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The Study of the Glass Fiber Surface Treatment Effect on the Base Strength Characteristics of the Composite Material

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Abstract. *This article examines the impact of surface treatment of glass fibers on the strength of a composite material with an unsaturated polyester polymer matrix reinforced with glass fibers. The strength tests conducted include tensile, bending, and impact strength tests, as well as weight loss measurements. The research was conducted in two stages: in the first stage, the time was kept constant while the fibers were treated with varying concentrations of an alkaline NaOH solution; in the second stage, the concentration was fixed and the variable parameter was the treatment time. The results of the study indicate that surface treatment of glass fibers significantly improves their adhesion to the matrix materials, resulting in improved strength test results for the composite samples. The impact resistance, bending resistance, and tensile strength values all increased compared to the reference samples. However, certain changes in the individual parameters of fiber processing led to a slight decrease in tensile strength when it fell below the reference values. Additionally, it was observed that as the concentration of the solution and treatment time increased, the weight of the fibers decreased.*

Keywords: *composite materials, polyester, fiberglass, surface treatment*

1. Introduction

Unlike other structural materials, polymer-based composite materials have a lightweight design with relatively high strength. Additionally, they exhibit resistance to various chemicals and corrosion.

Fiber-reinforced polymer materials, among the range of composite materials available, have found extensive application in the field of mechanical engineering. They are also widely utilized in automotive and aircraft manufacturing, shipbuilding, construction, as well as in the production of concrete and pipes [1, 2]

The properties of composite materials are directly influenced by the types of matrix material and reinforcing material they contain. The mechanical properties of the composite are greatly influenced by the process of bonding the reinforcing material with the matrix, and the binder also plays a crucial role in redistributing stress from external loads on the reinforcing material. As a result, several studies have been conducted to investigate the impact of surface treatment on fibers and their adhesion to the binder, as they bear the majority of the applied load [3, 4].

The study [5] demonstrates that treating Roystonea regia and banana fibers enhances the tensile strength, modulus of elasticity, and stiffness in composite materials. It also improves resistance to shock loads, thermal stability, and reduces the tendency to fracture under cyclic loading, as compared to materials containing untreated fibers.

The study [6] demonstrates that the mechanical properties of composite materials, such as tensile strength, elastic modulus, elongation at break, and adhesion between fibers and the matrix, are significantly improved after treating the surface of natural fibers using various physico-chemical methods, such as laser or plasma treatment.

The study [7] demonstrates that different surface treatment methods or chemical modifications of fiberglass can decrease the environmental impact on the composite material. This enhances the adhesion between the fiberglass and the matrix, reduces moisture absorption and corrosion, resulting in improved strength, stability, and preservation of mechanical properties during operation.

The objective of this study is to investigate the impact of surface treatment on glass fibers and its effect on the strength properties of a composite material. The composite material consists of an un-

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saturated polyester polymer matrix reinforced with glass fibers. The study will focus on conducting effect on the strength properties of a composite material. The composite material consists of an unsaturated polyester polymer matrix reinforced with glass fibers. The study will focus on conducting tensile, bending, and impact strength tests, as well as evaluating any weight loss that occurs.

2. Methods and materials

In this study, the main polymer material used for the matrix is unsaturated polyester resin manufactured by SIR (Kingdom of Saudi Arabia). Specifically, it belongs to the type of isophthalic polyesters and has the following mechanical properties: a longitudinal density of 1200 kg/m^3, elongation of 3.2%, tensile strength of 42 MPa, and a modulus of elasticity of 2.8 GPa. At room temperature, this polymer appears as a transparent viscous pink liquid and falls under the category of molten thermosets, with a 20% styrene monomer content.

2.1. Material reinforcement

Fiberglass (E-glass) (Qinhuangdao Guangyu Fiberglass Co. China) was utilized as the reinforcing material, in the form of continuous bundles cut to a length of 16 cm. It has a longitudinal density of 2500 kg/m^3, an average fiber diameter of 20.14 μm, elongation, 0.01%, tensile strength 2400 MPa, modulus of elasticity 85 GPa, and a volumetric density of 2.5 g/cm^3.

Additional substances:

For better hardening of polyester the following substances were additionally used:

Cobalt was used as a 2% weight accelerator in the polyester Akcobalt 12 (II ethyl hexanoate). from AKPA (Turkey), which has the following characteristics: Brookfield viscosity (at 20°C) - 57 mPa.s, density - 1.01 g/cm^3, metal content - 12%, solvent - toluene, flash point - 34°C.

The catalyst used as the initiator of the process was Butanox M50 (Methyl ethyl keton peroxide) from Azkonobel (Belgium) at 2% weight of polyester, with the following main characteristics: mixture density - 1.18 g/cm^3, dynamic viscosity at 25°C - 24 mPa•s, peroxide content - 30%, active oxygen - 9%, water content not exceeding 3%, self-accelerating decomposition temperature - 60°C.

NaOH was used as an alkaline material for fiber processing, with the following properties: molar mass - 39.997 g/mol, density - 2.13 g/cm³, melting point - 323°C, boiling point - 1403°C, enthalpy of formation - 425.6 kJ/mol, vapor pressure - 0 ± 1 mm Hg, solubility in water - 108.7 g/100 mL, dissolved in distilled water with different proportions

Distilled water was used, which has the main characteristics of complete transparency and colorlessness, elevated boiling point -110°C, freezing point - about -10°C, salt and trace element content ranging 2 mg/L, hardness (with a soft composition) – 1.5 mg-equiv./l, and is 1 g/mL, *p*H level ranging 5.5.

2.2. The fiber processing method and sample preparation

The fibers underwent a two-stage processing using an alkali solution, with each stage having varying parameters.

At the first stage, the concentration of the NaOH solution in distilled water was taken as a variable, meaning the *p*H was 9, 10,11 and 12 at a constant time of 3 h.

In the second stage, the processing time was varied. The durations used were 0.5, 1, 1.5, 2, and 2.5 h, while maintaining a constant *p*H value of 10. The *p*H level was monitored and controlled using a pH meter. The *p*H value indicates the concentration of active hydroxide ions in the solution. A higher *p*H value corresponds to a higher concentration of hydroxide ions and a higher reaction efficiency.

After processing the fibers in an alkali solution, they are rinsed under a water shower for 5 min and subsequently dried in a oven at a temperature of 70°C for two hours. Once dried, the fibers are ready for sample formation and preparation.

For various studies the samples were made by casting and were divided into the following groups:

- for tensile tests the samples were made with dimensions of 165x19x10 mm in accordance with ASTM 638;
- for bending tests the samples were made with dimensions of 160x15x8 mm in accordance with ASTM D 790;
- for impact strength tests the samples were made with dimensions of 10x10x55 mm in accordance with ASTM 256.

The molds were made of carbon steel coated with zinc according to the specified dimensions and treated with a special preservative for the convenience of removing the samples from the molds after the casting process.

Figure 1. Steel molds used for the manufacture of samples

The process of forming samples begins by adding 2% cobalt hardener to the unsaturated polyester and thoroughly mixing the mixture for 5-7 min. Then, a catalyst is added and mixed again until the mixture becomes homogeneous. The mixture is then poured into molds, ensuring even filling. To form each sample, fibers of a specific length are added. The pouring process is carried out in two stages, creating two layers of fibers in each sample. The weight ratio of the reinforcing material to the main material is 13%.

After pouring the mixture into molds, its viscosity increases until it reaches a gelatinous state, at which point its temperature rises to approximately 40°C. As time passes, the mixture begins to solidify, and its temperature continues to rise, reaching its peak at 110°C after approximately 25 min. The samples are removed from the molds using the ejection method approximately one hour after the casting process, once the polyester has fully hardened.

Figure 2. Some tensile and bending samples prepared for testing

2.3. Study methods

Initially, reference samples were prepared and tested to obtain corresponding results.

After that the samples were made, using glass fibers treated with an alkaline NaOH solution in distilled water with a variable *p*H (9,10,11 and 12) at a constant time of 3 h. These samples were then tested for tension and the results were obtained.

Then the samples were prepared in which the reinforcing material was treated with an aqueous alkaline NaOH solution with a constant *p*H 10 at a variable time: 0.5, 1, 1.5, 2 and 2.5 h, respectively. The samples were tested and the results were obtained.

The mass loss test was performed on samples with fibers that were treated with aqueous alkaline NaOH solution at varying *p*H (9, 10, 11, and 12) for a constant duration of 3 h. Additionally, samples were treated with an alkaline NaOH solution in distilled water at a constant *p*H of 10, but with varying treatment times: 0.5, 1, 1.5, 2, and 2.5 h. Throughout the experiments, the mass changes resulting from the alkaline fiber treatment were continuously monitored.

2.4 Conducting tests

To investigate the impact of surface treatment on the tensile strength of composite materials, both tensile strength tests and weight loss tests were conducted, accompanied by microphotography of the fiber surfaces.

2.4.1 The tensile test

The tensile strength tests were performed using the Olsen 50 kN tensile testing device, following the ASTM 638 standard. The device was connected to a computer program, which provided the "stress-strain" curve and the "strength-elongation" curve.

In addition to the calculation of the modulus of elasticity (E), the program also yielded results for the maximum stress the sample could withstand (S_{max}), yield strength, and elongation.

The durability of the sample (T) was calculated by the following equation:

$$T = \frac{\lfloor 0{,}5 \cdot \left(f_y + f_{\max} \right) \cdot \Delta L \rfloor}{V},$$

where:

f_y - yield load, N;

$f_{\max}$ - maximum sample load, N;

ΔL - total elongation, mm;

V - sample volume, cm^3.

2.4.2. The resistance moment test

The resistance moment test of the samples was conducted using the Olsen 50 kN Tensile Device in accordance with the ASTM D790 standard by the three-point bending method.

The maximum bending load was obtained from the «force – bending» diagram, and then the bending strength was calculated using the following ratio:

$$\sigma = \frac{F \cdot L \cdot 3}{2 \cdot w \cdot t^2},$$

where:

F- the load (force) at the fracture point, N,

L – sample length, mm;

t – the thickness of the sample, mm;

w – the width of the sample, mm.

2.4.3. The impact strength test

To conduct these tests, unlabeled samples were prepared in accordance with the ASTM 256 standard using a Sharpy device. The absorbed energy required for destruction was directly obtained from the impact device during the study.

The study of impact strength is a practical method that provides a descriptive understanding of material strength and resistance to destruction under high stresses. Impact strength is calculated using the following equation:

Impact strength = absorbed energy/sample surface area, J/cm^2.

2.4.4. The weight loss test

To observe the effect of surface processing of the reinforcing material on weight change, electronic scales were used with an accuracy of 0.0001, and weight loss was calculated according to the following dependence [8]:

$$\Delta W = \frac{W_1 - W_2}{W_1} \cdot 100,\%$$

where: W_1- fiber weight before processing, g; W_2- fiber weight after processing, g;

2.4.5. The electron microscopic imaging

In order to note the effect of surface treatment of the reinforcing material on the fiber surface, a microscopic image of some processed and untreated fibers was made using an electron microscope (Tescan, Czech)**.**

3. Results and discussions

3.1. The microscopy results

Figures 3 and 4a display the results of electron microscopy analysis of untreated fibers, revealing the presence of deposits and bumps on their surface due to production processes [6]. The surfaces of fibers processed at the *p*H9 (Figure 4b) seem softer and smoother compared to the untreated fibers (Figure 4a). This suggests that the treatment of fibers with a *p*H9 solution significantly improves the purity and softness of their surfaces [9, 10].

Microscopic imaging of fibers processed with a *p*H10 solution (Figure 5a) shows the appearance of pits and corrosion on the fiber surface as a result of the treatment, in contrast to untreated fibers. Increasing the *p*H to 12 leads to an even greater formation of pits. Figure 9b depicts a microscopic image of fibers treated with a *p*H12 solution, and it can be observed that the surface treatment causes a reduction in fiber diameter to less than 7 microns, which is consistent with previous studies [10, 11].

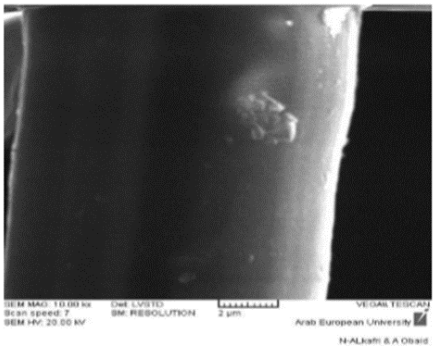
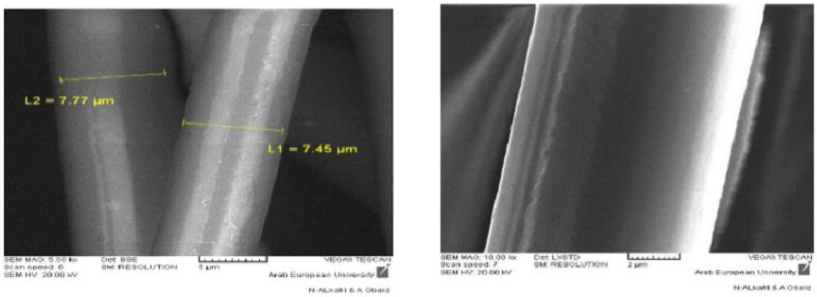
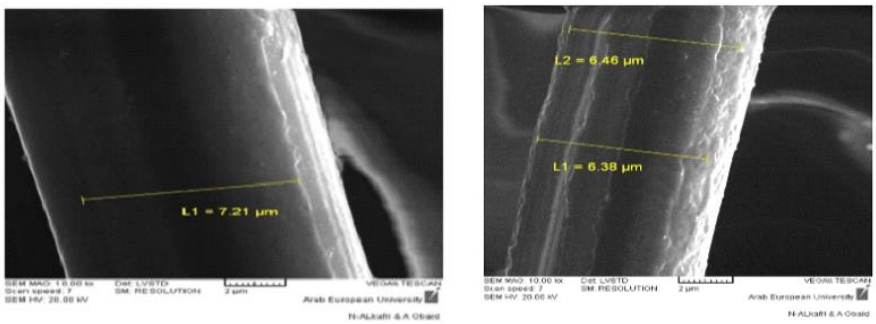


Figure 3. The micrographs showing untreated fiber surfaces at 10.000 times magnification



a) b)

Figure 4. a. The micrographs showing a) the diameter of the untreated fiber and b. the diameter of the fibers treated with a solution with the *p*H9 at an increase of 10.000 times



a) b)

Figure 5. The micrographs showing: a) surfaces of fibers treated with the solution of the *p*H10 concentration and b) treated with the solution of the *p*H12 at the increase of 10.000 times

3.2. The weight loss tests results

Figures 6 and 7 illustrate the relationship between the percentage of weight loss (ΔW%) and the *p*H solution and processing time. The presence of water in the solution facilitates the complete ionization of NaOH ions, resulting in a significant impact on the surface of the fiber. This effect includes the removal of deposits and the formation of pits on the fiber surface [12-14], as described by the following equation:

$$Fiber - OH + NaOH \rightarrow Fiber - O - Na + H_2O + (Surface\ impurities)$$

An increase in the *p*H of the solution and the processing time leads to a decrease in the weight of the fibers.

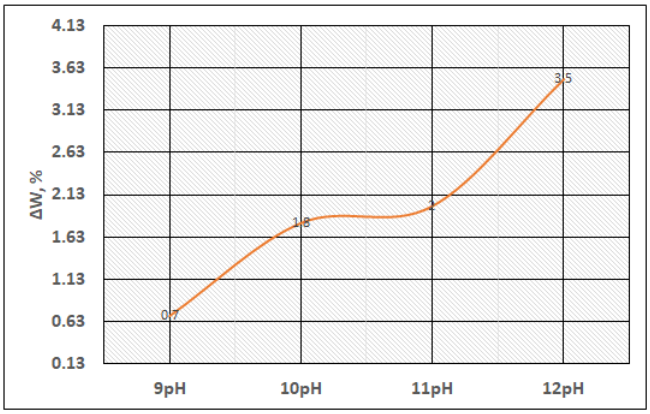


Figure 6. The weight loss effect for the fibers treated with the solutions of different *p*H at the fixed time (3 h)

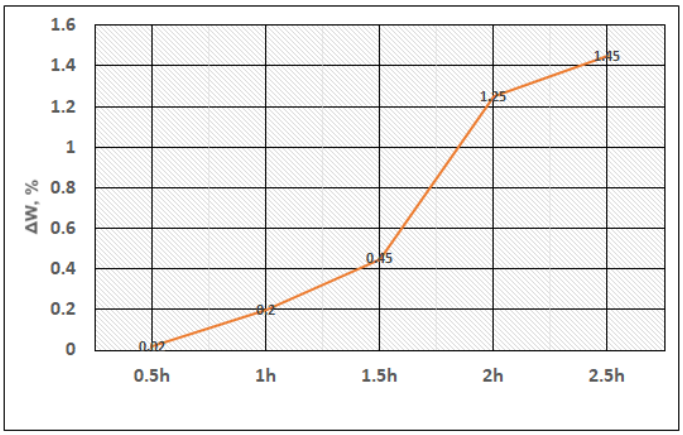


Figure 7. The effect of fiber treatment with the solution of constant *p*H10 at the variable time on weight loss

Reference samples

Table 1 shows the values of reference samples prepared with untreated fiber, which were based on the results of tensile, resistance moment and impact test. (Reference samples were prepared using the same conditions as the treated samples).

Table 1. Test results of reference samples for tension, bending and impact

Reference value				
N		1	2	Average
Tensile Strength	S_{max} (MPa)	89.5	9.4	90
	E (MPa)	88	85	86.5
	T (MPa)	7.7	7.24	7.47
Bending Strength (MPa)		174.1	17.5	171.9
Impact Strength J/cm^2		10	10	10

3.3. The tensile test results

Figures 8 and 9 demonstrate the impact of surface treatment on tensile strength properties. It is observed that:

- When the processing time is kept constant at 3 h, a change in solution (*p*H) results in a decrease in all values of tensile strength (Smax), durability (T), and modulus of elasticity (E) compared to the reference samples. The maximum decrease in values is seen at a *p*H12, with a 30% decrease, and at a *p*H10with a 10% decrease.

With a constant solution *p*H10 and varying processing time, the values of tensile strength, durability, and modulus of elasticity decrease until 1.5 h, then begin to increase at 2 h. At 2.5 h, all values are higher than the reference values, with a 15% increase in durability and a 10% increase in Young's coefficient and tensile strength.

The decrease in tensile strength with increasing (*p*H) is attributed to the higher content of hydroxide ions in the solution, which enhances the reaction efficiency and the formation of pits on the fiber surface. This explains the rapid effect of the solution on the fibers in two samples: one with *p*H10 at 0.5 h and the other with *p*H9 at 3 h, even though pits have not yet formed. Additionally, the solution acts as a cleaning agent, removing deposits and irregularities from the fiber surfaces resulting from manufacturing and packaging processes. This reduces fiber adhesion to the matrix material and facilitates sliding, aligning with previous studies [9, 10] and microscopy results Figure 3, 4b.

Comparing fiber samples treated with *p*H9 and *p*H10 solutions for a constant time of 3 h, a higher tensile strength is observed in the *p*H10 samples, despite the formation of pits as shown in microscopic images (Figure 5a). The pit formation increases surface roughness, improving the bond between the matrix and reinforcing material, but it remains below the reference value [10].

In the second case, as processing time increases, hydroxide ions start affecting the fiber surface and pit formation occurs, increasing roughness and surface area. This enhances contact interaction with the matrix material and leads to an increase in all values at 2.5 h [10, 13].

Furthermore, excessive pit formation reduces fiber diameter and unevenly decreases the cross-sectional area, resulting in areas with increased stress concentration. This is evident in the micrograph of a sample treated with a *p*H12 solution (Figure 5b) [9 - 11], where the effect on the fiber cross-section is more pronounced during tensile testing due to the perpendicular application of force to the fiber cross-section. This explains the low tensile strength (Figures 8 and 9), where the strength decreases at *p*H12.

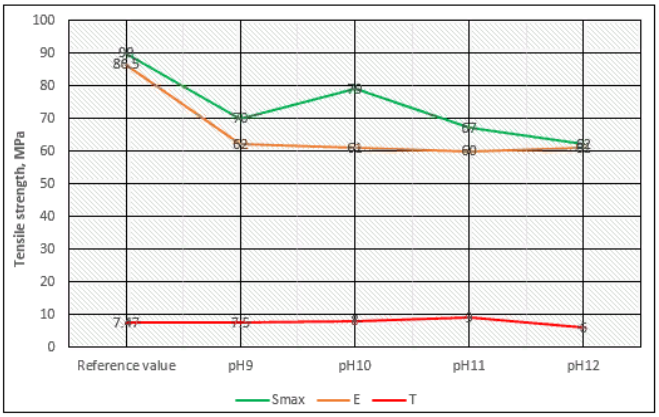


Figure 8. The effect of surface treatment of fibers with the different of *p*H solution at the constant time (3 h)

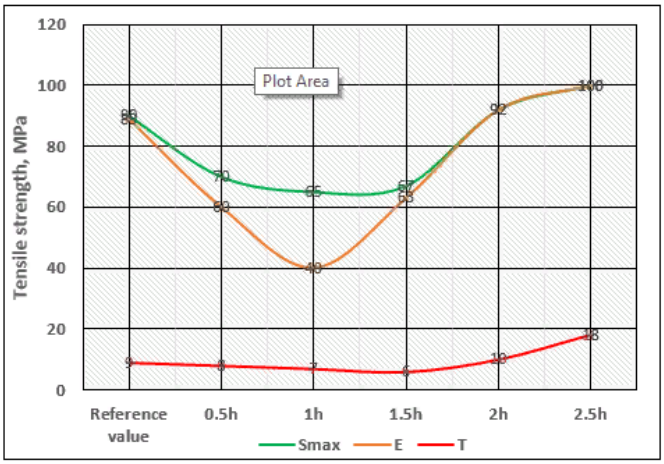


Figure 9. The effect of surface treatment of fibers with t he fixed of *p*H10 solution and variable time

3.4. The resistance moment tests results

Based on the analysis of Figures 10 and 11, it is evident that treating the fibers with a solution of varying *p*H concentration at a constant processing time improves the resistance moment of all samples. The maximum values are observed at *p*H10 and *p*H11, which are 55% higher than the reference value. However, increasing the *p*H12 results in a decrease in the resistance moment, although it remains 10% higher than the reference samples.

When treating the fibers with a solution of constant *p*H10 and varying processing time, there is no significant effect on the bending stress value at 0.5 h. The bending stress gradually increases with longer processing times and reaches its highest value after 2.5 h, with an increase of over 50%.

The improved resistance moment in both cases can be attributed to the alkaline solution treatment, which creates pits and enhances the roughness of the joint surfaces. This increases the surface area of

the mechanical joint between the matrix and the reinforcing material, improving stress transfer from the base material to the reinforcement, consistent with previous studies [1, 15].

The decrease in tension at *p*H12 and a processing time of 3 h is associated with an increased presence of pits, weakening the fiber surface and reducing its resistance to loads. Despite this, the tension value remains 10% higher than the reference value, as supported by themicrographs of the fibers in Figures 4a and 4b [1, 15, 16].

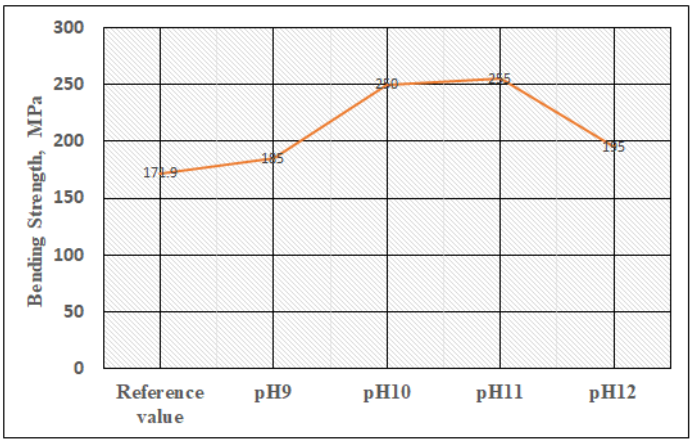


Figure 10. The effect of surface treatment of fibers on bending strength at the variable solution pH and the constant time

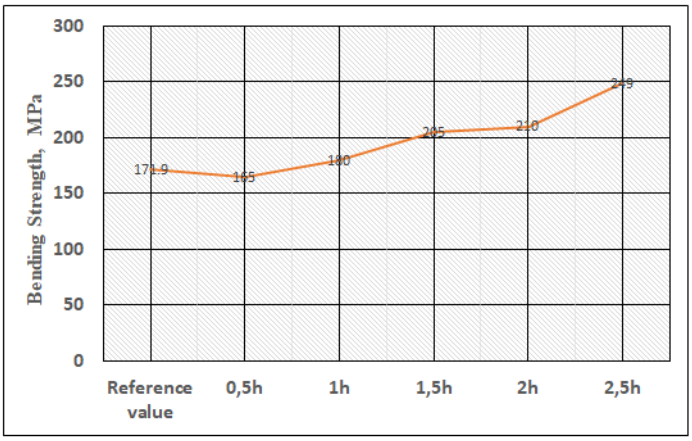


Figure 11. The effect of fiber surface treatment on bending strength at the constant solution pH and the variable time

3.5. The results of samples impact resistance tests

Figures 12 and 13 demonstrate that increasing the concentration of the solution at a stable processing time of 3 h results in an increase in impact resistance. The maximum absorbed energy is achieved when using a *p*H10 solution, with a 30% increase compared to the reference value. However, when the solution concentration is further increased to *p*H12, the absorbed energy starts to decrease and falls below the reference value by approximately 30%.

When keeping the *p*H10 constant and varying the processing time, there is no change in absorbed energy at 0.5 h. The impact resistance begins to increase at 1 h and continues to rise until reaching its maximum absorbed energy at 2.5 h, which is 50% higher than the reference value.

The improved impact resistance in both cases can be attributed to the formation of a high-quality interface resulting from the chemical treatment of the fiber surface. This treatment enhances adhesion between the fiber and matrix, leading to an increased ability of the fiber to transmit stresses [1, 16].

The decrease in impact resistance compared to the reference value at *p*H11 and *p*H12 is due to the deepening of pits on the fiber surface, weakening the fibers and reducing their diameter. This also leads to areas with a higher concentration of curing [1, 16].

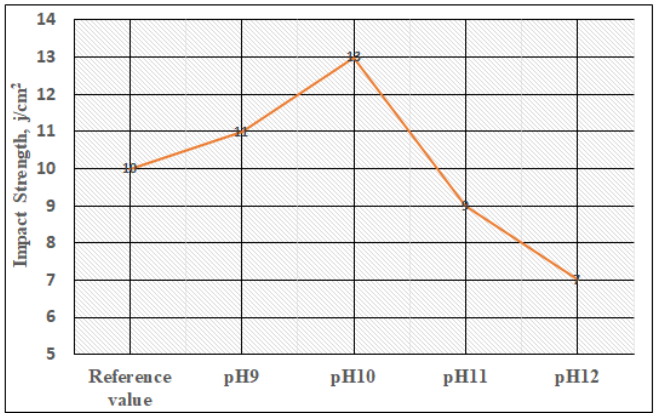


Figure 12. The effect of fiber surface treatment on impact resistance at the variable solution concentration and the constant time

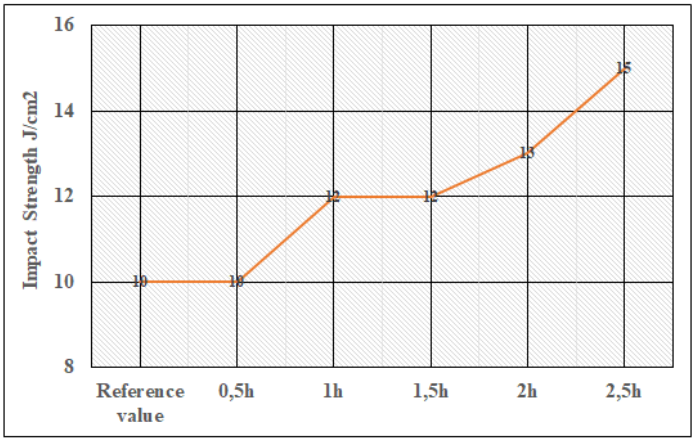


Figure 13. The effect of fiber surface treatment on impact resistance at the fixed solution concentration and the variable time

4. Conclusions

During the conducted studies, the following findings were made:

Treating fibers with an alkaline solution at specific concentrations and processing times resulted in a 10% increase in tensile strength and modulus of elasticity. Additionally, the impact strength improved by 50%.

The resistance moment showed improvement across all variable parameters. The highest improvement was achieved when fibers were processed with a *p*H10 solution for 3 h and a *p*H10 solution for 2.5 h. These results were closer to the reference and resulted in a 50% improvement in resistance moment.

Impact resistance also showed improvement in specific proportions. The largest improvement was observed in samples with fibers treated with a *p*H10 solution for 3 h, which showed a 50% improvement compared to the reference values.

The surface treatment of reinforcing fibers had a greater impact on moment and impact resistance compared to tensile strength.5. The results of some tests were very similar in values, despite the change in the time factor and the concentration value, for example, in samples treated with the solution with the concentration of *p*H9 at 3 h and the solution of *p*H10 at 0.5 h, which helps us to use the processing time by increasing the concentration.

The results of some tests were very similar in values, despite the change in the time factor and the concentration value.

Recommendations

The results of the conducted research should encourage the responsible persons and specialists in the field of composite materials manufacturing technology to develop production lines in the direction of introducing the fiber processing stage before manufacturing.

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