Experimental investigations on the moisture absorption and mechanical behaviour of basalt-aramid/epoxy hybrid interply composites under different ageing environments

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Abstract: The structural components made of polymer composites in aviation, automobile, and marine applications are subjected to various environmental conditions throughout their design service life. Furthermore, the examination of the effect of various ageing environments on moisture absorption and mechanical behaviour is essential to protect the structures from premature and catastrophic failures. This study evaluates the influence of three different ageing conditions, namely, ambient (25°C), sub-zero (−10°C), and humid (40°C and 60% relative humidity) on the mechanical properties of hybrid interply basalt-aramid/epoxy composites. The compression molding process was adopted to fabricate the specimens and the specimens were aged in a distilled water environment for a period of 180 days. The aged specimens were subjected to static and dynamic mechanical tests viz. tensile, flexural (3-point bending), short-beam shear (SBS), and Charpy.

PUBLIC INTEREST STATEMENT

Nowadays, researchers are focusing more on environmentally sustainable materials. Basalt fibres are one among such materials, which are eco-friendly and have good mechanical properties. However, Basalt fibres are brittle in nature and for impact applications, these fibres are generally- hybridized with more ductile materials, such as aramid to improve the impact strength of the composites. Furthermore, polymer composites in structural applications are subjected to different hygrothermal conditions. Epoxy resins are hydrophilic in nature, which absorb moisture from the environment and undergo swelling, affecting both physical as well as mechanical properties of the composites. This study investigates the effect of three different ageing conditions viz. ambient, sub-zero and humid ageing on the mechanical characteristics of the hybrid basalt-aramid/epoxy composites. Composite specimens were exposed to ageing environments till the moisture saturation and further, they are subjected to tensile, flexural, short beam shear strength and impact tests. Highest degradation in properties was observed in ambient aged specimens followed by humid and sub-zero specimens.
impact tests to study the behaviour, and then results were compared with the unaged specimens. Fourier Transform Infrared Spectroscopy (FTIR) was also performed to analyze the chemical changes within the composites due to the ageing process. It was witnessed that moisture absorption rate increases with - increase in ageing period and attains a state of saturation between 1.8% and 5.44% depending on the ageing conditions. Investigations revealed that moisture absorption has an unfavourable effect on the mechanical performance of the composites. The retention of mechanical strengths of aged composites is in the order of Unaged > Subzero > Humid > Ambient. Fractured tensile specimens were analyzed for microscopic observation using Scanning Electron Microscope (SEM) to study the damage morphology. Matrix decomposition, matrix cracks, and interfacial debonding were the major failure modes observed in aged composites.

**Subjects:** Mechanical Engineering; Mechanics of Solids; Testing; Manufacturing Engineering; Materials Science

**Keywords:** Hybrid composites; Compression molding; Hygrothermal ageing; Moisture absorption; Static and dynamic mechanical tests; FTIR; Scanning electron microscopy

1. Introduction

Composite Market is estimated to grow from USD 70.4 billion in the year 2020 to USD 112.8 billion by the year 2025, at a CAGR of 8.8% between 2020 and 2025, as predicted by the Markets and Markets (2020) Report (Markets and Markets, 2020). In recent years, conventional materials are being replaced by fibre reinforced composites such as aramid, carbon, and glass in many engineering sectors owing to their inexpensiveness, lightweight, improved strength, and stiffness. However, due to growing concern towards environmental issues, researchers are focussing on the use of natural fibre reinforcements extracted from plants and mineral origin (Faruk et al., 2012; Pai, Pai et al., 2021). In particular, basalt fibres are gaining popularity in the scientific community owing to their benefits in terms of biodegradability, inexpensiveness, and enhanced mechanical as well as thermal properties (Dhand et al., 2014; S et al., 2012). Basalt fibres have similar strength and modulus as that of glass fibres but they lack in impact properties due to their brittle nature which can be enhanced by hybridizing with the ductile aramid fibres (Bandaru et al., 2016; Sarasini et al., 2013). Basalt fibres hybridized with aramid fibres find application in aviation, automobile, sports, and other industries (Vasudevan et al., 2019). In these applications, during their service life, composites are usually exposed to moisture, extreme temperature, UV radiation, biological environments, etc., which results in the degradation of material properties (R. Kumar & Chandra, 2018; D. Kumar & Kumar, 2019). The durability of composite materials subjected to different ageing conditions has become a primary concern for the safety and reliability of polymer composite materials. Epoxy polymers have a strong affinity toward water due to their hydrophilic nature. This makes epoxy resins absorb a high amount of moisture. Generally, depending on the type of polymer, the moisture uptake at saturation may vary in the range of 1–7% (Soles & Yee, 2000). The moisture absorbed by the polymer matrix subsequently degrades the composite mechanical characteristics, such as strength, stiffness, and fibre matrix interfacial strength (Ishisaka & Kawagoe, 2004). The long-term exposure of polymer composite structures to the hygrothermal environment results in both physical as well as chemical variations of the matrix through various mechanisms, namely plasticization, hydrolysis and swelling. Plasticization refers to change in the structure of a polymer as a result of the interaction of moisture with polar groups of the resin (De’Nève & Shanahan, 1993; Prolongo et al., 2012). Many researchers have reported that plasticization primarily reduces the glass transition temperature and thereby affects strength and modulus of the epoxy polymer (Lu et al., 2001; Zanni-Defforges & Shanahan, 1995). Thermal stresses are also developed in the laminate due to differential thermal expansion at
polymer and fibre interface region, which results in residual stresses. These changes ultimately affect the overall strength, stiffness, and damage tolerance of the composites.

Several researchers have worked in the past on the mechanical durability of composites subjected to hygrothermal ageing. Cerbu et al. (Cerbu, 2010) explored the effect of long-term ageing on the tensile and 3-point bending characteristics of E-glass/epoxy composites. Laminates were aged in distilled water and marine water for about 300 days maintained at 20°C. Studies revealed that reduction in mechanical strength was higher for the distilled water specimens. Fang et al. (Fang et al., 2017) analyzed the degradation behaviour of glass fibre/epoxy composites by subjecting to accelerated ageing in water and marine water for about six months. The authors observed that glass transition temperatures were reduced by 2.9% and 2.5% for water and seawater-aged specimens, respectively. Menail et al. (Menail et al., 2009) examined the fatigue performance of aramid/epoxy fibre reinforced laminates subjected to seawater immersion for a duration of 100, 500, and 1000 h. Results showed that seawater absorption has a negative influence on the mechanical characteristics of the laminates. Tanaka et al. (Tanaka et al., 2002) experimentally examined the effect of prolonged immersion on the interfacial performance of aramid/epoxy laminates. It was observed that immersion in deionized water at 80°C for a period of 13 weeks resulted in a decrease in fibre fracture load and interfacial strength of the composites. Ray et al. (Ray, 2004) studied the influence of sub-zero environment on the apparent interlaminar shear strength (ILSS) characteristics of GFRP composites. Specimens were subjected to a hygrothermal environment of −6°C temperature and reported that freezing treatment has a negative effect on the apparent ILSS characteristics of the composites. Numerous studies are available in the field of ageing and its influence on the mechanical performance of polymer composites but most of the studies were limited to seawater ageing of composites made of either glass or carbon reinforcements. Limited literature are available on distilled water ageing and its effect on static and dynamic performances of hybrid composites reinforced with basalt and aramid fibres.

Composite structures in outdoor applications are exposed to seasonal climatic changes. The durability of these structures is of great concern while protecting them from damage caused due to mechanical loadings. Structures made from glass/carbon fibres have been studied extensively for such applications. In the current study, biodegradable basalt fibres have been used as a replacement for glass fibres. The impact performance of these fibres can be enhanced by hybridizing them with more ductile fibres, such as aramid. The previous investigations have revealed that hybrid interply composites with aramid fibres as surface plies are more efficient in resisting the damage due to delamination when compared to inner aramid plies when subjected to impact loads (Dorey et al., 1978). The central load-carrying basalt plies (0°/90°) can be protected from impact damages by incorporating aramid plies as surface layers (Vasudevan et al., 2018). During the impact, the outer aramid layer distributes the damage over a larger surface area from the point of impact and hence controls the damage due to the delamination of the plies. This work is an extended part of the previous work carried out by the authors where it is found that surface aramid fabrics oriented at (0°/90°) exhibited the highest mechanical properties compared to other orientations (Pai, Kini et al., 2021). Therefore, this research work is an effort to study the influence of different ageing situations on the mechanical characteristics and damage phenomena of interply composites with surface aramid fabrics oriented at (0°/90°). Five layered basalt-aramid interply composites were prepared with all the fabrics oriented at (0°/90°) using a compression molding process. The mechanical characteristics of the laminates were determined as per ASTM standards and compared with the results of unaged composites reported (Pai, Kini et al., 2021). Further, failure modes of fractured tensile and impact specimens were studied using SEM and optical microscope, respectively.
2. Experimental procedures

2.1. Materials and composite fabrication

The reinforcement materials and resin were purchased from M/S Composite Tomorrow, Gujarat, India. Bidirectional (2-D) plain basalt and aramid fabrics having the areal weights of 400 g/m² and 480 g/m² respectively were used as the reinforcement and CT/E 556 as the epoxy resin and CT/H 951 as the hardener for the current study (Pai, Kini et al., 2021). Five layered interply composites with a fibre volume fraction of 0.6 were prepared by placing three basalt layers as core, sandwiched between the surface aramid plies as shown in Figure 1. The thickness of basalt and aramid fabrics are measured to be 0.45 and 0.55 mm, respectively. The composite layup was prepared on a thick mild steel sheet using hand layup followed by the compression molding process. Curing was performed for 24 hours under ambient conditions. The thickness of the laminates was measured to be 2.8 mm. The void percentage of the laminates was measured as per ASTM D792 (D792 – 20, 2013) and reported in (Pai, Kini et al., 2021).

3. Ageing methods and testing procedure

Moisture absorption test was performed to observe the moisture uptake characteristics and to analyze the amount of moisture weight gained by the composites over a period of 180 days as shown in Figure 2. The test samples were cut as per ASTM standards and preheated at 50°C in
a drying oven before conditioning. Edges of the specimens were coated with resin before exposing them to ageing environments to avoid the exposure of cut fibres and to have uniform absorption of moisture throughout the specimen. Initial dry weights of the specimens before conditioning were measured as reference using an electronic weighing scale with an accuracy of ± 0.001 g. The following ageing conditions were chosen to study the influence of moisture on the mechanical behaviour of the composites.

(i) Ageing in distilled water at ambient temperature (25°C)

(ii) Ageing in distilled water at sub-zero temperature (−10°C)

(iii) Ageing in humid environment (40°C and 60% relative humidity in an environmental chamber)

ASTM D5229 (Materials, 2020) is followed to periodically monitor the extent of moisture absorbed by the composites. The percentage moisture uptake of the composite at various time intervals is determined by Eq. 1.

\[ M(t) \text{ (%) } = \frac{m_1 - m_0}{m_0} \times 100 \]  \hspace{1cm} (1)

In the above equation, \( M(t) \) represents the % of moisture uptake, \( m_0 \) is the weight of the unaged specimen, and \( m_1 \) is the specimen weight during ageing.

The coefficients of distilled water diffusion of the aged composites were evaluated from the Eq. 2 (Shetty et al., 2020).

\[ D_2 = \pi \left( \frac{h}{4M_2} \right)^2 \left( \frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} \right) = \pi \left( \frac{h}{4M_2} \right)^2 k^2 \]  \hspace{1cm} (2)

where, \( D_2 \) denotes the coefficient of moisture diffusion, \( h \) is the specimen thickness, \( M_1 \) and \( M_2 \) represent the amount of moisture absorbed at time intervals \( t_1 \) and \( t_2 \), respectively. \( M_2 \) is the amount of moisture absorbed at saturation and \( k \) denotes the gradient of the linear portion of the curve.

4. Mechanical characterisation

4.1. Tensile test setup

The basalt-aramid/epoxy hybrid laminates are widely used in many structural applications which are exposed to different hygrothermal environments. Therefore, determining ultimate tensile strength, modulus, and fracture strain were necessary from the durability perspective. Tensile test was performed using BISS, Indian make universal testing machine (UTM) equipped with a loading capacity of 50kN. To determine the stress-strain characteristics, ASTM D3039 (Materials et al., 2020) standard was employed. Span length was maintained at 150 mm and the testing was performed at a constant crosshead speed of 2 mm/min. The amount of load introduced and the deformation of the specimens was acquired from the Data Acquisition System.

5. Flexural tests setup

Flexural tests are carried out to measure the composite’s flexural strength and modulus under bending load. Flexural tests were conducted as per ASTM D7264 (ASTM D7264/D7264M-07, 2007) on UNITEK-9450, Indian make UTM with a loading capacity of 50 kN. The specimens were placed on two
supports with a span length equal to 32 times the thickness while keeping a standard width of 13 mm. The overall length is maintained at 1.2 times the span length. A concentrated load at 2 mm/min speed was applied at the centre of the beam and corresponding displacement were recorded. Total of five specimens from each ageing condition were examined and average values were obtained.

6. Short-beam shear (SBS) test setup
Delamination is one of the major reasons for the failure of composites in many engineering applications due to poor interlaminar shear strength. The capability of a composite material to resist the damage due to delamination is measured by the SBS test. The test was performed as per ASTM D2344 (ASTM D2344/D2344M-13, 2013) using Instron UTM equipped with a 50kN load cell. This test method aimed to minimize the bending stresses so that the specimen develops stresses only due to shear between the laminae during loading. As per the standard, the length was taken as 6 times the thickness, and the width was taken as twice the thickness. Specimen loading was similar to the 3-point bending test with 2 mm/min as the cross-head speed.

7. Charpy impact test setup
Structural components with low impact strength may fail when subjected to sudden impact loads. The Charpy test is performed to study the strength of the composite under the dynamic impact loading. Specimens were tested on pendulum impact tester equipment Zwick Roell HIT50P, German made as per the standard ISO 179–1(1. 60,812, “EN ISO 179–1, 2000). The un-notched specimens of length 65 mm, width 12.7 mm, and thickness of 2.8 mm were prepared from the composite panel. The impact striker was made to drop from a certain height to hit the specimen and the corresponding energy absorbed was recorded.

8. Results and discussion

8.1. Moisture absorption behaviour
The experimental measurements of moisture uptake of the composites for three different ageing conditions are presented in Figure 3. The graph shows that the rate of water absorption by the samples depends on immersion time. A higher rate of moisture absorption was seen in the initial period of ageing. The percentage of moisture absorbed by the specimens increases gradually with an increase in the duration of ageing for all the ageing conditions, until it reaches a saturation state after around 3500 hours (or 145 days). The moisture absorption tendency is accelerated due to the existence of voids, or micro openings, on the sample surface (R. Kumar & Chandra, 2018). In the initial period of the ageing process, the rate of moisture uptake follows Fick’s law, in which moisture percentage increases linearly with respect to the ageing duration.

The laminates aged in distilled water at ambient conditions absorbed the highest amount of moisture (5.44%) at saturation. The sub-zero temperature-aged composites absorbed 3.12% and the laminates aged at 40°C, 60% RH in an environmental chamber, absorbed the least, that is, 1.80% of moisture at equilibrium. The percentage moisture absorption for three different laminates and their diffusion coefficients are shown in Table 1. The different rate of moisture diffusion of ambient, sub-zero, and humid aged specimens is governed by Fick’s first law, which states that the weight of the moisture that diffuses through a given cross-sectional area, termed as flux, is directly proportional to the concentration gradient of moisture. The proportionality constant is called the diffusion coefficient (mm²/sec). This first law is most significant in case of fibre reinforced polymer composites where moisture concentration is a function of time. Concentration gradient is the main driving force behind the moisture absorption and it continues until the gradient is equalized. In ambient ageing condition, the specimens were exposed to distilled water which is equivalent to relative humidity (RH) of 100%. Since, the concentration gradient was higher, molecules diffused at a faster rate, and the specimen absorbed maximum amount of
However, in humid ageing conditions, specimens were exposed to 60% RH in an environmental chamber resulting in the reduced rate of moisture absorption and maximum moisture content compared to ambient specimens. In sub-zero ageing conditions, the temperature was below the freezing point of water. The specimens were surrounded by the frozen water which continuously releases the sub-zero vapour or moisture. As the specimens were exposed to the sub-zero environment for a longer duration, moisture diffusion occurred until the specimens reached saturation point.

Moisture uptake of laminates is a complex phenomenon, which is governed by many factors, such as type of fibre and resin used, void content of the laminate, quality of fabrication, and also type of ageing condition (Almeida et al., 2016). Also, strong adhesion between the matrix and the fabrics reduces the speed of molecular diffusion. The good adhesion between the reinforcement and epoxy leads to closer packing within the composite and consequently, reduces the mean free path between the water molecules (distance moved between the two successive collisions by the diffusing water molecules), and lower moisture absorption is attained. Furthermore, it is evident that, laminate stacking sequence noticeably influences the moisture diffusion behaviour, the existence of aramid fabrics as the surface plies not only protects the basalt fabrics from damage.

Table 1. Percentage of moisture uptake at equilibrium and diffusion coefficient of aged composites

| Ageing Condition | Maximum moisture absorption (%) | Duration of ageing | Diffusion Coefficient D_z (mm²/s) |
|-----------------|---------------------------------|--------------------|----------------------------------|
| Ambient         | 5.44                            | 180 days           | 1.705 × 10^-7                   |
| Sub zero        | 3.12                            | 180 days           | 7.949 × 10^-8                   |
| Humid           | 1.80                            | 180 days           | 2.290 × 10^-8                   |

Moisture uptake curves for different ageing conditions.
but also increases the resistance to moisture uptake as aramid fabrics act as a barrier to basalt layers avoiding direct interaction between basalt layers and moisture.

9. Fourier-Transform infrared spectroscopy (FTIR)
The chemical changes within the basalt-aramid/epoxy hybrid composites due to the ageing phenomena were evaluated by using JASCO FT/IR-6300 (type A), Japan made Fourier transform Infrared (FTIR) spectrometer. The spectra were acquired by scanning the samples in a transmittance mode between the region of 400 cm⁻¹ to 4000 cm⁻¹ with a resolution of 4 cm⁻¹ and 32 scans. Figure 4 shows the comparison of FTIR vibration spectra of three different aged composites with respect to unaged specimens.

The specimens aged in ambient conditions displayed the stretching vibration between the frequency range of 3300 cm⁻¹ to 3500 cm⁻¹. These peaks indicate the higher rate of moisture absorption compared to sub-zero and humid conditions which led to the increased intensity of stretching of the hydroxyl (O-H) groups. The oxidation of C-H group can be observed between the wavenumber 2700 cm⁻¹ to 2950 cm⁻¹ characterized by weak bending vibrations in all the aged specimens. Further evidence for the hydrolysis of the matrix material in all the aged specimens is observed by the increased intensity of C = O stretching at 1728 cm⁻¹. The vibrations at frequencies 1728 cm⁻¹ and 1510 cm⁻¹ are due to the stretching of carbonyl (C-O) and—C = C—groups of aramid fibres. The FTIR spectra of aged specimens clearly indicate the increased intensity of—C = O and—OH groups due to the hydrolysis of amide groups present in the aramid fibres.

10. Tensile strength
The tensile test result, obtained from basalt-aramid/epoxy composites subjected to various ageing conditions is shown in Table 2. It was observed that deterioration of the mechanical properties increases as the moisture gain percentage of the specimen rises. The degree of deterioration depends on the type of ageing the specimen has undergone. The deterioration of mechanical properties is in the following order of Ambient aged > Humid aged > Sub-zero aged > Unaged specimens. Figure 5 depicts the stress–strain curves of unaged and aged composites. Unaged specimens showed a maximum tensile strength and modulus of 190.36 ± 11.37 MPa and
6.32 ± 0.34 GPa, respectively. The immersion of hybrid specimens in distilled water, for a duration of 180 days at ambient conditions displayed an increased rate of degradation in tensile properties and the tensile strength reduction was 21.8% compared to unaged specimens. Specimens subjected to sub-zero ageing have shown the least reduction in tensile strengths among the three ageing categories and the reduction in tensile strength was 12.79%. The reduction in tensile modulus of aged composites was less noticeable compared to unaged specimens. Retention of tensile strength for various ageing conditions is shown in Figure 6. The ambient, sub-zero, and humid aged specimens retained tensile modulus of 78.12%, 87.20%, and 83.39% as compared to unaged specimens. The tensile modulus of the composite primarily depends on nature and alignment of the fibre used. It can be observed from the table that ambient and humid aged specimens experienced higher degradation in modulus due to increased moisture absorption. The large drop in tensile strength was witnessed in ambient aged specimens due to the higher percentage of moisture uptake, which resulted in swelling of epoxy resin. Since, the swelling of aramid and basalt fibres is comparatively low, the diffusion of water caused the fibre-matrix interfacial degradation which led to a decrease in tensile properties. Moisture absorption is a common phenomenon observed in all inorganic polymeric materials and it severely affects the mechanical properties (Dhakal et al., 2006). Improper fabrication may also develop weak interfacial bonding between the fibre and the matrix which assists the moisture to easily enter and wet the fibre completely and hence reduces the mechanical strengths.

The tensile test fractured samples were examined using a scanning electron microscope to identify the damage mechanisms as shown in Figure 7. SEM images revealed that, the ageing
condition and the amount of moisture absorption have significantly affected the matrix-dominated mechanical properties. Ageing process resulted in the increased amount of cracks at the micro-level in the matrix phase and which further led to the development of small fragments at the fibre and matrix interface. The ageing environment resulted in the matrix deterioration from the fibre surfaces. A similar kind of matrix decomposition was seen in the laminates aged at sub-zero temperatures. The SEM pictures also revealed that the hygrothermal ageing of the basalt-aramid/epoxy composites weakens the bonding between fibre–matrix interfaces and leads to fibre dominated failure during the tensile testing. The deterioration of tensile properties is associated with the plasticization and degradation of the resin structure as a result of moisture ingestion into
the polymer resin. The occurrence of multiple cracks, delamination, and interfacial debonding at the fibre and matrix are observed in ambient and humid specimens. In sub-zero aged specimens, the voids present in the specimens are occupied by the absorbed frozen moisture and resulted in an increased level of debonding at the interface of fibre and matrix during tensile testing. This resulted in increased strain at failure of the specimens as compared to other aged specimens (Padmaraj et al., 2021).

11. Flexural strength and apparent ILSS
The three-point bending test revealed that the flexural and SBS performance of basalt-aramid/epoxy hybrid composites are significantly influenced by the amount of moisture absorbed. Both the test results showed a similar trend, among the aged specimens, the higher strengths were displayed by the sub-zero specimens followed by humid and ambient specimens under the influence of flexural and pure shearing loads. Table 3 shows the results of three-point bending test for three different ageing conditions. In three-point bending, specimen failure occurs primarily due to bending and shearing between the layers. During the loading of the specimens, compressive forces are generated in the layers of loading surface and the tensile forces in the layers of the support surface. Since the extreme stresses occur at the outermost surfaces of the specimen, presence of aramid surface layers resulted in maximum flexural strength of 268.32 ± 13.53 MPa in unaged specimens. The central core basalt layers are subjected to shear and relatively unaffected by tension and compression. Vasudevan et al. (Vasudevan et al., 2018) have described the similar enhancement in bending strength for hybrid laminates having outer Kevlar layers and central core as the glass fibres. All the specimens failed due to bending action and broken fibres were observed at the bottom surface of the specimen.

Drop in flexural properties is due to moisture-induced fibre-matrix interfacial degradation and swelling action of polymer resin (Bian et al., 2015). Flexural load effortlessly destroyed the fibre and matrix bonding strength in case of aged specimens. The presence of higher void content makes the composite to absorb a higher amount of moisture and results in reduced interfacial bonding. Tensile and flexural properties are generally fibre dominant, and fibre attack by the moisture reduces the tensile and flexural strengths (Bank et al., 1995; Processing, 2007). The flexural strengths of ambient, sub-zero, and humid specimens were reduced by 26.74%, 10.62%, and 18.41%, respectively, compared to dry specimens. A maximum decline in the strength was witnessed for ambient aged specimens while the least decline in properties occurred in sub-zero aged specimens. The key reason for the decline in bending strength is believed to be the diffusion of water molecules, which led to the degradation of fibre/matrix interface. Figure 8 shows the retention of flexural strength of aged hybrid laminates with respect to unaged specimens. The bending strength retention of specimens is in the order of Unaged>Sub-zero>Humid>Ambient. This is due to interface bond weakening at the polymer matrix and fibre resulting in early fracture of basalt fibres in the core having low strain capacity than the outer surface aramid fibres. Kretsis (Kretsis, 1987) reported that the initial cracks are formed by the breaking of fibres having lower

| Ageing condition | Flexural Strength (MPa) | Flexural Modulus (GPa) |
|------------------|-------------------------|------------------------|
| Unaged           | 268.32 ± 13.53          | 31.41 ± 1.51           |
| Ambient          | 196.54 ± 15.29          | 23.92 ± 1.14           |
| Sub-zero         | 239.82 ± 12.47          | 28.26 ± 1.35           |
| Humid            | 218.89 ± 16.25          | 25.35 ± 1.27           |
strain and then followed by the fibres with higher strain. This allows the stronger fibre having a lower strain to reach near its ultimate strength.

The decline in mechanical strengths of aged laminates is attributed to the matrix cracking due to the osmotic phenomena arising at the interface along with hydrolysis (breaking up of hydrogen bond) by the occurrence of moisture leading to weakening of the adhesive bond. Absorbed moisture during ageing fills the cavities and cracks of the composite, which acts as a plasticizer and results in free movement of the molecules of the reinforcing fabrics. Moisture uptake can also cause volumetric expansion of the polymer, that is, swelling of the matrix (Arun et al., 2010; Saha & Bal, 2018; VanLandingham et al., 1999). A shell is formed around the dry part of the polymer resin by the swollen part of the polymer, thus resulting in a state of triaxial stresses on this dry part. Matrix fracture occurs when this stress developed due to swelling reaches a critical value and results in the direct rupture of the fibre/matrix interface (Costa et al., 2005). This enhances the polymer-chain flexibility and eases the segmental motion resulting in reduced flexural characteristics (Saha & Bal, 2018). Fractured flexural specimens are shown in Figure 9.
SBS test specimens are loaded such that tensile and compressive forces are eliminated which results in pure shear between the laminae. In this test, the specimen produces horizontal interlaminar cracks and delamination due to the shearing of parallel faces of the specimen in opposite directions. SBS test results of unaged and aged hybrid composites are shown in Figure 10. Unaged specimens showed the highest resistance to shear load which is 15.1 MPa. Among the aged specimens, sub-zero specimens displayed the least reduction in apparent ILSS at 5.16%, while a moderate reduction of 12.51% was displayed by humid specimens, which was followed by the highest reduction of 16.68% by ambient specimens. Retention of apparent ILSS of aged specimens is shown in Figure 11.

The primary reason for degradation of apparent ILSS of aged specimens was due to the swelling stresses generated within the matrix resin and/or at the interface due to the moisture uptake. Specimens mainly failed by micro buckling, fibre rupture, or interlaminar shear cracking as shown in Figure 12. The highest drop in shear strength was exhibited by ambient specimens due to the increased percentage of moisture uptake which led to higher interfacial degradation. The diffused moisture not only damages the material, but also causes the development of hygroscopic residual stresses. In case of sub-zero composite specimens, diffused water gets frozen and results in volumetric expansion and subsequently additional swelling stress development in the laminate. The diffused moisture enters the polymer matrix triggering it to swell, and leading to micro cracks or voids in the polymer matrix/fibre interface. Generally, the failure mechanism of polymer composites due to shear is described by a combination of plasticization, swelling of the material, and reduction in glass transition temperature of polymer resin as it absorbs moisture. Embrittlement of the composite specimens is also related to the deterioration of molecular structure at the macro level by osmotic cracking, hydrolysis, and localized damage at the interface of fibre and matrix (Ray, 2004).

12. Impact strength
The charpy test is an economical method to measure the amount of impact energy a composite material can absorb before the fracture. Only a small amount of the total impact energy is absorbed through the elastic deformation phase, and the plastic deformation phase is rarely seen in composites as they are brittle in nature. Most of the impact energy is utilized to fracture

Figure 10. Interlaminar shear strength of unaged and aged composites.
the material during the impact. The comparison of impact strength of three different aged specimens is shown in Figure 13 with respect to unaged composites. It is clear from experimental results that the ageing process directly affects the impact behaviour of composites. Unaged samples displayed the highest impact strength which is 128.86 kJ/m². The higher impact strength is due to the outer ductile aramid fibres, which absorb most of the impact shocks and protects the inner basalt layers. In unaged specimens, energy during impact is predominantly absorbed by the delamination at the fibre–matrix interface and the debonding of the fibres. Damage of fibres on the non-impacted surface is initiated by the tensile failure due to the tensile forces being
This indicates that the tensile properties of the material at the back surface control the damage phenomena of the composite.

Among the aged specimens, sub-zero specimens showed the highest impact strength, which is at 119.64 kJ/m², followed by humid aged specimens, which is at 112.20 kJ/m² and ambient aged specimens showed the lowest impact strength of 97.21 kJ/m². Impact strength of ambient, sub-zero, and humid specimens was decreased by 24.55%, 7.15%, and 12.92%, respectively compared to unaged specimens. Figure 14 shows the impact strength retention of aged specimens in contrast to unaged specimens. Sub-zero aged composites retained higher impact strength compared to humid and ambient aged composites. This is the result of frozen moisture trapped in the void regions thus inducing higher failure strain to impact, compared to humid and ambient specimens. Unaged samples displayed the highest deformation and dispersed around the impacted region. This is due to the presence of surface aramid fibres, which are also termed as high strain
capacity fibres. The surface aramid fibres absorb higher amount of impact energy compared to low strain basalt fibres. In aged specimens, the swelling of the polymer matrix results in reduced deformation at the impacted zone.

Optical micrograph images of failed impact specimens are presented in Figure 15. The impact behaviour of composite is primarily influenced by interfacial and interlaminar bonding between the fibres and the matrix (Yahaya et al., 2015). This is because, in composite materials, damage due to impact is initiated by the cracking of matrix, fibre/matrix debonding, and delamination at relatively low fracture energies (Strait et al., 1992). The fibre structure damage will offer additional passages for moisture to diffuse into the laminates, which will lead to further weakening of the fibre–matrix interface (Abd El-baky, 2019; Chow et al., 2007; Zhao et al., 2016). Therefore, the drop in impact strengths due to ageing phenomena can be described by the moisture initiating chemical degradation resulting in lower fracture energies. In general, ageing conditions reduce the durability and reliability of the composites due to the deterioration of mechanical properties.

The primary reason for the decrease in mechanical properties is attributed to fibre-matrix interfacial degradation. Unaged specimens have strong interfacial bonding between fibre and matrix interface. When the composites are exposed to ageing environments, they absorb moisture and interfacial bonding weakens. Generally, fibres do not absorb moisture or fibres have zero moisture expansion coefficient, on the other hand epoxy matrix is hydrophilic in nature and has a very high moisture expansion coefficient. Due to this mismatch in the moisture expansion coefficients, when the water molecules are ingressed, epoxy matrix tends to swell and overall thickness of the specimen increases. Due to this, swelling stresses are generated and reduces or weaken the fibre-matrix interfacial bonding which ultimately decreases the load carrying capacity by deteriorating the matrix dominated properties. Furthermore, moisture absorption makes the polymer more pliable and changes the structure of the polymer as a result of the interaction of moisture with the polar group of the resin, which eventually affects the strength and modulus.

Figure 15. Optical microscope images of fractured specimens after impact (a) Unaged (b) Ambient (c) Sub-zero and (d) Humid aged composites.
13. Conclusion
In the current investigation, the influence of different ageing conditions on the mechanical behaviour of basalt and aramid reinforced epoxy composite was experimentally evaluated and compared with the unaged composites. The following conclusions may be drawn based on the results obtained:

- The ageing environmental conditions significantly influence the moisture content and moisture diffusion behaviour of the basalt-aramid/epoxy laminates. Moisture uptake primarily weakened the matrix dominated properties of the composites.
- Composites subjected to ambient, sub-zero, and humid ageing conditions absorbed 5.44%, 3.12%, and 1.8% of moisture, respectively.
- Sub-zero aged composites exhibited maximum retention of mechanical properties followed by humid and ambient composites.
- The specimens aged in ambient conditions experienced the highest reduction in tensile strengths, which is 21.8% when compared to unaged specimens. Sub-zero specimens showed the least reduction in tensile strength at 12.79%.
- Maximum degradation in flexural and interlaminar shear strength were observed in specimens aged in ambient conditions followed by specimens aged in humid and sub-zero conditions.
- Retention of impact strength was highest for sub-zero aged composites (92.84%), moderate for humid aged (87.07%), and lowest for ambient aged (75.44%) specimens.
- SEM analysis of fractured specimens indicated that the mode of failure was primarily due to matrix degradation, fibre pull-out, debonding, and micro cracks.
- Results revealed that moisture has unfavourable effects on the overall properties of the composites. However, confirming other design factors are also necessary in a case-to-case basis before realizing the complete potential of the hybrid laminates for structural applications.

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