

Research Article

Effect of Aging Process in Different Solutions on Kenaf Fibre Structure and Its Interfacial Adhesion in Epoxy Composites

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Interfacial adhesion of kenaf fibres in epoxy composites was investigated using single fibre pull-out test. Several aged kenaf fibres were tested in this work. Two types of kenaf fibres were used in the work, those treated with 6% NaOH and those untreated kenaf fibres. Kenaf fibres were aged in engine oil, water, salt water, and diesel. The pull-out tests were performed using microtensile tests. The tests were performed at 1 mm/min loading rate. Scanning electron microscopy was used to observe the damage on the fibres and the effect of the treatment. The general results revealed that aging of the fibres reduced their strength and interfacial adhesion. Salt water showed the least effect on the strength of the fibres. At most cases, the breakage in the fibres is the main failure. In other words, there is no remarkable effect of aging on the interfacial adhesion since the most impact was on the structure of the fibres.

1. Introduction

Fibre polymer composites are increasingly replacing the conventional materials in several industrial applications due to their low weight, low cost, and high specific strength compared to conventional metal materials. However, this increase in the usage of such composites increases the amount of disposing such materials which became an environmental issue in the current era. Reducing the impact of polymer composites is the ambition of many current researchers and environmental activities.

Nowadays, natural fibres are becoming very attractive alternative candidate to the synthetic fibres. Natural fibres have the advantages of the biodegradability, availability, low cost, and ease of processing compared to the syntactic fibres [1–4]. Kenaf fibre is one of the most popular natural fibres due to its high growth, high strength, and good structure compared to other natural fibres, [5–8]. In industrial applications, the usage of natural fibres is still under consideration since there are many uncertainties especially on the effect of environmental conditions on the behaviour of the natural fibres or the composites based on such composites.

On the other hand, no much work has been reported on the influence of environmental conditions on the natural fibres behaviour [9]. Meanwhile, some other works have been reported on the effect of the water absorption on the strength of polymeric composites based on natural fibres [10–12]. In recent review article [13], it has been reported that water absorption significantly affects the structure of the natural fibres and attacks the interfacial adhesion of the fibres with the matrix.

2. Material Preparation

2.1. Fibres Preparation. Raw kenaf fibres were obtained from Malaysian farm. The fibres fist was cleaned and extracted. The extracted fibres were cleaned with water, combed, and then dried. The fibres were cut in 50 mm length. The quality of the fibres was ensured by optical microscopy observation. Diameter of the fibres was in the range of 0.31 mm 0.35 mm. Part of the fibres was chemically treated with NaOH at concentration of 6% for 24 hrs at room temperature. In the treatment process, the fibres were immersed in the solution and then washed with water. The fibres were then dried in

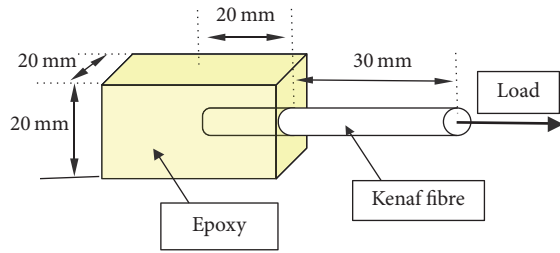


FIGURE 1: Schematic drawing of pull-out samples.

oven at temperature of 50°C for 24 hr. A mould of $20\text{ mm} \times 20\text{ mm} \times 40\text{ mm}$ was fabricated from a metal. Epoxy resin was used as the matrix with a hardener with a ratio of 50%.

2.2. Fabrication of the Samples and Experimental Procedure. In the fabrication process, the mould was coated with a thin layer of release agent to ensure the ease of the sample removal after curing. The fibres were fixed in vertical direction using string fixed in the bottom of the mould. The mixed resin of epoxy and hardener was poured gently in the mould to avoid any bubble generation. Figure 1 shows schematic drawing of the prepared samples and the dimensions. The volume fraction of the fibres in the composites is the most important key in determining the amount of the fibres in the composites. Accordingly, the weight the fibres used in the composites is considered before fabricating the composites. However, in terms of the number of fibres in each weight, the number is determined before conducting the test to determine the strength and the pull-out of the composite accordingly.

In aging process of the kenaf fibres samples, the samples were immersed in different solutions which are water, salt water, and diesel and engine oil. The samples were kept in the solution for three months and then removed from the solution. The samples were dried from the solution by using high absorbed materials. The samples were then placed in the machine for testing. In the testing process, the epoxy part of the samples was gripped on the universal tensile machine. The tensile machine was at low capacity of 10 kN only since the failure of the fibres is below the maximum capacity of the machine. The gripper is special for fibres. It should be mentioned here that the difficult part in the work is fixing the fibres in the holder of the machine. More than 5 attempts have been made for each test to gain the accurate value of the test. Average of three matching results was reported. In addition, due to the difficulties in fixing the samples on the machine, bundle of the fibres was used at different amount of fibres. 0.02 g–0.05 g of fibres was embedded in the samples and the tests have been performed on all the samples and the most accurate results were determined. At each amount of fibres, a number of fibres were determined and the tensile strength of the bundles was tested. Accordingly, the single fibre pull-out test was determined.

3. Results and Discussion

The results of the current study are divided into two parts as untreated kenaf fibres and treated kenaf fibres. The stress

versus deformation is presented associated with the average values of the each set of tests. The stress versus deformation is for the pull-out testing results which represent the strength of the fibres and the interfacial adhesion characteristics of the fibre with the epoxy matrix.

3.1. Untreated Aged Kenaf Fibres. The stress versus strain of aged untreated kenaf fibres is given in Figures 2(a) and 2(b). In Figure 2(a), the stress is very scattered. However, elastic region of the tensile result can be seen. After reaching the peak of the elastic stress, fluctuation can be seen which is due to the breakage of several fibres in the bundle. Figure 2(a) shows the results of 0.04 grams of the kenaf fibres aged with different solutions of water (w), salt water (SW), diesel (D), and engine oil (EO). V represents the virgin fibres which have not been aged or treated. Maximum strength can be seen when virgin fibres were tested which achieved about 110 MPa. In Figure 2(b), the average of the strength of the fibres after the pull-out test is presented. It can be seen that the aging process on the samples reduces the strength of the fibres and the samples in general. This is very obvious in the case of the engine oil aging. In the case of the water and salt water, the effect was not that bad. It seems that the diesel and the engine oil acted as lubricant in the interface of the fibres with the matrix which assist in the putting out mechanism resulting in low strength. In the case of the salt water, it seems that some of the sodium particles were formed around the fibres which interlocked the fibres in the samples. However, due to the presence of the water, there is deterioration in the structure of the fibres rather than the interfacial bonding area. This has been reported previously in composite aging of coir fibres [14].

3.2. Treated Aged Kenaf Fibres. Kenaf fibres were treated chemically with 6% NaOH solution. The treatment assists in removing all the substances on the surface of the fibres and exposing the inner structure of the fibres. The results of the aged fibres after pull-out testing are given in Figures 3(a) and 3(b). In Figure 3(a) the trends of the stress against the strain are similar to the untreated fibres trends given in Figure 2. However, the scattered values are much less than the ones given in Figure 2 which could be due to the removal of the undesirable substances on the surface of the fibres. In addition, it can be seen that the virgin treated fibres have the highest strength of about 155 MPa which is much greater than the untreated fibres. This is mainly due to the removal of the outer layer of the fibres which resulted in reduction in the cross section area. In other words, the outer layer has no strength at all and including it in the calculation of the strength resulted in reduction in the strength of the fibres. With regard to the effect of aging process on the strength of the samples, Figure 3(b) shows the averages of the strength of the aged samples. Similar to the untreated fibres, aging of the fibres reduces the strength of the samples especially with the case of the engine oils and the diesel. There could be built-up boundary around the fibres in the composites while it is under aging process in engine oil or diesel. This can reduce the interfacial bond between the fibres and the epoxy which resulted in poor strength. On the other hand, the treatment

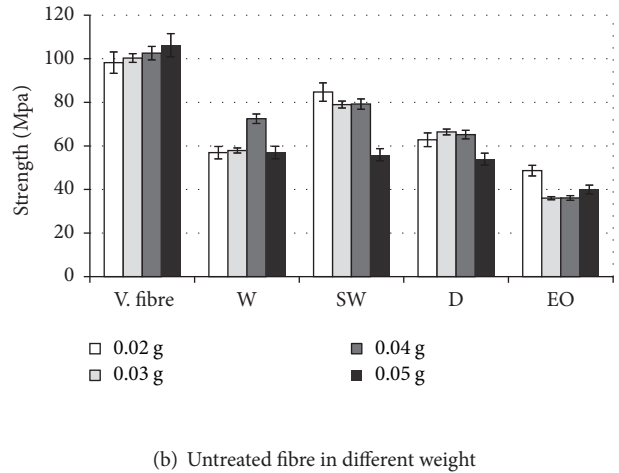
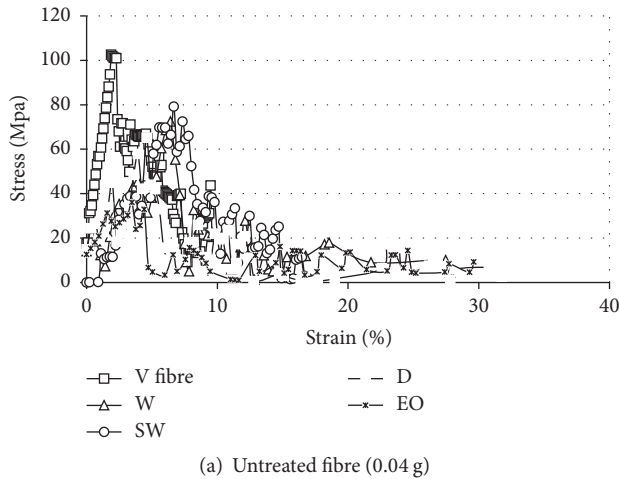


FIGURE 2: Tensile behaviour of untreated kenaf fibres with different solution.

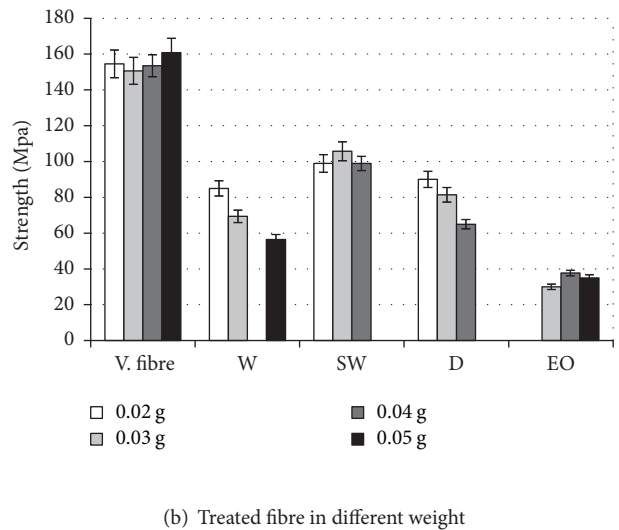
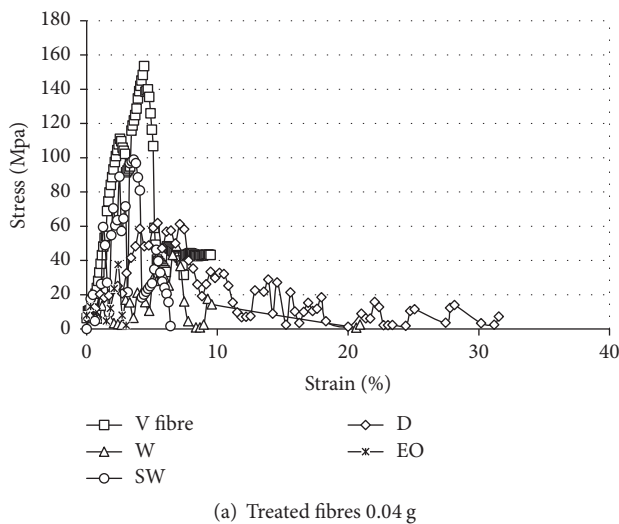


FIGURE 3: Tensile properties of treated fibres and aging in different solutions.

of the fibres assisted in dropping the effect of the aging since the reduction in the strength of the treated aged fibres is much less than the untreated fibres. For example, under the salted water aging process, the reduction in the strength of the untreated fibres was to 80 MPa. Meanwhile, the reduction in the cases of the treated fibres was about 100 MPa. In other words, treating the fibres gives the opportunity to use the composites of epoxy based on kenaf fibres in salted environment.

The above results showed that adding of the composites in solution reduces the strength of the composites after three months. Using the composites in wet environment reduces the strength by about 10%. In other words, in component design a factor of environment should be considered. This fact is about 0.6, 0.8, 0.6, 0.6, and 0.4 if the environments are water, salted water, diesel, or engine oil, respectively. Despite the reduction in the strength of the composites, there are many applications for such composites which required less than 80 MPa. Aluminum alloys materials have a strength of

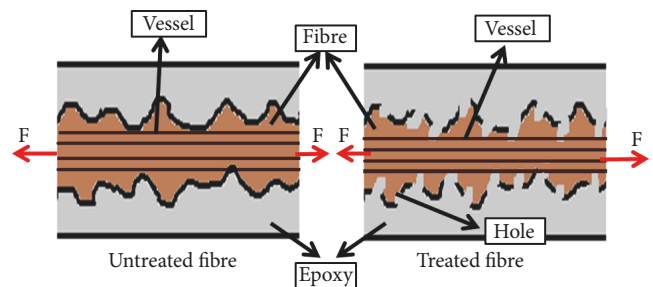
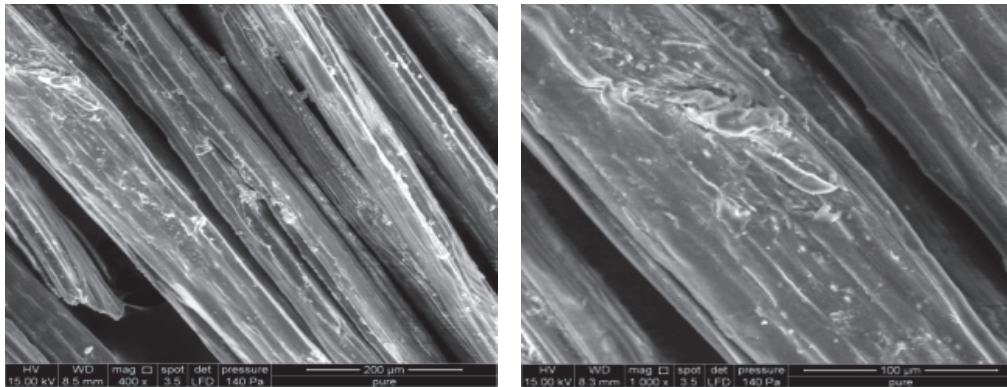


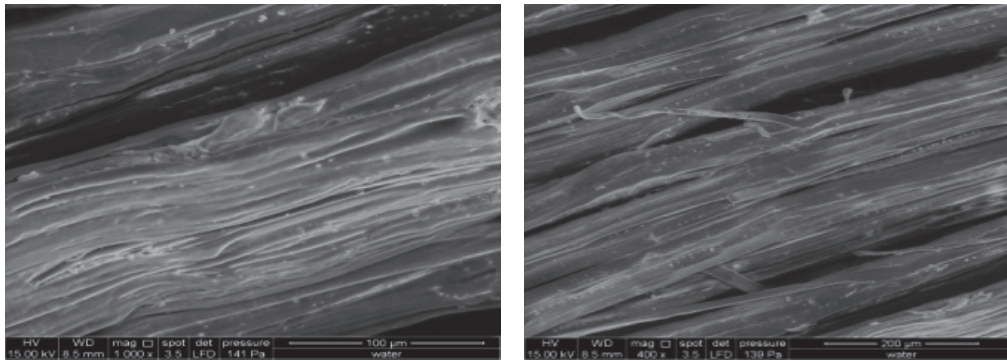
FIGURE 4: Behaviour of fibre in composites.

about 70 MPa which is used for tank, vessel, ships, and other marines' applications. Using kenaf fibres reinforced epoxy composites for similar applications is possible.

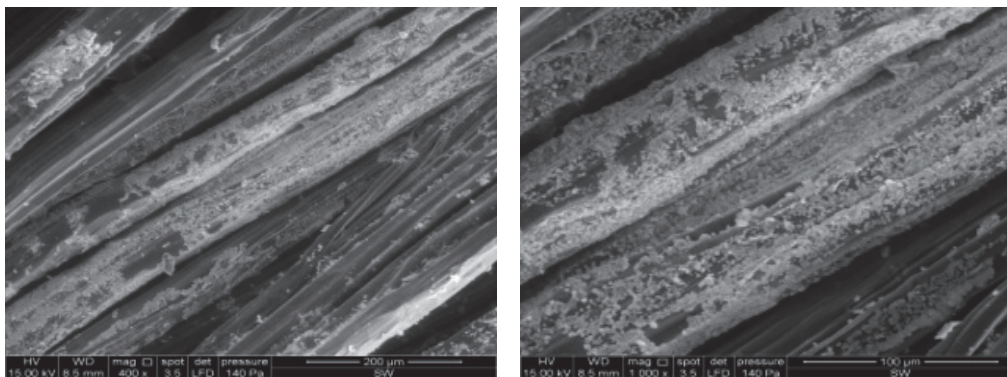
With regard to the effect of the fibre weight bundle in the strength of the single fibre and the pull-out characteristics, both treated and untreated fibres did not show a remarkable



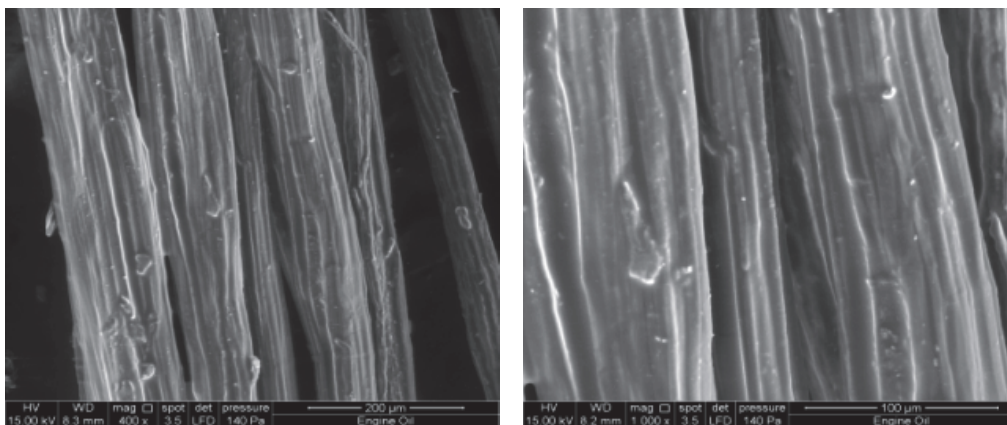
(a) Untreated virgin fibre



(b) Untreated fibre aged with water

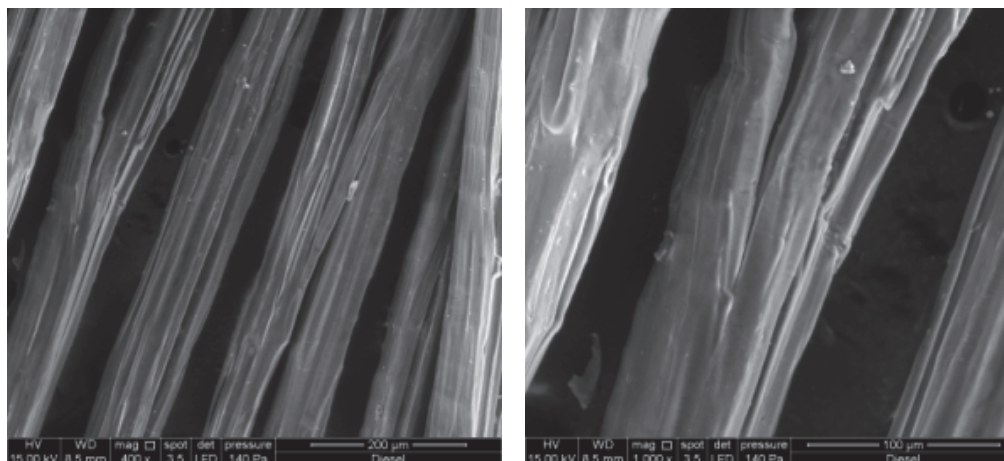


(c) Untreated fibre aged with salt water



(d) Untreated fibre aged with diesel

FIGURE 5: Continued.



(e) Untreated fibre aged with engine oil

FIGURE 5: SEM micrograph of aged untreated kenaf fibre sample.

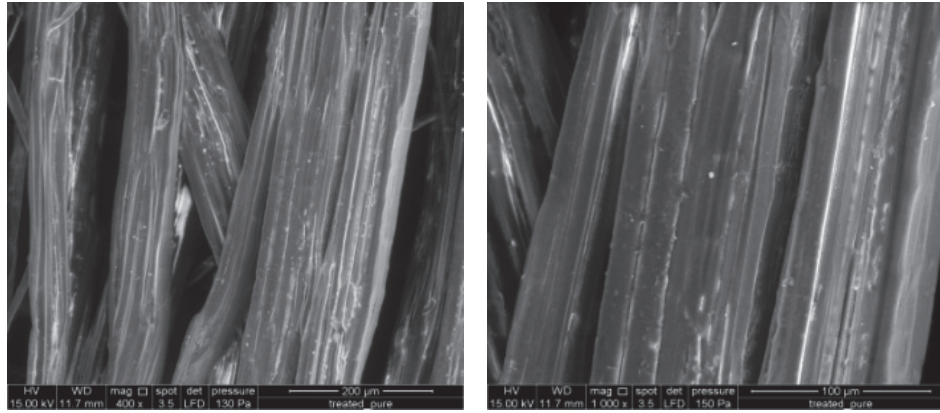
influence of bundle weight on the strength of the single fibre or the pull-out results. There is a bit of increase in the overall strength of the composite when the composite was not aged. In the cases of the aged composites, there is reduction in the overall strength of the composites with the increase of the weight of the fibre bundle. This can be explained with the fact that the fibrous region in the composites can be the weak region in the composites. Under the aging process, the fibres are weakened and the region of the fibres/matrix is deteriorated as well. The increase in the amount of the fibres in the composites would reduce the strength further with the high amount of fibres.

The failure mechanisms of the samples seem to be controlled by the treatment of the fibres. Figure 4 shows the possible failure in the case of treated and untreated fibres. In the case of untreated fibres a waxed layer is used to cover the fibres and prevent the epoxy to enter the fibres during the curing process. Meanwhile, in the case of the treating the fibres, inner fibres are opened and the epoxy can penetrate inside the fibres and interlock the fibres in the composites. For the aging process, the untreated fibres showed poor results since there is double action in the interface between the fibres and the matrix which is the presence of the waxy layer and the solution as well which resulted in very poor interfacial adhesion of the fibres with the matrix.

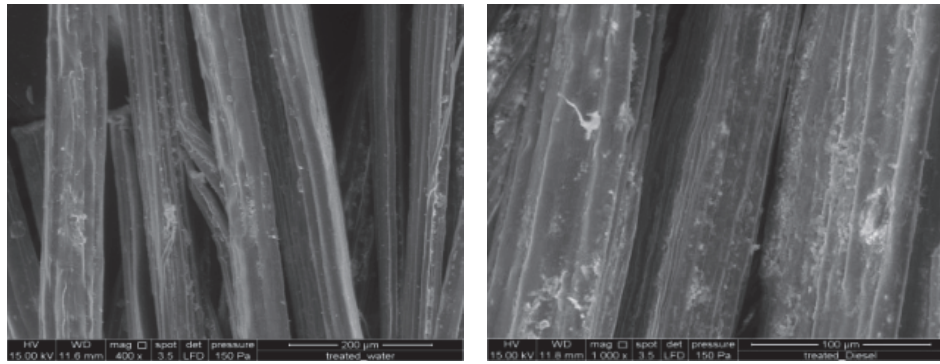
3.3. SEM Observation. The scanning electron microscopy was used to observe the structural changes resulting from the aging effect on the samples focusing on the fibres. Figures 5 and 6 show the SEM micrographs of the untreated and treated kenaf fibre samples in different aging solution, respectively. In Figure 5(a), the untreated kenaf fibres seem to have a covered layer on the surface of the fibres associated with loss substances. Also, some of the fibres seem to have inner fine fibres which are in bundle form. This has been observed in the literature as well with other natural fibres such as coir [15], date palm [16], and oil palm fibres [17]. This layer on the kenaf surface would prevent the penetration of the epoxy during the curing causing some debonding around the interfacial area of

the fibre with the matrix. This can be the reason of the poor performance of the untreated fibres. With the aging process with water, Figure 5(b) shows the exposed fibres were slightly cleaned with the splitting of the fibres into fine fibres. This can be due to the degradation process taking place on the fibres during the aging duration. In other words, there is some decomposition to the fibre itself which would weaken it and result in lower performance of the aged composites in water compared to the unaged samples. With the case of the salted water aging, Figure 5(c) shows a covered layer of salt on the surface of the fibres. It may be proposed that some of the salt may enter the fibre structure which can interlock the fibres in the matrix. This may result in better performance in the case of the salted aging process compared to the pure water. Aging in diesel and engine oil has dramatically impacted on the fibre structure as can be seen in Figures 5(d) and 5(e). It is strongly not recommended to use the kenaf fibres in an environment which has either diesel or engine oil.

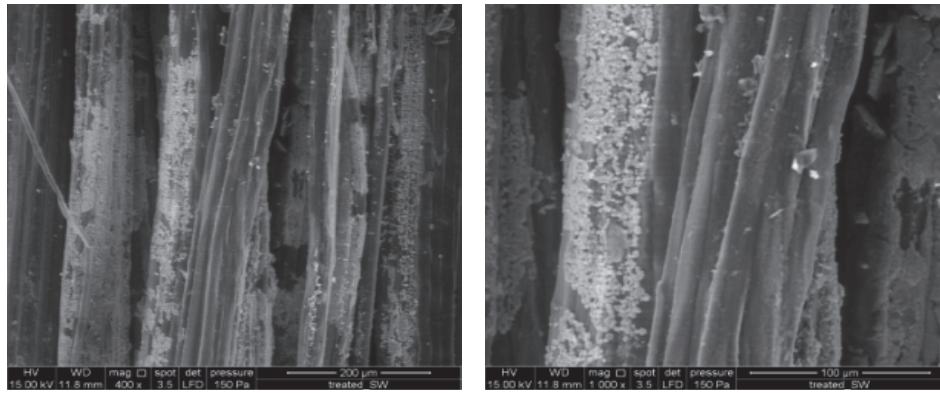
With regard to the influence of the 6% NaOH treatment on the aged samples, Figures 6(a)–6(e) show the micrographs of the samples with different aging processes. Firstly, Figure 6(a) in comparison to Figure 4 shows that the treatment of the kenaf fibres significantly improved their surface since the undesired substances were completely removed and the inner fibres exposed. This would allow the epoxy to enter the bundle of the fibres during the curing and strengthen the interfacial adhesion of the fibres with the matrix. However, Figure 6(b) shows that aging the samples with water damaged the structure of the fibres which weakened the strength of the fibres and this can explain the poor performance of the composites. Aging the treated samples with salted water exhibited similar effects to those of the untreated ones as can be seen in Figures 5(c) and 6(c). In both cases, the salt was adhered to the surface of the fibres which can help to strengthen the interfacial adhesion of the fibre with the epoxy. Diesel and engine oil damaged the structure of the treated fibres as can be seen in Figures 6(d) and 6(e).



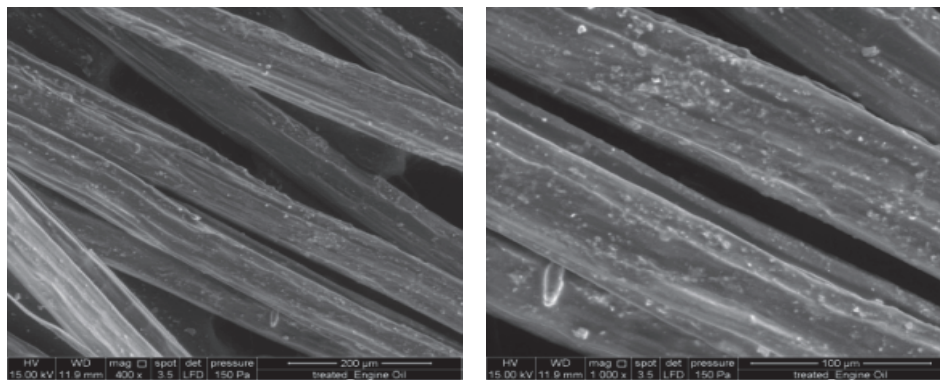
(a) Treated virgin fibre



(b) Treated fibre aged with water

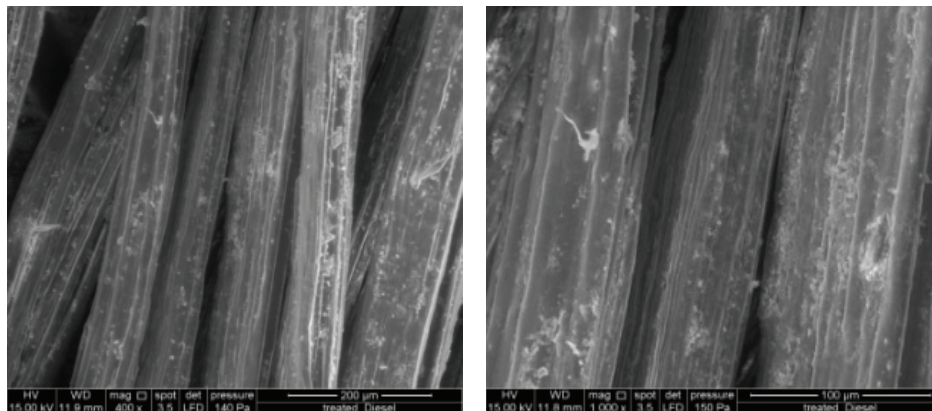


(c) Treated fibre aged with salt water



(d) Treated fibre aged with diesel

FIGURE 6: Continued.



(e) Treated fibre aged with engine oil

FIGURE 6: SEM micrograph of aged treated kenaf fibre sample.

4. Conclusions

After conducting the pull-out tests on the treated and untreated kenaf fibres reinforced epoxy and analyzing the results, the main findings can be concluded as follows:

- (i) Treating the fibres with 6% NaOH significantly improved the interfacial adhesion of the fibres with the matrix.
- (ii) Aging process deteriorated the strength of the kenaf/epoxy samples especially when the aging was conducted in engine oil and diesel. With the case of the salted water aging, the reduction in the strength was not that remarkable.
- (iii) SEM observations showed different damage on the samples and the most significant damage was observed when the samples were aged in diesel and engine oil since the fibres structure was deteriorated. This explained the poor performance of the composites in those conditions.
- (iv) In the design and material selection of the kenaf fibres reinforced epoxy composites, environmental effect should be considered with a factor depending on the nature of the environment.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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