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MECHANICAL BEHAVIOUR OF INFLORESCENCE/GLASS-FIBRE-REINFORCED HYBRID EPOXY COMPOSITES

MEHANSKO OBNAŠANJE HIBRIDNEGA EPOKSI KOMPOZITA OJAČANEGA S KOMBINACIJO NARAVNIH IN STEKLENIH VLAKEN

Athithanambi Anna Anban[1*], Manomani Krishnaswamy[2], Rajesh Appasamy[3]

[1]Nehru Institute of Engineering and Technology, Coimbatore, Tamil Nadu
[2]Government College of Engineering, Tirunelveli, Tamil Nadu
[3]Sri Krishna College of Engineering and Technology, Coimbatore, Tamil Nadu

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The biodegradability and environmental friendliness of natural fibres makes them suitable for implementation in a circular economy. As a result, several natural fibres and processing methods have evolved. The hydrophilic nature of ligno cellulose fibrils restricts the effective adhesion at the interface of fibre and matrix. The hybridization of natural fibres with synthetic fibres leads to promising characteristics of the resulting composite materials. This paper deals with the hybridization of conventional glass fibre and natural fibre extracted from coconut inflorescence. The effect of hybridization on the tensile and flexural strengths of surface-modified inflorescence fibre with glass fibres was investigated. The composites were fabricated using a hand-layup technique by varying the inflorescence fibre and glass-fibre reinforcement composition by (5, 10, 15 and 20) %. A notable improvement in the tensile and flexural strengths of 193.65 MPa and 240.69 MPa was observed for 85 % of glass and 15 % of benzoyl-chloride-modified inflorescence-fibre-reinforced hybrid composites. The elimination of amorphous constituents in the inflorescence fibres was checked by XRD and FTIR analyses. A surface-morphology analysis of unmodified and benzoyl-chloride-modified inflorescence fibres revealed pores and cavity formation on the fibril walls. These composites with superior mechanical properties can be an alternative to synthetic fibre composites and ensure the implementation of a circular economy and sustainable manufacturing.

Keywords: inflorescence fibres, hybridization, X-ray diffraction, Fourier-transform infrared, scanning electron microscope

V novejšem času je bilo razvitih in izdelanih več vrst procesnih metod za izdelavo kompozitov z ojačitvijo iz naravnih vlaken z namenom ekonomičnega in krožnega gospodarjenja za ohranjanje okolja in zmanjševanje njegove degradacije. Hidrofilna narava ligno-celuloznih vlaken omejuje učinkovito adhezijo amidov na mejah med vlakni in matrico. Hibridizacija naravnih vlaken s sintetičnimi vlakni vodi do obetavnih lastnosti izdelanih kompozitnih materialov. V tem članku avtorji opisujejo hibridizacijo konvencionalnih steklenih vlaken z naravnimi vlakni, pridobljenimi iz kokosovih cvetov. Analizirali so učinek hibridizacije na natezno in upogibno trdnost naravnih vlaken, ki so jih površinsko modificirali s steklenimi vlakni. Kompozite so izdelali s preprosto ročno tehniko ter pri tem spreminjali vsebnost naravnih in steklenih vlaken (5, 10, 15 in 20) %. Ugotovili so znatno izboljšanje natezne in upogibne trdnosti epoksi kompozita (193,65 MPa in 240,69 MPa), ki je vseboval kombinacijo 85 % steklenih in 15 % z benzoil-kloridom modificiranih naravnih vlaken iz kokosovih cvetov. Odstranitev amorfnih sestavin v naravnih vlaknih so potrdili z XRD in FTIR analizami. Analize površinske morfologije nemodificiranih in z benzoil-kloridom modificiranih naravnih vlaken so odkrile porozno in jamičasto tvorbo na stenah vlakenc. Opisani kompoziti z odličnimi mehanskimi lastnostmi so lahko alternativa kompozitom, ki vsebujejo samo sintetična vlakna pri zagotavljanju implementacije krožnega gospodarjenja in trajnostno naravnane proizvodnje.

Ključne besede: naravna vlakna, hibridizacija, rentgenska difrakcija, spektroskopija na osnovi fourierjeve transformacije infrardeče svetlobe (FTIR), vrstični elektronski mikroskop (SEM)

1 INTRODUCTION

Composite materials are replacing conventional materials owing to their higher specific strength, improved stiffness, decreased weight and better thermal properties. A composite material can be described as a material consisting of two or multiple distinct phases in which one is the reinforcement material (known as the load-carrying element) and other is a matrix material. For the past two decades composite materials have been emerging as dominant materials. The application of composite materials is increasing steadily in terms of volume and numbers, finding a prominent place in newer markets. Contemporary composite materials aggregate a momentous rate of the engineered materials starting from day-to-day products to highly complicated applications. Over the years, composite materials have been proven to be lower-weight materials, but the present goal is to make them economically viable. The possibility of manufacturing composite materials with low cost resulted in innovative fabrication techniques that are currently employed in composite industries. To overcome the cost factors in the development of composite materials there

*Corresponding author's e-mail:
aathithanambi@live.com (Athithanambi Anna Anban)

must be a unified effort on the design, processing, tooling and manufacturing for the composite material to be competitive enough with metals. The business scale of composite materials is found to be promising in all fields compared to aerospace applications. Polymer matrix materials and reinforcement, such as synthetic/natural fibres, have shown a significant result in terms of use and quantity (volume). The behaviour of composite materials can be modified based on the control of design parameters. Proper selection of the reinforcement materials results in developed composite materials' properties that can be customized enough to fit any application. The materials' selection relies on important factors such as product complexity, operating conditions of the product, and the fabrication skills of the person to achieve the optimal characteristics of the composites. For a better understanding on the behaviour of the developed composites, it can be used in association with traditional materials.

A recent literature survey showed that several works reported by researchers on different natural fibre-reinforced polymer composites for wide assortment of uses. The survey also covers how natural fibres can be extracted, different surface treatments made on the natural fibres, thereby the resulting natural-fibre-reinforced polymer composite exhibits better mechanical, tribological and other related properties in comparison to artificial-fibre-fortified polymer composites. The outcome of soluble base execution on the mechanical and other related characteristics of ensete stem fibre fortified with unsaturated polyester matrix showed improvement in the fibre properties.[1] They found that alkali-treated fibres contribute to an improvement in the composite properties in contrast to untreated fibre-reinforced composites. In addition, they found 5 % of NaOH treatment resulted in better mechanical characteristics of the ensete stem fibres. The possibility of using Acacia planifrons fibres[2] as a possible reinforcement material with polymer matrices was tested. The fibres were extracted by the process of retting. The extracted fibres were subjected to alkalization with different percentages of sodium hydroxide. It was found that the alkalization leads to an improvement of the crystallinity index, thermal stability and removal of amorphous substances present in the fibres. The optimal alkali treatment was found to be 5 %. The outcome of fibre content over flexural behaviour of malva fibre[3] reinforced with an epoxy matrix was investigated. The composites are manufactured via the compression-moulding technique by assorting the fibre's volume fraction. The flexural behaviour of the composites was found to be good for a 30 % volume fraction of continuous and well aligned malva fibres. The feasibility of using coffee hull[4] as a reinforcement member with high density polyethylene matrix revealed it could improve the impact strength of the composites. The mechanical, moisture characteristics of hydrophilic treated jute fibre fortified unsaturated polyester composites[5] revealed composites have a better role to play in different sectors. The silane coupling agent's influence on the tribological behaviour of corn-stalk-fibre-fortified polymer composites[6] revealed an improvement in the hydrophilic tendency of fibrils.

Hybridization in which two or more fibres either synthetic/natural fibre or both combined together to improve the mechanical properties of the resulting composites.[7,8] Hybridization of natural fibres with glass fibres offers numerous advantages, like reduced moisture-absorption characteristics[9] of the resulting composites with improved tribological characteristics.[10] From the existing literature it is concluded that the hybridization of glass fibre with coconut inflorescence fibres is not yet explored. This has been the motivation for this research work.

Based on the extensive literature survey it was found that several natural fibres have been explored and used as an alternate to synthetic fibre that has a prominent role to play in fibre-reinforced polymer composites for a wide variety of application components, ranging from automobiles to aerospace applications. The application of natural fibrils as reinforcement materials results in composites that are partially or fully degradable in nature. However, the natural fibre extracted from a coconut tree is limited by the application. Thus, there arises a situation in which natural fibre with superior properties is to be identified and extracted from a coconut tree.

Therefore, the present research work focusses on the hybridization of inflorescence fibre with glass fibre and epoxy resin as the matrix. Before the hybridization, the inflorescence fibre is to be surface treated with 5 % of NaOH, KOH, benzoyl chloride solution to remove the amorphous constituents present in the fibres. The untreated and surface-modified fibres are taken for the manufacturing of hybrid composites. The hybrid composites are to be fabricated using the hand layup technique and curing by compression moulding.

2 EXPERIMENTAL PART

The coconut tree is a prominent source of natural fibre, from which the fibrils are weeded out from the various elements of the tree. In this investigation, one such part of the coconut tree is identified, which is named inflorescence,[11] shown in **Figure 1**, and the extracted fibre, as shown in **Figure 2**, and efforts were made to extract fibre from the inflorescence There are different methods to extract natural fibres from which a process called retting a simple biodegradable treatment of subjecting the fibres by placing it in water for a specific time interval is adopted in this research work. As a result of retting, the outer layers of the coconut inflorescence are softened, and then the fibres can be extracted from coconut inflorescence by malleting.

The presence of an amorphous substance in the coconut inflorescence fibre hinders effective adhesion between the interface of the fibre and the matrix. There-

Figure 1: Coconut inflorescence

fore, the inflorescence fibre after extraction is exposed to a surface treatment with 5 % *w*/*v* of NaOH, KOH and benzoyl chloride for one hour. Then the fibrils are washed completely with distilled water to separate the chemical contents from the coconut inflorescence fibre. The effect of surface treatment on the fibre's properties improvement can be inferred from XRD and FTIR analyses.

Commercially available glass fibre mats, epoxy resin LY556 and hardener HY951 were used for the hybridization of the glass fibre with coconut inflorescence fibre. Sixteen different composite samples were prepared by varying the coconut inflorescence fibre content of (5, 10, 15 and 20) % for untreated, NaOH-treated, KOH-treated and benzoyl-chloride-treated coconut inflorescence fibres. The fabricated samples were sized as per the ASTM D638 and ASTM D790 standards for tensile and flexural tests. **Figure 3** shows the fabricated composite sample.

Figure 2: Extracted inflorescence fibre

Figure 3: Fabricated glass/inflorescence hybrid composite sample

3 RESULTS AND DISCUSSIONS

For the present research work, glass and inflorescence fibres were reinforced with epoxy matrix for the development of composites. An increasing volume fraction of inflorescence fibres was used to analyse the influence on the tensile and flexural strengths. The tensile and flexural strengths recorded for the composite samples are listed in **Table 1**.

Table 1: Tensile and flexural strengths of glass/inflorescence fibre composites

Sample No.	Fibre type	Glass fibre (%)	Coconut inflorescence fibre (%)	Tensile strength (MPa)	Flexural strength (MPa)
1	Untreated fibre	95	5	178.84	214.48
2		90	10	174.49	215.14
3		85	15	171.63	218.63
4		80	20	161.21	207.93
5	NaOH treated fibre	95	5	181.32	219.39
6		90	10	179.68	221.65
7		85	15	176.03	224.87
8		80	20	163.74	210.11
9	KOH treated fibre	95	5	183.65	225.97
10		90	10	182.96	228.32
11		85	15	179.68	233.06
12		80	20	169.82	212.86
13	Benzoyl treated fibre	95	5	189.35	231.67
14		90	10	191.64	233.00
15		85	15	193.65	240.69
16		80	20	176.21	219.38

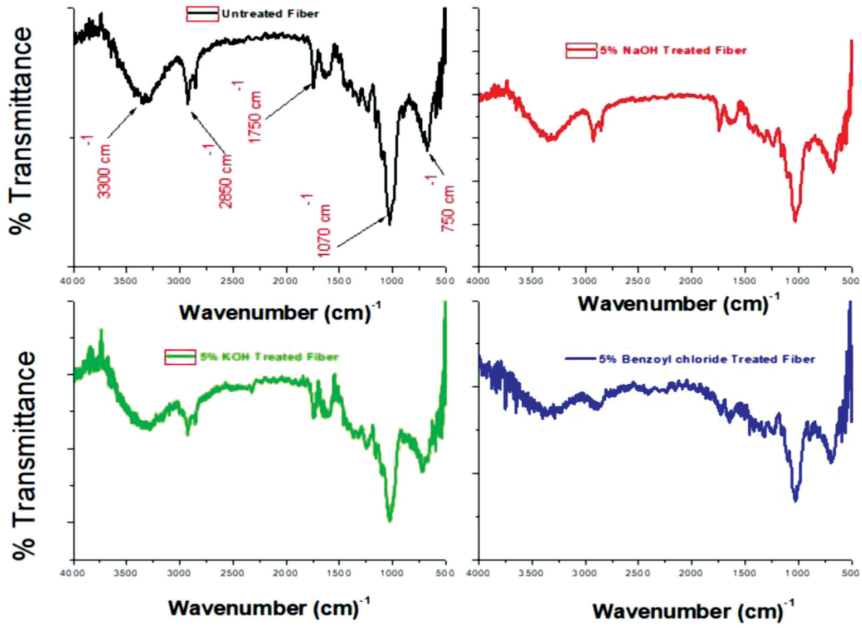

Figure 4: FTIR spectra of untreated and surface-treated inflorescence fibre

3.1 FTIR characterization

A KBr pellet technique was adopted to characterize the coconut inflorescence fibres subjected to different surface treatments. The wave numbers were varied from 500 cm^{-1} to 4000 cm^{-1}. The corresponding functional groups and their elimination can be seen in **Figure 4**. The wave number 3300 cm^{-1} corresponds to O–H stretching vibration of hemicellulose,[12] whereas the peak narrow down at 5 % benzoyl chloride which clarifies the elimination of hemicellulose, the peak at 2850 cm^{-1} corresponds to C–H symmetrical stretching,[13] the peak disappeared in 5 % benzoyl-chloride-treated fibre, which concludes the elimination of lignin and wax constituents. The amorphous peak at 1750 cm^{-1} relates to carboxyl group stretching vibration[14], the corresponding peak evaded at 5 % benzoyl-chloride-modified fibre, which concludes the elimination of hemicellulose and lignin. The peak at 1070 cm^{-1} belongs to the CO–O–CO stretching[15] whereas the disappearance of the peak at 5 % benzoyl chloride fibre confronts the elimination of wax and other oil-covering constituents. The peak at 750 cm^{-1} corresponds to the C=C bending of alkene,[16] which confirms the elimination of hemicellulose in coconut inflorescence fibres. Finally, from the FTIR characterization studies it is concluded that a 5 % benzoyl chloride modification has a major influence on the elimination of amorphous substances in the coconut inflorescence fibres.

3.2 XRD characterization

X-ray characterization studies were performed on untreated and alkali-treated inflorescence fibres and revealed an increase in the crystallinity size and the index of the fibres. **Figure 5** shows the XRD peaks of the untreated and surface-treated inflorescence fibres. The value of the 2θ angle of amorphous and crystallin peaks are mentioned below. The crystalline and amorphous peaks of the untreated fibres were observed at 29.63° and 21.85°, whereas for 5 % benzoyl-chloride-treated fibres the crystalline and amorphous peaks observed at 27.32° and 19.81°. The percentage of crystallin index for the untreated and 5 % benzoyl chloride were found to be 35.6 % and 37.92 %. The cementing materials of inflorescence fibres had a greater interaction with the 5 % benzoyl chloride solution, which means the 5 % benzoyl chloride had a prominent effect on the elimination of amorphous constituents. This is the reason behind the increase in crystallinity and the swelling of fibres is visible from SEM morphology studies.

The benzoylation of coconut inflorescence fibre results in fibre cells rearrangement ahead the direction of

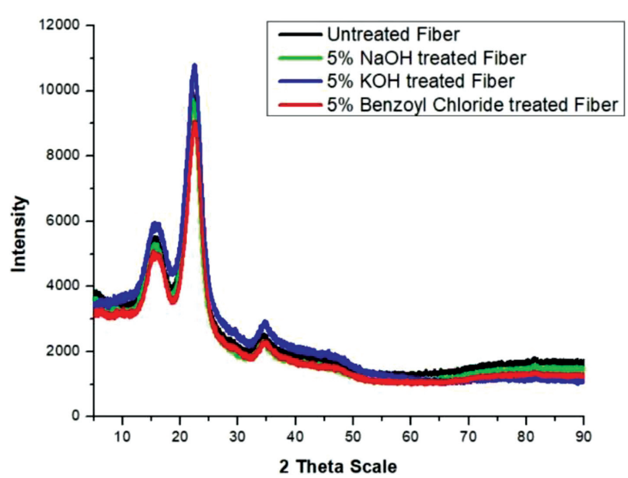

Figure 5: XRD peaks of untreated and surface-treated inflorescence fibres

tensile deformation, which enhances better dispense of the load in the fibre as a result the stress concentration of the fibre reduces. Therefore, the tensile strength of the coconut inflorescence fibre increases. In addition, the diameter of the fibre is found to reduce because of the axial splitting of the fibrils. The diameter of the fibre is determined using an image analysis technique. As a result of the benzoylation of the coconut inflorescence fibre, the hydroxyl groups present in the fibres are broken down, which further reacts with water molecules and becomes eroded from the fibre surface. The elimination of hemicellulose, lignin and other functional groups' existence in the fibrils were tested by the FTIR analysis. The XRD analysis tested that the crystallinity size increases as a result of the orientation change of tightly packed crystalline cellulose structure. Finally, the surface of the fibres becomes cleaner as a result of micro voids' elimination and also the resistance to moisture and stress transfer among the coconut inflorescence fibre becomes improvised.

3.3 Tensile strength

From the tensile-test experiment, it is evident that hybridization of inflorescence fibre with glass fibre attributed to an increase in the tensile strength of the composites. A gradual increase in the tensile strength was observed, as shown in **Figure 6**. The maximum tensile strength of 193.65 MPa was observed for 15 % benzoyl-chloride-treated inflorescence fibre hybridized with glass fibre. A sudden drop in the tensile strength was observed for a 20 % volume fraction of inflorescence/glass fibre composites. This can be the result of a higher weight percentage of inflorescence fibre in the composites. From the tensile test results it can be concluded that 15 % of benzoyl chloride treated inflorescence fibre contributed to the maximum tensile strength. This can be due to better interfacial adhesion between the glass/inflorescence fibre. The benzoyl-treated fibres enhance the better dispersion of load in the fibre as a result the stress concentration of the fibre reduces, thereby hybridization with glass fibre contributed to the maximum tensile strength.

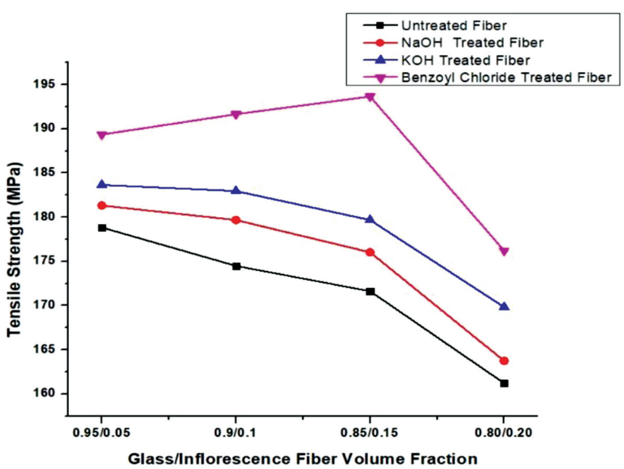

Figure 6: Tensile strength of hybrid glass/inflorescence-fibre-reinforced epoxy composites

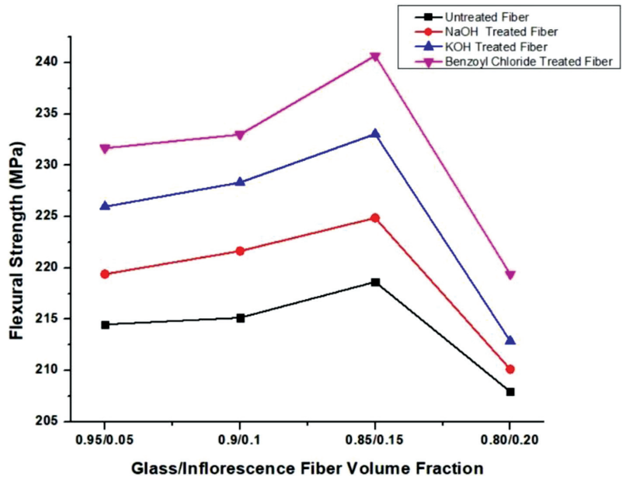

Figure 7: Flexural strength of hybrid glass/inflorescence-fibre-reinforced epoxy composites

3.4 Flexural strength

Figure shows the flexural strength of glass/inflorescence-fibre-reinforced hybrid epoxy composites. The flexural strength of the glass/inflorescence-fibre hybrid composites was found to be increasing with an increase in the volume fraction of inflorescence fibre, as shown in **Figure 7**. An increase in the flexural strength was observed for inflorescence fibre volume fractions of (5, 10 and 15) %. When the inflorescence fibre volume fraction is increased to 20 % a sudden drop in the flexural strength was observed, like with the tensile strength results. The maximum flexural strength of 240.69 MPa was observed for 15 % reinforced inflorescence fibre hybridized with 85 % of glass fibre. The phenomenon can be governed by benzoylation of inflorescence fibre, which makes the surface of the fibres cleaner as a result of the micro voids' elimination and also the resistance to

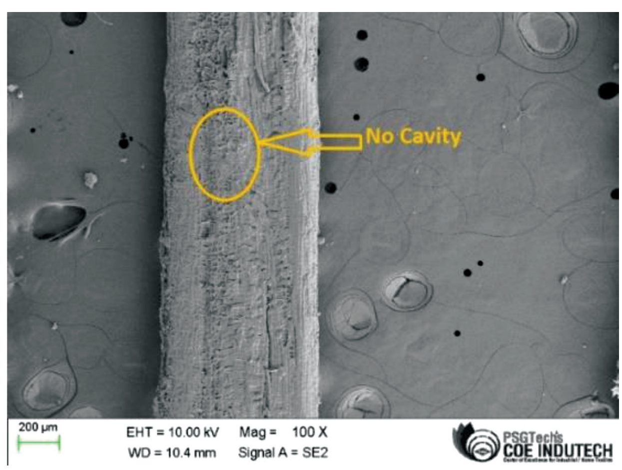

Figure 8: Untreated fibre

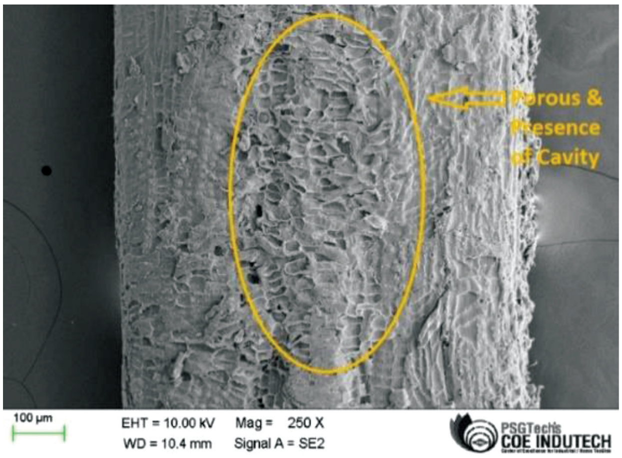

Figure 9: Benzoyl chloride modified inflorescence fibre

moisture and the stress transfer among the coconut inflorescence fibre is improved.

3.5 SEM characterization

The surface morphology of untreated and 5 % benzoyl-chloride-modified inflorescence fibres was examined using scanning electron microscope, which is shown in **Figures 8** and **9**. The surface of the untreated inflorescence fibres was nonporous and the absence of any cavity were recorded. The 5 % benzoyl-chloride-modified inflorescence fibres revealed the surfaces of the fibres were more porous and cavities were observed on the surface. Thereby during reinforcement with epoxy matrix contributed to maximum tensile strength for 15 % benzoyl chloride modified coconut inflorescence fibres.

4 CONCLUSIONS

Coconut inflorescence fibres and glass-fibre-reinforced hybrid composites were fabricated using a hand lay-up technique and cured by compression moulding. The inflorescence before reinforcement were surface treated with 5 % *w*/*v* of NaOH, KOH and benzoyl chloride. The extensive experimentation draws the following conclusions:

1) Among the surface treatments, benzoyl chloride has a prominent effect on the removal of amorphous constituents existing in the inflorescence fibres. Benzoyl chloride modification contributes to a better load distribution along the tensile deformation direction, as a result the tensile strength of the inflorescence fibre increases.

2) X-ray diffraction of benzoyl-chloride-modified inflorescence fibres revealed an increase in the crystallinity size and index of the fibres.

3) Fourier-transform infrared spectroscopy analysis of the benzoyl-chloride-modified inflorescence fibres revealed the elimination of amorphous constituents present in the fibres.

4) A maximum tensile strength of 193.65 MPa was observed for a 15 % reinforced benzoyl-chloride-modified inflorescence fibre hybridized with glass fibre.

5) A maximum flexural strength of 240.69 MPa was observed for a 15 % reinforced benzoyl-chloride-modified inflorescence fibre hybridized with glass fibre.

6) A scanning electron microscope analysis of unmodified and benzoyl-chloride-modified inflorescence fibre revealed surface-modified fibres were porous, and cavities were observed, which contributed to better interfacial bonding during reinforcement with glass and epoxy matrix.

From the results it is concluded that the hybridization of glass fibres with inflorescence fibres would result in composites that exhibit better properties than pure synthetic fibre-fortified composites. It is also concluded that inflorescence fibre could be one potential alternate material for the development of partially degradable hybrid composites.

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