IJIE

Journal homepage: http://penerbit.uthm.edu.my/ojs/index.php/ijie
ISSN : 2229-838X e-ISSN : 2600-7916

The International Journal of Integrated Engineering

Synthesis and Characterization of Coir and Luffa Cylindrica filled with $CaCO_3$ hybrid composites

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DOI: https://doi.org/10.30880/ijie.2019.11.01.029
Received 01 December 2018; Accepted 13 February 2019; Available online 30 April 2019

Abstract: Usage of natural fiber hybrid composites is increasing in the present days because of its eco-friendly and biodegradable nature. Coir fibers were extracted from the plant bark and Luffa Cylindrical were extracted from the vegetable fruit. Later both the fibers were treated with alkaline (5% NaoH) solution to remove the greasy nature over the surface. In the present study, composites were fabricated by using Coir and Luffa Cylindrical along with $CaCO_3$ natural fiber hybrid composites. Each composite is tested for tensile, flexural and impact tests as per the ASTM standards. For sample no. 10, maximum tensile strength of 57 MPa is obtained, whereas sample no.15 has got highest flexural strength of 635 MPa and for impact strength, sample no. 13 has got highest strength of 68 kJ/m^2. The composite specimens were characterized using Fourier transform infrared spectrophotometry (FTIR), scanning electron microscopy (SEM) and thermo-gravimetric analysis (TGA/DTA). SEM illustrated a good bonding between matrix and fibers. TGA illustrated the amount of residue leftover in the composite. FTIR illustrated which compounds are present in the composite.

1. Introduction

In the present era, usage of natural fiber hybrid composites has gained decent interest throughout the world. The main driving force for using of this natural fiber composites includes the light weight, eco friendly, biodegradability, strength to weight ratio, high specific modulus etc [1-3]. Moreover, the costs for manufacturing of these composites are also less when compared with the synthetic fiber composites [4]. These are the major reasons to consider this material in the field of structural, automobile, construction and engineering applications [5-9]. Natural fibers as reinforcements along with matrix for the preparation of composites; in addition to that, the inclusion of filler material into the matrix improves the properties of the composites. To attain better properties, the surface of the fibers is chemically treated in order to remove the hydrophilic nature of the fibers [10]. The chemical treatment (NaoH) influences not only the cellulosic components inside the plant fiber but also the non-cellulosic components (hemi-cellulose, lignin, and pectin). An enormous amount of work has been conducted in terms of mechanical properties, characterization and fiber modification [11].

Cao *et al.* [12] investigated and compared the mechanical properties of bagasse fiber-reinforced polyester composites with and without alkali treatment and concluded that there is an improvement of 13% in tensile strength, 14% in flexural strength, and 30% in impact resistance, respectively, due to surface modification by alkaline treatment. [13, 14] investigated on the Coir fibers and its alkaline treatment by observing the scanning electron microscopy (SEM) studies and stated that there is a progressive change in the surface of Coir fibers due to the removal of greasy nature from the surface of the Coir fiber. From the thermo-gravimetric analysis it was observed that there is a slight decrease in thermal stability due to alkali treatment. Not only alkali treatment improves the mechanical properties but also

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delingnification, the effect and influence of delignification and alkali treatment to modify the fiber surface of Coir fibers was examined by [15]. Tanobe *et al.* [16] characterized Luffa Cylindrica fibers by modifying the surface with metha-crylamide and NaOH. From the study it was observed that the Metha-crylamide treatment severely damaged the fibers with that poor results were obtained, whereas alkali treatment showed a good result due to the enhancement of surface area and thermal stability. The surface modification of kenaf fibers by chemical treatments was studied by Ederozey *et al.* [17]. Fibers are treated with different concentrations of NaOH and their morphological changes were observed by SEM. Mutje *et al.* [18] investigated the hemp fibers along with the effect of maleated PP as coupling agent and concluded that the addition of the coupling agent improved the tensile strength by 4% and flexural strength by 38%. The interface among the surfaces of hemp fibers and the coupling agent was also observed by FTIR spectroscopy. Topography of the Coir fibers and their crystallographic structure can be modified by mercerization. The physical and chemical behaviour of Coir fibers was characterized by FTIR spectra [19]. Boynard *et al.* [20] explored the flexural properties of luffa fiber-reinforced polyester composites. The authors observed mercerization treatment produced strong morphological changes on the surface of Luffa fibers but poor flexural properties than the NaOH treatment. Krishnudu *et al.* [21] investigated on natural fiber hybrid composites on mechanical properties by comparing it with treated and untreated composites. Optimizing mechanical properties were also carried out by varying fiber and filler contents [22-24]. Investigations on natural fiber hybrid composites were studied by many researchers by varying its fiber weight content and its effects on the mechanical properties and their characterization [25-28].

In this context, it has come to know that there are many new natural fibers were investigated by many researchers and academicians with an ease in their usage as reinforcement for polymer composites and also developing as an eco friendly materials. In the present study, the authors made an attempt to explore the possibility of using Coir and Luffa Cylindrica as new reinforcing materials in polymer green composites along with $CaCo_3$ filler. The effect of filler on the mechanical properties is studied along with its characterization. The suitability of the fibers based on its properties for different applications is suggested.

2. Materials and method

2.1 Materials

Epoxy (LY-5052), Hardener (HY-5052), NaOH and $CaCO_3$ are purchased from Sri Lakshmi Chemicals, Hyderabad, INDIA. Coir fibers are extracted from Coir bark and Luffa Cylindrica from vegetable fruit.

2.2 Alkaline Treatment

Fibers of both Coir and Luffa Cylindrical are taken in a tray which contains water mixed with 5% NaOH (50gms for 1 litre of water) and washed thoroughly with the solution for 2 hours. Later the fibers are again washed with distilled water in order to remove the hemi cellulose and greasy nature of the fibers. The fibers are cut with a length of 200 mm and weights of around 10 kgs were placed over it for a period of 24 hrs.

2.3 Composite Fabrication

Glass mould of size 200 x 200 x 3 mm^3 was prepared so that the composite slabs of required dimensions are prepared. Initially the glass mould is coated with wax to remove the composite slab easily from the mould. Matrix (mixture of Epoxy and hardener) of required proportion (10:1) are mixed thoroughly with different proportions of $CaCO_3$ (0, 2, 4 gms) and poured into the glass mould which contains reinforcements of varying loadings. It is allowed to cure the composite at room temperature for 24 hrs and after that these composites are placed in the oven for 80^oC for 30 min to remove the moisture content over the surfaces of the composite slabs.

2.4 Mechanical Properties Evaluation

For tensile test, the specimens were cut as per ASTM D 3039 with dimensions of 150 x 15 x 3 mm^3 from the composite slab. INSTRON 3369 were used for tensile test and the test was conducted with a cross head speed of 10 mm/min and at a temperature of 18^oC with 50% relative humidity. For flexural test, the specimens were cut as per the ASTM – 07 with dimensions of 150 x 12.7 x 3 mm^3 from the composite laminate. Again the same INSTRON machine was used to conduct three point bend test (flexural test) with a span length of 50 mm. The test was conducted with a cross head speed of 5 mm/min and at a temperature of 18^oC and 50% relative humidity. The specimens required for the impact test were cut from the composite laminate with dimensions of 62.5 x 12.7 x 3 mm^3 as per ASTM 256 – 06. For Impact testing, machine used in the present study was IZOD impact tester.

2.5 Characterization of the hybrid composite

Scanning Electron Microscopy (SEM), Thermo-Gravimetric Analysis (TGA) and Fourier Transform Infrared Spectroscopy (FTIR) analysis are carried out on the fabricated samples. Optical microscopic interpretations of Coir and Luffa Cylindrica fibers were carried out using a polarized optical microscope. The interfacial bonding between the fibers and matrix was observed using MIRA3 LMU scanning electron microscope at an accelerating voltage of 10 kV. The samples were sputtered with gold coated to testimony the micrographs. The thermal stability Coir and Luffa Cylindrica fibers filled with $CaCO_3$ was established using a thermo-gravimetric analyzer NETZSCH model of STA-2500. An amount of 2.59 mg sample is taken for each measurement in an aluminium oxide crucible up-to a temperature limit of 900ºC at 10 K/min. The FTIR spectra of all the samples were run on Perkin Elmer RXI FTIR spectrophotometer. The samples were powdered with KBr and the pellets were used for recording the spectra in transmission mode. All the spectra were recorded in the 4000–500 cm^{-1} region with 32 scans in each case at a resolution of 4 cm^{-1}.

3. Results and Discussion

3.1 Mechanical Properties

The Coir and Luffa Cylindrica fibers contain cellulose (40% wt.) and lignin (45% wt.), in order to remove the moisture content and hemi cellulosic nature of these fibers. These are treated with 5% Sodium Hydroxide solution, such that the surfaces of the fibers are modified and a good bonding has taken place as seen from the SEM microscopes. The prepared samples along with the composition are shown in Table.1. A total of 18 samples were prepared based on the literature study and each sample with three repetitions (three samples for each test) is tested for tensile, flexural and impact tests. The average values of all the three tests for the total 18 samples are shown in Fig.1. The strength of the composite samples mainly depends on the bonding nature between the fibers and matrix. The filler content in the composite samples improves the bonding between the fibers and matrix. So the filler content is maintained up to certain limit in order to stabilize the mechanical properties. The variations of the filler content in the composite sample varied the strengths.

Table 1 Composition of the fabricated composite samples

Sample No.	Coir Wt. in gms	Luffa Cylindrica Wt. in gms	Filler $CaCO_3$ Wt. in gms
Sample 1	10	5.7	0
Sample 2	10	5.7	2
Sample 3	10	5.7	4
Sample 4	20	5.7	0
Sample 5	20	5.7	2
Sample 6	20	5.7	4
Sample 7	10	6.2	0
Sample 8	10	6.2	2
Sample 9	10	6.2	4
Sample 10	20	6.2	0
Sample 11	20	6.2	2
Sample 12	20	6.2	4
Sample 13	30	5.7	0
Sample 14	30	5.7	2
Sample 15	30	5.7	4
Sample 16	30	6.2	0
Sample 17	30	6.2	2
Sample 18	30	6.2	4

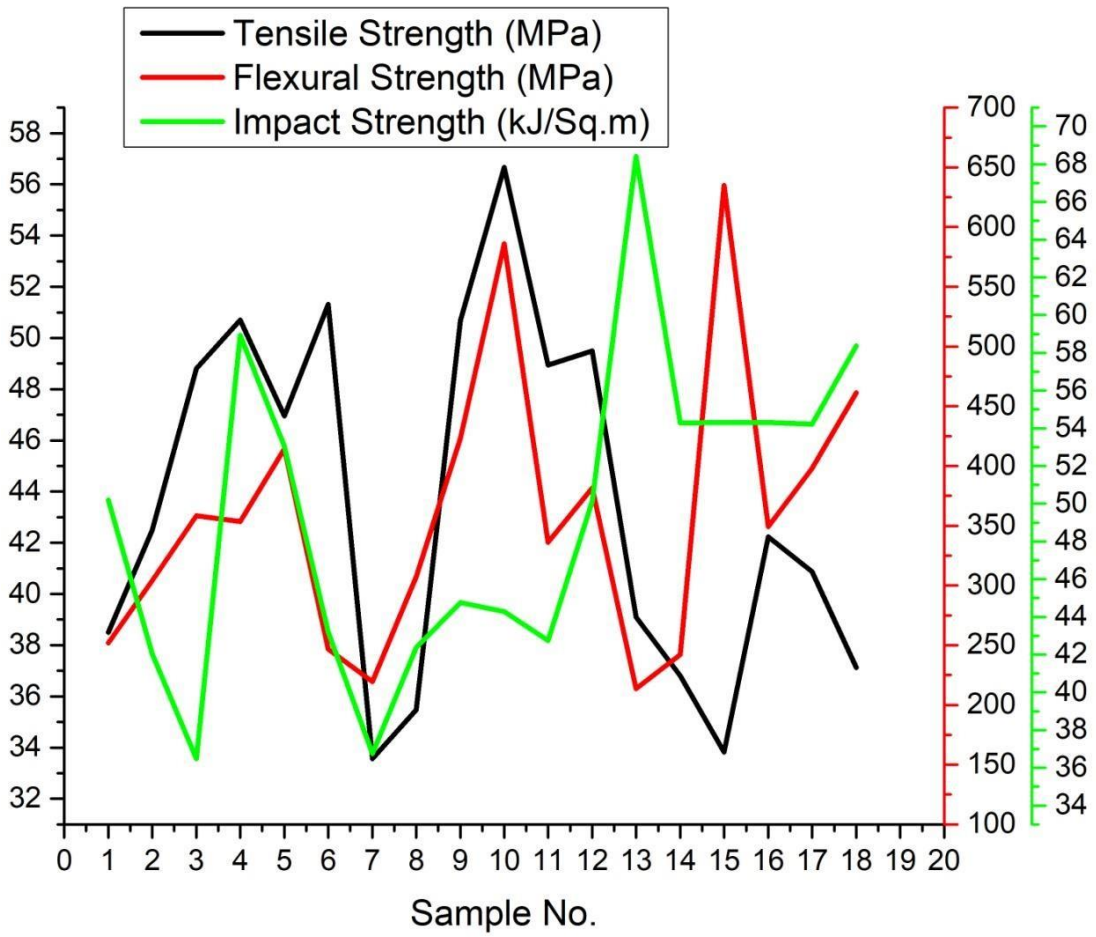

Fig. 1 Mechanical Properties of test samples

From Figure 1 it was observed that the tensile stress is higher for the sample No. 10 with a tensile stress of 57 MPa, which is having a composition of high Luffa Cylindrica weight and average Coir fiber weight. For flexural stress, sample No. 15 is having higher value of 635 MPa with a composition of higher Coir and filler content with low Luffa Cylindrica content. For impact strength, sample No. 13 is having higher value of 68 kJ/m^2 with a composition of higher Coir with low Luffa Cylindrica and filler content. Similar results were obtained for prosopis juliflora reinforced compoites, that is higher fiber content in the composite sample improved the mechanical properties [29].

3.2 Scanning Electron Microscopy (SEM)

The morphology of the fractured samples was studied using Scanning Electron Microscopy (SEM). The fractured surfaces were coated with gold sputtering by electro-deposition to impart electrical conduction before recording SEM. SEM micrographs of fractured surfaces at different magnifications were taken using MIRA3 LMU equipment at CeNS Bangalore. Scanning Electron Micrographs of the tested specimen of fractured Coir- Luffa Cylindrica filled hybrid composites, which resulted in better tensile and flexural strength, are shown in Figure 2 & 3. The SEM images of the tensile and flexural fracture revealed the uniform distribution of fibres as well as $CaCO_3$ fillers are well dispersed in the epoxy matrix. The attainment of higher mechanical properties is because of the good bonding between matrix and reinforcement. Fillers filled the voids present between the matrix and reinforcement as is observed from the figures.

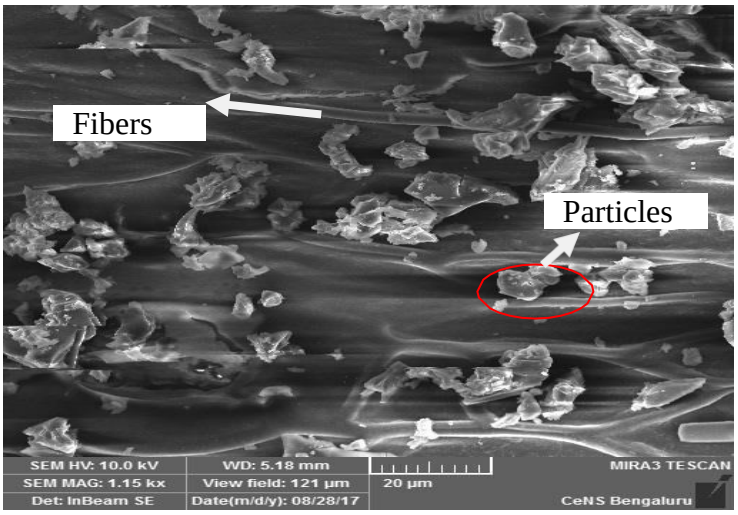

Fig. 2 SEM image of tensile test sample at 20 µm

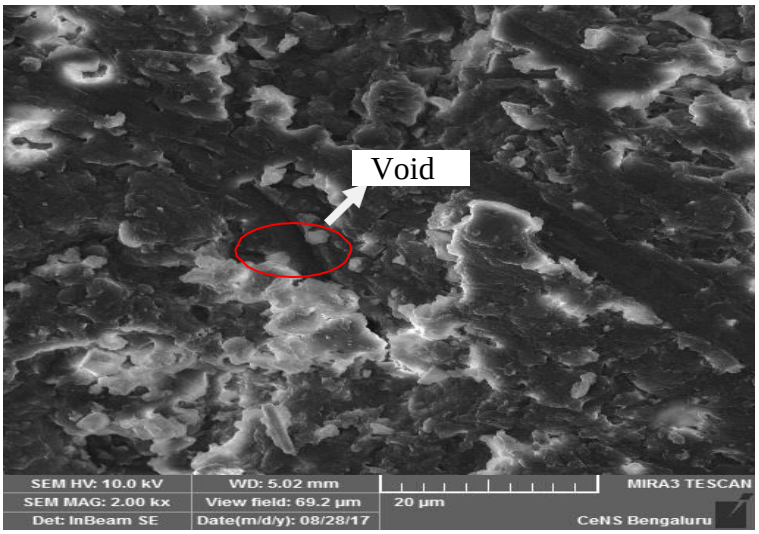

Fig. 3 SEM image of flexural test sample at 20 µm

3.3 TGA Analysis

TGA test was performed on NETZSCH model of STA-2500 for 2.691 milligrams of powder in an aluminium oxide (Al_2O_3) crucible with temperature raised up to 900ºC at 10ºC/min. TGA is used to evaluate the thermal stability and to understand the degradation characteristics of the considered specimens. TGA was used in the analysis of ceramics and thermally stable polymers. Ceramics usually melt before they decompose as they are thermally stable over a large temperature range, thus, TGA is mainly used to study the thermal stability of polymers. Most polymers melt or degrade earlier than 200°C. However, there is a class of thermally stable polymers that are able to bear up temperatures of at least 300°C in air and 500°C in inert gases without structural changes or strength loss, which can be analyzed by TGA [30, 32]. All the measurements were conducted under inert gas (nitrogen), keeping a constant heating rate of 10ºC/min and using an aluminium crucible with a pin hole. In TGA analysis as a change in thermal stability was examined in terms of percentage weight loss as function of temperature. The samples were placed in aluminium oxide crucible, while the tests are carried out in nitrogen atmosphere.

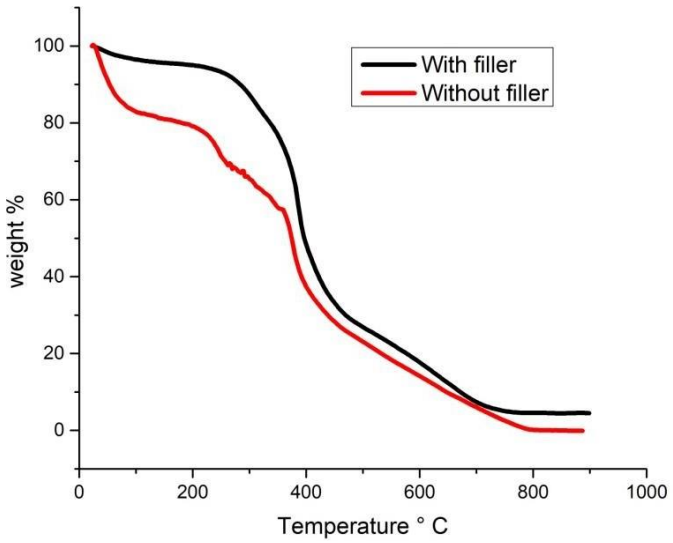

Fig. 4 TGA Graph of hybrid composite

Figure 4 indicates tensile and flexural strength of the weight loss of particle filled with and without hybrid composites at temperature between 0ºC to 900ºC. Degradation occurred in 4 stages, where evaporation of moisture content, water soluble hydroxyl and carboxyl groups present in the composite is legible around 270°C, degradation was Initiated at 270°C and completed around 390°C which signifies the glycosidic bonds and thermal depolymerisation of non fibre groups such as hemicelluloses and pectin, it can be observed that Degradation of fibre & filler starts around 400°C and ending at 800°C which signifies the weight loss due to decomposition of α-cellulose constitutions, a difference wt % loss for with and without filler can be specified here.

3.4 FTIR Studies

The particles filled hybrid fiber composites exhibited spectral patterns of sharp absorption bands which confirmed the heterogeneous structure of the fibres. The particle filled with and without particles filled FTIR spectra of samples at wave numbers between 500 and 4000 cm^{-1} are shown in the Figure 5. The peak bands between 3700 and 3000 cm^{-1} confirm the presence of hydrogen-bonded OH stretching in Coir - Luffa Cylindrica hybrid composites. The sharp band at 2923.73 cm^{-1} and 2922.15 cm^{-1} indicated the subsistence of C–H stretching which often notorious with methyl and ethyl-methyl groups with and without hybrid composites. The pointed absorption band at 1734.81–1609.37 cm^{-1} and 1608.1-1505.38 substantiate the amount of water molecules absorbed by non crystalline cellulose with and without particle filled hybrid composite. The peak band at 1456.58 cm^{-1} and 1459.09 reveals the aromatic structure of lignin exhibiting lingo cellulosic characteristic in particles filled with and without composites. The sharp band at 1038.61 cm^{-1} and 1036.95 cm^{-1} tells the existence of C–O–C symmetric glycosidic stretch, which arises from polysaccharide components. The peak obtained from FTIR shown in Table 2 accomplishes that particles filled and without particle filled composites has a cellulosic structure with fair amount of cellulose and lignin content and fit in to the natural fibres which could be used as reinforcement material in composites.

Table 2 FTIR spectra regions

S. No	*Wave number(cm−1)*	*FTIR peak region*
1	3700-3000	Hydroxyl group and OH stretching
2	2923.75	C-H stretching
3	2858.28	CH aliphatic stretching
4	1734-1609.37	C-O stretching vibrations
5	1456.58	Lignin components
6	1038.61	c-o-c glycosidic stretch

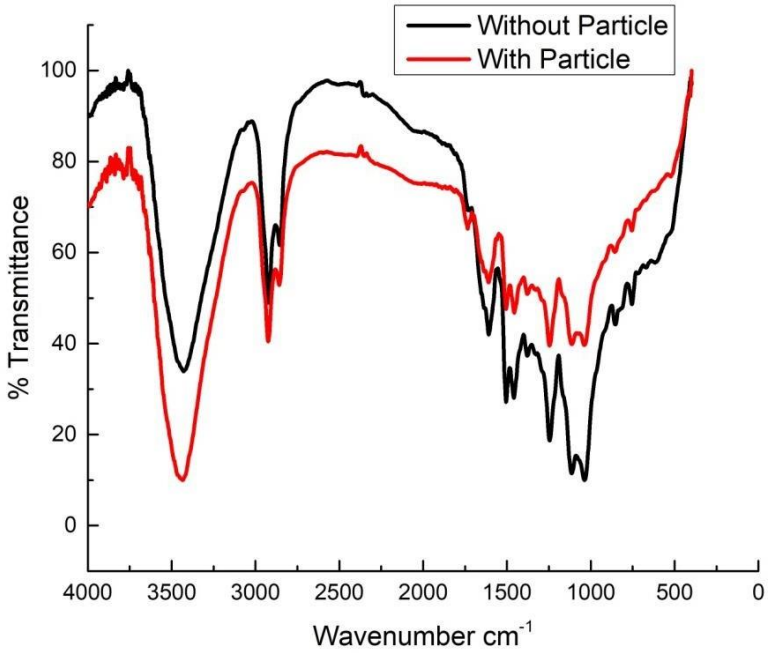

Fig. 5 Coir Luffa Cylindrica FTIR spectra

4. Conclusions

Coir and Luffa Cylindrica filled with $CaCO_3$ hybrid composites were fabricated by treating the fibers with NaOH. The composite samples were tested for mechanical properties. SEM, TGA and FTIR studies were also carried out on the fabricated samples. The following conclusions were drawn from the present work.

1. From the mechanical tests it was observed that maximum tensile stress as 57 MPa, flexural stress of 635 MPa and impact strength of 68kJ/m^2.
2. Thermal stability of both the filled and unfilled composite sample have a left over residue of 7.36% and 8.4% respectively
3. SEM graphs shows that there are no fiber pullouts and voids which is a sign of good bonding between matrix and fibers because of alkali treatment.
4. FTIR studies in the composite samples shows the presence of –OH, -COO, C-O-C and C=O chemical functional groups in both filled and unfilled composite samples.
5. The composite samples can be used in applications like door panels, window panels, partition boards, and fall ceiling.

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