

Low Velocity Impact Response of Kenaf/Epoxy Composites Exposed to Corrosive Solutions

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ABSTRACT

Kenaf fibres are one of the natural fibres that have been widely explored since the fibres are low in cost, have excellent mechanical properties and biodegradability features. Recently, there have been numerous efforts to employ kenaf fibres reinforced composites in a wide range of applications. One of the concerns is that these composites may be exposed to several ranges of corrosive conditions during their in-service life and their durability performance is extremely necessary. Therefore, the aim of this work is to study the mechanical response subjected to flexural and low velocity impact tests of kenaf/epoxy composites with a variety of corrosive solutions, after being exposed to hydrochloric acid (HCl) and sodium hydroxide (NaOH) solutions for durations of 12, 24 and 36 days. The experiment result demonstrated that the corrosive solutions have a detrimental effect on the flexural properties and impact response of composites. The degradation effect on composites is also found to be dependent on the exposure period in the corrosive solutions. On the other hand, acidic environments have been shown to have a more aggressive effect on the performance of composites than an alkaline environment.

Keywords: *Kenaf Fibre Reinforced Composite; Corrosive Condition; Flexural Properties; Low Velocity Impact*

Introduction

A worldwide desire for more efficient and less costly ecologically friendly goods has increased interest in natural fibres. This has prompted industries

such as furniture, automotive, construction, and packaging to look for new fibre-reinforced that can replace traditional composite materials that rely on synthetic fibres (glass fibre, carbon fibre) for reinforcement [1]. Among natural fibres (kenaf, flax, jute etc), kenaf fibres scientifically known as *Hibiscus Cannabinus* L. from the Malvaceae family is one of the potential materials as reinforcement in polymer composites because of their fast growth, mechanical properties and low prices [2]. The performance of the kenaf fibre reinforced in polymer composites has been extensively studied. For instance, Tharazi et al. [3] explored the processing parameters of composite manufacturing using design of experiment (DOE) on the kenaf/PLA composites while other authors such as Razali et al. [4] investigated the damage behaviour of misaligned kenaf fibres during composites fabrication.

The application of kenaf reinforced composites may involve highly corrosive environments such as tanks, boat hulls, containers and pipes. Hence, the durability of this material under such corrosive environments i.e. alkaline and acidic conditions is important. Mechanical properties and behaviours of fibre reinforced polymer composites after immersion in alkaline solutions have been openly reported in the literature. Stamenovic et al. [5] compared the tensile properties of glass/polyester composites after immersion in alkaline solutions. In comparison with control specimens, they reported the tensile properties of alkaline immersion were reduced substantially. Deterioration in composites was more pronounced in the tensile modulus than in the modulus of elasticity. Similar studies were taken into consideration in the case of natural fibre composites. Immersion of natural fibre polymer composites in sodium hydroxide (NaOH) resulted in the loss of tensile properties of coir/polyester composites [6]. A prolonged immersion test in the 5% NaOH solution for up to 365 days caused flax/epoxy composites to lose 21% of flexural strength and 25% of flexural modulus, respectively [7].

Many efforts have also been made to study the influence of acidic environments. Mahmoud et al. [8] studied the influence of strong acidic concentrations of glass/epoxy composites in hydrochloric acid (HCL), phosphoric acid (H_3PO_4), nitric acid (HNO_3) and sulfuric acid (H_2SO_4). They found the hardness, flexural, and impact properties remained consistent for 30 days of immersion in comparison with control specimens. However, the mechanical properties decreased after immersion in acidic environment for a duration of 60 days and 90 days. However, in the case of the Stamenovic et al. [5] experiment, the tensile properties of glass/polyester composites showed a gradually increased with increasing immersion time, 10, 30, and 50 days in the phosphoric acid (H_3PO_4) and nitric acid (HNO_3) solutions. A similar tendency was also reported for natural fibre polymer composites, the tensile strength of coir/polyester increased gradually with higher immersion time in acidic environments [6].

A material needs to absorb and dissipate energy in the event of impact loadings while maintaining its structural integrity. Impact damage can be

initiated by a crash, falling debris or an object of a foreign body. Low velocity impact is characterized by the impact incident that took place below 10 m/s [9]. The impact damage can be manifest either in a complete perforation or show no sign of damages resulted by the impactor rebounding on the specimens. The study of low velocity impact response on both corrosive acidic and alkaline environments on fibre reinforced polymer composites was reported by several authors. Amaro and co-workers [10] found that impact resistance was severely compromised with immersion in NaOH solutions as compared to the HCL solutions. Similar work was carried out by Mortas et al. [11] on the kevlar/epoxy composites and carbon/epoxy composites with further attention being given in terms of NaOH and HCL concentrations. These authors concluded the impact response and flexural properties are heavily dependent on the solution concentrations and temperatures.

Current literature has shown that kenaf fibre reinforced polymer composite is a potential material for a wide range of applications. However, there have been limited studies on the low-velocity impact response of composites reinforced with natural fibres like kenaf exposed to corrosive conditions. Therefore, the aim of this study is to evaluate the low velocity impact performances and flexural properties of woven kenaf/epoxy after immersion into hydrochloric acid (HCl) and sodium hydroxide (NaOH) solutions where a comparison was made with the control samples. The flexural test was chosen in addition to the low velocity impact test which is due to the sensitivity of the bending test towards the changes in exposure conditions [12]. Various of exposure duration on the kenaf/epoxy composites was also considered to provide real environmental conditions.

Methodology

Materials

The natural fibre fabric used in this study was woven kenaf out of courtesy of the Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia (UPM). The fabric was initially cut into 290 mm by 200 mm before composites fabrication. Low viscosity epoxy resin system was selected as a matrix material containing Miracast 1517 Part A epoxy resin which was mixed with Miracast 1517 Part B hardener by Miracon Sdn. Bhd. The epoxy resin system had a mixing ratio of 100:30 by weight content.

Composite laminates fabrications

Composite laminates were manufactured by resin infusion method using an aluminium mould. The process began by stacking 3 layers of kenaf fabric into the mould and a vacuum bag was securely applied on the laminates. Air was subsequently withdrawn from the laminates using a vacuum pump to create a vacuum condition. In the meantime, epoxy and hardener were mixed and

stirred with a thin stick for three minutes in an open container, followed by approximately another 5 minutes of degassing in a vacuum chamber. The process of degassing was carried out to eliminate any trapped air in the epoxy and hardener mixture. With the aid of a vacuum pump, the fabric was infused until the resin filled the laminates. The laminates were left for curing at room temperature overnight. The cured composite panel has a dimension of 290 (wide) × 470 (long) × 3 mm (thickness). Using a horizontal band saw, specimens were cut according to their respective mechanical test.

Corrosive solutions

Two types of corrosive solutions were selected to simulate the possible application of kenaf/epoxy composites in mechanical engineering which may be subjected to harsh environments such as acid rain and alkaline chemical spills. An alkaline solution was prepared by using sodium hydroxide (NaOH) while the acidic solution was prepared by employing hydrochloric acid (HCL). The solutions presented a concentration of 10% by volume (vv%) and pH values is 12.0 and 1.5, respectively. In many applications, only one side of composites is usually exposed to the corrosive environment unless there are cracks in the resin-rich region [12]. In this investigation, the specimens were fully submerged and exposed to the corrosive liquids for the following durations: 12, 24, and 36 days at room temperature. A 12-day interval was sufficient for the solution to diffuse into the composites. Lastly, all the samples were rinsed with tap water, wiped with a clean towel and left to dry at room temperature. All the samples were then sealed in a plastic bag before mechanical testing.

Flexural test

Flexural tests were conducted utilizing a servo hydraulic dynamic fatigue testing machine outfitted with a 5 kN load cell and software at a displacement rate of 2 mm/min following ASTM D790-03. Samples were cut into a rectangular shape (100 mm in length, 15 mm in wide and 3 mm in thickness). The 80 mm support span was used for all 3-point bending tests, which were likewise completed at room temperature. Five (5) specimens were used for each test and condition.

Low Velocity impact test

Using a drop weight testing equipment, the low velocity impact tests were conducted on samples with a square shape (50 x 50 mm and 3.0 mm in thickness). A 3.4 kg impactor with a 10 mm diameter was employed in the low velocity impact test. The impactor stroke at the centre of the samples was acquired by centrally supporting the specimens. The impact used 10 J of impact energy. Five (5) samples were evaluated at room temperature for each circumstance. The specimens were visually examined after the impact testing to determine the damage size and shape.

Results and Discussion

Flexural test

Figure 1 shows representative flexural stress-strain curves for control samples, HCl and NaOH specimens that were immersed for 12, 24, and 36 days. Each curve portrays a linear elastic trend where flexural stress proportionally increases with increasing flexural strain. The flexural stress finally reached the peak and then falls abruptly, indicating the composites have failed in a brittle manner. The influence of corrosive solutions and varying exposure periods on the flexural properties of kenaf/epoxy composites are better illustrated in Figure 2a to Figure 2c.

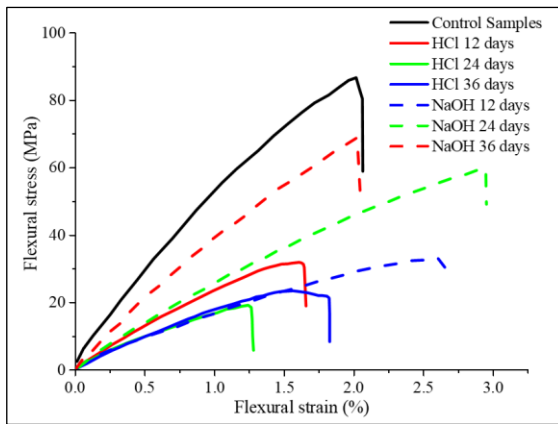


Figure 1: Stress-Strain curves for control samples, HCl immersed for 12, 24, and 36 days, and NaOH immersed for 12, 24, and 36 days

Figure 2a shows that the flexural strength of HCl samples is lower than that of the control samples. The flexural strength is reduced by about 50% after 12 days of immersion in HCL solution. Further decrease is seen for specimens that submerged for 24 days for which the flexural strength is at 20.9 MPa. Interestingly, the specimen has attained a plateau region because there is no further reduction of flexural strength when the specimens were exposed to 36 days of HCL solution. For specimens that have undergone the NaOH immersion procedure, as expected, the flexural strength is lower as compared to the control samples. The flexural strength was reduced by about 17% after being exposed to NaOH solution for 12 days. The increased exposure period in the NaOH solution in 24 and 36 days has resulted in a downturn behaviour to the flexural strength with minimal values of 39 MPa. From the chart, it is obvious that the HCL solution is an aggressive solution which a greater loss in

flexural strength than the NaOH solution, indicating the HCL solution could possess substantial damage to the composites.

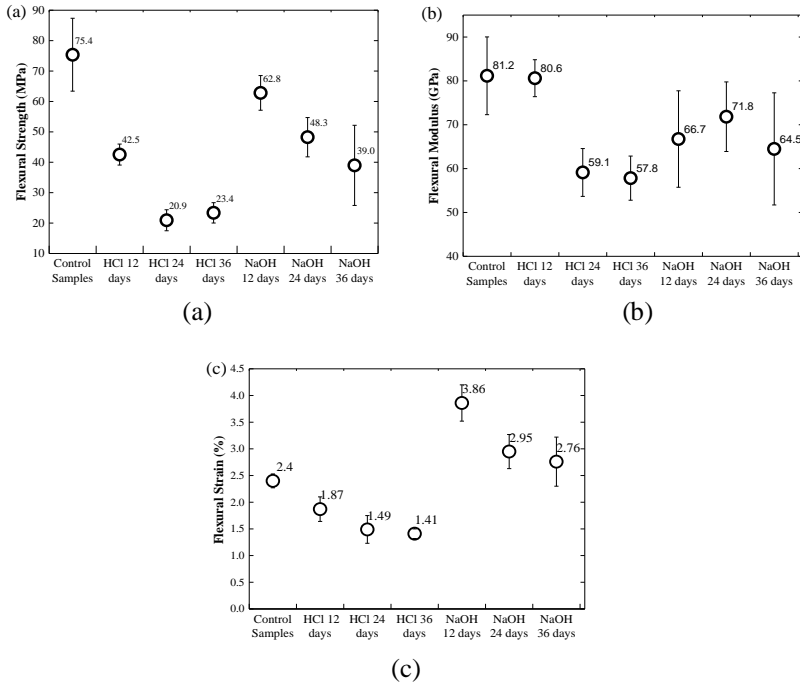


Figure 2: (a) Flexural strength, (b) flexural modulus, and (c) flexural strain of composites.

Variation of the flexural modulus of kenaf/epoxy composites against corrosive solutions and varying exposure periods is presented in Figure 2b. The most striking result to emerge from the data is that the flexural modulus of the control sample closely matched that of HCL samples at 12 days of immersion period at around 80 GPa, suggesting the strength of the composite may not be affected during this period. However, the flexural modulus reduced significantly another subsequent 12 days down to 59.1 GPa. It appears that prolonged exposure to HCL solution for up to 36 days has resulted in minimal effect on the flexural modulus. To confirm this, the mean flexural modulus between HCL 24 days and HCL 36 days was validated with a student two-tail t-test ($\alpha = 0.05$ level of significance). The obtained p-value was calculated at 0.5887 and the null hypothesis was rejected. Therefore, there is enough evidence to show that the differences in the mean flexural modulus between HCL 24 days and HCL 36 days are statistically insignificant. In Figure 2b, it

is observed that all NaOH immersed samples have a lower flexural modulus than the control sample, with a decrease ranging from 12% to 20%. With the increasing exposure period in NaOH solution, there is a decreasing trend of flexural modulus, but it is very minimal. Figure 2c compares the results obtained from the flexural strain in kenaf/epoxy composites subjected to different solutions and exposure times. Relative to the control sample, it is apparent from this data that the HCL specimens have lower flexural strain. However, that is not the case for NaOH specimens, the flexural strain of these composites is higher than the control samples, indicating the specimen takes more bending deformation before breaks. The number of exposure periods has a significant role here where a downtrend of flexural strain is noticeable for both HCL and NaOH specimens following the increase of exposure time; 12, 24 and 36 days in the alkaline and acidic solutions.

It has been previously demonstrated that alkaline environments tend to negatively affect mechanical properties more than acidic environments [10]-[11], [13]. However, in the current investigation, the samples immersed in acid (HCL) environment exhibited the lowest flexural strength and flexural modulus when compared to those immersed in alkaline (NaOH) solution. Another interesting observation from the literature is that Stamenovic et al. [5] and Sindu et al. [6] reported there is an increased in tensile properties after immersion in acidic solutions on glass/polyester composites and coir/epoxy composites. However, this is not the case in the present research, where all of the samples immersed in acidic solution exhibit lower tensile properties than control samples as shown in Figure 2. Therefore, the results of this study may suggest that both HCL and NaOH solutions reacted differently in the case of kenaf/epoxy composites.

The degradation of flexural properties is mainly due to diffusion, penetration or reaction that occurs in the matrix, fibre/matrix interphase quality and occurrences of microcracks. Damage to the fibre strength by the aggression of NaOH and HCL solutions is also possible [14]. The presence of hydroxyl group in kenaf fibres and epoxy resins may react with alkaline solution [15]. The composites may undergo plasticization and swelling of both fibre and polymer, resulting in a degradation of the fibre/matrix interphase structure [16]. The plasticization effect can be evidently seen from the increased flexural strain in comparison with control samples as shown in Figure 2c. In the case of HCL immersion specimens, the degradation of the composite can be explained by hydrolysis of the matrix in contact with this acid similar to alkaline. It is shown that composite degradation strongly depends on acid concentrations [11], [17]. The possible explanation is that the acid concentration used (10% by volume) in this investigation may be excessive and cause severe hydrolysis reactions on the polymer chain and fibre/matrix interfacial bond, resulting in more damage to the composites.

Characteristics of impact events

Impact load–deflection curves

Figure 3a and Figure 3b show impact load versus deflection for the impact test conducted with 10 J of energy after exposure to HCL and NaOH solutions. In all experiments, the curves are suggested to be an open form because the descending part of the curves increases with the deflection. The impactor adhered to specimens and perforations occurred, indicating that the maximal impact energy was sufficient to achieve penetration. The first ascending curve is a linear form, but significant changes in gradients were noticeable in the second ascending of the curves up to the peak height of impact load. This ascending part in the curves is thought to be an important indicator of damage resistance which is related to the impact bending stiffness, particularly in the occurrence of delamination [18]. The onset of gradient changes in the curves typically in the second ascending traces suggests the impact bending stiffness has been compromised before a complete perforation in the composites.

In Figure 3a, the HCL samples for 24 and 36 days exhibit the same inclination in the first ascending part as the control sample. However, it is interesting to observe that the HCL samples have lower bending stiffness in the second ascending part which takes place at a 50% lower impact load (~300 N) as compared to the control sample, ~600 N. The same situation also applies to NaOH immersed specimens as shown in Figure 3b where it is likely the bending stiffness in the first ascending part is the same. The bending stiffness at the second ascending curve is slightly lower than the control sample. When comparing the HCL and NaOH samples in Figure 3a and Figure 3b, it appears that the NaOH samples have a higher peak height in the second ascending curves, indicating higher impact load.

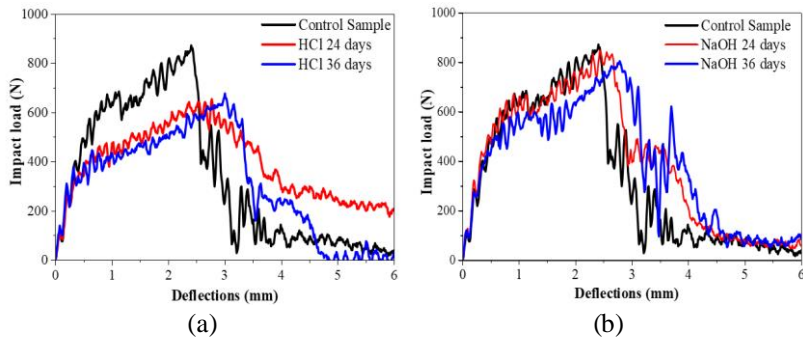


Figure 3: Impact load-deflection traces impact event of (a) HCL immersed and (b) NaOH immersed samples

The impact load-deflection of the kenaf/epoxy composites reveals the impact load are affected by the type of corrosive solutions as shown in Figure

4. As expected, the control sample has the highest impact load at 875.8 N in comparison with NaOH and HCL samples. Between the solutions, it seems that the HCL solution cause a more severe deterioration effect on the impact load than the NaOH solution. A relative reduction of 24% for NaOH and 9% for HCL are determined in comparison with the control sample. This finding is consistent with that of Amaro et al. [10] and Mortas et al. [11] that NaOH and HCL solutions have detrimental effects on the impact load on glass, Kevlar and carbon fibre reinforced composites. In the case of the immersion periods, it appears the number of exposure times has a limited effect on the impact loads. The NaOH and HCL specimens have attained similar impact loads through the exposure time (24 and 36 days) measured about 660 N and 790 N, respectively.

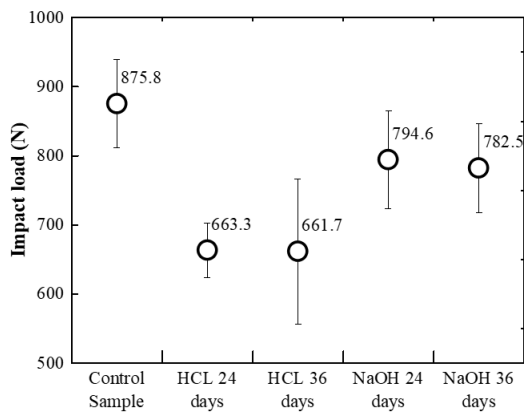


Figure 4: Impact load of kenaf/epoxy composites against the type of solutions and exposure time

Absorbed energy

The energy absorbed by the sample will be equal to the energy generated to create the damage according to the principle of energy conservation. Figure 5 shows the energy and load curves profile against the time of the control sample. The determination of absorbed energy was acquired by examining between energy and load curve profiles as shown in Figure 5. Following the method described by Zike et al. [19], the absorbed energy where perforation occurred during the impact loading was determined at the perforation point on the load curves as shown in Figure 5. The perforation point is considered when the load reaches the plateau region in the descending part.

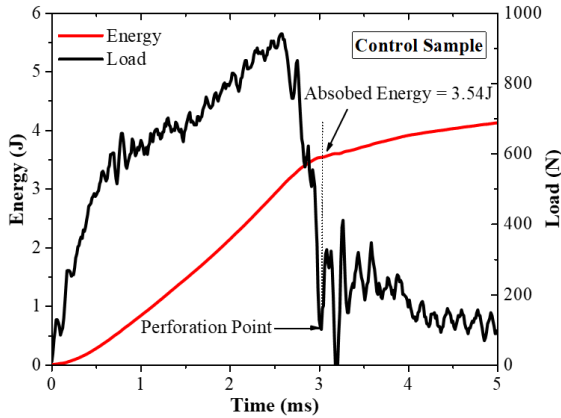


Figure 5: Energy and load curves against the time of the control sample.

Figure 6a to Figure 6d show the typical energy-time and load-time curves of HCL and NaOH after being immersed for 24 and 36 days. Except for that HCL 24 days and HCL 36 days in Figure 6c and Figure 6d, the energy-time profile of the control sample, NaOH 24 days and NaOH 36 days keeps on gradually increasing after perforation. The gradually increased load-time curves may suggest the development of friction between the edges of perforation holes in the composites against the lateral surface of the impactor [20]. In other words, HCL 24 days and HCL 36 days experienced more area of impact damages.

Comparing the values of absorbed energy against the composites as shown in Figure 7, a profound effect in energy absorption capability is manifest between the control sample, HCL and NaOH samples. It is clearly seen that the control sample has the highest absorbed energy, 5.86 J in comparison with HCL and NaOH. It is evident that the amount of absorbed energy varies depending on the type of immersion. The immersion in HCL solution causes more detrimental effects to the absorbed energy than immersion in NaOH solution. In addition, the effect of the immersion period can be clearly observed with the HCL solution. In Figure 7, the amount of energy absorbed by HCL samples decreased as immersion time increases. The absorbed energy decreased to 4.08 J and 2.86 J when the exposure period increased to 12 and 36 days, respectively. However, the same immersion period may not give any significant effect on the NaOH solution.

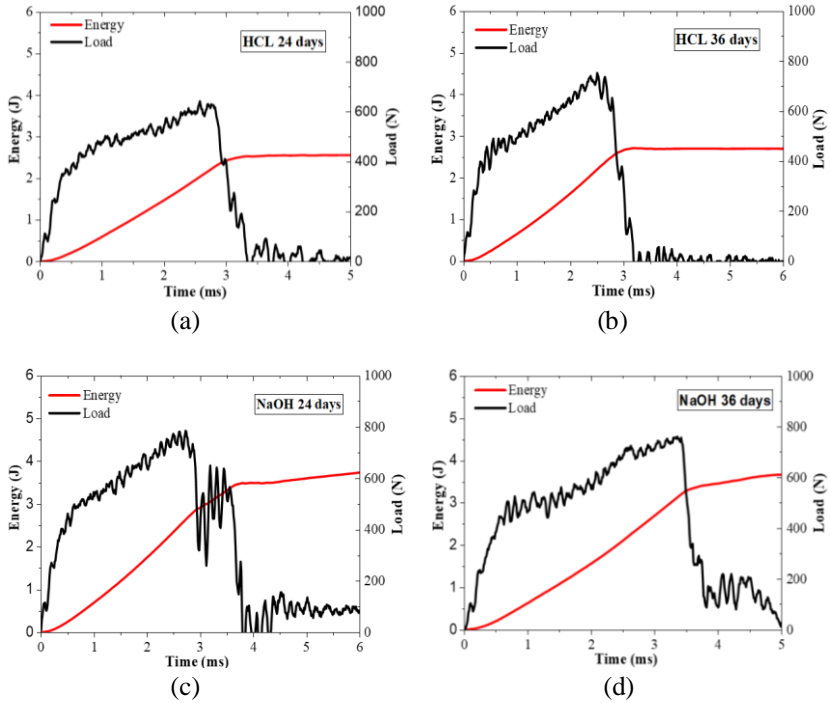


Figure 6: Energy-time and load-time curves of (a) HCL 24 days, (b) HCL 36 days, (c) NaOH 24 days and (d) NaOH 36 days

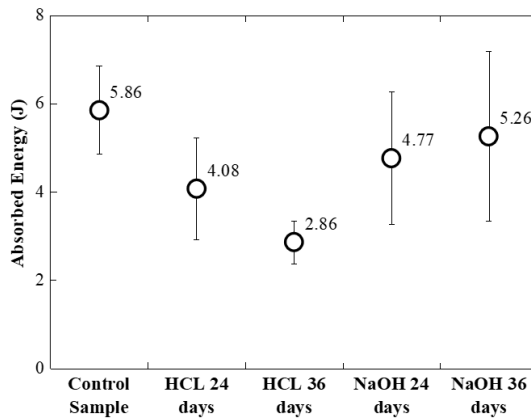


Figure 7: The energy profiling curves of kenaf/epoxy composites

Velocity-time curves

Figure 8 gives a comparison the velocity-time profile for different kenaf/epoxy composites. The changes in impact velocity and its directions as the impactor passes through the specimens may reflect the energy by the fracture process [21]. A rebounding impactor on the specimens during impact incidents causes a zero reading of residual velocity. In the case of a fully penetrated specimen by the impactor, the curve traces may present an almost similar reading of striking and residual velocity throughout the impact incident. In Figure 8, it is obvious that the residual velocity is decelerated between 1.55 and 1.6 ms from the given striking velocity of 1.85 ms. Dhakal et al. [21] explained the differences between striking and residual velocity are related to the energy absorption experienced by the impacted specimens. Since there are relatively small differences between the striking and residual velocity of the HCL sample, this would mean that the impact resistance of HCL samples was very low.

Another interesting feature of the curves is that HCL samples are still undertaking some residual velocity whereas the NaOH and control samples displayed a straight residual velocity following the rapid deceleration of the curves. This may imply the evidence of friction development between the lateral surface of the impactor and perforation holes for which a similar finding was noticeable with the energy-time and load-time curves in Figure 6c and Figure 6d. In other words, the HCL may have experienced a different failure mechanism than NaOH and control sample during the impact incident.

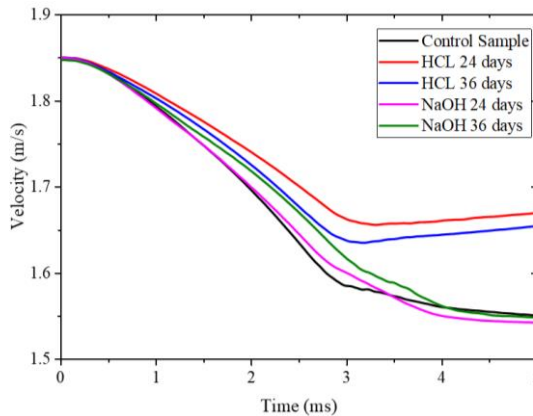


Figure 8: Velocity against time profile

Observation of damage following low velocity impact

Typical impact damage of test specimens is shown in Figure 9a to Figure 9e for the front face and in Figure 10a to Figure 10e for the back face. The

damaged area in the front face appears to be smaller than the damaged area in the back face, corresponding to a pyramid-shaped fracture. This pyramid shaped fracture is likely caused by a through-thickness damage formation that is associated with the combination of delamination, matrix cracks due to shear and bending failure, fibre breakage and in-compression fibre buckling.

Nonetheless, there are different mechanisms of energy absorption in NaOH, HCL and control samples in the front face. The damage characteristic of control samples exhibits a circular area together with extended delamination as shown in Figure 9a. The damaged area of HCL samples in Figure 9b and Figure 9c is larger than of that control samples and NaOH samples as shown in Figure 9a, Figure 9d, and Figure 9e. A closer inspection indicates that the damaged area of NaOH and the control sample are confined by the diameter of the impactor (10 mm in radius). The exposure time presented a considerable effect on HCL composites where the HCL 36 days showed about 20% increases in damage area in comparison with HCL 24 days. As expected, the damaged areas increased with the decreasing absorbed energy which is consistent in Figure 8. The reason is that the stiffness of these HCL composites is lower than control and NaOH samples and hence, it has suppressed the damage process.

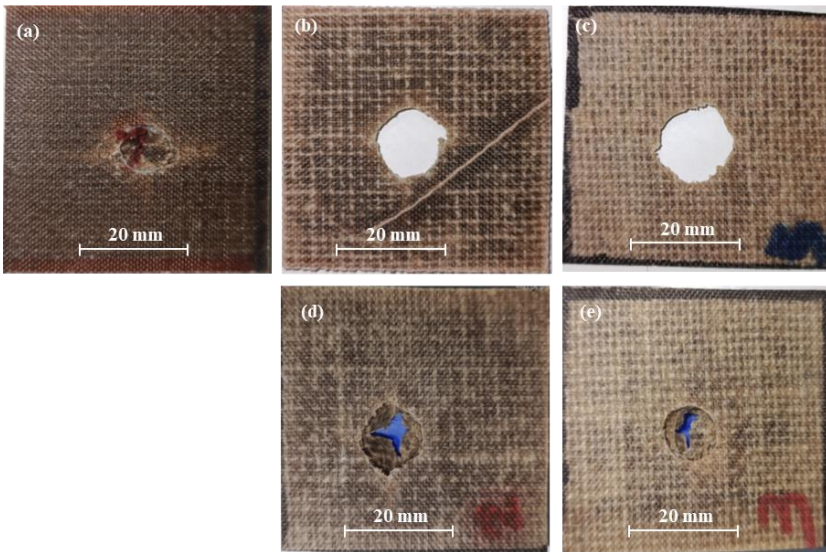


Figure 9: Damaged occurred on the front face; (a) control sample, (b) HCL 24 days, (c) HCL 36 days, (d) NaOH 24 days, and (e) NaOH 36 days

The damaged pattern on the back face between the composites involved several damage characteristics. The back face of the control sample in Figure

10a consists of both circumferential and radial fracture in the vicinity of the perforation point. Beyond the perforation area, the damage is presented by vertical and horizontal crack lines propagating away from the impact hole which is consistent with the warp and weft direction in the fabric. These cracks lines in outer impact zones are possibly due to fractures in the fibre and the matrix [22].

The situation is not the same in the case of specimens that were immersed in HCL solution. The HCL 24 days and HCL 36 days were completely fragmented upon impact loading, leaving a complete and clean perforation hole in the back face as shown in Figure 10b and Figure 10c. It is estimated that there is about a 15 mm round diameter damaged area in the front face while a 20 mm round diameter damaged area in the back face of specimens. It is unlikely that the effect immersion time may not be distinguished for the back face since the round diameter is almost comparable between 24 and 36 days of HCL immersion.

In contrast, the damaged pattern for 24 and 36 days of NaOH specimens is considered partially fragmented as shown in Figure 10d and Figure 10e. There is evidence of circumferential and radial fracture traces on the samples. Additionally, there are no indications of vertical and horizontal crack lines beyond the perforation hole area for NaOH and HCL samples, unlike the control sample.

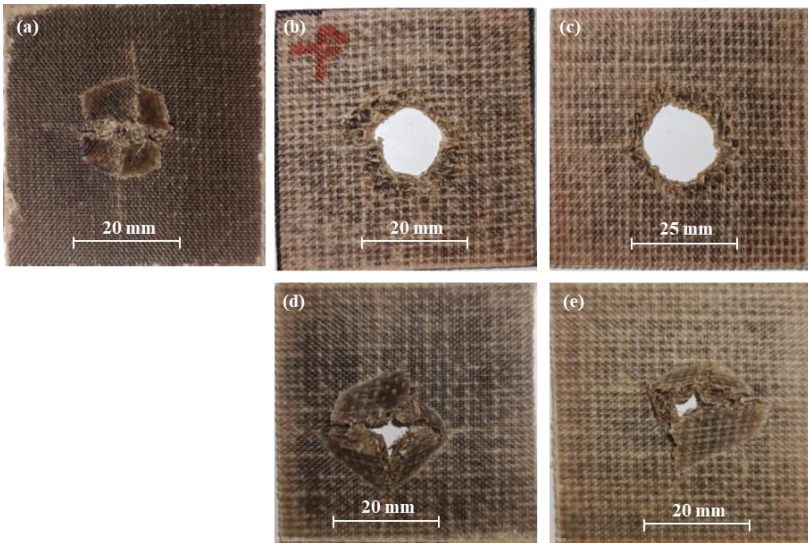


Figure 10: Damaged occurred for samples on the back face; (a) control samples, (b) HCl 24 days, (c) HCl 36 days, (d) NaOH 24 days, and (e) NaOH 36 days

Conclusions

The performance of kenaf/epoxy composites after immersions in hydrochloric acid (HCl) and sodium hydroxide (NaOH) solutions on the flexural properties and low velocity impact response were determined. The investigation also considered the number of exposure periods in these aggressive solutions, 12, 24 and 36 days. The presence of corrosive solutions has a significant effect on the flexural strength and flexural modulus, with lower values of these properties observed in comparison with control samples. The flexural properties were also highly dependent on the number of exposure durations in the corrosive solutions. There is evidence of a plasticization effect on the NaOH immerse samples that is due to the hydrolysis reaction of alkaline solution. In terms of the low velocity impact test, it was concluded that impact bending stiffness, impact load and absorbed energy decrease when exposed to the NaOH and HCL solutions. However, the residual impact velocity shows an inverse tendency which is higher than the control samples. The increased exposure period of composites in these solutions is found to lower the impact load and absorbed energy of NaOH immerse samples but it has limited effect on the HCL immerse samples. The study also indicates the HCL to be a more aggressive solution than the NaOH, with a larger damage area and a complete fragmentation occurrence was observed on these samples upon impact loading.

Contributions of Authors

The authors confirm the equal contribution in each part of this work. All authors reviewed and approved the final version of this work.

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Conflict of Interests

All authors declare that they have no conflicts of interest.

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