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Investigation on Mechanical and Morphological Characteristics of Ramie/Silk with Epoxy Hybrid Composite of Filler OMMT Nanoclay

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Abstract

When subjected to mechanical characterization, natural fibre-reinforced composites perform better. Ramie/silk reinforced with epoxy hybrid composites have been used to enhance the quasistatic characteristics of natural fibre composites. Yet, research has been done on the synergistic effects of the design factors on ramie and silk reinforced with epoxy resins and filled with nanofillers. In this study, the impact of the stacking order on the mechanical characteristics and moisture absorption of ramie and silk with montmorillonite nanoclay (OMMT) particles has been examined. Five different laminates were prepared using the manual hand layup method with a filler percentage of 3%. The prepared laminates are cut according to the ASTM standard for conducting different mechanical tests. The result shows that the incorporation of pure ramie fibres enhances the mechanical characterization. The hybrid composite of laminated RSSR with 3% OMMT nanoclay produced the highest tensile strength compared to the other two hybrid laminates. The highest flexural strength and modulus are found in the laminate RRRR. The moisture absorption percentage was maximum for ramie laminate RRRR and minimum for silk laminate SSSS. The fractured and debonding tensile specimen was analysed by scanning electron microscopy.

Keywords: Ramie, Silk, Epoxy, OMMT Nanoclay, Mechanical properties, and SEM.

1. Introduction

Natural fibres like banana, jute, sisal, hemp, etc. can be used as substitutes for synthetic fibers. To name a few of them, glass, carbon, boron, and Kevlar are the conventional fibers, and these fibres are not biodegradable and have a higher cost contrasted with natural fibres that have several advantages, such as lower cost, lighter weight, ease of processing, and biodegradability. In regard to the several publications relating to natural fibre hybrid composites, the mechanical properties have been studied. It was found that better mechanical strength is obtained for the hybrid composites than the conventional natural fibre composites [1, 2]. Natural fibres are increasingly being used due to their biodegradability, abundance, affordability, and low processing energy requirements [3, 4]. In polymer composites, natural fibres are used as reinforcement, and they are plant, animal, and mineral fibres to the matrices such as thermoset and thermoplastics, due to their exceptional qualities, including high specific modulus, great resistance to wear and tear, and lightweight[5, 6]. This analysis focuses mainly on natural fibres made from plants because the research community and business have recently turned their attention to this source of fibre. However, in order to give readers a comprehensive understanding of natural fibre reinforced polymer composites, bagasse, sugar palm, bamboo, wood, pineapple, hemp, cotton, jute, and kenaf fibres have been widely employed in various polymers to improve the characteristics of the composites, especially from the standpoints of the environment and biodegradability. [7, 8] Due to the variation in the diameter, length and their specific gravity, the mechanical and physical properties vary in the fibres more importantly. However, the fibres can be compared only to their chemical constituents only [9]. Although natural fibres have been thoroughly investigated as polymer reinforcing materials, numerous other natural fibres, including those from banana, abaca, ramie, and pine, have comparable chemical compositions and have not yet been thoroughly investigated. [7] The lack of research on these fibres as reinforcing agents for composites shows that there is opportunity for additional investigation in the sector and that there are endless opportunities to advance this technology. In order to improve the composite qualities, hybrid composites are created by employing multiple reinforcing agents in the same polymer matrix. When numerous reinforcing materials are combined, it is possible to achieve either a material's synergistic or antagonistic effects. [10] Compared to single-fiber reinforced composites, the integration of numerous reinforcements in a matrix gives a wider range of properties. As a result, the scientific community has given the combination of reinforcing agents with natural and synthetic origins a great deal of attention. [11] Because synthetic fibre composites' properties are regarded to be better than those of natural fibre, their usage in the manufacturing and production sectors is unavoidable. [12] The natural protein fibre that has been found to be the best for biodegradability and biocompatibility is silk, which is frequently utilised for

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implantable purposes. The silk properties include strong strength (650-750 MPa), high elastic modulus (16GPa), density (1.3-1.38 g/cm3), elongation at break (18%-20%), and excellent resilience. [13] Due to their substantial crystallinity and strong hydrogen bonding, silk fibres have improved environmental stability. [14] The silk fibres are made up of two proteins: fibroin and sericin. In contrast, sericin was taken out of silk by using an implanted application during the degumming process. A fibrous substance called silk fibroin has an ordered structure and a repeating sequence of amino acids that are high in alanine and glycine. [15, 16] Particularly helpful in biomedical applications, such as tissue engineering, wound treatment, and implant materials made of silk fibroin. [17] Due to its intriguing qualities, silk was a suitable reinforcement material for composite preparation. The purpose of using natural fibres is to displace synthetic fibres. A type of natural fibre called ramie fibre is obtained from the bark. Additionally, it has the lowest lignin level and the highest cellulose content. [18] As a result, there is enormous strength to be developed as a reinforcement material for composites. Crude ramie fibre has 9.25 % lignin, 9.8-16.7 % hemicellulose, 68.7-70.1% cellulose, 6.95 % moisture content, 1.25 % ash, 0.3-0.6 % waxes, and 4.8 % pentosan as chemical constituents. Ramie fibre is compatible with a variety of substances, making it simple to combine as it has better tensile strength, Young's Modulus, and resistance to biological degradation. [19, 20, 21]The production of sheets for decorative purposes uses polymers. In this investigation, the wear resistance and water absorption tests were analysed. To improve the lower water absorption qualities, a higher amount of Bakelite was put in. [33]the use of a copper sulphate solution is characterized as an electroless way of coating copper on basalt fibres. The purpose of this study is to research how electroless coating parameters in electroless solution affect the coating morphology of basalt short fibres and to optimize the coating process parameters using genetic programming[34]. Due to their extensive variety of mechanical qualities and applications, composite laminated structures have received a lot of attention lately. However, since the latter is a key factor in the layers' delamination in an eight-layer laminated, simply supported Graphite/Epoxy FRP composite beam, this study investigates the impact of temperature as well as the effect of fibre orientation on mid-plane transverse deflection and interlaminar shear stress[35]. However, none of the researchers fully concentrated on natural fibres like ramie and silk fibre reinforced hybrid polymer composites. In this study, the tensile, flexural, moisture absorption, density, impact strength, and hardness characteristics of silk and ramie reinforced hybrid polymer composites are examined to know their behavior.

2. Materials and methods

2.1. Materials

ULTRANANOTECH India Ltd., Bengaluru, supplied the Araldite epoxy resin with the grade name LY556 and the hardener HY-951 for curing the resin, and the OMMT nanoclay of 100 nm. Vruksha Composites Guntur, Andhra Pradesh, India has supplied the woven Silk Fibre and Ramie fibre of 250 GSM (Gram per square meter).

The natural fibre, ramie, belongs to the Urticaceae family and was hence extracted from Bohemian nivea bast. From the outer section of the stem, usually the fibres will be harvested, which are now being used as textile fabric. The properties of fibers and matrix are represented in Table 2.

Table1. Laminate Designation

Sl no	Laminates
1	Epoxy
2	S+S+S+S
3	R+R+R+R
4	R+S+R+S
5	S+R+R+S
6	R+S+S+R

R-Ramie fibre, S-Silk fibre

2.2. Ramie Fiber

Table 2. Physical characteristics of the fibers and epoxy [32]

Description	Ramie fiber	Silk fiber	Epoxy
Density (g/cc)	1.5	1.38	1.1
Tensile strength (MPa)	1000	650	35 - 135
Tensile modulus (GPa)	61.4 - 128	16	3.4
Elongation (%)	2-4	18-20	1-8.5

2.3. Silk Fiber

Silk fiber is one more fiber that offers properties like low density, flame resistance and high elongation and hence silkfibreportrays higher mechanical performance than plant fiber.

Figure 1. A. ramie fabric **B.** silk fabric **C.**OMMT nano clay and **D.** epoxy resin

2.4. OMMT nano clay

The nanoclay called montmorillonite is made up of the phyllosilicate group of minerals which, when induced to go suddenly from water solution, form microscopic crystals. The montmorillonite falls under the smectite group. It is a 2:1 nanoclay, which implies that it has the octahedral sheet of alumina put between the two tetrahedral sheets of silica. The median diameter is around 1 μm and the thickness appears to be 9.6 mm. However, the nanoclay particles can be named by utilizing the scanning electron microscope with a magnification of about 25,000 times. Saponite is also a member of this group.

2.5. Composite Preparation

The hand layup process was used for developing hybrid nanocomposites using ramie, silk fibre, and nanoclay as depicted in Figure 1. The nanoclay was dispersed at 3%wt using a sonication process for about 20 minutes at 20 kHz and then mixed with the known quantity of resin. However, the mixture is then poured for composite fabrication. To make the 300x300x3mm3 laminate, 40% of the ramie and silk fibre mixture is manually mixed with the resin. While the process is taking place, all the bubbles which are entrapped are removed by means of a cotton roller. Then, the composite laminates are cured at room temperature for about a day before being put into the oven. Soon after this, the laminates are machined using the water abrasive jet machine as per the requirements of ASTM standards for further testing.

3. Experimental Studies

3.1. Density Test

The density and voids in the laminates are determined experimentally as per the requirements of ASTM D2734-94. The experimental density is calculated by calculating the weight of the specimen by means of air and liquid. The experimental density is calculated using the following equation (1) [22].

$$\rho_{exp} = (w_a/w_a - w_l) * \rho_l \quad (1)$$

Where ρ_{exp}= experimental density, w_a= weight of the specimen in air, w_l= weight in liquid, and ρ_l= liquid density.

For each laminate calculation for the theoretical density will be made for the all trials and values was recorded. Henceforth, the theoretical density will be calculated using the equation (2) [23].

$$\rho th = \frac{100}{\frac{Wm}{\rho m}+\frac{Wf}{\rho f}} \quad (2)$$

Where W_m= the weight fraction% of matrix phase, ρ_m= the density of the matrix phase, W_f =weight fraction% of fabric, and ρ_f= the density of the fabric. The voids generally take place during the fabrication process that too, in the manual layup process, which is unavoidable, and can be due to human error. Generally speaking, a composite laminate with less than 5% will be considered good. As the percentage of void increases, the property deteriorates, i.e., the composite cannot be used for commercial use. The tabulated values of the density and void fraction are given in Table 3. The determination of the void in terms of percentage was found using theoretical and experimental densities in Equation (3). [24] And Table 3 shows the experimental and theoretical densities and voids of the specimens.

$$Vp = \frac{\rho th - \rho exp}{\rho th} \quad (3)$$

Where Vp = the void percentage, ρ_{th} = the theoretical density and ρ_{exp}= the experimental density.

Table 3. Density and void fraction of five laminates

Laminates	Theoretical density ρ_{th}(g/cm³)	Experimental densityρ_{exp}(g/cm³)	Void (%)
SSSS	1.233	1.226	0.56
RRRR	1.360	1.352	0.58
RSRS	1.266	1.255	0.86
SRRS	1.195	1.184	0.92
RSSR	1.282	1.275	0.54

3.2. Tensile test

The laminate is stacked, and weight is added bit by bit until a fracture occurs under tensile loading. By this, the tensile properties of silk-ramie and nanoclay (OMMT) built up polymer hybrid composite will be examined. An electronic information data system is utilized to keep a nonstop record of load versus deflection. Figure 2 shows the composite laminates SSSS, RRRR, RSRS, SRRS, and RSSR displaying the tensile characteristics.

3.3. Flexural test

The flexural strength and modulus will be found as for the dimension of 90 x 10 x 3 mm3, which is as per ASTM D790 in the Universal Testing Machine. Using the flexure test, the three-point bending method will be used with a continuous strain rate of 2 mm/min applied to the five identical samples from each laminate. The application of the load will be on the mid-point of the gauge length, and jaws are used to support the ends of the device. Consequently, the graphs get generated for the breaking load vs. laminates and flexural strain vs. laminates.

3.4. Impact strength

The impact strength will be found out as for the dimension of 63 x 12.7 x 3 mm3, which is as per ASTM D256 using the computerised impact tester machine. The composite laminate was loaded into the impact tester's grippers and the test resulted in the energy absorbed by the laminate after its failure, finally measured in joules.

3.5. Micro- Hardness

The digital Shore-D hardness durometer is used to measure the specimen's hardness. The durometer with a step of 0.5 HD has a range of 0–100 HD and is hence used to find out the hardness of rubber, polymers, as well as plastics. While testing, the durometer is used to press onto the exterior part, where the indenter pin will get penetrated onto the sample, thus displaying digitally the resistance to indentation [27]. Suppose the value of HD is more than 60, it is found that it is a good resilience material or else a poor resilience material.

3.6. Moisture Absorption Test (MAT)

The water absorption test will be carried out by immersing the laminate in normal water as well as in distilled water for nearly 30 days. However, the laminates were machined enough as per the ASTM D570 standard, for the dimensions of 30×28×3 mm^3. The weights of the laminate were frequently measured at intervals of 10 days. The water molecules should be removed from the surface of the specimen using the cotton cloth and weighed with a digital balance. The percentage of moisture absorption was calculated by using Equation (4). [26]

Moisture absorption (%) = W_b-W_a/W_a *100 (4)

Where W_b is the final weight of the laminate after 10 days of immersion and W_a is the initial weight of the laminate before immersion.

3.7. Scanning Electron Microscopy (SEM)

Scanning electron microscopy is a device that will be used for observing the morphology of the composite laminates. This test will be used for analysis of the interfacial properties, internal cracks, and inward design of the broken surfaces of the laminate. The conducting material will be coated on the exterior part of the laminate, preceding the Scanning electron microscope assessment of the surfaces.

4. Results and Discussions

4.1. Density

The void fraction (%) in composite laminates was calculated using the differences in experimental and theoretical densities, which showed some amount of variation (from Table 4). Due to the close compatibility of the fabric, matrix, and filler material, the laminate RSSR had fewer voids (0.54 per cent) than that of other laminates. The void content of laminate SSSS is 0.56 % which is slightly higher than that of laminate RRRR. Furthermore, because RSRS laminate is entirely composed of natural ramie and silk reinforcement, it has a void content of 0.86 percent. The void fractions of all the five different configurations of laminates range from 0% to 1%, which indicates that the composites were properly manufactured and the void percentages obtained are acceptable. It is observed that a higher percentage of filler material showed a lower void fraction, while natural fabrics contained a greater number of voids.

4.2. Tensile test

The tensile properties of laminate designations SSSS, RRRR, RSRS, SRRS, and RSSR were investigated using a Universal Testing Machine (UTM) as per the standards of ASTM. The stress-strain analysis was used to determine the tensile strength and tensile modulus for various laminates. From the figure, it is observed that the composite having only ramie reinforcement (RRRR) has a higher tensile strength of 60.05 MPa and a modulus of 2.74 GPa. It is natural for composites to gain more developed mechanical qualities as the number of layers increases because the load acting on the matrix will be distributed homogeneously. [25] The SSSS laminate consists of only natural silk reinforcement and obtains a tensile strength of 27.69 MPa and a modulus of 3.42 GPa. The hybridised composites of ramie and silk with 3% filler, the designation of laminates RSSR, obtained a strength of 52.37 MPa and a modulus of 2.29 GPa. Figures 2A and 2B represent the different values of tensile strength and tensile modulus of different laminates, respectively. The hybrid laminate of 3% OMMT filled composite has shown a lower tensile strength of 38.14 MPa and a tensile modulus of 4.02 GPa when compared to those of RRRR and RSSR. To move further, the SRRS composite displayed a declined strength value of about 38.14 MPa and 2.29 GPa for the tensile modulus values. So, as per the observations made so far, it can be concluded that the highest tensile and modulus values are exhibited in the case of laminate RRRR, whereas laminates RSSR and RSRS with 3% OMMT filler showed a moderate increase in tensile strength and modulus values. Henceforth, the tensile strength and the tensile modulus values of natural ramie fibre can be improved by the addition of a 3% weight concentration of filler material.

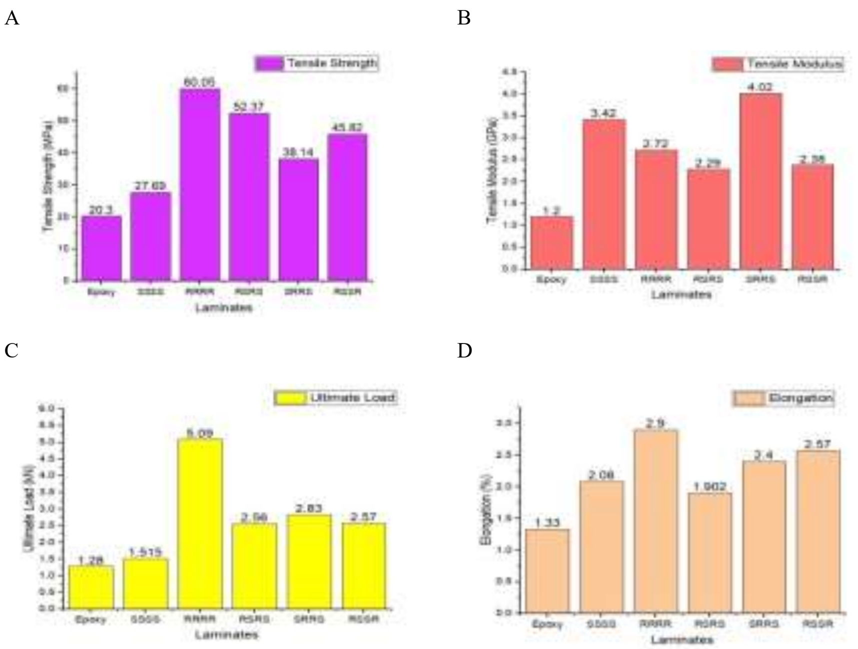

Figure 2. Tensile property of the samples: **A.** Tensile strength and **B.** Tensile Modulus **C.** Ultimate load **D.** Elongation

4.3. Flexural test

The three point bending method is used to test the flexural properties of the composites for flexural specimens and is thus depicted in Figure 3. The highest flexural load of 203.02N laminate is obtained for RRRR and the least flexural load for SSSS laminates is 61.35 N. When it was hybridised with 3% OMMT to the ramie and silk reinforcement, laminate RSSR displayed the highest flexural load of 101.26 N, compared to the other two laminates of RSRS at 93.77 N and SRRS at 88.09 N. With the increase in filler concentration of 3% OMMT, all the laminates have gained the momentum of strength to withstand higher loads. The characterization for the various laminates for the flexural strength and flexural modulus is shown in Figure 3. The better stress transfer has taken place between the fabric–matrix and the filler material, indicating the better flexural strength. The laminates with 3% of OMMT filler RRRR indicated the highest flexural strength of 116.45 MPa and a modulus of 3.42 GPa when compared to the pure silk laminates SSSS, with a flexural strength of 61.86 MPa and a modulus of 1.874 GPa, respectively. The hybrid laminates of ramie and silk with 3% filler, RSSR, show the highest flexural strength of 92.77 MPa and a modulus of 2.208 GPa. However, when compared to the other two laminates of the same designation, SRRS, it depicted a value of 76.5 MPa and 2.185 GPa, respectively. In view of all these observations, it is concluded that the flexural strength and modulus were elevated by the incorporation of natural ramie fabric.

4.4. Impact strength

The bonding strength of the fibre and the matrix along with the filler material will be determined by the impact studies. Figure 4 shows the impact strength (kJ/m^2) of the different laminates. The test on the impact relies on many variables, like fibre-matrix bonding and the nature of the fibre material. Equation 6 gives us the calculation of the impact strength. The laminate RRRR with 3% filler has a higher impact strength of 56.58 kJ/m^2 when compared to other laminates. The reason is the stiffness of the Ramie fabric. The pure silk fibre reinforcement laminate SSSS depicts the lower impact strength. This is due to the presence of hemicellulose, which reduced the impact strength. The reasonable impact strength of 36.75 kJ/m^2 was observed in the Silk and Ramie reinforced composite laminates (RSSR). Furthermore, when the filler material of 3% OMMT was added to the hybrid composites, it led to a decrease in the impact strength of 30.5 kJ/m^2 in RSRS and 27.30 kJ/m^2 in SRRS subsequently. The reduction in the strength was attributed to the fact that the filler material reduced the compatibility and the adhesive nature between the reinforcement and the matrix phase.

$$Impact\ strength\ (I.S) = \frac{Impact\ Energy\ in\ joules}{Area\ of\ cross\ section\ in\ m^2} \quad (5)$$

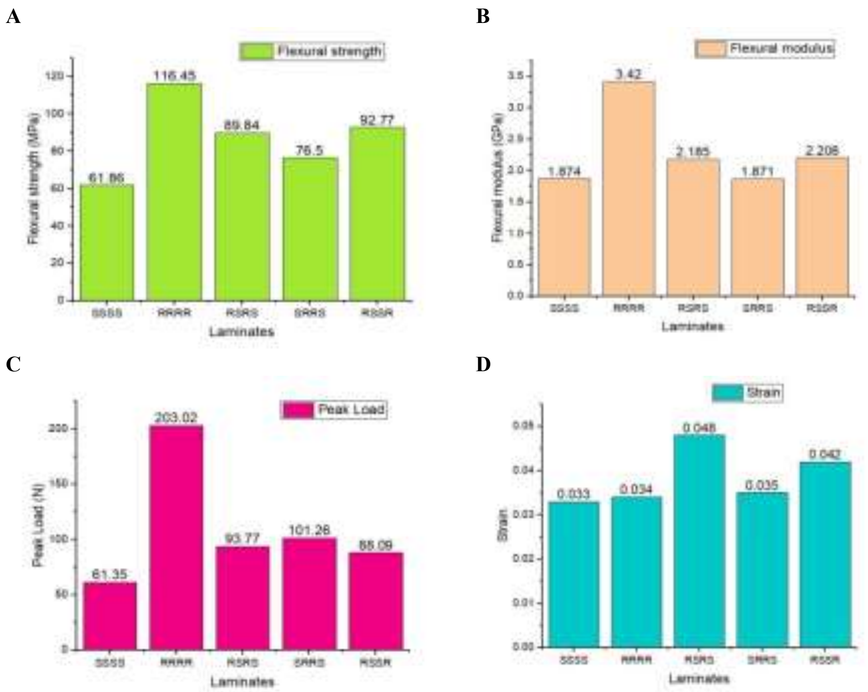

Figure 3. Flexure property of the samples: **A.** Flexure strength B**.** Flexure Modulus **C.** Peak load and **D.** Strain

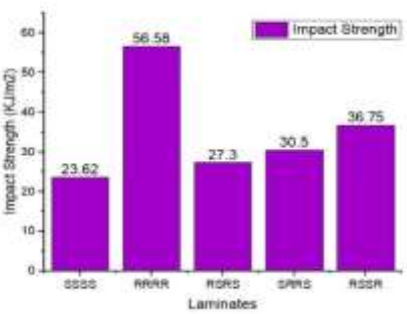

Figure 4. Impact strength of composites

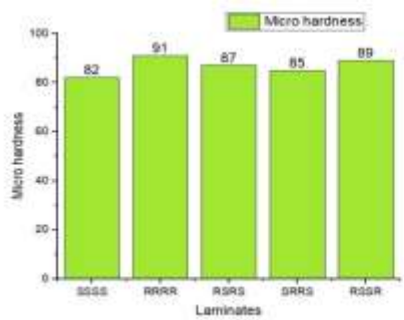

Figure 5. Micro Hardness values of different laminates

4.5. Moisture Absorption Test (MAT)

The specimens, after absorbing the water, will gain weight. The experiment continued for 30 days and was repeated frequently at an interval of 10 days in both normal and distilled water. Tables 4 and 5 show the weights before and after the duration in both conditions. The results showed that the water absorption percentage is higher in the RRRR laminate, a pure natural ramie fibre. Water absorption is less in the case of laminate SSSS, which had four layers of natural silk fibre. Because natural fillers are cellulose-based, plane hybrid composites have lower absorption capacity than filler-filled laminates. The specimen absorbed more water in normal water than in distilled water.

Table 4: Moisture absorption % in distilled water.

Laminates	Weight of the samples before absorption (g)	% increase in weights		
		Day 10	Day 20	Day 30
SSSS	3.656	5.61	9.35	12.25
RRRR	4.451	8.62	14.32	18.36
RSRS	4.125	7.27	12.98	17.16
SRRS	3.961	6.78	11.32	14.65
RSSR	4.213	8.86	12.65	15.23

Table 5: Moisture absorption % in normal water

Laminates	Weight of the samples before absorption (g)	% increase in weights		
		Day 10	Day 20	Day 30
SSSS	3.556	5.21	9.55	13.25
RRRR	4.466	8.82	14.62	18.96
RSRS	4.115	6.97	13.19	17.28
SRRS	3.861	6.98	11.62	14.70
RSSR	4.220	9.02	12.74	15.86

4.6. Scanning Electron Microscopy (SEM)

The laminate fractured surfaces are used here to carry out the analysis of reinforcement–matrix blended interfaces. Initially, the surfaces are well coated with gold sputtering before image capturing to know the morphology. Figure 6 depicts images of Silk/ramie hybrid epoxy composites with and without filler. A solid interface of the laminate allows more stress transfer through filaments than a weak interface, though a weak interface produces a sleek bond between the fibres and the matrix. [27] At the point when the power applied to the composite surpassed the interface bonding, a weak bonding basically empowered the filaments to pull out and break effectively, making the matrix split. [28] Case matrix break is presently the most prevalent failure mode, and it is in all likelihood associated with the brittle failure of SSSS composites delivered by huge fiber-to-fiber contact, as shown in Figure. 6A. [29]another issue is inadequate fibre scattering, which brings about unequal fibre breaking when the stacked fibres are broken since they are not symmetric and equal. The tensile properties of plain ramie composites are the most reduced (RRRR). However, due to its single fibre properties, the morphological construction of RRRR (Figure. 6B) shows a greater number of fibre pull-outs, suggesting a weak interfacial association among fibres and matrix, bringing about diminished tensile strength. The fibre breakage on the fractured surface shows the less fibre pulled out in the hybrid composite RSRS, as found in Figure 6C. Due to the extent of inclusion of ramie fibre, the tensile strength and modulus of the hybrid composites have expanded. Individual reinforced fibre characteristics have the biggest effect on hybrid composite properties. [30] Besides, various investigations have discovered that the hybrid composites' mechanical properties might be fundamentally influenced by various stacking arrangements. This review supports previous findings that substituting stacked fibres in composite materials increases tensile strength. [31] This is because of the way that the top and base layers of a composite are skin-covered. Since the skin factor decides the rigidity of the essential loadbearing part, picking a high-strength material as the skin layer can bring about higher tensile strength. As an outcome, axial stress might be productively conveyed by the ramie fibre's external layers prior to being passed to the core layer.

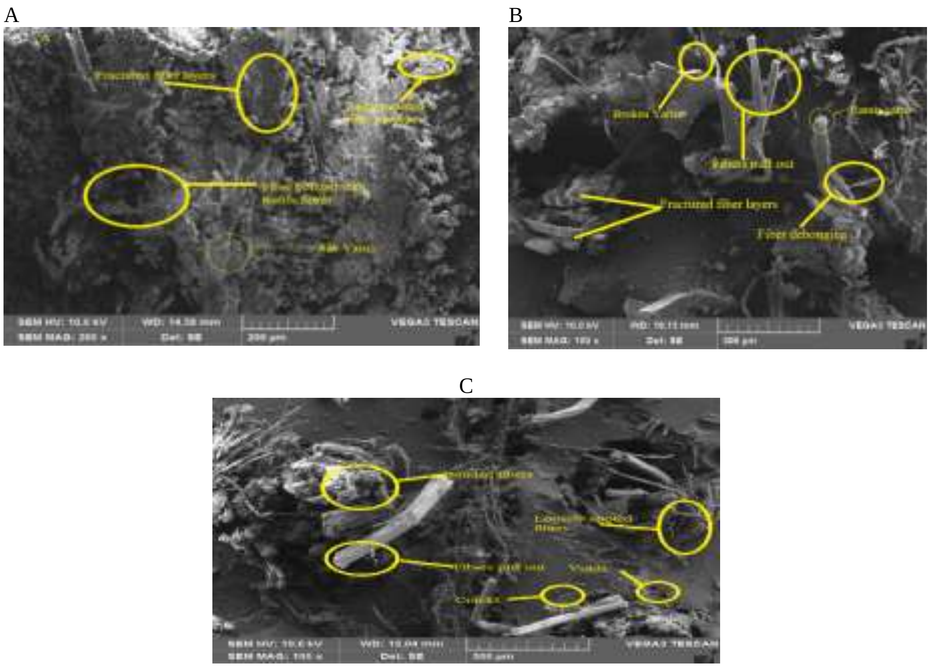

Figure 6. Scanning electron microscopy (SEM) micrographs of tensile fractured surfaces: **A.**SilkFibre, **B.** Ramie Fibre, and **C.** Ramie/Silk Fibre Hybrid Composite.

5. Conclusion

In these investigational studies, successful fabrication took place with and without filler (3% of OMMT) for silk/ramie fibre reinforced epoxy matrix hybrid composites in the field of natural hybrid composite materials. The composite laminate being used is inspected for its different physical, mechanical, and microstructural properties. The results yielded only fewer voids in the case of RSSR laminates due to the incorporation of nano clay material in hybrid composites. Better properties were achieved in tensile, flexural, impact, and hardness tests on nano clay OMMT filled silk/ramie reinforced hybrid composite laminates and can be validated for their use in some medium-load structural applications. The behaviour of filler-filled hybrid composites affected the resistance to moisture considerably due to the presence of OMMT. The solid bonding in OMMT filler-based composites showed less void content that revealed the better adhesion properties between fabrics and epoxy matrix in SEM analysis. Henceforth, all the tests conducted above indicate that the hybrid composite with 3% filler OMMT gives better results. To ascertain the usage in large commercial applications, a few more tests are required to evolve the mechanical properties of the composites.

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