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The effect of salt water on the properties of basalt fibre reinforced composites

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Abstract

The use of natural or naturally derived reinforcing materials in polymer composites is also increasing, thanks to the growing importance of sustainable economy and environmental consciousness. The most promising natural reinforcing material is basalt fibre, which has a very similar chemical structure to glass fibre, which is widely used. However, due to the difference in chemical structure, basalt fibre may be more resistant to more aggressive environments, such as seawater. In this research, it was analysed the effect of salt water on basalt fibre and its composites. It was treated unimpregnated basalt, glass and carbon fibre as well as impregnated composites with different concentrations of saltwater solutions for different durations. The effect of salt water was studied by mechanical and morphological tests.

Keywords: *basalt fibre, salt water, ageing, polymer composite.*

1. Introduction

Modern materials science offers excellent material combinations for the production of parts that are exposed to high loads. Structural materials can basically be divided into three groups, one of which is polymer materials. A special group of polymers is the polymer composites, which are characterized by high specific strength and designable anisotropy. Polymer composites are a combination of a reinforcing material and a matrix material, the reinforcing material is used to provide the structural strength.

The reinforcing material is typically in the form of fibres, the most used are glass fibre and carbon fibre. Nowadays, due to the growing environmental awareness and sustainable economic approach, the use of natural or naturally derived reinforcing materials is becoming more and more popular.

One of the most promising natural reinforcing materials for polymer composites is basalt

fibre. Basalt is a naturally frequent igneous rock with a very similar chemical composition to glass fibre, whose main constituents are SiO_2 , Al_2O_3 , CaO , MgO , FeO és Fe_2O_3 [1].

The mechanical properties of basalt fibre are similar to glass fibre, but it also has the advantage of natural, bioinert, non-irritating and environmentally friendly properties [2], and is resistant to UV and high-energy electromagnetic radiation [3]. The excellent resistance of basalt fibre also extends to its chemical resistance [4]. Continuous basalt fibre with improved mechanical properties can be produced by two-step fibre pulling [5]. The tensile strength of basalt fibres is influenced by many factors besides the manufacturing technology, including chemical composition, fibre diameter, fibre structural inhomogeneities. Basalt fibres are less sensitive to fibre ageing because the iron oxides they contain behave as nucleating elements and promote the formation of a fine and relatively homogeneous crystalline structure [6].

Thanks to its beneficial properties, basalt fibre is expected to become more widely used in areas where it is exposed to more aggressive environmental conditions. The environmental exposure can vary widely depending on the application [3].

The impact of seawater on the properties of basalt fibre composites has been evaluated by several researchers [7], but the analyses were not comprehensive.

Wei et al. [8] investigated the effect of seawater on the properties of glass and basalt fibre composites. They showed that the basalt fibre composites were more resistant to seawater than the glass fibre composites. Bonsu et al. [9] analysed the behaviour of glass and basalt fibre in composite [10] and in hybrid composite [11] at constant solvent concentration and different holding times. It was shown that the decrease in mechanical properties is mainly caused by a significant degradation of the fiber-matrix adhesion and the plasticizing effect of water.

In addition, Fourier transform infrared spectroscopy (FTIR spectroscopy) was used to examine the surface of the embedded fibres before treatment and 305 after aging. The results of the tests showed that the seawater treatment changes the chemical composition of the fibre surface.

Davies et al [11] analysed the properties changes of basalt reinforced fibre composites in response to soaking in salt water at different temperatures using static and cyclic tests. They found that the behaviour of the basalt fibre composites was similar to that of the glass fibre composites. The interlaminar shear strength decreased by around 20% in both cases, caused by degradation and moisture absorption of around 1.5%.

Kaybal et al [12] attempted to compensate for the saltwater-induced delamination by addition of a halloysite nanotube (HNT). They investigated the damage mechanisms in the composites due to applied loads. It was demonstrated that HNT reduced moisture absorption, resulting in higher residual strength in the composites. Several researchers have investigated the effect of holding time [13], medium temperature [14] on morphological and mechanical properties [15][16], but the effect of concentra-

tion has not been analysed and the property changes of reinforcing materials without matrix material have not been further investigated.

In my research, I analysed under laboratory conditions the effect of salt water on the properties of basalt fibre and composites with epoxy resin matrix using basalt fibre fabric. As a reference, I used the most commonly applied reinforcing materials (glass fibre, carbon fibre) and their composites made with their fabrics and carried out their treatment too.

2. Materials, methods and equipments

In this chapter I have summarised the materials used in the research, the procedure for preparing the samples, the test methods and the equipments used for testing the specimens.

2.1. Materials

During my research, I used two of the most commonly used reinforcement materials, fibreglass and carbon fibre, besides basalt fibre fabric (**Table 1**). The fabrics were plain woven, and all were surface treated to epoxy resin.

Table 1 Main properties of used reinforcements

Materials	Producer	Type	Areal density
Basalt fabric	Basaltex (Belgium)	BAS 220 P	220 gsm
Glass fabric	UNIQUE textiles (Czech Republic)	UTE 220 P	220 gsm
Carbon fabric	SGL Group (Germany)	SIGRATEX C W200-PL1/1	200 gsm

For the preparation of the polymer composites, I used a general-purpose laminating epoxy resin type MR3009 bisphenol A ("Component A") with an amine type harder MH3120 ("Component B"), prepared by IpoX Chemicals Ltd. The basic properties of the matrix material are summarized in **Table 2**.

Table 2 Main properties of used matrix

Component A	Component B	Mixture	
Dynamic viscosity		Mixing ratio	Pot life
2000 mPas	300 mPas	100:20	45 min

I made the composite laminates by hand lay-up. I ensured proper mixing of the matrix material components by two-step mixing. In the first step, the components were mixed with an IKA RW 16 Basic stirrer at a speed of 5000 rpm for 3 minutes at room temperature. The mixture was then rested for 2 min and the first step was repeated. After impregnating each layer during the laminating process, I used a roller to remove air voids and also remove unnecessary resin from the fabric. The composite sheets were built up using 6 layers of reinforcements. In all cases, I used the manufacturer's recommended post curing heat treatment of 4 hours at 60 °C in a Heraeus UT20 dryer oven.

For easier identification, I have abbreviated the names of the materials, where GFEP stands for glass, BFEP for basalt and CFEP for carbon fibre reinforced epoxy resin-based composites.

2.2. Test methods and equipments

The properties of the fabrics were analysed by performing tensile tests according to MSZ EN ISO 13934-1:2013 using a Zwick Z020 universal testing machine. The samples used for the test were 250 mm long and 50 mm wide. The tensile strength and elastic modulus of the fabrics were calculated based on the number and diameter of the rovings that build up the strip, the number and diameter of the elementary fibres that make up the rovings and the tensile strength of the reinforcing fabric. 10-10 samples per fabric were tested at room temperature.

Tensile testing of the composites was carried out according to MSZ EN ISO 527-4:2023 on a ZWICK Z020 universal testing machine using 250 mm long and 2×25 mm cross-section specimens. Tensile strength, elongation at break and Young's modulus were determined from the force-displacement curve recorded during tensile tests. The grip length was 150 mm, and the test speed was 2 mm/min. To make the results more accurate, I also used a video extensometer to measure the elongation during the measurement. I examined 5-5 specimens at room temperature for each composite.

Three-point bending tests of the composites were carried out on a ZWICK Z020 tensile testing machine according to MSZ EN ISO

14125:1999 at ambient temperature. The test specimens of 2×10 mm cross section used for the measurements were tested with a support distance of 64 mm and a test speed of 5 mm/min. The specimens were tested to a limiting deflection of 6.4 mm. From the force-deflection curves, bending stress and bending elastic modulus were calculated. I examined 5-5 specimens for each composite.

Charpy impact test of composites was carried out according to MSZ EN ISO 179-2:2020 using CEAST Resil Impactor Junior machine with 2×10 mm cross section test specimen without notch. The applied impact energy was 25 J, the velocity of the impact 3.3 m/s, and the support distance 80 mm. During the measurement, the energy absorbed by the test specimen was recorded and the Charpy impact strength was calculated. The measurements were carried out on 5 specimens of each composite at room temperature.

The Fourier transform infrared spectroscopy (FTIR) measurements were performed on a Perkin Elmer Spectrum 400 in reflection mode. The light source in the instrument allows measurements in the wavelength range 4000-6500 cm⁻¹ and 2500-15385 nm, respectively.

The ultraviolet (UV) spectroscopic studies were carried out using a Hewlett Packard 8452A spectrometer with diode array detector in absorption mode. The instrument uses a visible UV light source operating in the wavelength range 190-820 nm with a resolution of 2 nm.

Microstructural analysis of the samples was performed with a JEOL JSM-6380LA scanning electron microscope. During the tests, I made images of the fracture surfaces of the broken specimens to characterise the quality of the interfacial adhesion. The examined surfaces were coated with a conductive layer before the measurement to avoid electrostatic charging.

3. Results and discussion

In this chapter I have summarised the results and conclusions of the measurements carried out in the experimental part of the research.

3.1. The effect of salt water on the properties of reinforcements

Saltwater corrosion is also a real potential risk for the polymer composites that are used to build the blades of offshore wind turbines. In addition to moisture's already identified softening and adhesion damaging effects, in the case of a possible damage, the reinforcing fabric could also come into contact with seawater, which could directly damage the reinforcing fabric.

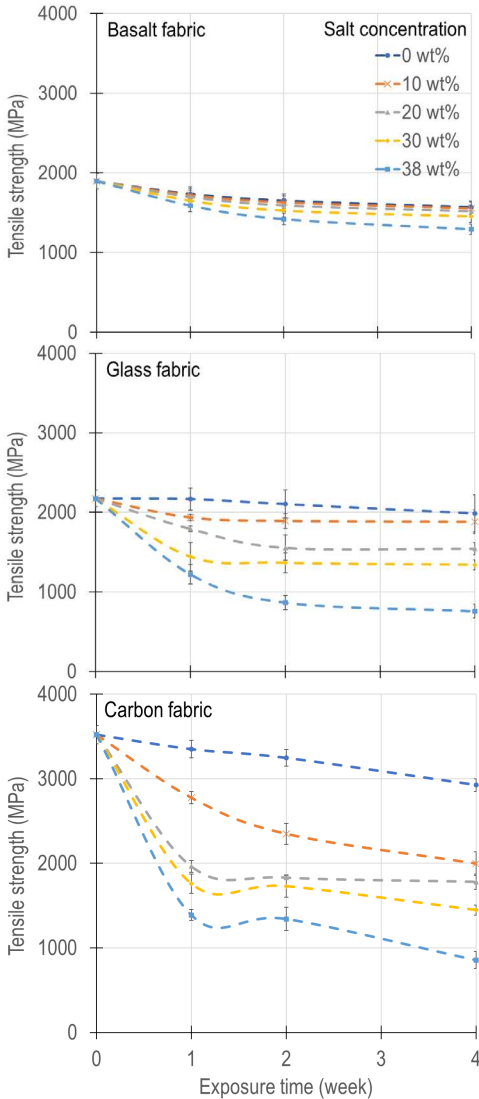


Figure 1 The effect of salt-water on the tensile strength of reinforcements

To analyse this influence, I prepared treatment solutions simulating seawater at different concentrations (0, 10, 20, 30, 38%) and placed the reinforcements and composites in them for different durations (1, 2, 4 weeks). To ensure a constant concentration, the treatment was carried out in a closed container, isolated from light. Treatment with a saturating solution (38%) was justified by the possibility of localised increases in concentration on the wind turbine blades. I flushed the surface of the samples with distilled water before each test. The effect of the treatment on fabrics was analysed using strip tensile tests, the results of which are summarised in **Figure 1**. The results of the tensile test showed that the tensile strength of each reinforcements decreased steadily with increasing solution concentration and treatment exposure time. This decrease was highest for the carbon fibre (76%), followed by the glass fibre (65%) and the most resistant was found to be the basalt fibre (32%). The strength degradation in basalt fibre fabric is caused by the interaction between salt water and the elementary fibres of the basalt fabric, as demonstrated by scanning electron microscopy images shown in **Figure 2**.

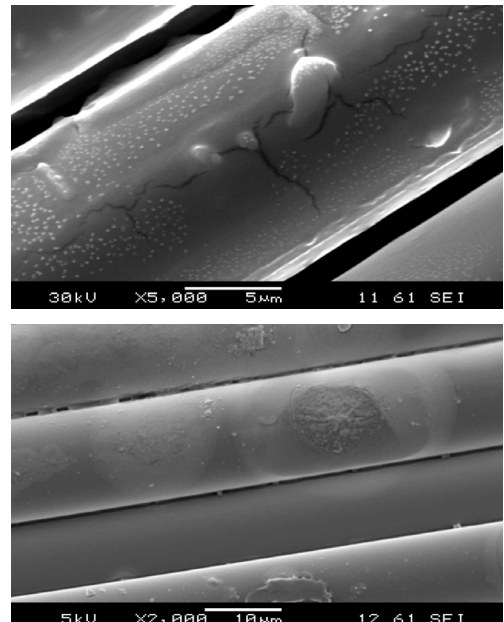


Figure 2 Damage of elemental basalt fibres by salt water treatment; 38% concentration 4 weeks (upper), 30% concentration 4 weeks (lower)

The images show that the elementary fibres of the basalt fabric are damaged, cracks and indentations can be observed on their surface.

It is assumed that the treatment has led to the dissolution of a component from the elementary fibres, and to confirm my theory I took samples from the distilled water, and the prepared solution of 38% concentration, which did not contain any reinforcement, and from the solution in which the basalt fibres were left for 4 weeks.

The liquids were analysed by Fourier transform infrared spectroscopy. I performed 3 to 3 measurements for each sample and the spectrograms of the individual spectral curves are displayed (Figure 3).

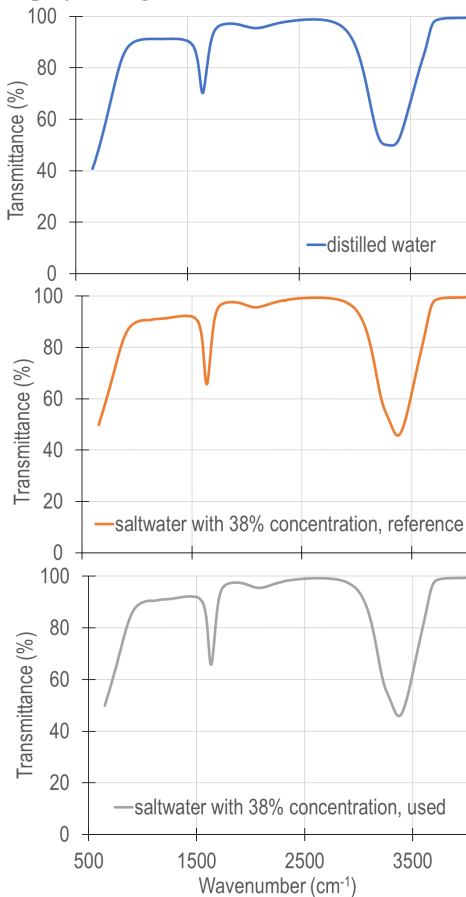


Figure 3 Results of Fourier transform infrared spectroscopic measurements

The results show that the difference is only visible between the distilled water used as a

reference and the saltwater solution with 38% concentration. The detectable difference is represented merely by a change in transmittance and not by a wavelength difference. The two characteristic peaks are the characteristic signal of aqueous solutions. It is assumed that the wavelength range limits the identification of the leachable elements, so UV spectroscopy was used, which operates in a shorter wavelength range. The average spectra of 3-3 individual spectrum curves were plotted for each sample (Figure 4).

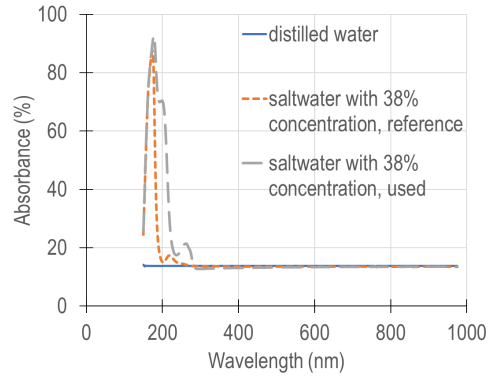


Figure 4 Results of ultraviolet spectroscopic studies

The absorbance of the distilled water used as reference is negligible compared to the 38% saltwater solution. The characteristic wavelengths determined for the saltwater solutions are summarised in Table 3. Based on the literature [17][18][19], the values between 190-220 nm (199.76; 218.10) recorded for the used saltwater solution are for sodium and potassium, while the higher peak value (262.62 nm) falls in the range of the characteristic peak for iron and iron oxide.

Table 3 Characteristic peaks of the recorded UV spectra

Type of saltwater with 38% concentration	Characteristic peaks (nm)		
	1.	2.	3.
reference	173.57	218.1	–
used	178.81	199.76	262.62

Spectroscopic studies confirmed that the mechanical properties of the basalt fibre fabric were reduced due to leaching caused by the interaction of the iron or iron oxide that builds up the basalt with salt-water.

3.2. The effect of salt water on the properties of composites

After the reinforcement fabrics, I analysed the effect of salt water on the mechanical properties of basalt mono- and hybrid composites by tensile, three-point bending and Charpy tests. Manufactured edges of the specimens were sealed with wax to avoid direct penetration of salt water into the composite material. The results of the tensile tests are shown in **Figure 5** and **Figure 6**.

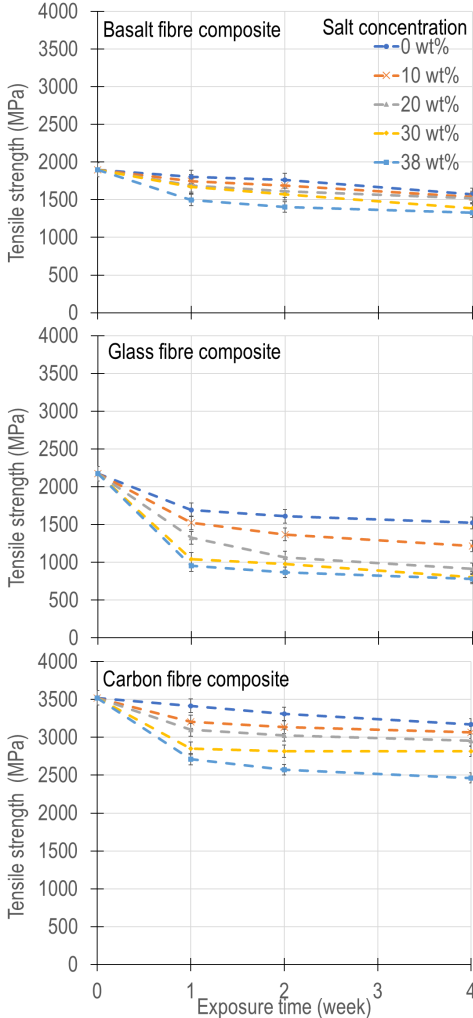


Figure 5 Tensile strength values of composites determined during the tensile test

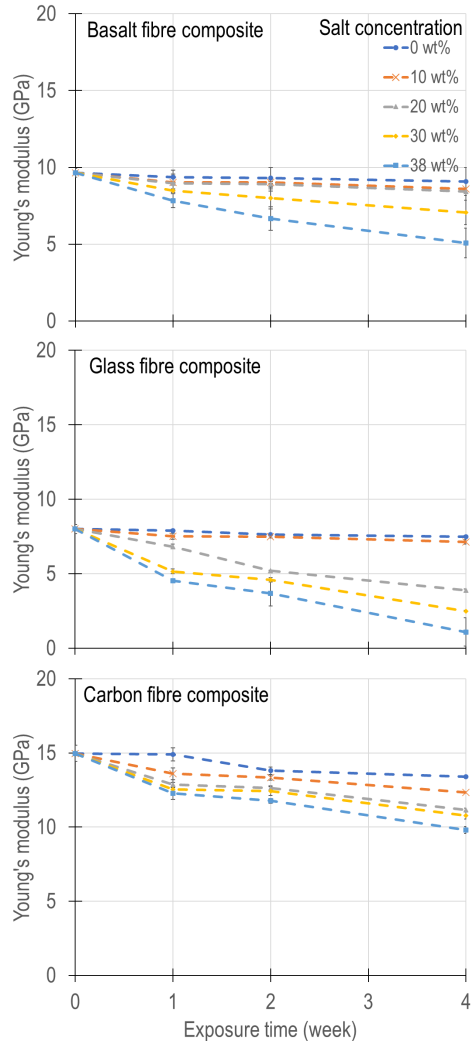


Figure 6 Tensile modulus of elasticity values of composites determined during tensile tests

The results of the tensile tests show that the composite reinforced with basalt fabric was damaged the least by salt water, due to the better resistance of the basalt fibre to saltwater penetrating through the micropores and microcracks of the matrix material, which is a consequence of the metallic elements that compose the basalt. Compared to glass fibre fabric, basalt fibre is approximately 30% more resistant. The results for the basalt and carbon fibre fabrics show no significant difference compared to the reference. The studies show that replacing glass fibre fabric with basalt fi-

bre fabric in a saltwater environment can lead to a significant increase in lifetime, which also leads to a cost reduction, so the use of basalt fabric in offshore wind turbines can have a positive impact on the lifetime of turbine blades. The flexural strength and flexural modulus values of the composites determined in the flexural testing are summarised in **Figure 7** and **Figure 8**.

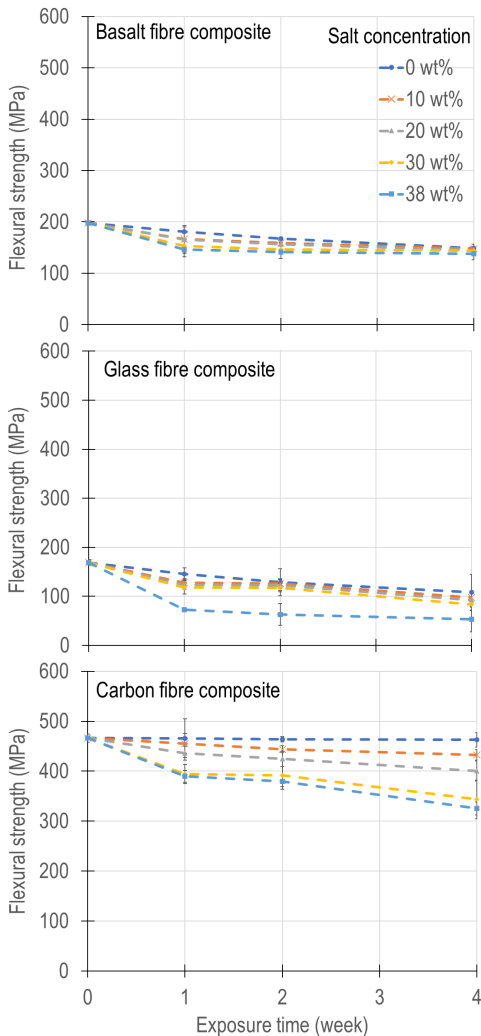


Figure 7 Flexural strength values of composites determined during bending tests

The trend determined from the tensile test is consistent with the results of the flexural test, with the basalt fibre reinforced composites proving less sensitive to saltwater treatment than the glass fibre reinforced composites.

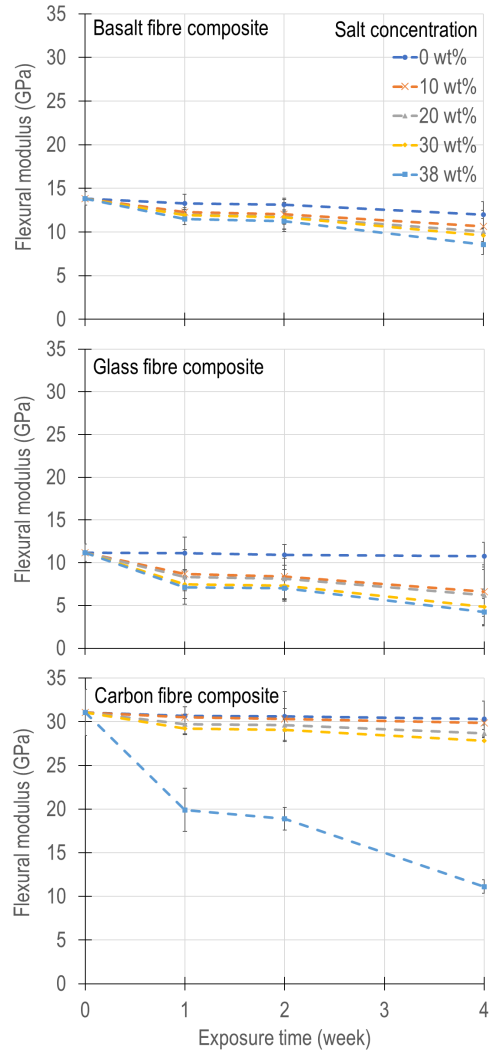


Figure 8 Flexural elastic modulus values of composites determined by bending tests

The basalt fabric was 36% more resistant in flexural strength and 26% more resistant in flexural modulus of elasticity after 4 weeks of exposure, compared to the glass fibre fabric. The results of the bending tests show that the basalt fibre can be used effectively even when the structural elements are subjected to bending stress.

In conclusion, the use of glass fabric can be substituted by basalt fabric. According to my test results, basalt fibre is more resistant to the saltwater environment. The results of the Charpy impact test are shown in **Figure 9**. The

results of the Charpy test also support the results of the tensile and bending tests. The salt-water treatment also degraded the matrix material as well as the elementary fibres that compose the reinforcing materials, as evidenced by the 50% impact strength loss of the glass fibre reinforced composite after 4 weeks in 38% solution.

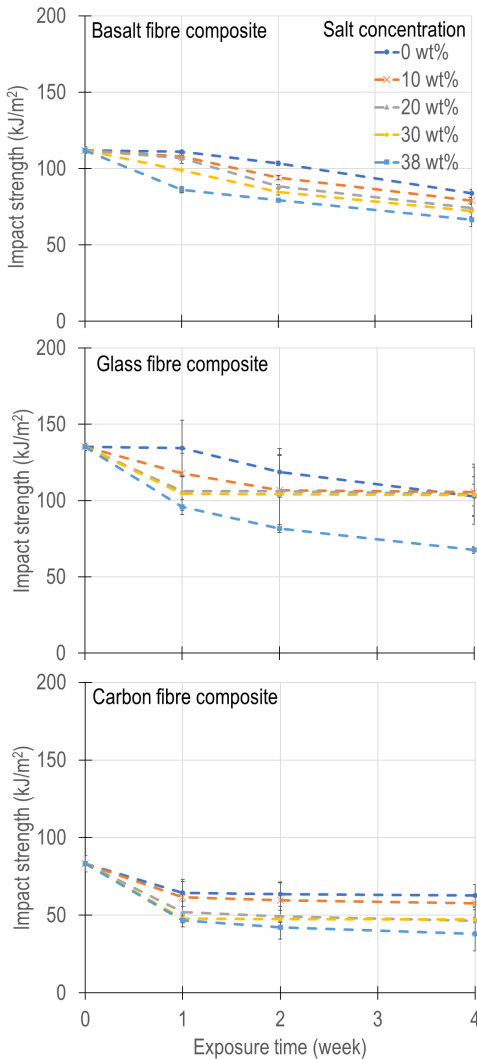


Figure 9. Impact strength values of composites determined by the Charpy impact test

4. Summary

The results of the tests carried out show that a significant degradation of reinforcement fabrics and composites occurs as the result of salt-water treatment, irrespective of the reinforcing material used to produce them. It can be seen, however, that the extent of degradation is already significantly influenced by the constituents that compose the reinforcing material. The results of the tensile, flexural and Charpy tests show that the basalt fibre reinforced specimens are the most resistant of all those tested, due to the metallic elements and their oxides that build up the basalt fibre. Due to this behaviour and resistance, the basalt fibre fabric could become the base material for some components of composite structures operating in different seawater environments, such as offshore wind turbines, and thus its further expansion is predicted.

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