American Journal of Materials Science 2016, 6(1): 19-34
DOI: 10.5923/j.materials.20160601.02

Evaluating the Efficiency of Basalt and Glass Fibres on Resisting the Alkaline, Acid, and Thermal Environments

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Abstract Over the past few years, basalt fibre has been used extensively as a reinforcement material in the construction industry, and close attention has been paid to its qualities, especially its chemical and thermal resistance. In view of the significance of basalt fibre as a strengthening material, and to evaluate the long-term durability of basalt fibre, four different experimental works were performed, namely: resistance to alkalinity and acid environment tests, thermal resistance tests on fibres, measuring weight change of fibres, and observing the microstructure and element change of fibres using the Scanning Electron Microscope (SEM) and Energy Dispersive X-ray (EDX). In the past, there have been few studies on the chemical and thermal resistance of basalt fibres, most researchers focused on the effect of the strongest alkaline and acid chemicals such as Hydrochloric acid (HCl), and Sodium hydroxide (NaOH) as well as water on basalt fibres, but paid little attention to the effect of different alkaline and acid chemicals containing different pH levels. This research aims to fill this gap in the literature by evaluating the efficiency of basalt fibres in resisting different strong and weak alkaline and acid chemicals containing different pH levels, and also evaluates the capability of basalt fibres to resist several thermal degrees. For comparison purposes, glass fibres were also tested. The results showed that basalt fibres were more capable of restoring their strength than glass fibres when immersed in different alkaline and acid environments, and were more capable of resisting high heat, in particular resisting temperatures of 300° and 500°. Whereas glass fibres were more capable of resisting the low heat, in particular resisting temperatures of 50° and 100° better than basalt fibres. In this context, the chemical and thermal resistance of basalt fibres becomes an important parameter as a strengthening material for the construction industry.

Keywords Basalt Fibre, Glass Fibre, Chemical and Thermal Resistance

1. Introduction

Basalt fiber resistance to aggressive media acid, alkaline or thermal is determined by the strength change after some time of exposure in these media. Chemical resistance of basalt fibres depends mainly upon their chemical composition, nature of the aggressive medium, and temperature and time influence on the fiber [22]. In addition, the rate of silicion, aluminum, calcium, and iron oxide in the basalt composition is of high importance. In particular, the presence of the iron oxide in the silicate carcass of the basalt fibre imparts to them higher chemical and thermal resistance when compared with glass fibres [22].

In general, there is no material that is naturally durable because the material's interaction with the environment changes the properties of the materials. Therefore, the addition of basalt fibres to concrete has a significant effect on the properties of the basalt fibre and, subsequently, it is essential to develop a concrete reinforced by basalt fibres which can resist various in-service environments.

In particular, in-service durability is defined as the ability of fibres to resist chemical attack, thermal heat and abrasion while maintaining its desired properties [23]. And Civil Engineers have traditionally focused on enhancing the strength characteristics of the material. However, in recent years, the civil engineering industry has started to view fibres from a different perspective, with an emphasis on the whole-life cycle of the structure, focusing mainly on enhancing the fibres and concrete durability. Hence the durability of fibres is now viewed as equally important as its mechanical properties. [24-26].

Since their occurrence, basalt fibres demonstrated higher alkali resistance compared with majority of glass fibres. This feature has specified the attempts of using them as a reinforcement material of Portland cement concrete.

2. Research Methodology

To investigate the chemical durability and performance at the cement basalt interface, several types of experimental tests were undertaken. These included; measuring fibres' weight change, determining the tensile strength of fibres after being boiled in different alkaline and acidic

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Published online at http://journal.sapub.org/materials

environments, and measuring the thermal resistance of fibres. Surface studies were also carried out by scanning electron microscopy, while microanalysis with complementary X-ray diffraction analysis (SEM/EDS) was also used to ascertain the durability of basalt fibre.

The resistance to the alkalinity and acidic environment tests were carried out by boiling the basalt fibres in different alkaline and acids for 1, 5 and 24 hours. Three alkaline chemicals were used (KOH, NaOH, NH_3) and three acids (HCl, H_2SO_4, CH_3COOH). The concentrations of the chemicals used were 2 Mol/Litre. The durability test was conducted in accordance with BS EN ISO 175:2010 [17] and similarly to research undertaken by [7, 8, 14], whereby the basalt and glass fibres were boiled in the chemical solutions for 24 hours, and the initial and final mass of the fibres was determined. After boiling and measuring the mass change, the tensile strength of the fibre was then measured. Similarly, the thermal resistance of fibres was performed by heating basalt fibres in a preheated oven at different degrees (50°, 100°, 300° and 500°) for 1, 3 and 6 hours, and the tensile strength was then measured. After each treatment, the tensile strength of basalt fibres was tested by a single fibre testing machine (INSTRON 3369 Dual Column Testing Systems for Tensile, Compression, Flexure testing) [1] in accordance with ISO 5079:1995 [2]. Using a 5kN capacity, tensile load was applied on the fibres at 20mm clamp distance under a constant tensile speed of 3000 mm/min. Finally, the Scanning Electron Microscope (SEM) and energy dispersive x-ray (EDS) were used to monitor the surface shape changes in fibres after being subjected to different chemical and thermal environments, as well as to determine the element change of fibres. The same specimens analysed for the (SEM) are also analysed for (EDS) using 4kV energy. All specimens were coated by carbon, and the results were taken from an average of several fibres. Glass fibres were also tested for comparison purposes.

3. Results and Discussion

3.1. Weight Change of Fibres

To understand the durability of basalt fibres, a durability test to determine the weight change of basalt fibre boiled in different chemical solutions was conducted. The durability test was conducted by boiling basalt and glass fibres in different chemical solutions: (KOH, NaOH, CH_3COOH, HCl, H_2SO_4, NH_3) for 24 hours, and the initial and final mass of the fibres was determined. All results are presented in figure 1.

The difference of mass before and after immersion, expressed as a percentage of one hundred (%) is calculated using equation 1, and rounded off to two significant digits. However, both types of test specimen shall be dried and their mass measured until their weight is constant [3].

$$\alpha = \frac{w0 - we}{w0} \times 100 \tag{1}$$

Where
α: Change in mass (%)
we: Mass after immersion (g)
w0: Mass before immersion (g)

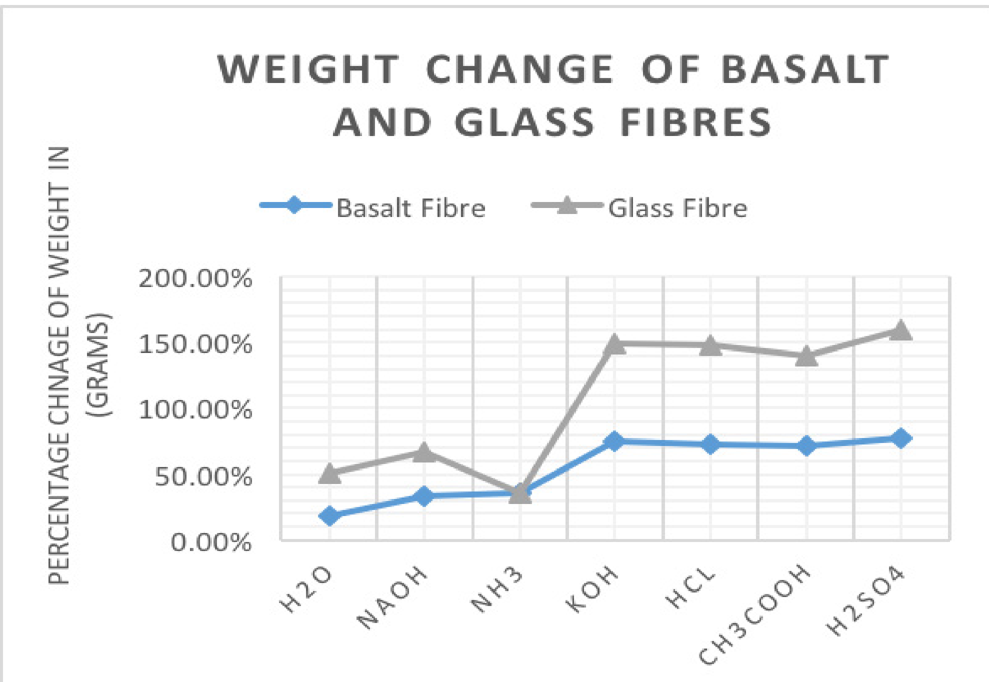

Figure 1. Weight Change of Basalt and Glass Fibres

The results from figure 1 show that there is a weight gain in fibres after being boiled. This is due to the ability of both types of fibres to absorb the chemical solution which resulted in the weight of fibres being slightly heavier. According to the results from figure 1, the change in weight of basalt fibres after being boiled in different chemical solutions seems less than that of glass fibres. However, boiling both types of fibres in NH_3 had similar result. The increasing weight of basalt fibre in alkaline and acid solutions is in line with the results from former research which showed that basalt fibre increased in weight from day 1 to day 3 more promptly in aggressive alkaline solutions [4].

When fibres are boiled in different chemical mediums, the chemical molecules penetrate into the material which causes the sample mass to increase. It is believed that the higher void content in the matrix and fibre–matrix interface, the higher weight change that can be achieved [27]. The results confirmed that glass contain higher voids than basalt fibres, as it gained more weight that basalt fibres. Both types of fibres have gained more weight in the acid solutions compared to the alkaline solutions. This due to the penetration of the small molecular hydrogen ion of the acids into the structure of the fibre. To investigate this further, more experiments were carried out in this research paper such as tensile strength tests, (SEM) and (EDS) to study the effect of the acid and alkaline chemicals on the structure of the fibres.

3.2. Acid Resistance

An acid is a substance that donates hydrogen ions (Chemical symbol H^+). When dissolved in cement or water, there are more hydrogen ions than hydroxide ions in the solution, and as a result the hydroxide ions will move. When mixed with fibres, metal ions on the fibre surface are replaced by hydrogen ions from the acid. Once exposed to acid mediums, the fibres containing alkalis (Na^+, K^+, Ca^{2+}, Al^{3+}) become prone to chemical attack [5]. An ion exchange mechanism between the fibres and the acid solution starts and is shown by equation (2).

$$SI - OM + H^+ \xrightarrow{\quad} SI - OH + M^+ \qquad (2)$$

The relationship between fibre composition and chemical stability in water and acids depends mainly on three factors, which are (1) the chemical agent in which the fibre is exposed, (2) the pH level of the chemical, and (3) the microstructure of the fibre. Figures 2 and 3 shows the tensile strength of basalt and glass fibres after being boiled in three different acids (HCL, H_2SO^4, and CH_3COOH) for periods of 1, 3 and 24 hours. The pH levels of the three chemical solutions are 0 for the HCL, 2.1 for the H_2SO_4, 2.4 for CH_3COOH and 7 for the water. The objective is to test the acid resistance of the basalt fibres to different strong and weak acids. Figure 2 shows that in a 2 Mol/L chemical solution, the tensile strength of basalt fibre seems to decrease when it is kept in the solution for long periods. In particular, basalt fibres lost more strength when immersed in H_2SO_4 and CH_3COOH solutions due to both chemicals having higher pH levels, but gained strength when immersed in HCl. The same result was shown by (EDS) using table 1, basalt fibre element change, as the element of the fibres have increased when boiled in HCl compared to the original elements of the pure basalt fibres. As expected, increasing the pH level of the solution led to a dramatic decrease of the fibre tensile strength.

The variation in the result is due to the chemical composition of the solutions and concentration of the hydrogen ions, as it is known that the lower the pH, the higher the concentration of hydrogen ions in the solution. Strong solutions such as HCl have a pH level of 0, hence contain a high concentration of hydrogen ions. Therefore, when basalt fibres were bolied in HCl, a high level of the hydrogen ions moved from the solution to the fibre. On the other hand, the lower concentration of hydrogen ions in H_2SO_4 and CH_3COOH has effected a move of the hydrogen ions to the basalt fibres, as a result reducing the tensile strength of the basalt fibres after being boiled for 24 hours, as shown in figure 2. In general, basalt fibres showed excellent tensile strength retention in the acid environment, and capability to resist alkaline chemical attack.

In addition, using glass fibres also showed a major effect on the tensile strength of fibres. From figure 3 it can be seen that the strength of glass fibre have increased only for the initial period (after 5 hours). This is due to chemical exchange between the fibre and the medium which can increase the elements content within the fibre, as a result enhance its microstructure and strength. This result also indicates that glass fibre can resist acid attack for a short period. However after longer periods (24 hours), the presence of the acid solutions starts to affect the glass fibre, hence reducing its tensile strength value. The same results is shown by the (EDS) using table 2, glass fibre element change, as the elements of the fibres tend to decrease after 24 hours. This decrease is caused by the replacement of the key metal elements such as sodium (Na), Aluminium (Al), and Calcium (Ca) by hydrogen ions from the acids. As the hydrogen ions are much smaller than the metal elements, tensile stress is induced on the surface of the glass fibre, and eventually cause stresses, and then cracks [5]. This replacement will destroy the fibre's network structure and reduce the fibre's strength. In addition, the microstructure of the glass fibre and the different pH levels play essential roles in effecting the glass fibre strength. This may explain the high reduction in the tensile strength value when boiling glass fibres in different solutions after 24 hours, as can be seen from figure 3. Not only this, but also when glass is exposed to an acid environment, it forms a film that protects it from future damage. The same result was achieved using Picture 16, glass fibre boiled in HCl for 24 hours, which shows clearly the formation of this film. After the formation of the film, the major elements of the glass fibre are replaced by the hydrogen ions such as Aluminium (Al), and Calcium (Ca), and recent investigation has shown that zirconia glass, which does not contain either element, does not corrode when exposed to an acid environment [5].

To understand the effect of the acids on fibres and to compare the performance of the basalt and glass fibre after being boiled in different acid solutions, a tensile strength retention formula, equation (3) was used and shown below: see strength retention [3]

$$R_{ett} = \frac{f_{u0\ f} - f_{ue\ f}}{f_{u0\ f}} \times 100 \quad (3)$$

Where:
R_{ett}: *Tensile strength retention (%)*
$f_{u0\,f}$: *Average value for tensile strength before immersion* (N/mm^2 *or MPa)*
$f_{ue\,f}$: *Average value for tensile strength after immersion* (N/mm^2 *or MPa)*

Figure 4 shows the tensile strength percentage change of basalt and glass fibres after being boiled in different acid solutions for 24 hours. On average, the tensile strength retention achieved for the basalt fibre after being boiled in different acid solutions is around 93%. This means that basalt fibres lost on average around 7% of the tensile strength from their original shape after being boiled. On the other hand, on average the tensile strength retention achieved for the glass fibre after being boiled in different acid solutions is around 43%. The glass fibre lost around 57% of its original tensile strength shape. The big differences in the values between basalt and glass fibres are due to the differences in the microstructure of the fibres as well as the chemical reactions between the surface of the fibres and the environment. Glass fibres were effected significantly by the acid environment while basalt fibres showed an excellent resistance to the acid attack.

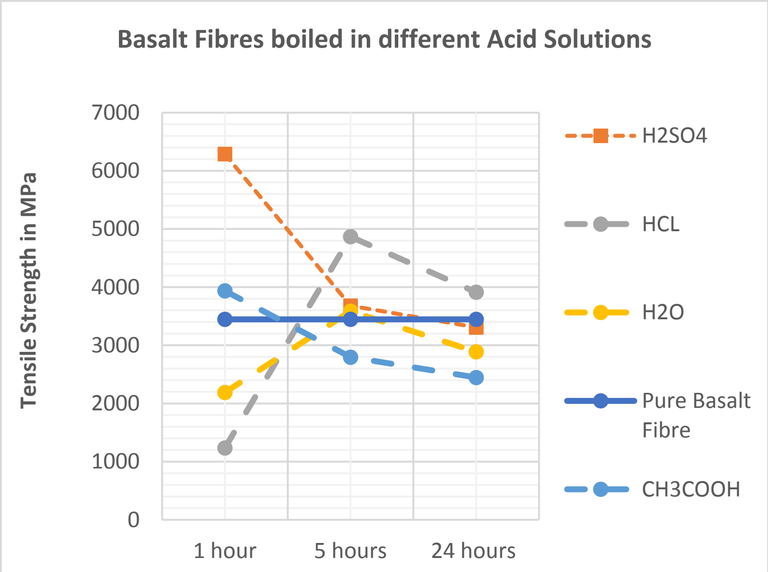

Figure 2. Basalt Fibres boiled in different Acid Solutions

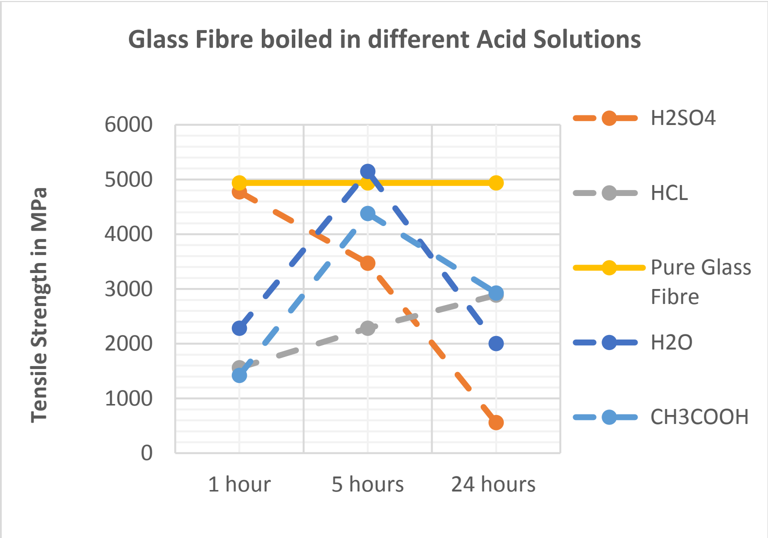

Figure 3. Glass Fibres boiled in different Acid Solutions

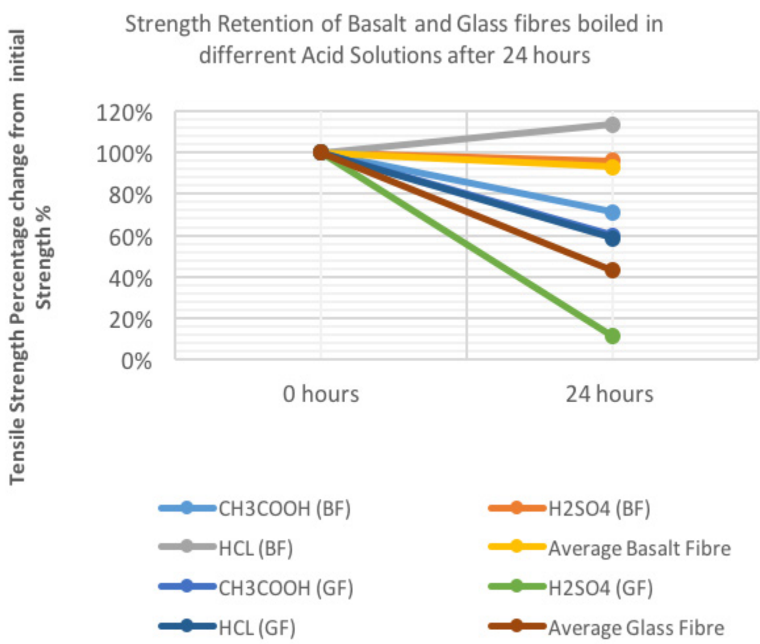

Figure 4. Strength Retention of Basalt and Glass Fibres boiled in different acid solutions

3.3. Alkaline Resistance

An alkaline is a substance that donates hydroxide ions (Chemical symbol OH^{+}) and has a pH level of more than 7. When dissolved in cement or water, there are more hydroxide ions than in the hydrogen ions solution, as a result of the move of the hydroxide ions. An example of an alkaline is NaOH which gives $Na^{+}_{(aq)}$ and $OH^{-}_{(aq)}$ ions in water. When mixed with fibre, a corrosion layer is formed on the surface of the fibre, see picture 17 basalt fibre boiled in NaOH, and the chemical composition of the fibre surface starts to change due to its reaction with an alkaline medium. According to [6] alkali treatment leads to a decrease of sodium, potassium, aluminium and silicon (Na^{+}, K^{+}, Si^{+}, Al^{3+}) on the surface of all basalt fibres. In the alkaline solution, an alkaline attacks the silica network directly and the hydroxide ions of the alkali breaks the Si-O-Si linkage which is shown in Equation (4)

$$\text{Si ----O ---------- Si------- OH + } \rightarrow \text{ SiOH + OSi} \quad (4)$$

Figures 5 and 6 show the tensile strength of basalt and glass fibres after being boiled in three different alkalines (NaOH, KOH and NH_3) for a period of 1, 3 and 24 hours. The pH levels of the three chemical solutions are 14 for the NaOH, 11 for the KOH, 10.6 for NH_3 and 7 for the water. The objective is to test the alkaline resistance of the basalt fibres to different strong and weak acids. Alkali resistance of the fibres was determined by the tensile strength after alkali treatment. From Figure 5 it can be seen that mixing fibres with alkaline solutions leads to a decrease in the tensile strength of the basalt fibres. This severe degradation is due to the high ability of OH^{-} ions to break the fibre network and reduce its strength. This explains why the tensile strength of the basalt fibres have been affected the most when subjected to the NaOH solution, which possess a pH level of 14 and contain a high percentage of hydroxide ions. The tensile strength of the basalt fibre from 1 hour to 5 hours seems to increase slightly; however it reduced dramatically after 24 hours. This is due to the slow chemical reaction of the solutions which takes time to have an effect on the fibres. The tensile strength of the basalt fibres have no detectable change expected when immersed in the NaOH medium, and the strength variation of the basalt fibre seems much better when the reaction is performed after 24 hours.

In view of investigating the influence of the alkaline solutions on fibres, it is remarkable to note that the effect of using glass fibre is quite significant in comparison. From figure 6 it can be seen that when the glass fibre is boiled for a long period (24 hours) there is a big decrease in the fibre strength. The strength of the fibre seems to increase slightly between 5 hours and 24 hours, but remains very low when compared with the original strength of the glass fibre. This reduction becomes clearer when boiling glass fibres in strong alkaline, such as NaOH and KOH. As shown in figure 6, it is clear that using alkaline has a significant effect on reducing the strength of the glass fibres.

Figure 7 shows the strength retention of glass and basalt fibres after being boiled in different alkaline solutions for 24 hours. Based on statistical analysis of the graph, there is a reduction by approximately 40% in the strength of both basalt and glass fibres after being boiled in the very strong alkaline sodium hydroxide (NaOH). In contrast, there was an enhancement in the strength retention of both types of fibres when immersed in two alkaline mediums namely NH_3 and KOH. From the graph it can be seen that there was a slight reduction in the fibre strength retention, by approximately 90% for both types of fibres when being boiled in ammonia NH_3, and an average reduction by 70% on the fibre strength when KOH was used. The enhancement in the strength retention of both fibres may be attributed to the decrease of the pH level of KOH and NH_3, around 11.6 – 12, which contain less hydroxide ions needed to reduce the strength of the fibres. On average both types of fibres performed well in resisting the alkaline attack and restoring their strengths; however, basalt fibres performed much better to maintain their strength compared to glass fibres.

3.4. Comparison between Fibers Resistance to the Acid and Alkaline Solutions

Basalt fibre resistance to an aggressive media (acid or alkaline) is determined by the strength change of the fibre after some time of exposure in the media. The ratio of silicon, calcium, magnesium and in the microstructure of the basalt fibre, is of high importance, in particular the chemical interaction between the fibre surface and the chemical environment. In determining if basalt and glass fibres are more capable of resisting the alkaline environments or the acid environments, figures 8 and 9 were drawn. Figure 8 shows the results obtained for boiling basalt fibres in different high and low acid and alkaline solutions as well as the average tensile strength of the basalt fibres in both types of solutions, while figure 9 shows the same attributes using glass fibre.

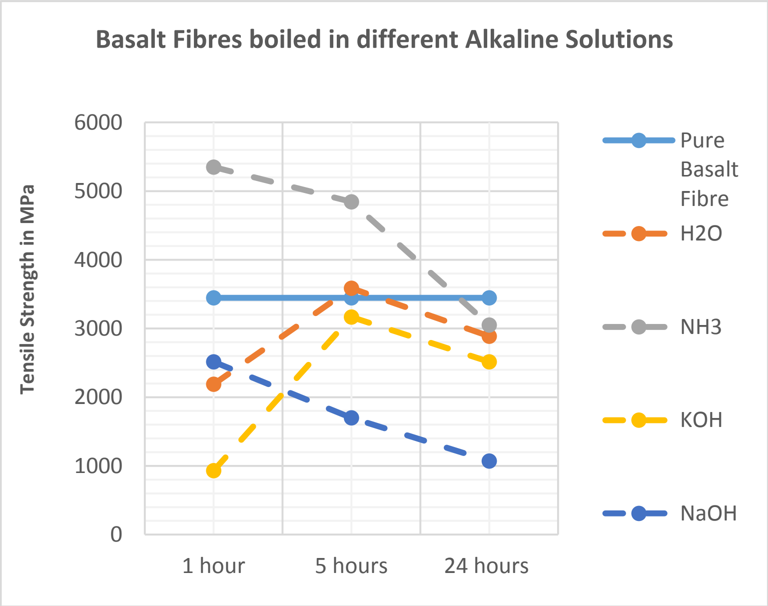

Figure 5. Basalt fibres boiled in different alkaline solutions

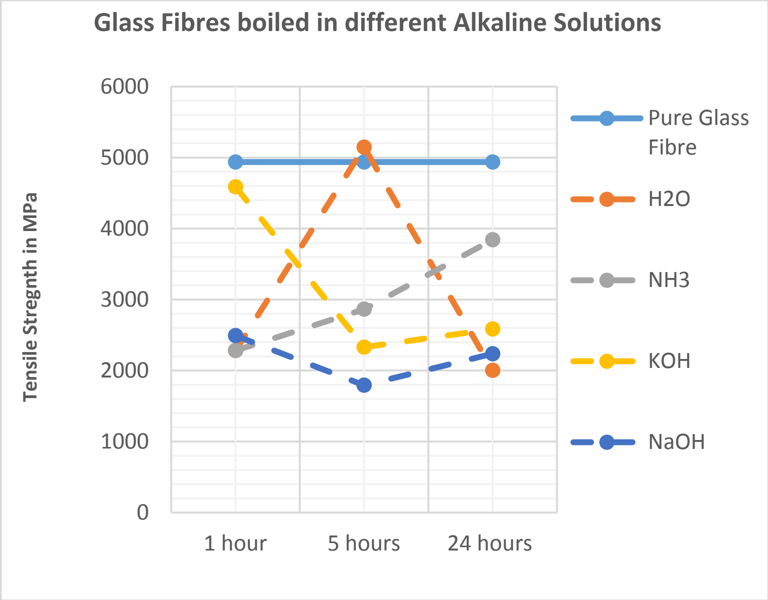

Figure 6. Glass fibres boiled in different alkaline solutions

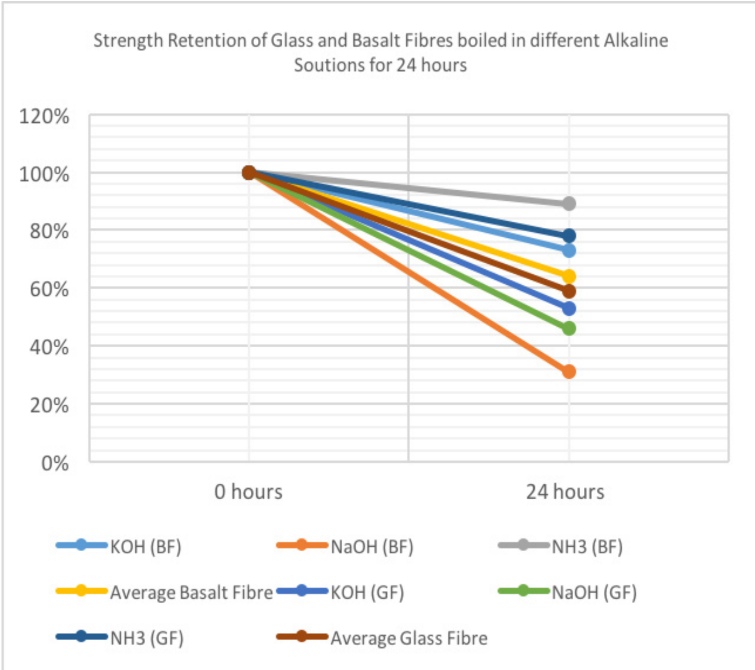

Figure 7. Strength Retention of Basalt and Glass Fibres boiled in different alkaline solutions

From Figure 8, it can be seen that after 24 hours of boiling fibres in different solution, the values of the tensile strength reduced significantly, which highlights the presence of hydrogen and hydroxide ions, which reduced the tensile strength of the fibres. The reduction values ranged from the highest after immersing basalt fibres in NaOH, to the lowest after immersing fibres in HCL. This result was expected as HCL is a strong acid with a pH level of 0, and NaOH is a strong alkaline with a pH level of 14. On average, basalt fibre performed marginally much better in resisting the acid environment in comparison to resisting the alkaline environment. In contrast, the same result was achieved by [7] as he discovered that the strength of the basalt fibre remains approximately unchanged after being boiled in an acid medium, while the basalt fibres strength declines gradually when boiled in an alkaline medium. Although basalt fibre performed much better in resisting the acid more than the alkaline, its resistance to alkaline remained excellent as highlighted in the previous section. Those results confirmed the results obtained from the first part of the experimental work carried out in this study that highlighted the slight reduction in the tensile strength value by acid chemicals, and an average reduction by the alkaline chemicals.

In comparison to the basalt fibre, the use of the glass fibre in different chemical mediums seem to have a different result. It is observed that glass fibres are more capable of resisting alkaline solutions than acid chemicals, as can be seen from figure 9. The reduction in the tensile strength of glass fibre after being bolied in alkaline solution NaOH, due to the high mobility of OH ions, is similar to that reported by another researcher [8]. The strength decrease of glass fibre at an initial stage is obvious when boiled in alkaline solutions as shown in figure 9; however, fibres show a better strength retention after treatment for a long time. On the other hand, after boiling fibres for 5 hours in acid solutions such as HCL and CH_3COOH, there is an increase in the value of the tensile strength compared to its value when it was boiled for 1 hour. After 5 hours of boiling glass fibre in HCL and CH_3COOH, the tensile strength values seem to reduce by 52% and 10% respectively from the strength of pure glass fibre, whereas after 24 hours, the reduction of the tensile strength is 48% and 42% respectively from the original strength of the pure glass fibre. Moreover, after 24 hours the tensile strength of glass fibres seems to be mostly affected by the high acid solution H_2SO_4, to reduce the pure glass fibre tensile strength value by 90%, and less by the high alkaline NH_3 to reduce glass basalt tensile strength value by 24%. This result confirmed that glass fibre is more capable of resisting the alkaline environment than the acid environment. In addition, the average result achieved for glass fibre to resist alkaline solutions is higher than the average result achieved for glass fibre to resist the acid environment, as can be seen in figure 9.

The significant findings of this section of the paper are due to the differences in basalt and glass fibre capabilities to resist chemical attacks from different mediums. Some mediums possess a high pH value with high ion hydroxide or hydrogen content, while others contain low pH values. The reaction of fibres to different mediums differ greatly in acid and alkaline environments, therefore this section of the paper evaluates the chemical durability of glass and basalt fibres in different alkaline and acid environments. The performances of glass and basalt fibres were compared in terms of weight change, acid resistance, alkaline resistance, strength retention, and comparing both fibres' performance when resisting different alkaline and acid environments. Basalt fibre seemed to have gained less weight than glass fibres when boiled in alkaline and acid solutions. In terms of acid resistance, basalt fibre perfectly resisted all acids except HCl, while glass fibre suffered greatly and could not perform well to resist acid attack, in particular H_2SO_4. In terms of the alkaline resistance, both fibres performed well to resist the alkaline attack. However, basalt fibre seems to be affected mostly by the high alkaline NaOH, which reduced its tensile value significantly. On average, the strength retention of basalt fibres boiled in acid solution was significantly much higher than glass fibres. However, this gap was reduced when boiling both types of fibres in alkaline solutions. In general, the results showed that basalt fibres are more capable of resisting the acid environment, while glass fibres are more capable of resisting the alkaline environment. The next section will investigate whether glass or basalt fibres are more capable of resisting thermal heat, and carrying out surface observations and element change of fibre composition using scanning electron microscopy and microanalysis with complementary X-ray diffraction analysis (SEM/EDS).

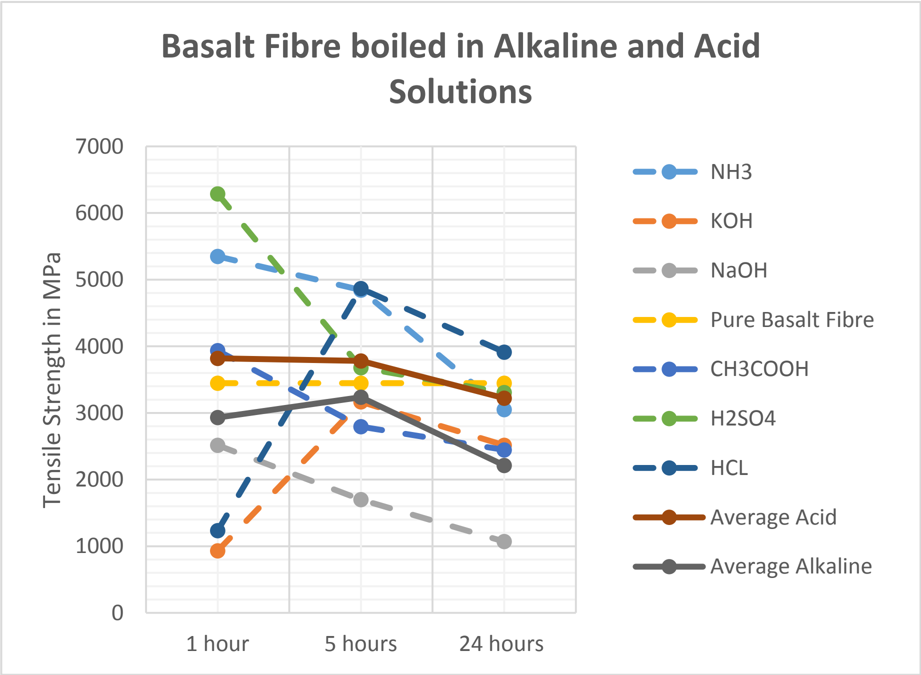

Figure 8. Performance of Basalt Fibres in different Alkaline and Acid Solutions

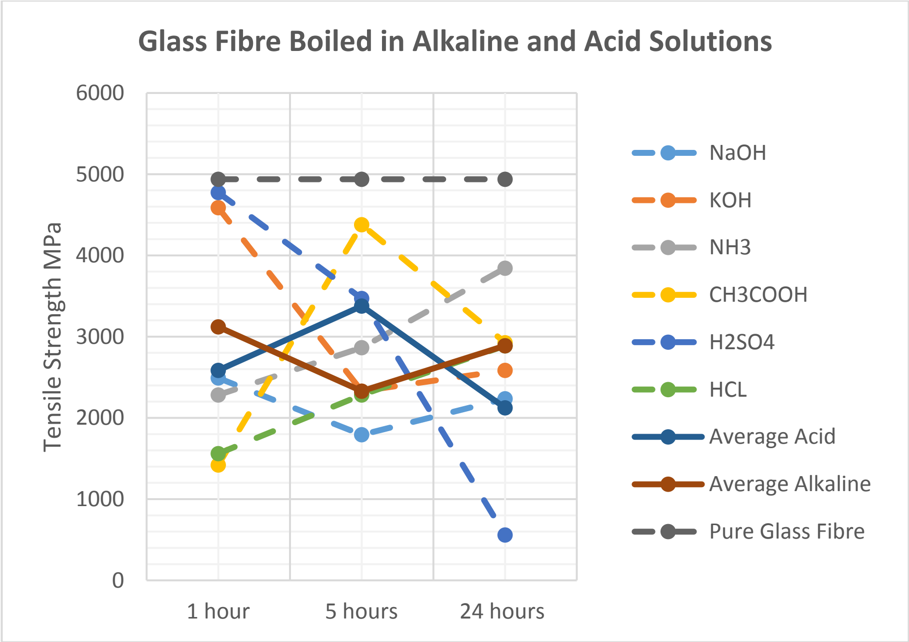

Figure 9. Performance of Glass Fibres in different Alkaline and Acid Solutions

3.5. Thermal Resistance of Fibres

When subjecting fibres to elevated temperature, the microstructure of the fibres degrades at different rates depending on the type of the fibre, atmospheric conditions and time of exposure. Fibres can normally resist heat above their normal operating temperatures, but elevated heat will begin to degrade the fibre. This degradation has the effect of reducing the tensile properties of the fibre and ultimately destroying its integrity [9].

To investigate the thermal stability of basalt fibres, the tensile strength of basalt fibres after they were subjected to elevated temperatures (50°, 100°, 300° and 500°) were measured after heating fibres for 1, 3 and 6 hours respectively. The thermal stability of glass fibres were also measured for comparison. Figure 10 shows the basalt fibres' tensile strength variation after heating fibres for 1, 3 and 6 hours at different temperatures. Before high temperature treatment, the tensile strength of pure basalt fibres were measured. From figure 10 it can be seen that, when basalt fibres were heated for 1 hour at different temperatures, there were no significant strength variation changes in the initial stage, but they then showed some differences after longer periods. When the heat was kept for 3 hours, at low temperatures (50°, 100°) the tensile strength of basalt fibre reduced significantly, whereas at higher temperatures (300°, 500°) the strength of the fibres reduced slightly. When the heating time increased to 6 hours, the strength of the fibres heated at different temperatures remained approximately unchanged. Basalt fibres had better strength retention at high temperatures (300°, 500°) than heating fibres at low temperatures (50°, 100°).

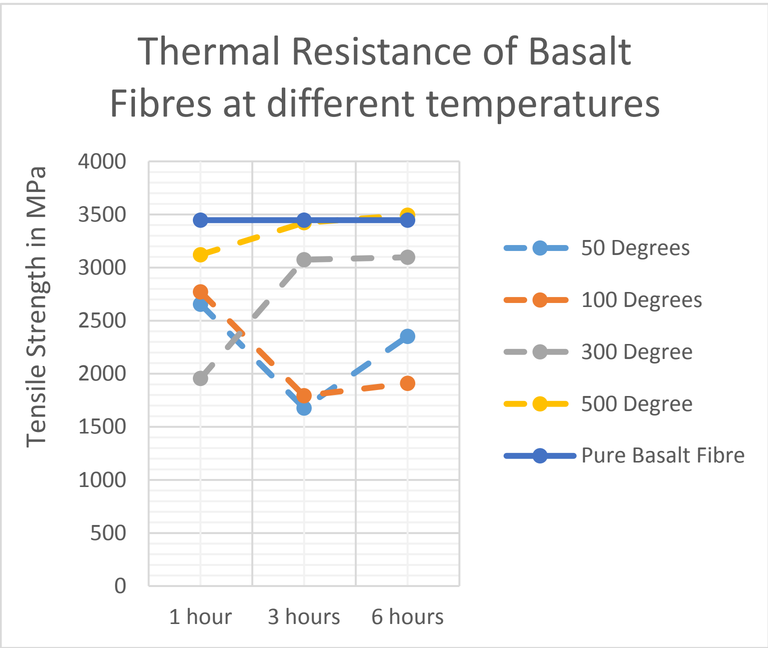

Figure 10. Thermal resistance of basalt fibres at different temperatures

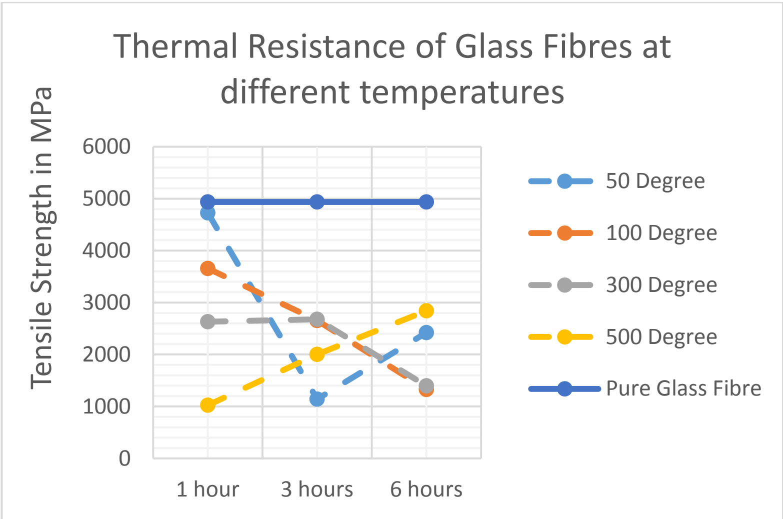

Figure 11. Thermal resistance of Glass fibres at different temperatures

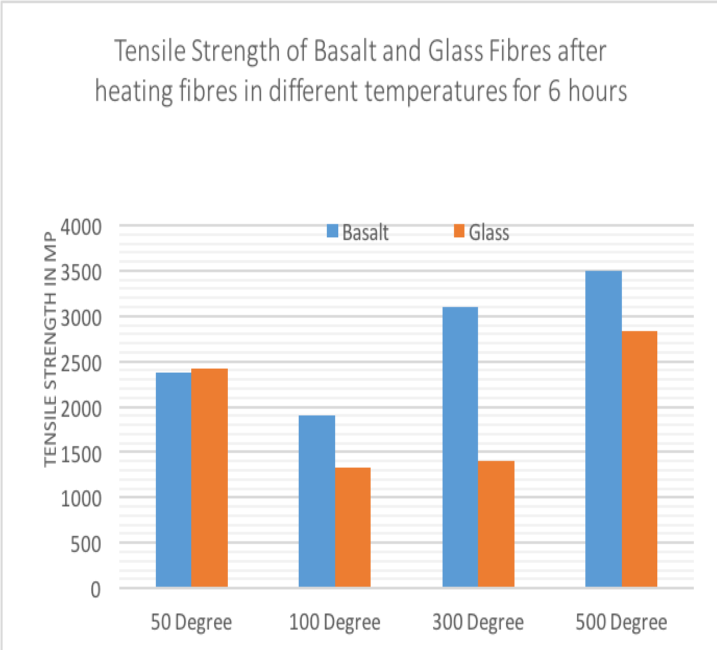

Figure 12. Tensile Strength of basalt and glass fibres after heating fibres for 6 hours

In contrast, heating glass fibres at different temperatures generated different results. Figure 11 shows that, after heating glass fibres for 1 hour, there was a significant variation in the strength values of the fibres heated at different temperatures. At low temperatures (50°, 100°) the strength of fibres seem to have been affected less than heating fibres at high temperatures (300°, 500°). After a longer period of heating (3 hours), the tensile strength values increased when heating fibres at high temperatures (300°, 500°), whereas this value reduced when heating fibres at low temperatures (50°, 100°). When heating time increased to 6 hours, glass fibre showed good strength retention from heating fibres at 500°, but showed weak strength retention to heating fibres at 50°, 100° and 300°.

To investigate basalt and glass fibres capabilities and compare their thermal resistance, figure 12 was drawn. Figure 12 shows the tensile strength of basalt and glass fibres after heating fibres in different temperatures for 6 hours. The significant findings in the thermal resistance experiment, as can be seen from the figure 12 below, is that, basalt fibres are more capable of resisting elevated heat, and maintaining their strength than glass fibres, in particular resisting temperatures of 300° and 500°. Whereas glass fibres are more capable of resisting low heat, in particular resisting temperatures of 50° and 100°, than basalt fibres. This result was also achieved by another researcher as he quoted [10] that basalt outperforms glass in the 300°-500°C range, and also by [11]. This is due to, the material characteristics of natural basalt rock, as it is capable of resisting high heat. The temperature stability of basalt fibres is controlled mainly by the ratio of Fe_2O_3:FeO, and also by the rate of oxidation. When fibres were heated below 300°, the ratio of Fe_2O_3:FeO tends to increase, which resulted primarily in the formation of magnetite (Fe_3O_4), and reforming the fibre structure which ensured the thermal stability of basalt fibres [12]. However when fibres are heated at a high temperature above 300°, the formation of hematite (Fe_2O_3) takes place as a result of oxidizing magnetite (Fe_3O_4), which improve the thermal resistance of the basalt fibre [13].This result confirmed that basalt fibres have a better thermal resistance than glass fibres.

In contrast, the correlation between boiling and heating basalt and glass fibres for 1 hour at 100°C is discussed. By using figure 8 and 10, its noticed that basalt fibre tensile strength value when heated for 1 hour at 100°C is 2771 MPa, however this value increased when boiling basalt and glass fibre in acid and alkaline solutions values to be 3819MPa and 2913MPa respectively. These results indicate the capability of basalt fibres to resist the chemical environment more than thermal environment. One the other hand, by using figure 11 and 9, it can be seen that glass fibres are more capable of resisting the thermal environment more than the chemical environment. Overall, it can be concluded that when the performance of basalt and glass fibres are compared, basalt fibre seem in general to have higher resistance to the elevated heat and to resist the acid attack more than glass fibres.

3.6. Fibers Surface Observation

Figures 13 to 20 show the SEM after basalt and glass fibres were boiled in three chemicals (Water, NaOH, HCl) for 24 hours. The appearance of basalt and glass fibres boiled in water and in the strong acid HCl change slightly compared to their original shape. As can be seen from figure 15 and 16, the presence of the defects on the surface of basalt fibres boiled in HCl is much less than glass fibres. The basalt fibres seem to be more compacted and smoother in comparison to the glass fibres. Although the glass fibres boiled in the HCl have a larger fibre diameter in comparison to basalt fibres boiled in the same solution, basalt fibres seem to have less voids, as can be seen from figures 17 to 18. The remaining shape of basalt fibres confirms the brittleness of basalt fibres in the acid environment. The same results were achieved by [7] as the researcher observed slight surface change to basalt fibres when immersed in the acid solution HCl. The combination of the basalt fibres' compaction and interaction with the matrix made its structure stronger to resist the acid environment.

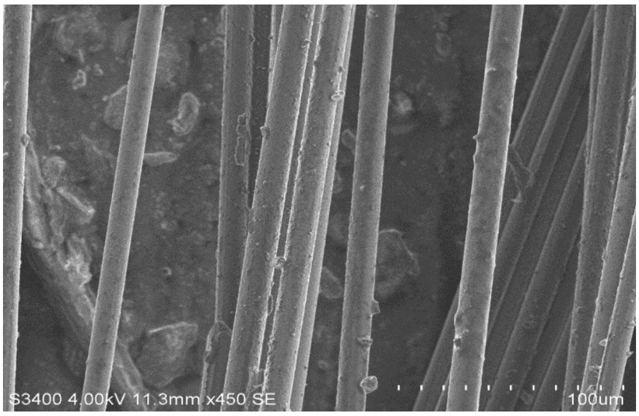

Figure 13. Pure Basalt Fibre

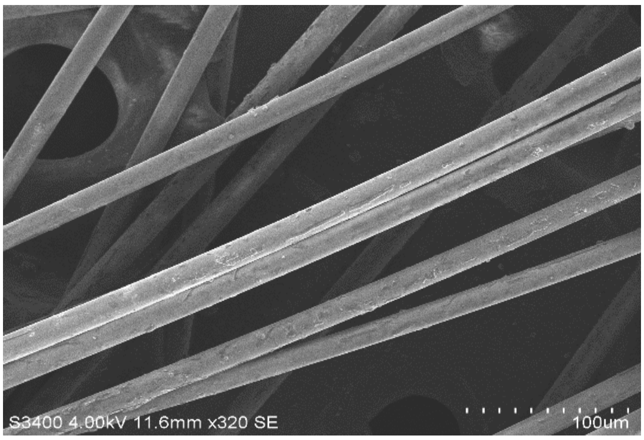

Figure 14. Pure Glass Fibre

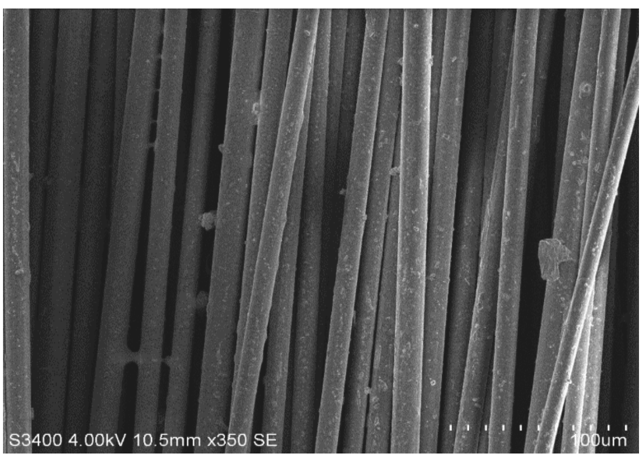

Figure 15. Basalt fibre boiled in HCl for 24 hours

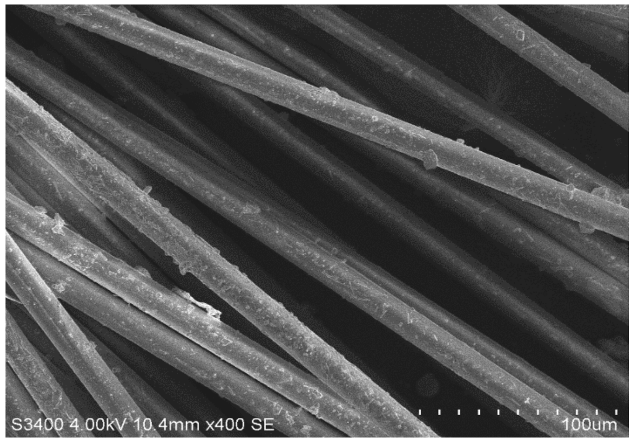

Figure 16. Glass fibre boiled in HCl for 24 hours

In the case of the sodium hydroxide (NaOH), it is very different. Figures 17 and 18 show the effects of the sodium hydroxide solution on basalt and glass fibres. As the alkaline solution penetrates basalt fibre, it breaks the Si-O-Si network due to the high presence of the hydroxyl ions which result in the creation of micro cracks and pores on the surface of the fibres [14]. As the hydration reaction continues, a hydroxide film is formed on the surface of the fibre, which repairs some of the surface defects and prevents fibres from further damage to some extent. However the fibre strength still reduces as a result of hydration, due to the creation of cracks which reduce its strength. With the passage of time, reaction products developed and fell apart, and consequently the strength of the fibres decreased dramatically. Research [7] found that the cracks on basalt fibres form only on its surface and do not penetrate to its core. Thus, the strength of basalt fibre boiled in alkali reduces slightly, while the appearance changes considerably as can be seen from figures 19 and 20. The same results were achieved, as shown in previous section of this research, as the fibres' strength reduced gradually when boiled in different alkaline solutions for long periods. In acid, although the appearance of the fibres looked smooth without the formation of a layer, the fibres' strengths were affected by the stress formed through numerous ion exchanges.

On the other hand, figures 17 and 18 show the hydroxide film that was formed on the surface of the basalt and glass fibres. Although glass fibre on average resisted the alkaline solutions more than basalt fibres, its physical appearance seems to have been affected by the hydration process. As a result, we see the formation of a thicker hydroxide layer on its surface in comparison to basalt fibres. The increase of the fibre strength of glass fibre compared to basalt fibre could be attributed to the thick protective film that was formed, which filled up the cracks on the surface of the fibres and prevented further damage [11].

In contrast, no traces of etching were detected on the surface of both fibres when boiled in water for 24 hours as can be seen in figures 19 and 20. The degradation of both fibres in water seem to have no significant effect on the fibres, and the appearance of both types of fibres when immersed in water seem unchanged. Meanwhile, the microstructure analysis shows that glass fibres have a slightly larger diameter dimension when compared to basalt fibres.

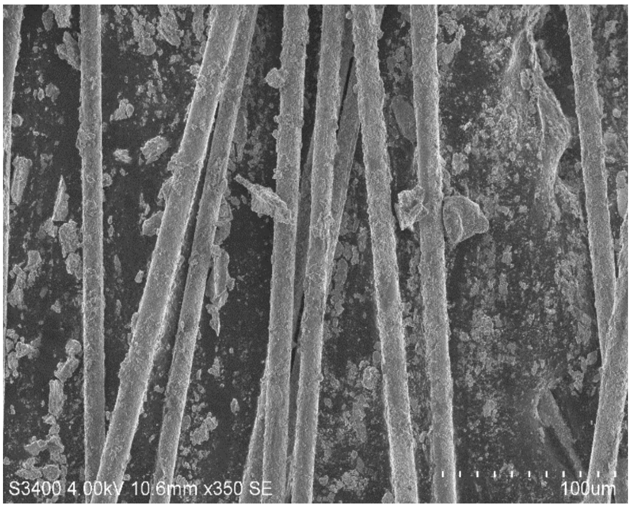

Figure 17. Basalt fibre boiled in NaOH for 24 hours

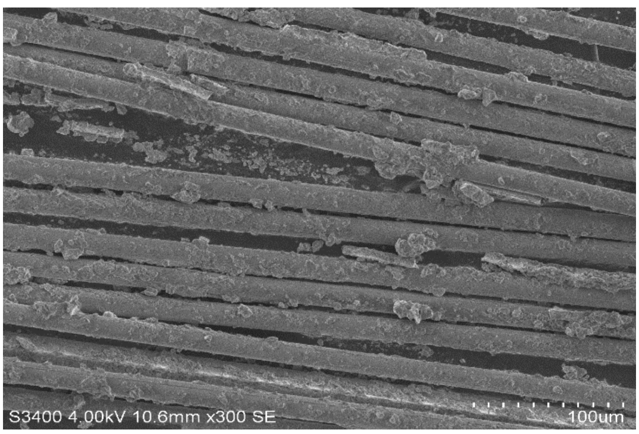

Figure 18. Glass fibre boiled in NaOH for 24 hours

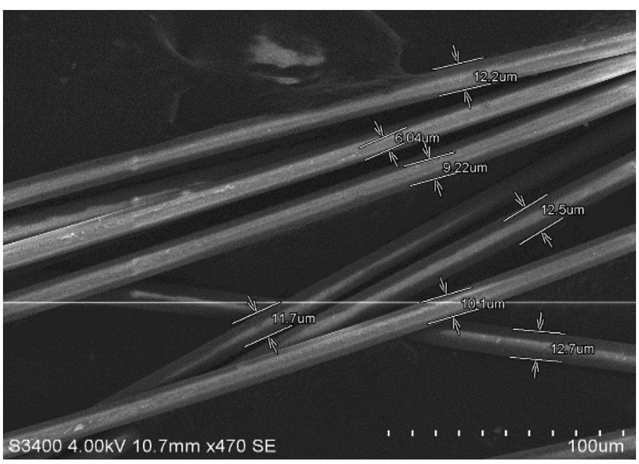

Figure 19. Basalt fibre boiled in Water for 24 hours

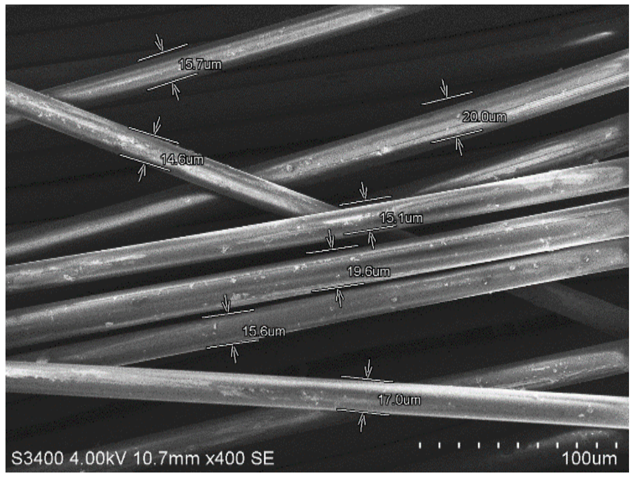

Figure 20. Glass fibre boiled in Water for 24 hours

At an elevated temperature of 500°, basalt fibres were able to maintain their original geometric shape, as can be seen in figure 21. In particular, the basalt fibre surface seems to be very clear and a thinner coating layer is formed when compared to the basalt fibres boiled in NaOH for 24 hours as can be seem in Figure 15. This confirms that the presence and chemical nature of the matrix had no significant influence on the phase formation of the basalt fibres. This is attributed to the formation of a nano-crystalline layer on the surface of the fibres as a consequence of the oxidation of Fe2+ to Fe3+. The layer remains after the bulk crystallisation process has been completed. The thickness of the layer increases with increasing the initial content of Fe2+ and Mg2+ in the fibres [15]. At the same elevated temperature, glass fibres seem to degrade and break into pieces, as can be seen from figure 22. This may be attributed to the fibres' lack of the nano-crystalline surface layer, as well as being due to an undetected crystallisation process in glass fibres at elevated temperatures [16]. These results indicate that basalt fibres have higher thermal stability than glass fibres. Nonetheless, further investigations of the high-temperature influence on fibre stability and strength is needed.

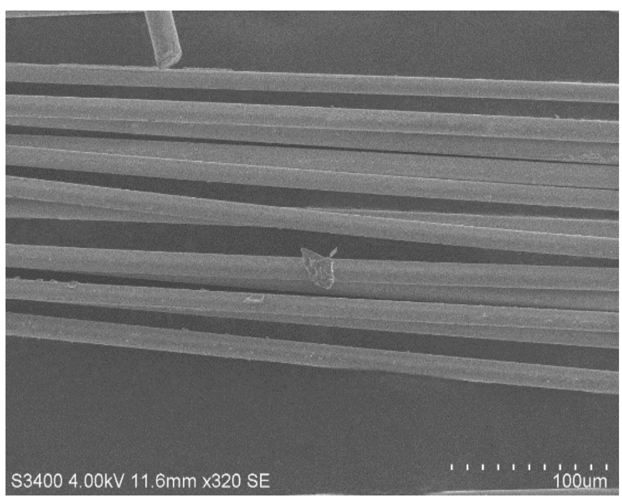

Figure 21. Basalt fibre heated at 500°

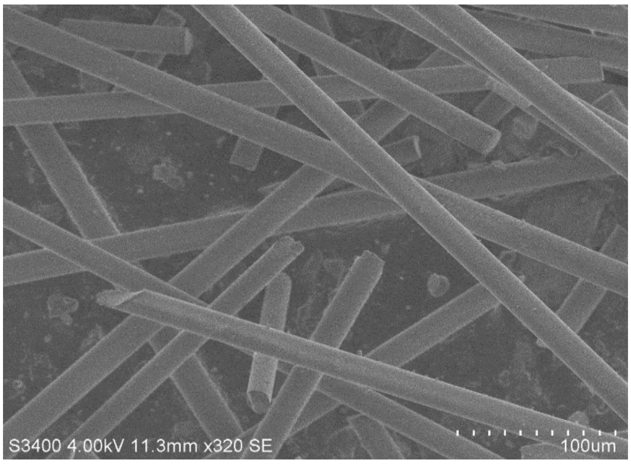

Figure 22. Glass fibre heated at 500°

3.7. Element Change of Basalt and Glass Fibers

In recent years, transition metal oxides have been studied with a view to understanding their effect on the fibres' strength. Therefore, the element content of basalt fibre after being boiled in water, HCl and NaOH were analysed using EDS and the results are shown in table 1. The EDS analysis showed that basalt fibre consisted of a number of oxides which have an essential impact on its properties. Dominant O_2 represented around 38% of its weight, followed by carbon (C) 26%, Silicon (Si) around 15%, and other oxides form just below 5% of its weight. Chemical properties in, particular Si, influences the mechanical properties of the fibres, especially when it reacts with an acid or an alkaline solution. From table 1 it can be seen that the element change of basalt fibre boiled in water for 24 hours stays almost the same as the element of the original fibre. In contrast, some elements' content, such as Ca, K, Ti and Fe, decreased slightly, but the content of carbon (C) increased rapidly. The slight change in the content of basalt fibre boiled in water confirms the excellent resistance of basalt fibre to water.

The element changes in acid and alkaline are different. In acid, the content of metal elements C and Ti decreased rapidly. However there is a high increase in the content of Silicon (Si). This is due to the replacement of the metal atoms by hydrogen ions (H+) and Chloride (Cl) from the acid [7]. This replacement will have an impact on the SI – O – Si structure, as a result increasing the content of Si and improving the fibre strength. This result was also achieved in previous section of this research-using figure 4, as the basalt fibre gained tensile strength when immersed in HCl compared to the original basalt fibre strength.

In an alkaline medium, the decrease of the Si element is observed when boiled in NaOH and notably the Na increases in the basalt fibre boiled in NaOH. This is due to the network being destroyed directly by OH. The reaction can be expressed as in equation (5):

$$\text{Si - O - Si + OH -------}\rightarrow \text{SiOH + OSi} \qquad (5)$$

As a result, the high amount of Si depletion might reduce the stability of the basalt structure and thus reduce its tensile strength. In general, the EDS analysis reveals the superior resistance of the fibre to acids, alkaline and water mediums. Figures 23 to 24 show the graphical full scale of element change of basalt fibres when boiled in, HCl, and NaOH.

The element content of glass fibre after being boiled in water, HCl, NaOH were analyzed using EDS and the results are shown in Table 2.

With regard to the chemical composition of glass fibres, it is very similar to the basalt fibre chemical composition, as can be seen from tables 1 and 2. This result confirms the potential ability of basalt fibres to replace glass fibres in several composites. However this similarity in the chemical composition can change when both fibres are exposed to different mediums such as an acid or an alkaline.

Table 1. Basalt Fibre Element Change

	The Element Change of Basalt fibres boiled in different mediums							
	Basalt fibre		Boiled in Water		Boiled in HCL		Boiled in NaOH	
Element	Mass ratio %	Atomic ratio %	Mass ratio %	Atomic ratio %	Mass ratio %	Atomic ratio %	Mass Ratio %	Atomic ratio %
O	38.87	42.52	33.81	31.00	49.25	65.54	35.64	33.05
Na	1.14	0.87	0.60	0.38	1.31	1.21	7.06	4.56
Mg	1.44	1.03	0.97	0.58	2.02	1.77	1.47	0.90
Al	5.43	3.52	5.66	3.08	5.66	4.46	4.21	2.31
Si	15.64	9.74	10.10	5.27	25.31	19.19	6.47	3.42
K	0.78	0.35	-	-	0.81	0.44	-	-
Ca	3.59	1.57	-	-	4.27	2.27	-	-
Ti	0.45	0.17	-	-	-	-	-	-
Fe	6.42	2.01	-	-	7.82	2.98	-	-
C	26.24	38.23	48.86	59.68	-	-	45.15	55.77
Cl	-	-	-	-	3.57	2.14	-	-

Table 2. Glass Fibre Element Change

	The Element Change of Glass fibre boiled in different mediums							
	Glass fibre		Boiled in Water		Boiled in HCl		Boiled in NaOH	
Element	Mass ratio %	Atomic ratio %	Mass ratio %	Atomic ratio %	Mass ratio %	Atomic ratio %	Mass ratio %	Atomic ratio %
O	39.96	41.92	50.86	66.71	32.55	31.00	36.86	34.63
Na	-	-	-	-	0.36	0.24	5.37	3.51
Mg	1.11	0.76	1.83	1.58	-	-	-	-
Al	3.96	2.46	5.80	4.51	2.96	1.67	1.76	0.98
Si	16.45	9.83	24.45	18.26	11.53	6.25	7.38	3.95
Ca	9.02	3.78	17.05	8.93	6.64	2.52	4.48	1.68

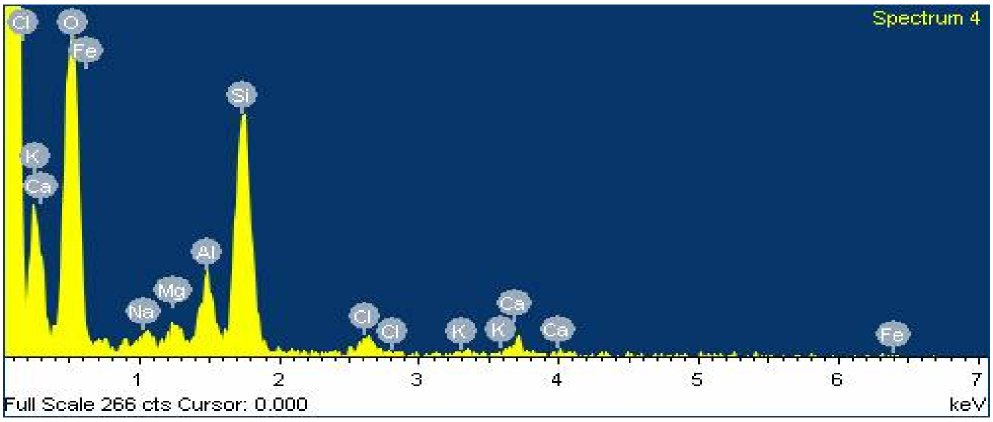

Figure 23. EDS analysis for Basalt Fibre boiled in HCl for 24 hours

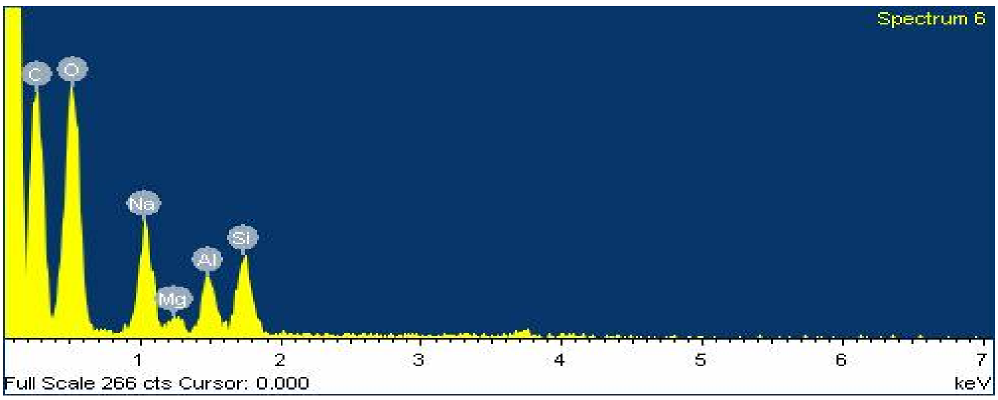

Figure 24. EDS analysis for Basalt Fibre boiled in NaOH for 24 hours

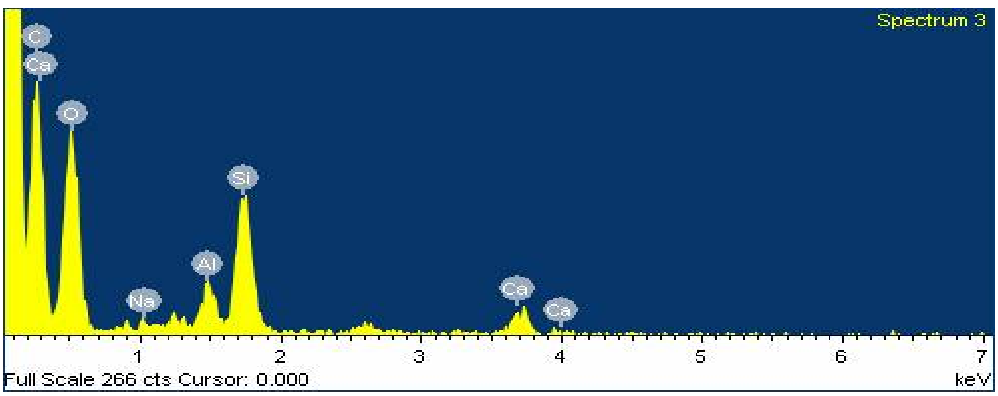

Figure 25. EDS analysis for Glass Fibre boiled in HCl for 24 hours

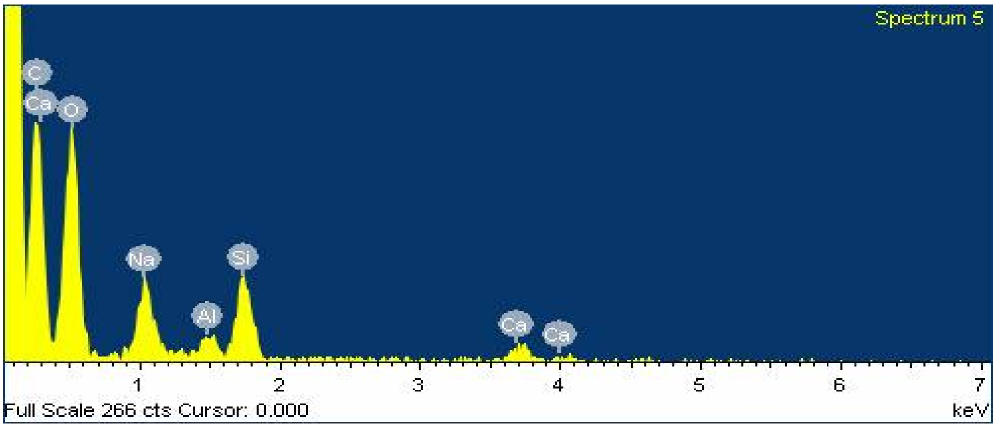

Figure 26. EDS analysis for Glass Fibre boiled in NaOH for 24 hours

From table 2 it can be seen that the chemical composition of glass fibre is dominated by Oxygen (O) representing 39% of the total weight, followed by Carbon (C) 29%, silicon 16% and other oxides representing up to 3%. The effect of the strong acid HCl on the surface of the glass fibre was clear as a result of an ion exchange reaction, which caused large quantities of magnesium, aluminium, silicon and carbon to be leached out from the glass fibre surface. This has a direct effect on the strength of the fibres, as the reduction of the silicon content has a large effect on the bond strength of the fibre structure network. In contrast, samples treated with strong base NaOH were found to have large quantities of sodium. This came from the sodium hydroxide that was used to treat the fibres. In alkaline solutions, the reaction of hydroxyl ions with Si–O–Si groups of the glass network leads to the formation of hydrated surfaces and dissolved silicate, aluminium and magnesium. The corrosion mechanism changed dramatically when glass fibre was boiled in water. The element content of the glass fibre increased when it was exposed to water. This indicates that glass fibres have greater resistance to water. However this resistance reduced when glass fibres where boiled in alkaline and acid solutions, as there was a reduction of the fibre element content. Figures 25 to 26 show graphically the full scale of element change of glass fibres when boiled in, HCl, and NaOH.

4. Conclusions

In this paper, a new method of evaluating the efficiency of fibres to resist the alkaline, acid, and thermal environments is proposed. The proposed method is applied by boiling basalt and glass fibres in different strong and weak alkaline and acid chemicals containing different pH levels, as well as subjecting fibres to several thermal temperatures. This comprehensive investigation of fibres' durability has outlined the capability of fibres to resist several chemical environments, and shown the ultimate benefits that can enhance composite durability. Basalt fibres were tested to resist temperatures achieved during typical cement hydration which is approximately 74°C according to [18], also being tested at fire level temperatures, as concrete begins to degrade above 300°C [19]. Another issue occurs when constructing concrete bridges. Due to carbonation, the alkalinity of the pore fluid drops from a pH value exceeding 12.6 to a value about of 8.0 [20]. On the other hand, concrete used in waste water treatment such as waste water tanks is exposed to harsh chemicals such as acids, salts and sulphates, ph level between 2 – 6, and if these chemicals have had a chance to get inside the concrete, they will cause damages [21]. As a result, fibres were tested against several heat degrees and different chemicals containing different ph levels. The results show that basalt fibres seem to have gained less weight than glass fibres when boiled in alkaline and acid solutions. In addition, basalt fibres have performed much better in resisting the acid environment than the alkaline environment. On the other hand, the average result achieved for glass fibre to resist alkaline solutions is higher than the average result achieved for glass fibre to resist the acid environment. The paper also confirmed that basalt fibres are more capable of restoring their strength than glass fibres, more capable of resisting high heat, and can maintain their strength better than glass fibres, in particular resisting temperatures of 300° and 500°. In contrast, glass fibres are more capable of resisting low heat, in particular resisting temperatures of 50° and 100° better than basalt fibres. In addition, the scanning electron microscope showed that basalt fibres seem to be more compacted and smoother when boiled in HCl compared to glass fibre, and a film was formed on the surface of the basalt and glass fibres when immersed in NaOH. There was a variation in the element change of basalt and glass fibres when boiled in different alkaline and acid solutions, especially for the glass fibre as it lost many of its original elements.

ACKNOWLEDGEMENTS

I would like to thank Dr. Gareth Whittleston for his contributions to this study, and David Simmons for supplying the basalt fibres required to do this research.

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