Chapter 3. Materials and Methods of Characterizing Sandwich Composites Components and Panels

This chapter describes the material and techniques utilized to develop and characterize the composites under investigation. It provides the route to surface treatment of fly ash and utilizing it as foam core particulate reinforcement. It explains the tests conducted on the components of sandwich composite and sandwich composites. Different types of characterization have been undertaken to quantify the manufactured face sheet, foam core, and sandwich composite specimen's physical, mechanical and microstructural characteristics.

3.1. Materials

Sandwich composites are composed of two face sheets and a core. In this study, fiber-reinforced laminated face sheets and particulate-reinforced PU foam core are used to fabricate sandwich composites.

3.1.1. Face Sheet Material

3.1.1.1. Face Sheet Matrix

Metals, ceramics, and polymers are just a few examples of the various types of matrix materials. When compared to metal and ceramic matrices, polymer matrices have excellent room-temperature properties. They are more frequently utilised due to their cost effectiveness, ease of producing complex parts with little tooling costs, and cost-effectiveness. There are two types of polymer matrices: thermoplastic and thermoset. The resin undergoes an irreversible chemical transition that results in an amorphous cross-linked polymer matrix, which is how thermoset matrices are created. Thermoset resins have strong molecular structures, which help them offer effective thermal and electrical insulation. Due

to their low viscosity, they can wet out fibers well and exhibit excellent thermal stability and higher creep resistance[190].

The most widely used thermoset resins are phenolics, vinyl ester, polyester, and epoxy. Among the above mentioned thermoset resin, Epoxy is having outstanding fibre adhesion to a variety of fibres exceptional mechanical and electrical characteristics, and efficient functioning at high temperatures. They are used extensively for many sophisticated composites. They also have strong chemical resistance and minimal shrinkage after curing. Epoxy (LY 556) is chosen as the matrix material for the current research work due to its various advantages over other thermoset polymers, as previously discussed. It is a member of the "epoxide" family chemically. Figure 3.1 depicts the molecular chain structure of Bisphenol-A-Diglycidyl-Ether, also known as BADGE or DGEBA in abbreviation. When paired with the aliphatic primary amine hardener tri-ethylene-tetramine (TETA), which has the trade name HY 951, it offers a solvent-free room temperature curing system (Figure 3.2).

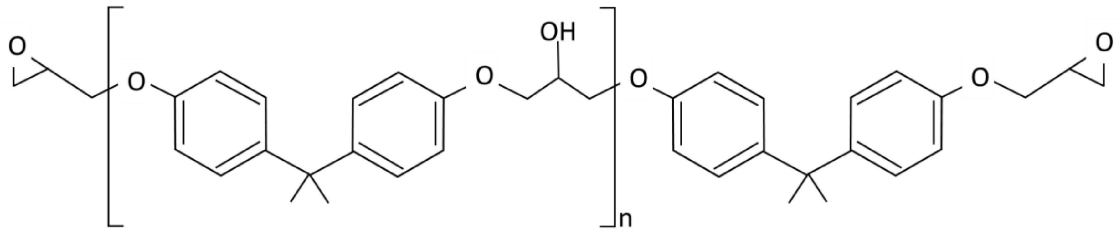


Figure 3.1 Unmodified epoxy resin chain (‘**n**’ denotes number of polymerized unit)

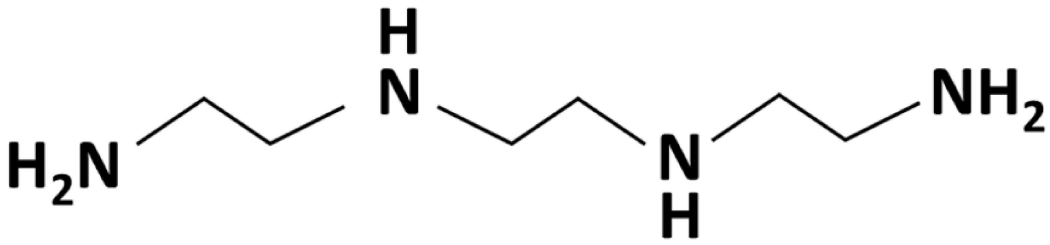


Figure 3.2 Tri-ethylene-tetramine (hardener used for epoxy matrix)

The HY-951 hardener and the LY 556 epoxy resin (Figure 3.3) are procured from excellence resins, Meerut, Uttar Pradesh. Some of the crucial characteristics of epoxy are shown in

Figure 3.3 Epoxy resin (LY 556) and hardener (LY 951)

Table 3.1 Physical and Mechanical properties of epoxy

Characteristic Property	**Inferences**
Density	1.1 gm/cc
Compressive strength	90 MPa
Tensile strength	58 MPa
Micro-hardness	0.085 GPa
Glass transition temperature	104°C
Coefficient of Thermal expansion	62.83 ppm /°C

3.1.1.2. Face Sheet Reinforcement

Long filaments are not always required as reinforcement. They come in a variety of shapes, including flakes, sheets, short fibres, whiskers, and particles. Because materials are stronger and stiffer in their fibrous form than in any other form, it turns out that the majority of reinforcements employed in composites have this form.

We are particularly interested in the so-called advanced fibres in this area because they have very high strength, very high stiffness, and very low density. While many naturally existing fibres can be employed to interior of the vehicle where, low stresses exist like door panels, dashboard parts, parcel shelves, seat cushions, backrests, cable linings, etc. Applications to vehicle exterior are limited due to the high demand of mechanical strength. The main benefit in this situation is undoubtedly the low price. Actually, the main source of fibre materials is the vegetable kingdom. In the textile industry, cellulosic fibres such as cotton, flax, jute, hemp, sisal, and ramie have been utilised, while wood and straw have been used in the paper sector. Other natural fibres like hair, wool, and silk are made of various protein types. Due to their high work of fracture, the silk generated by many spiders in particular appears to be particularly appealing[191].

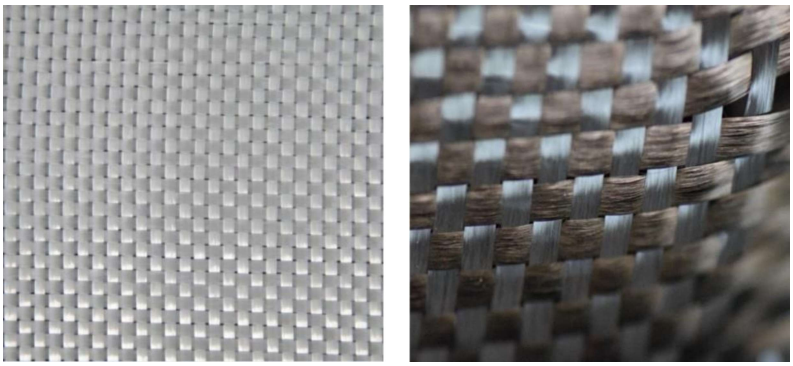


Figure 3.4 Bi-directional plain interwoven glass fibre and carbon fibre

In this study, we have used interwoven glass fibre and carbon fibre as reinforcement of laminated face sheet. The E Glass fiber bi-directional plain interwoven of 200 gsm was

supplied by the R.P. Industries, Aligarh, Uttar Pradesh and carbon fiber bi-directional plain interwoven 200 gsm was procured from the Arrow Technical Textiles PVT. LTD. Mumbai, Maharashtra.

3.1.2. Foam Core Material

Sandwich laminates used in high-performance lightweight structures consist of two strong and stiff thin sheets on the face side with a low-density material as the core in between the face sheets. Such design schemes provide outstanding weight specific bending stiffness and strength properties, thereby replacing the requirement of monolithic materials in aerospace, avionics, marine and automobile applications. In this study the foam core is reinforced with FA particulates. Therefore, the core of the sandwich structure is combination of FA particulate reinforcement and PU matrix.

3.1.2.1. Foam Core Matrix

In this study the core matrix is synthesised by mixing polyol and methylene diphenyl diisocyanate (MDI). A diisocyanate (or polyisocyanate) and a diol (or polyol) are typically used in the polycondensation procedure to successfully create PUs[192, 193]. In 1937, the PU industry was born thanks to the introduction of the diisocyanate polyaddition technique[194]. There are two main PU synthesis routes that are used in industry.

A prepolymer technique, which consists of one or two processes, can be used to create PUs[195]. Both techniques rely on polyaddition in the presence of aliphatic or aromatic diisocyanates and oligomeric diols as a chain extender, catalyst, or other additive (e.g., colouring, fillers, moisture scavengers, crosslinking agents). Figure 3.4 shows two steps of conventional PU production. The creation of urethane prepolymers containing isocyanate groups results from the interaction between diisocyanates and polyols in the first stage. The

prepolymer chains are then extended by low molecular weight chain extenders, such as diamines or diols.

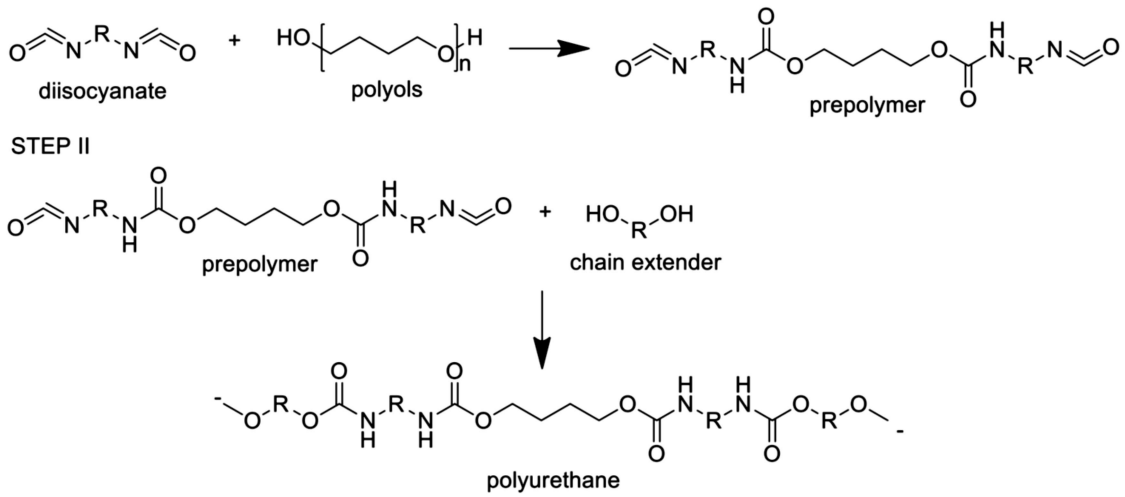


Figure 3.5 Traditional PU synthesis by a two-stage process using diisocyanate, polyol, and a low molecular weight chain extender

PUF is formulated from two ingredients: Polyol and Methylene Diphenyl Diisocyanate (MDI). In this study rigid PU foam has been fabricated with te help of polyol: CRISTOL-P-112/IS, and MDI: CRISTOL MDI, procured from Krishna Antioxidants Pvt. Ltd. Mumbai. Figure 3.6 shows the package of polyol and MDI received.

Figure 3.6 Small packaging of Polyol and MDI

Table 3.2 Properties of MDI and Polyol

Polyol		MDI	
Colour & Appearance	Pale Yellow Liquid	**Appearance**	Clear Dark Liquid
Viscosity @25°C, CPS	838	**NCO Content**	31.5
Hydroxyl Value mg, KOH/gm	380-400	**Specific Gravity@25°C**	1.23
Water	< 0.2%		

3.1.2.2. FA Particulate Reinforcement

Fly ash was gathered from different thermal stations. They are made up of tiny particles with significant concentrations of SiO_2 and Al_2O_3. To choose the appropriate fly ash that could be employed as reinforcing material in the creation of the reinforced PUF core with improved mechanical properties, chemical composition and particle size analysis were conducted. Table 3.3 presents the fly ash composition with weight percentage of chemical compound as obtained from X-ray fluorescence spectroscopy (XRF) and X-ray diffraction (XRD) studies. Fly ash is mostly composed of SiO_2 and Al_2O_3. Other essential compounds present in the fly ash are Fe_2O_3, K_2O, TiO_2, CaO apart from traces of other oxides, as mentioned in Table 3.3. The pictorial view of micro-sized FA powder used in this work is given in Figure 3.7

Table 3.3 Fly ash composition.

Elements	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**K_2O**	**TiO_2**	**CaO**	**P_2O_5**	**MgO**	**SO_3**	**Na_2O**
Weight %	53.82	29.28	7.76	1.82	1.54	1.38	0.53	0.48	0.03	0.03

Figure 3.7 Micro-sized fly ash particles used as filler in PU foam core

3.2. Fabrication of Sandwich Composites

Sandwich composite is fabricated in three steps:

1. Fabrication of the FRP laminated face sheets.
2. Fabrication of the Neat and FA-PUF core.
3. Fabrication of sandwich composites.

3.2.1. Fabrication of FRP Laminated Face Sheets

Face sheets of woven glass fiber and carbon fiber were fabricated separately with epoxy matrix (LY 556 and HY 951) by hand layup technique. Four plies of bi-directional plain glass fiber and carbon fiber were stacked to fabricate the face sheet laminate. The mould release agent was applied on the plane surface for the ease of removal of the finished laminates. A layer of epoxy was coated, and then a layer of bi-directional fiber was laid, and again epoxy was coated

on the fiber layer, and further rest of the layers were laid likewise and co-cured for 24 hours at room temperature. This hand lay-up procedure was adopted for both GFRP and CFRP face sheets fabrication. The steps followed in the fabrication of GFRP by hand lay-up technique is summarized in the Figure 3.8. The same procedure was followed for the fabrication of CFRP laminated face sheet.

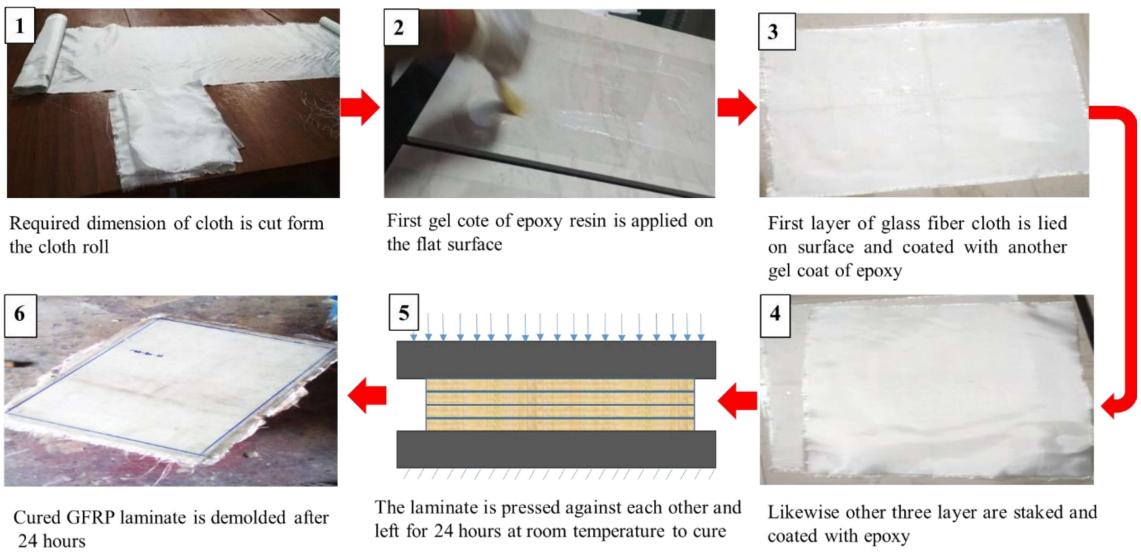
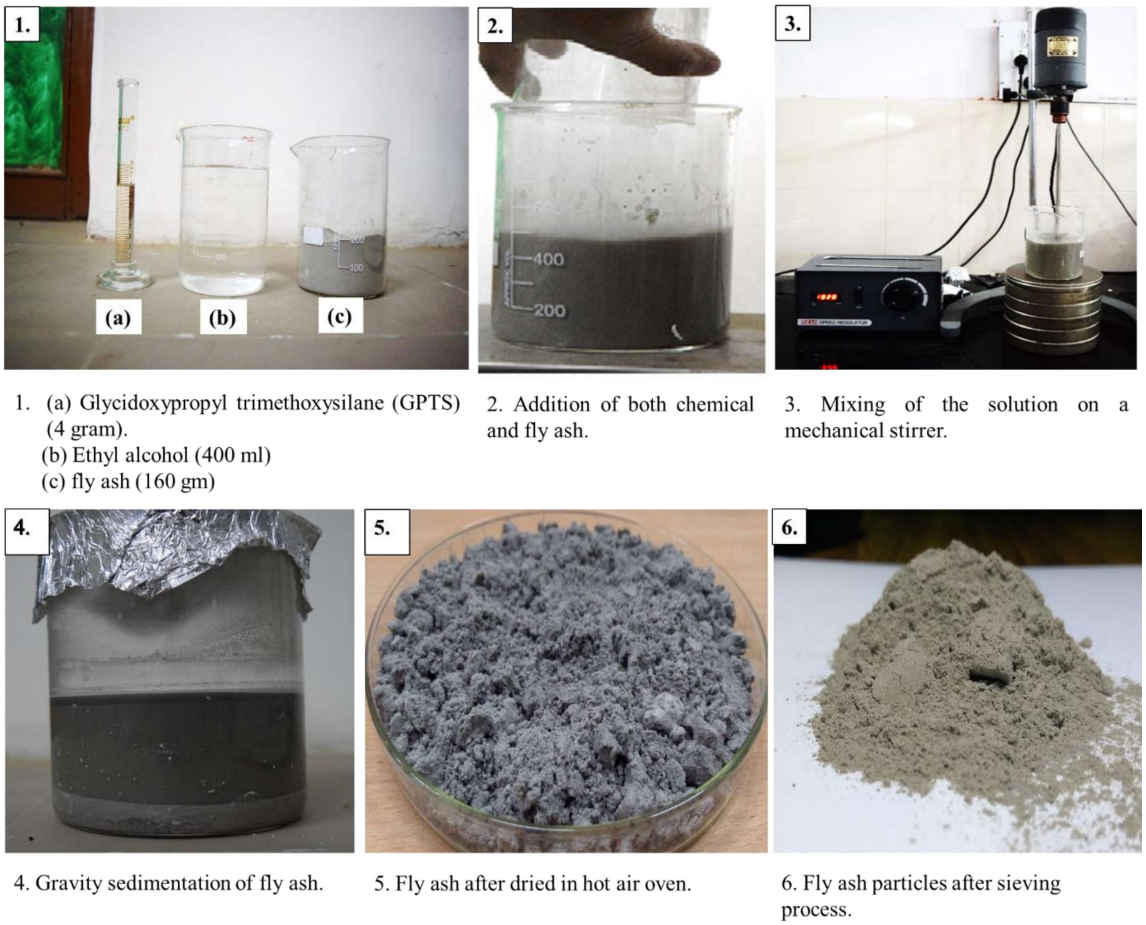


Figure 3.8 Hand layup technique for the fabrication of GFRP laminate face sheet.

3.2.2. Fabrication of the Neat and FA-PUF Core

The neat PUF core can be fabricated by mixing polyol and MDI in the ratio of 100:105. But FA particulates needs to be incorporated as reinforcement in the PUF matrix. One of the main obstacles encountered with attempted surface modification of inorganic filler materials, which includes procedures like chemical modification, chemical grafting, and silane treatment[21, 22]. There is a significant difference between the matrix and the fibre properties in terms of density, elastic modulus, thermal expansion, and surface energetics. The interfacial adhesion between the matrix and the fibres is governed by intermolecular or physical forces, while the production of the composite occurs as a result of chemical bonding during the composites curing process. It is preferable to have higher interfacial adhesion for

the effective transmission of stresses from the matrix to the fibre, which enhances the composites overall material properties[27].



1. (a) Glycidoxypropyl trimethoxysilane (GPTS) (4 gram).
(b) Ethyl alcohol (400 ml)
(c) fly ash (160 gm)

2. Addition of both chemical and fly ash.

3. Mixing of the solution on a mechanical stirrer.

4. Gravity sedimentation of fly ash.

5. Fly ash after dried in hot air oven.

6. Fly ash particles after sieving process.

Figure 3.9 Steps involved in the surface treatment of the FA

Due to inadequate interfacial contacts between the hydrophilic cenospheres surface and hydrophobic polymer, the mechanical characteristics of cenospheres (Fly ash) reinforced polymer composites are subpar. Silane coupling agents are typically employed to facilitate adhesion between an organic matrix and an inorganic filler. The fly ash particles might be integrated into the PU Foam's walls owing to the hydrolytic reaction of the silane groups. In the current study, fly ash's surface was treated with the silane coupling agent GPTS to strengthen its interfacial binding with the PUF matrix. The silane coupling agent GPTS (4 g) was mixed to 400 ml of ethyl alcohol and 160 g of fly ash. With the use of a mechanical stirrer, the mixture was stirred vigorously to provide an even distribution of the coupling

agent to the filler. At 1000 rpm, the mixing was done for roughly 45 minutes. Fly ash settled naturally after mixing, and it was then filtered via filter paper. The filtrate was then dried for 12 hours in a hot air oven. After that, the FA was sieved using a 300 mesh sieve to remove any big particles that may have otherwise compromised the reinforcing properties. The above mentioned procedure is shown figure 3.9 in steps.

3.2.2.1. Fabrication of fly ash reinforced polyurethane foam

The treated fly ash was mixed with MDI with varying weight of 5, 10, 15 and 20 percentage of total weight of MDI and Polyol. The measurement accuracy of the Contech Instrument weighing scale was up to 0.001 gm. The four different compositions of MDI and fly ash, as described above, were mixed with the help of stirrer at 1000 rpm for 10 minutes. A mould release agent was applied to the mould cavity so that the foam after curing can easily be taken out from the mould. Polyol was added to the mixture with the weight ratio of 100:105 that of MDI and stirred with stirrer for 27 seconds before pouring into a mould cavity of dimensions 305mm length, 175mm width and 10 mm depth. Clamps were tightened up to entrap the mixture in the mould cavity. After one hour of pouring the mixture of fly ash, MDI and polyol inside the mould cavity and subsequent curing, the cultivated polyurethane foam (FA-PUF) was taken out.

FA is mixed with Polyol at 1000 rpm for 10 minutes. This mixing distributes the FA particle uniformly throughout the Polyol. Subsequently, MDI is added to the Polyol-FA mixture, which leads to the polymerization and foaming of polyurethane due to chemical reaction. FA addition provides more nucleation sites in the bubble formation. So, the density of bubbles is increased, and the cell aperture is decreased. The FA powder acts as a nucleating agent generating more cells and thereby increasing the cell density. This increase in cell density with the addition of FA means more cells form within the same amount of polyurethane. More cells give rise to more surface area generation for the same polyurethane

volume inducing a decrease in the cell wall thickness. Since polyurethane foam is fabricated in a closed rectangular mould; therefore, polyurethane has the same volume to expand, before and after the addition of FA. The mould cavity dimension is 305x175x10 mm^3. For the dimensional stability of PUF, the shrinkage is measured with the dial gauge vernier and the shrinkage of PUF with respect to mould cavity is found to be 1% and is considered acceptable within the limit. With the addition of FA, no significant change in shrinkage percentage was observed.

3.2.3. Fabrication of Sandwich Composite

Once the fly ash reinforced polyurethane foam and fiber reinforced face sheets were fabricated, these two components were coupled together with the help of adhesive, epoxy LY 556 and HY 951 and put under pressure and left to cure for 24 hours. The ASTM standard specimens, as per the test requirement and dimension, were prepared from the finished sandwiched composite. ASTM C393 and ASTM D6264 has been followed to fabricate sandwich specimen for bend test and indentation test respectively.

3.3. Physical Characterization

3.3.1. Density Measurement of Laminated Face Sheet and PUF Core

Density has a substantial impact on the mechanical performance of the material. Therefore, it become essential to evaluate the density of fabricated face sheet and foam core.

3.3.1.1. Density measurement of face sheet

The density of the GFRP and CFRP laminates have been determined as per ASTM standard D792-13[196]. Since the specimen volume should not be less than 1cm^3 as per the standard, accordingly, the laminate thickness has been taken to be 1.2 mm, with the specimen cross-section being 30 x 30 mm^2. The measurement least count of the dial gauge vernier is 0.1 mm. The recommended weight of the laminate specimen should be 1 to 5 grams for density

measurement. The digital weighing balance used for the same can measure with a precision of 10^{-4} gram (0.1 mg). Fig. 3.10 shows the specimens used for density measurement. The typical measurement of the specimen mass and the calculated density based on the principles of Archimedes.

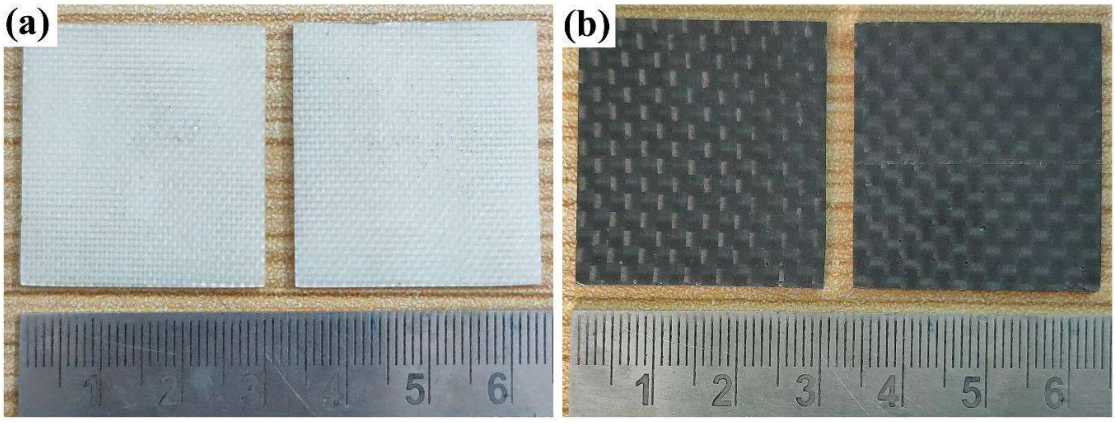


Figure 3.10 (a) GFRP (b) CFRP specimen for density measurement.

3.3.1.2. Density Measurement of Foam Core

The core density of the PUF and reinforced FA-PUF sandwich composite was calculated according to ASTM D1622/D1622M [197]. As per the standard, the volume of the specimen should not be less than 16.4 cm^3. The maximum allowed cross-sectional area perpendicular to the measured dimension should be 25 cm^2, and the maximum allowed length of the longest perpendicular dimension should be 10 cm. The core density was calculated by measuring the specimen dimensions with a digital Vernier Calliper and weight on a high precision weighing scale having the least count of 1mg. The core density changes with the fly ash inclusion because the mold cavity has a fixed volume to expand. The test specimen dimension chosen for the density calculation in this work is 5x5 cm^2, having 1 cm thickness.

Figure 3.11 PUF specimen for density measurement.

3.3.2. Scanning Electron Microscopy

The micro-structural features of the various particulate filled composite specimens were examined by Scanning Electron Microscope (SEM) (Serial no.- EVO18- 20-45, ZEISS EVO 18 RESEARCH, Germany). The specimens were mounted on stubs with silver paste. To improve the penetration of light and for better surface micrographs, a thin film of gold is vacuum-evaporated onto the samples before the pictures were taken. The fabricated and failure under indentation samples were characterized using SEM. The morphological and reinforcement study has been conducted by SEM images.

3.4. Mechanical Characterization

3.4.1. Tensile Testing of Face Sheets

The tensile testing of the CFRP and GFRP face sheets only were performed as per ASTM standard D3039/D3039M-17 [198] to determine the in-plane tensile properties. Tensile test specimens of 250mm x 25 mm x 1.2 mm of length, width and thickness, respectively, were prepared. The tensile test is done on an INSTRON 8801 servo-hydraulic UTM testing system with the strain rate of 0.5mm/min at room temperature. Five samples of each are tested to calculate the tensile strength and modulus of elasticity. Figure 3.12 shows Fig. (a) and (c) are the GFRP and CFRP tensile specimen of 250 mm x 25 mm x 1.2 mm (b) and (d) are the GFRP and CFRP specimen clamped in the tensile grips.

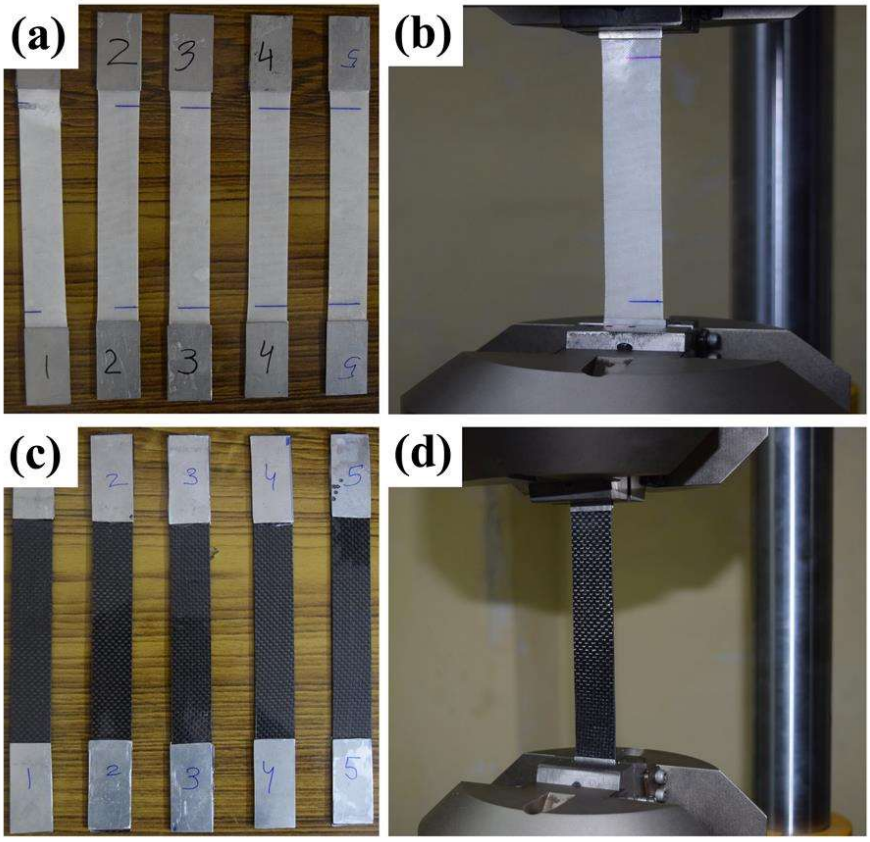


Figure 3.12 (a) GFRP tensile test specimen (b) GFRP tensile test held in tensile grips (c) CFRP tensile test specimen (d) CFRP tensile test held in tensile grips.

3.4.2. Compression Testing of Sandwich Core

The compression test on neat PUF and reinforced FA-PUF were performed separately according to the ASTM standard C365/C365M [199]. The flatwise compressive properties of the sandwich core are evaluated. The effect of reinforcement on the modulus of the core is calculated. The dimensions of the specimen PUF core as per mentioned ASTM standard should not exceed 10,000 mm^2. The minimum facing area of the specimen should be 625 mm^2 for continuous bonded surface, which is applicable for this case. Hence, in this study, the specimen dimension is 50mm x 50mm, making 2500 mm^2 well within the limit as aforementioned. The crosshead speed as per the standard is 0.5mm/min at room temperature. The neat PUF and reinforced PUF specimens are shown in Figure 3.13 (a) while, (b) shows

the PUF test specimen between the compression platen during the compressive properties evaluation.

Figure 3.13 (a) PUF core compression test coupons arranged in increasing wt.% of FA reinforcement neat, 5%, 10%, 15% and 20% respectively (b) PUF specimen under compression test configuration.

3.4.3. Shear Test of Core

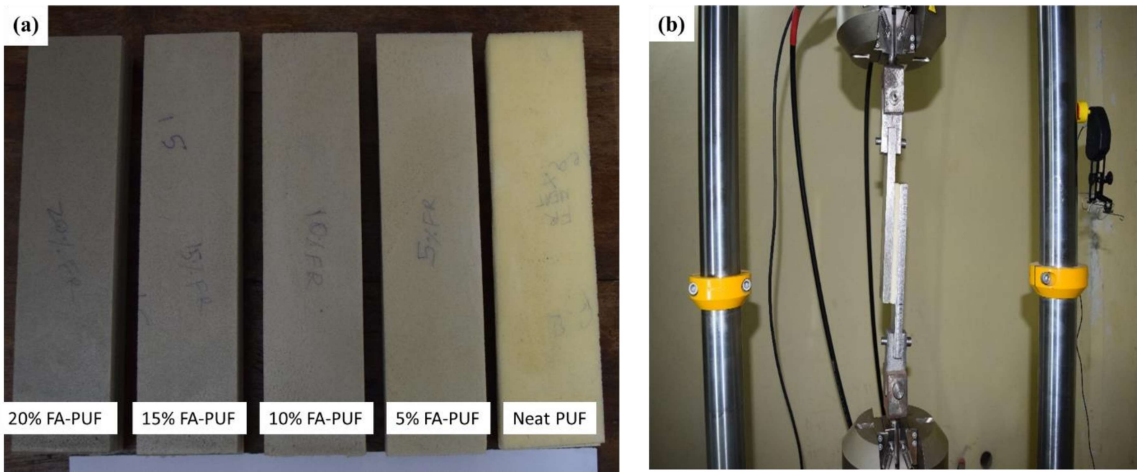


Figure 3.14 (a) PUF core compression test coupons arranged right to left in increasing wt.% of FA reinforcement neat, 5%, 10%, 15% and 20% respectively (b) Shear test arrangement of Fly ash reinforced core on INSTRON 8801.

The shear test of the core was done according to ASTM standard C273/C273M-16 [200] using INSTRON UTM at strain rate of 0.5mm/min at room temperature. This test determines the shear properties of sandwich core materials associated with shear distortion of planes parallel to the facings. The shear test determines the shear strength and shear modulus. Here, the thickness of the specimen is the thickness of the sandwich core, i.e., 10 mm; the width is 50 mm, and the length is 150 mm. Adhesive or cohesive failure in between core to load plate

is not an acceptable failure until unless core shear failure. Figure 3.14 shows the bonding and experimental set up for the PUF specimen.

3.4.4. Bend Test of Sandwich Composites

The flexural testing of the three-point bend GFRP and CFRP sandwich composite specimens were carried out as per ASTM standard C393/C393M-16 [201]. Figure 3.15 describes the test arrangement for the sandwich specimens. The standard speed of the crosshead displacement is 6 mm/min. The dimensions of the specimen used to test under bending loading are 200mm length, 50 mm width and 12.4 mm of thickness (1.2 mm of each face sheet and 10 mm of the core) according to the ASTM standard mentioned.

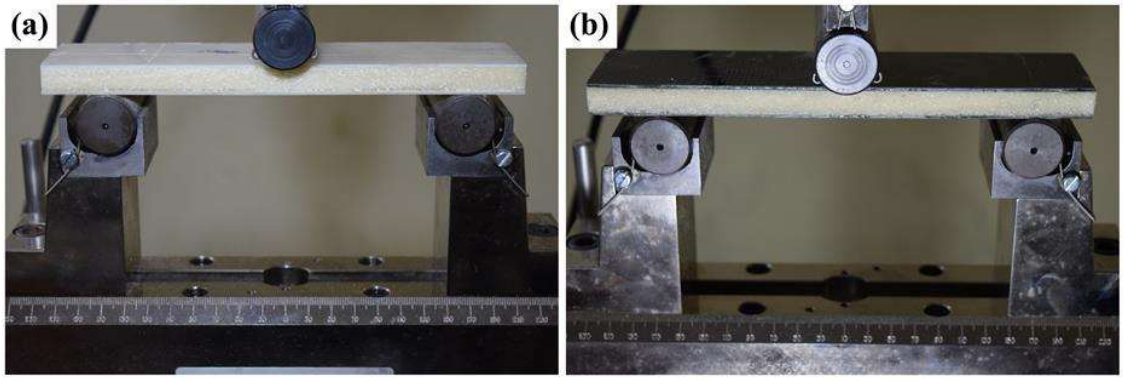


Figure 3.15 Bend test on (a) PUF core GFRP sandwich composite (b) PUF core CFRP sandwich composite.

3.4.5. Quasi-static Indentation Test

3.4.5.1. Quasi-static Indentation of PUF core:

The testing was conducted to examine the damage resistance and deformation mechanism of the neat and reinforced PUF core. Flat-circular, hemispherical, and conical indenters prepared in this investigation are shown in Fig. 3.16 (a). The indenters were mounted on the INSTRON UTM 3367, shown in Fig. 3.16 (b). The load rate and other parameters for the testing are well aligned with D6264[202]. Rigidly backed specimens are used to study the damage resistance in this case. Therefore, the specimen is placed directly on the rigid steel

plate, having a thickness of more than 12.5 mm, as stated in the standard. The width and length of the specimen forming area are 50*50 mm2, and the thickness is 10 mm. Flat-circular, hemispherical, and conical indenters are fabricated to investigate the effect of nose profile geometry on indentation resistance. The indenter geometry that has been used in this research is listed in table 3.4.

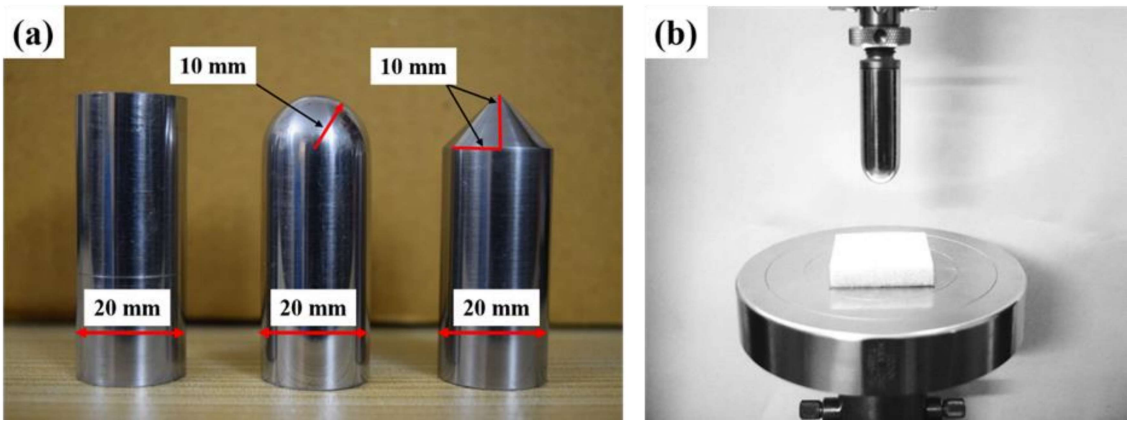


Figure 3.16 (a) The geometry of three types of indenter and (b) test configuration of indentation test on PUF with hemispherical indenter.

3.4.5.2. Quasi-static indentation of sandwich composite

GFRP and CFRP PUF sandwich composites are machined to required dimensions as per ASTM standard. The length and width of square specimen is 50 mm each and thickness is equal to the thickness of the sandwich panel. The sandwich specimen ready to test under indentation is shown in Figure 3.17 (a) and quasi-static indentation test arrangement is shown in Figure 3.17 (b). The load rate and other parameters for the testing are in conformance with ASTM D6264[202]. The indentation response varies with the inclusion of fly ash in the PUF core of the sandwich composites.

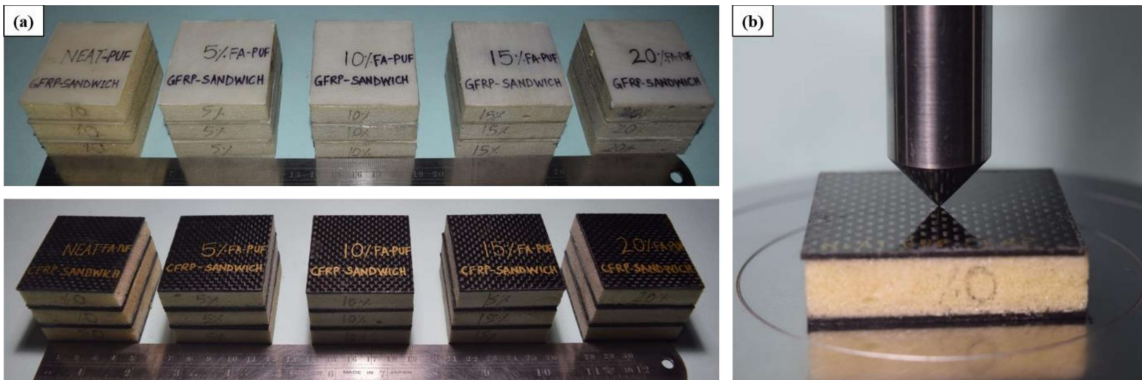
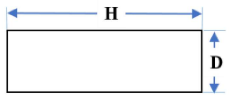
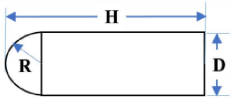
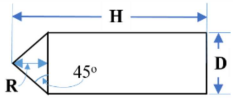


Figure 3.17 (a) Test specimen with varying fly ash wt. % to GFRP-PUF and CFRP-PUF sandwich composite and (b) Indentation test configuration on CFRP-PUF sandwich with conical indenter.

Table 3.4 Nose profile geometry of the indenters.

Indenter	Nose geometry	Nose shape	D	R	H
1.		Flat-circular	20	-	50
2.		Hemispherical	20	10	50
3.		Conical	20	10	50

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