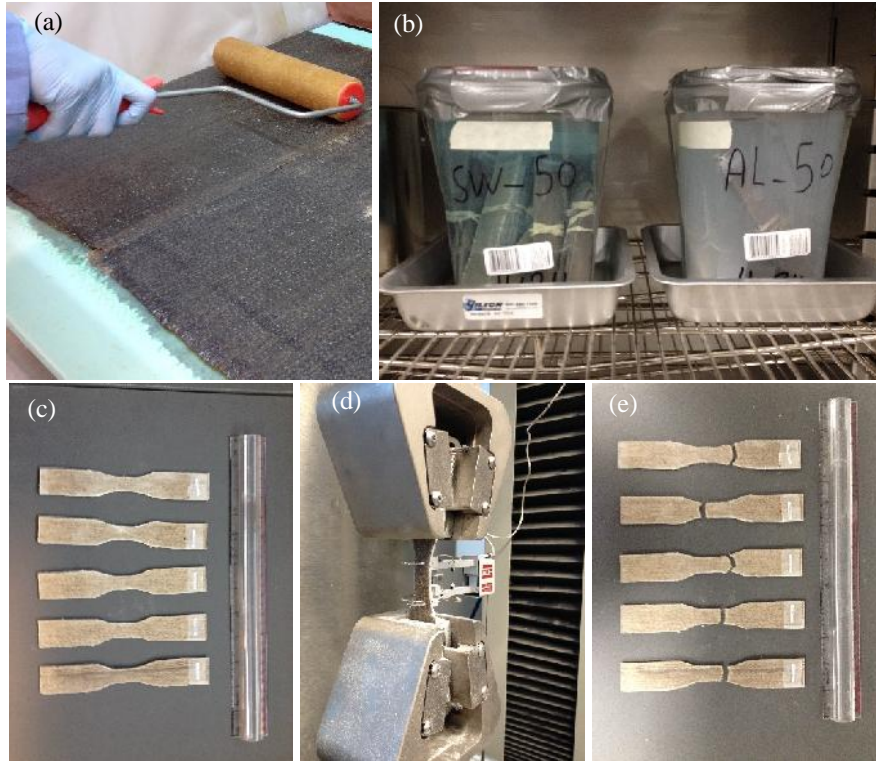
Figure 1. Specimen fabrication and testing: (a) wet lay-up method; (b) aging in solutions; (c) specimens before test; (d) test set-up; and (e) tested specimens.
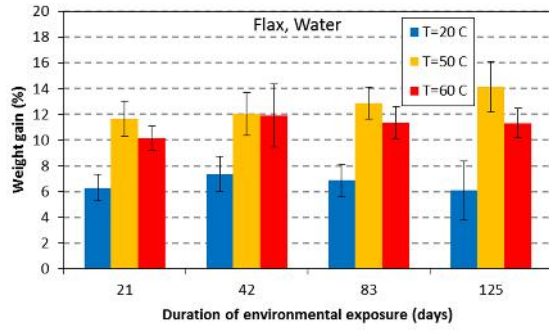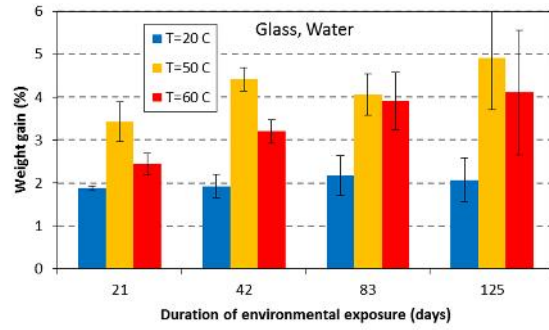
Figure 2. Variation in weight gain of (a) flax specimens and (b) glass specimens in water solution.
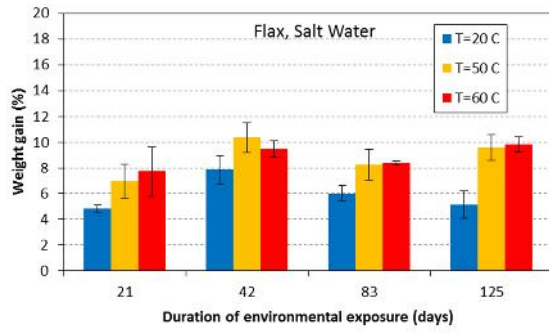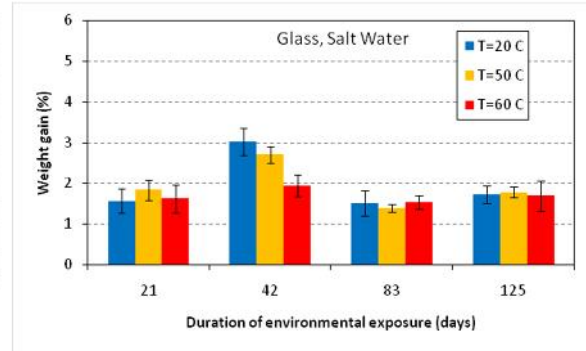
(a)   (b)

Figure 3. Variation in weight gain of (a) flax specimens and (b) glass specimens in salt water solution.
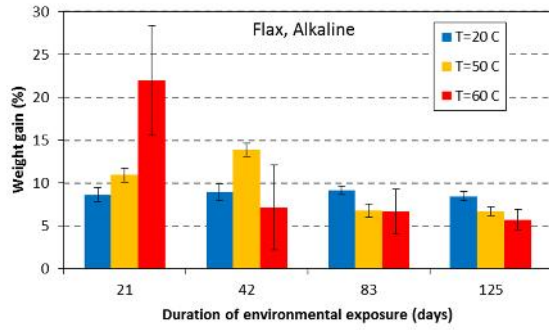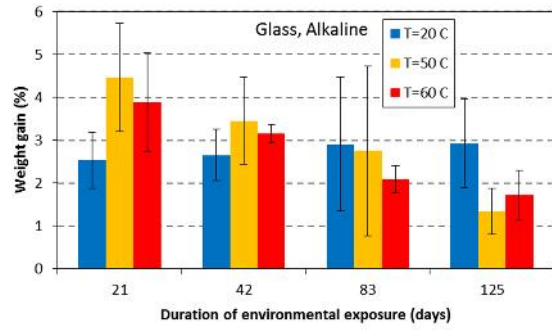
(a)   (b)

Figure 4. Variation in weight gain of (a) flax specimens and (b) glass specimens in alkaline solution.
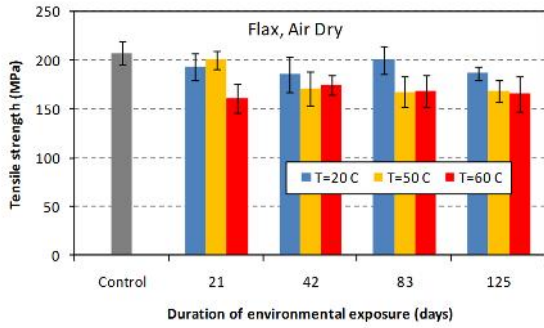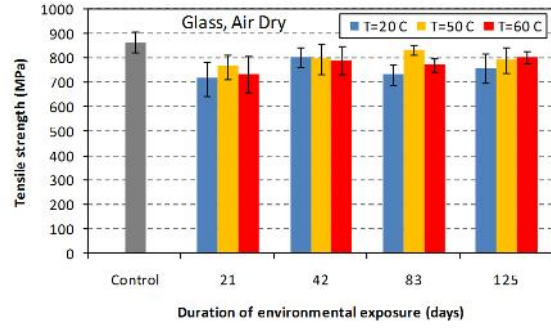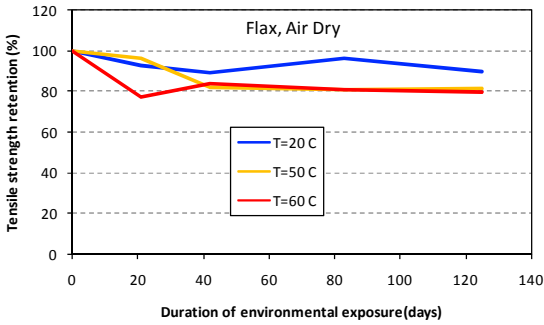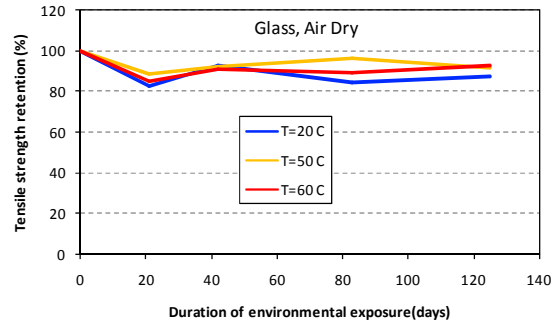
Figure 5. Effect of air day condition on: (a) tensile strength of flax specimens; (b) tensile strength retention of flax specimens; (c) tensile strength of glass specimens; and (d) tensile strength retention of glass specimens.
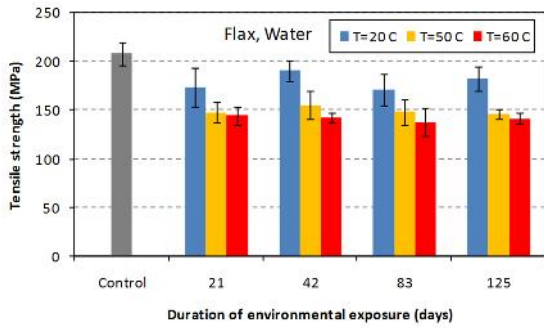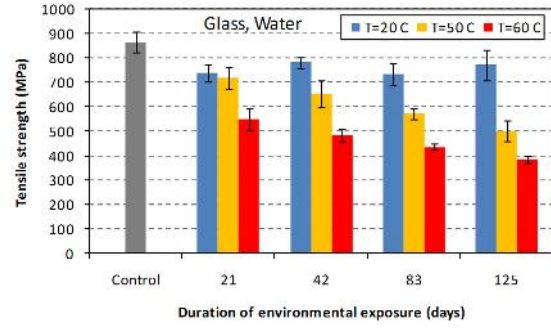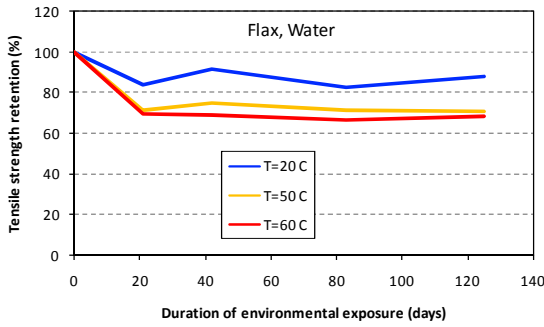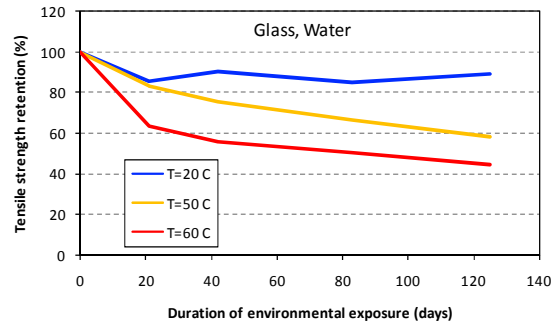
Figure 6. Effect of water solution on: (a) tensile strength of flax specimens; (b) tensile strength retention of flax specimens; (c) tensile strength of glass specimens; and (d) tensile strength retention of glass specimens.
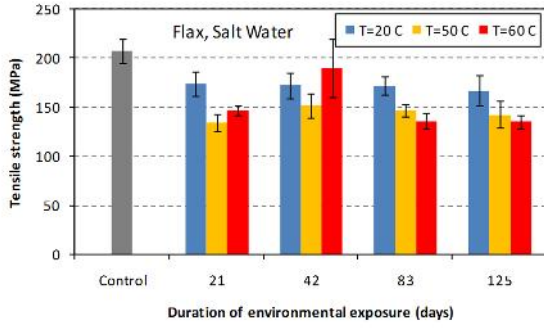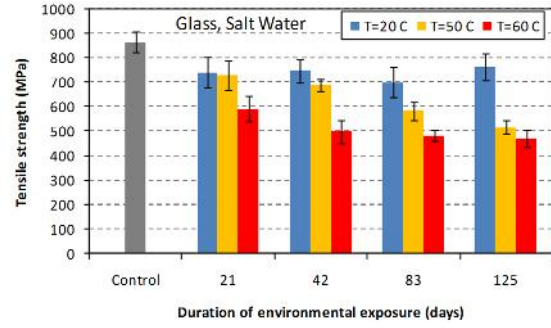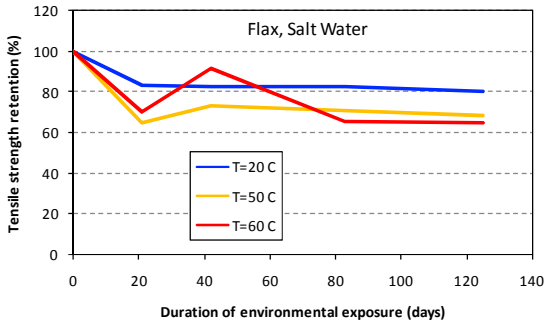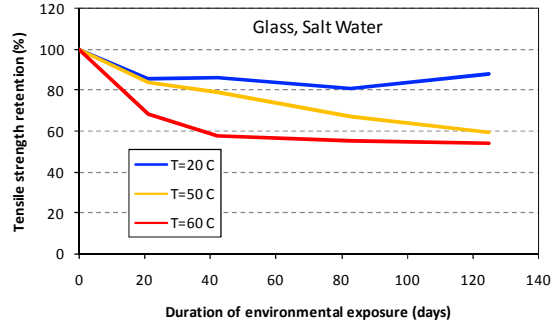
Figure 7. Effect of salt water solution on: (a) tensile strength of flax specimens; (b) tensile strength retention of flax specimens; (c) tensile strength of glass specimens; and (d) tensile strength retention of glass specimens.
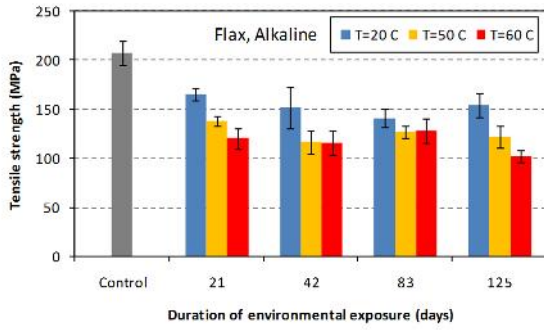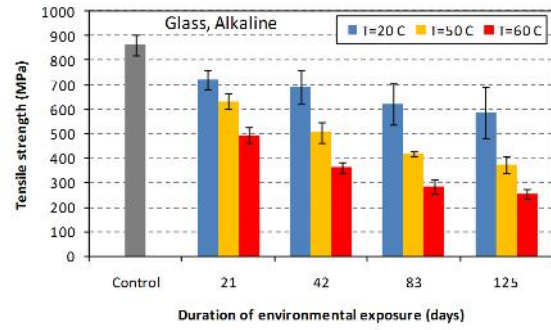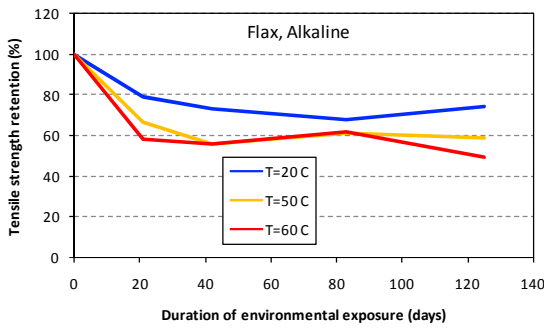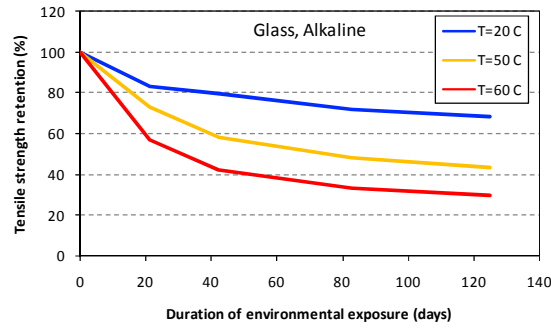
Figure 8. Effect of alkaline solution on: (a) tensile strength of flax specimens; (b) tensile strength retention of flax specimens; (c) tensile strength of glass specimens; and (d) tensile strength retention of glass specimens.
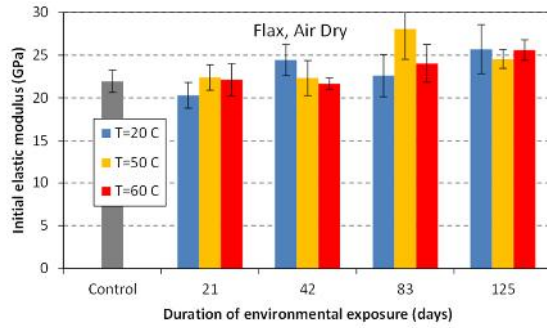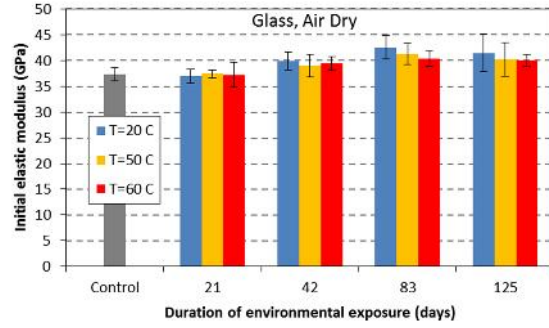
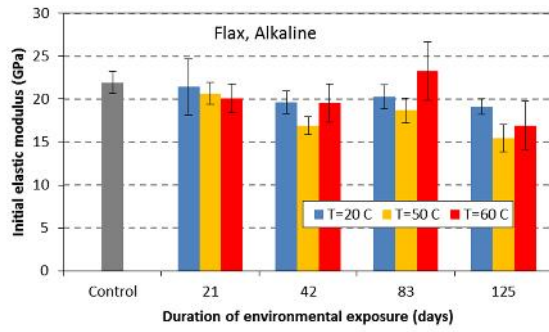(a)   (b)

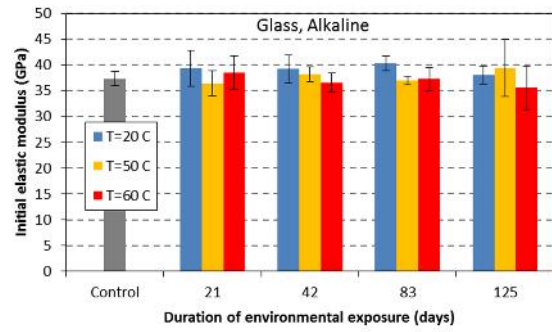**Figure 9. Variation in elastic modulus of (a) flax specimens and (b) glass specimens aged in air dry condition.**
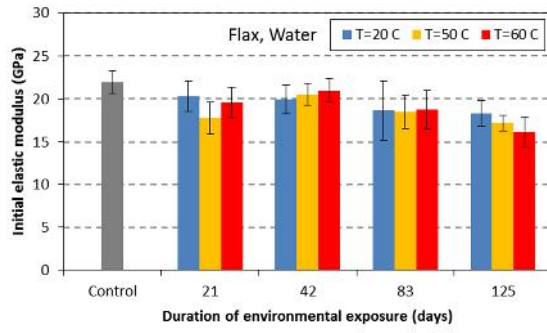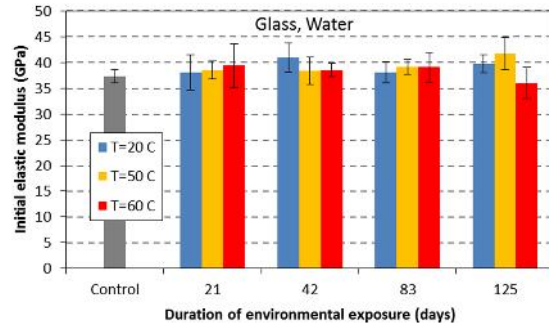
Figure 10. Variation in elastic modulus of (a) flax specimens and (b) glass specimens aged in water solution.
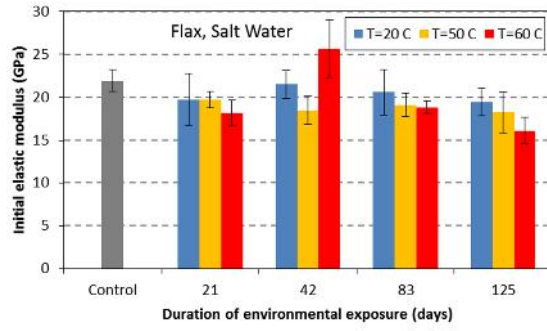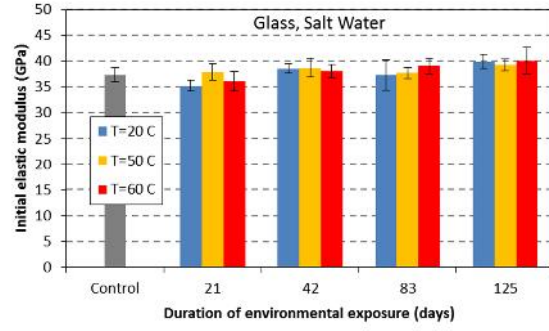
Figure 11. Variation in elastic modulus of (a) flax specimens and (b) glass specimens aged in salt water solution.
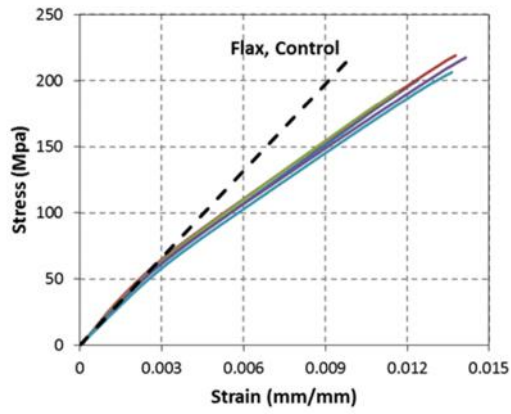
(a)
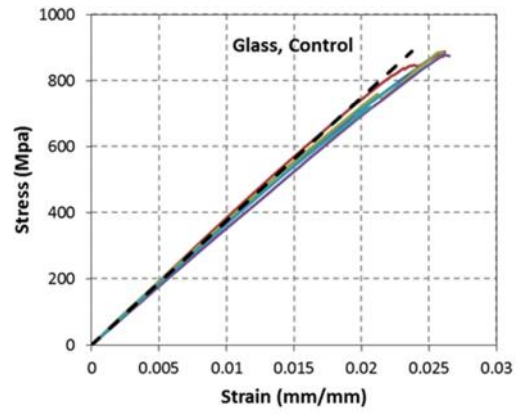

(b)

Figure 12. Variation in elastic modulus of (a) flax specimens and (b) glass specimens aged in alkaline solution.

(a)   (b)

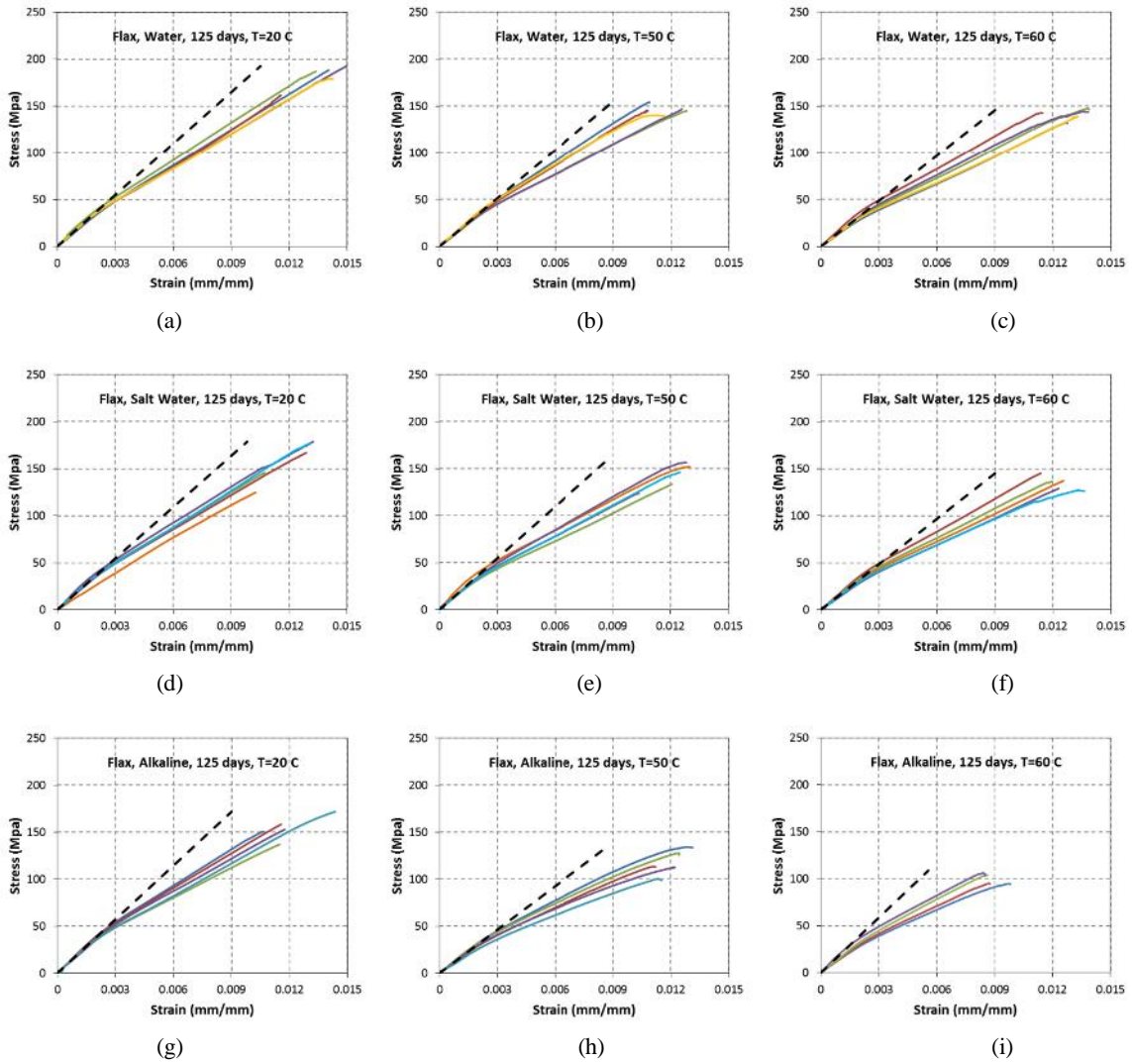**Figure 13. Stress-strain behavior of control (a) flax and (b) glass specimens.**

Figure 14. Stress-strain behavior of flax specimens aged 125 days in: (a) 20 °C water solution; (b) 50 °C water solution; (c) 60 °C water solution; (d) 20 °C salt water solution; (e) 50 °C salt water solution; (f) 60 °C salt water solution; (g) 20 °C alkaline solution; (h) 50 °C alkaline solution; and (i) 60 °C alkaline solution.
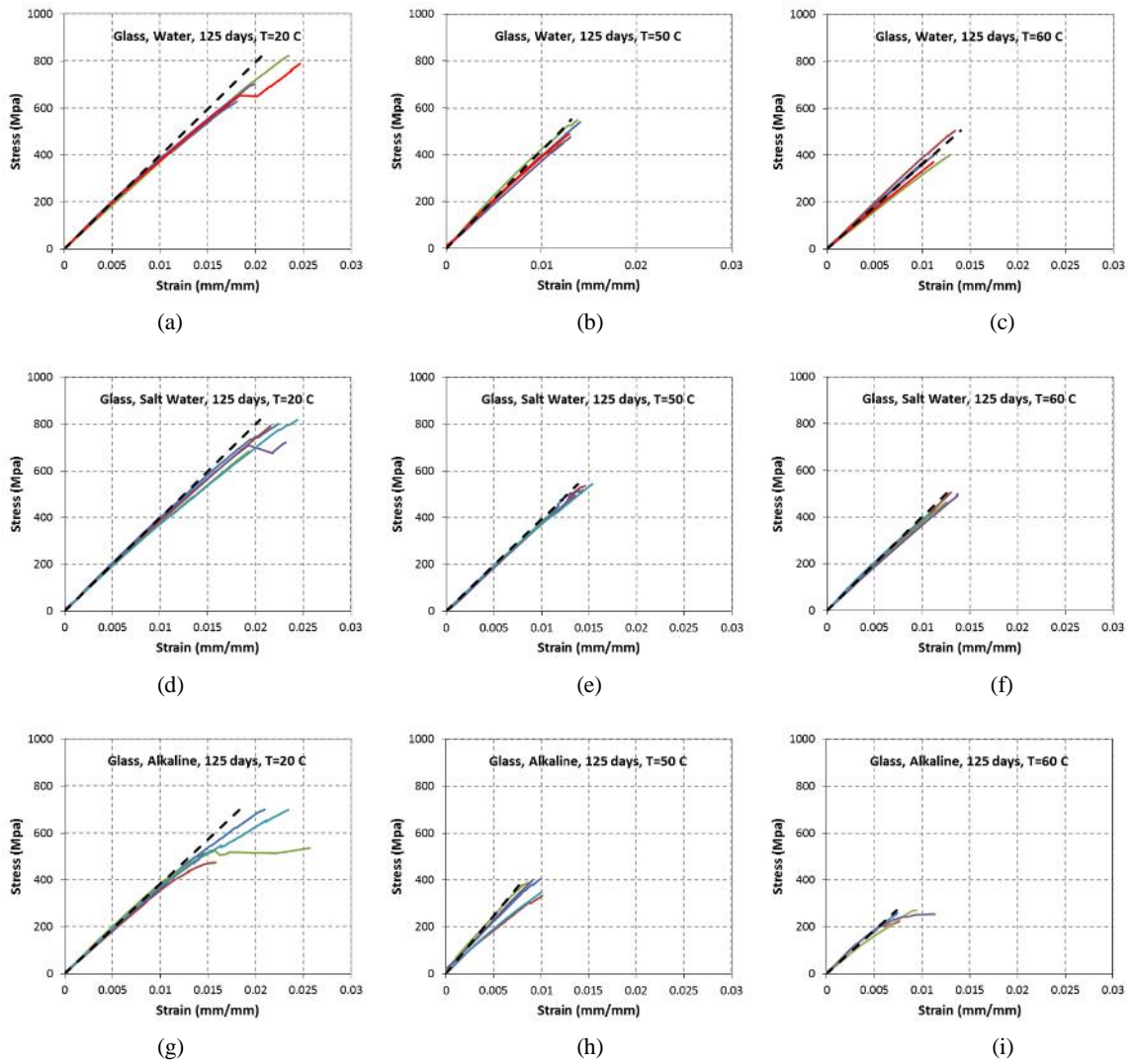
Figure 15. Stress-strain behavior of glass specimens aged 125 days in: (a) 20 °C water solution; (b) 50 °C water solution; (c) 60 °C water solution; (d) 20 °C salt water solution; (e) 50 °C salt water solution; (f) 60 °C salt water solution; (g) 20 °C alkaline solution; (h) 50 °C alkaline solution; and (i) 60 °C alkaline solution.