

MECHANICAL, THERMAL, AND MORPHOLOGICAL BEHAVIOUR STUDIES ON COCONUT SHELL AND PALM KERNEL FILLER BIOCOMPOSITE

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ABSTRACT

In the present work, the composite materials were prepared from coconut shell powder, palm kernel powder, and epoxy resin. The addition of coconut shell powder was considered when preparing the composite samples, and mechanical properties such as tensile strength, hardness, impact, bending strength, physical behavior water absorption, as well as morphological tests, were conducted using Fourier Transform Infrared Spectroscopy, Scanning Electron Microscope, and Thermogravimetric Analysis for both the prepared composite material boards and chipboard. The minimal variation of tensile stress and percentage of elongation between the 50 % coconut shell powder composite material and the wooden chipboard material is 4,44 MPa and 1,00 %, respectively, according to the findings of experimental tests. The lowest compressive stress and hardness variations between coconut shell powder composite material and wooden chipboard are found to be 0,14 MPa and 3,2 MPa, respectively. It is determined that the composite materials made from waste shell powders and epoxy resin are suitable for applications such as panel boards, automotive interior dashboards, roof sheets, and doors.

Keywords: Biocomposite material, chipboard, mechanical properties, waste shell fillers, water absorption.

INTRODUCTION

In recent years there are environmental changes drastically increased by the usage of non-degradable polymer materials and it causes environmental degradation (Laycock *et al.* 2017). Deforestation will affect the eco cycle due to the increase of wood-based products in engineering applications (Jiang *et al.* 2018). The weakness of one material is reduced by adding another one (Mariano *et al.* 2014). Different natural plant

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fillers used for composite production, such as coconut, palm kernel straw, and bamboo, can be found in the literature and industrial applications (Kai *et al.* 2016, Saba *et al.* 2014). Improve the strength of the polymer by incorporate the fillers with epoxy resin (Naghmouchi *et al.* 2015, Muraliraja *et al.* 2021). The natural materials have low mechanical properties, water resistance properties, and thermal resistance while comparing to wood filler-epoxy composite materials used in automotive, structural, etc. applications (Nagarajan *et al.* 2020). Availability and renewability of the waste organic filler material reduce the production cost (Arevalo-Gallegos *et al.* 2017). Wastages of crushed shell fillers are utilized for lightweight applications such as panel boards, automotive dashboards, etc. (Kaur *et al.* 2020, Dinesh *et al.* 2018). Lignocellulosic materials from the waste shell are now used in many applications for producing valuable products in engineering fields like chipboard and modified density fiberboard (Collins *et al.* 2019). The incorporation of fillers with a polymeric matrix is interested nowadays in biochemical and energy industries due to its availability and compatibility (Khalil *et al.* 2017, Wen *et al.* 2021). The economic analysis shows that the demand for waste shell fillers raises the agriculture market (H Silva *et al.* 2019, Rajinipriya *et al.* 2018). It is found that waste shell fillers price is Rupees 20 per kilogram it is justified that the low-cost composite products are manufactured (Zhang *et al.* 2016, Lebreton and Andraday 2019). Waste shell fillers are incorporated with thermosetting or polymer instead of wood fillers are found in various literature studies (Zaaba and Ismail 2019, Koçhan 2019, Santulli *et al.* 2020). The epoxy resin using agricultural waste fillers is effective in most trials. (Salasinska *et al.* 2018, Patil *et al.* 2019, Chen *et al.* 2020). The flexural strength and tensile strength of Date palm wood particles-based composite were decreased by increasing the filler content while the flexural modulus was increased (Kumar *et al.* 2018). Major advantages of using waste wood composites are easily disposed of at the end of their life cycle (Gu *et al.* 2017, Monteiro *et al.* 2017). Natural fibers have gained popularity as a suitable reinforcement in polymeric matrices in recent years due to their biodegradability, low cost, environmental friendliness, and renewable nature (Yildirim and Acay 2021, Kumar *et al.* 2021). This research aims to develop and characterize epoxy resin composites that are highly filled with readily available and inexpensive coconut shell and palm kernel shell ground waste material in the form of fillers, as well as investigate their mechanical behavior and percentage of filler weight, and compare them to chipboard (f) shown in Figure 1.



(f)
Figure 1: Chipboard.

Morphological properties of the composite materials are done through Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric analysis (TGA) test, and Scanning Electron Microscopic (SEM) test.

MATERIALS AND METHODS

Materials and fabrication

The shells of fully matured coconuts and palm kernel were collected from Nariyal Shell Powder manufacturers in Coimbatore, India. The following procedures are done before the manufacturing of shell powders. It is first cleaned and crushed into smaller grains. These smaller grains were then subjected to repeated grinding in a pulverizing machine, after passing through cyclones and vibratory sieves fitted with phosphor-bronze mesh with average particle size 25 nm to 30 nm and density 1,0462 kg/m³.

Eswar chemicals Limited, Trichy provides a low temperature curing Epoxy Water Clear Casting Resin that belongs to the 'epoxide' family of chemicals. In this test, five sample proportions are taken and blended in a 3:1 by weight ratio, as shown in Table 1.

The epoxy filled with waste shell powder is then mechanically stirred and gradually poured into the wax-coated rectangular tray of dimension 225 mm × 40mm × 10 mm. The whole mixture is cured for 24 - 48

hours at room temperature and prepared samples (a, b, c, d, e) are shown in Figure 2.

Table 1: Composite Sample preparation.

Samples	CSP %	PKP %	Epoxy Resin Ratio in weight
a	0	100	3:1
b	25	75	3:1
c	50	50	3:1
d	75	25	3:1
e	100	0	3:1

CSP: Coconut shell powder PKP: Palm kernel powder.

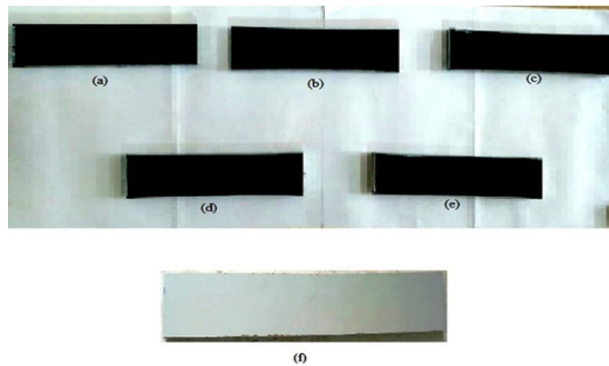


Figure 2: CSP Composite samples.

Testing of samples

The various mechanical properties such as tensile strength, hardness, impact strength, bending strength, and water absorption tests are conducted for prepared materials according to the American Society for Testing and Materials (ASTM 2021) international standard data.

Tensile test

The tensile test was carried out using the universal testing machine with a gripping capacity of 100 kN. The testing was performed at an ambient temperature of 24 °C and relative humidity of 53 %. The specimen was mounted by its ends into the holding grips of the universal testing machine. The machine is designed to elongate the specimen at a uniform rate and using an extensometer the instantaneous applied load and the resulting elongation of break are measured continuously until the break and results are shown in Table 2.

Table 2: Tensile stress and percentage of elongation of the materials.

Materials	Tensile stress (MPa)	Elongation (%)
Chipboard	23,62	1,78
0 % CSP	15,94	1,36
25 % CSP	23,1	1,59
50 % CSP	23,56	1,67
75 % CSP	15,82	1,46
100 % CSP	17,49	1,52

Bend Test

The three-point bend tests of composite specimens are carried out using a universal testing machine and results are shown in Table 3.

Table 3: The compressive stress of the materials.

Materials	Compressive stress (MPa)
Chipboard	16,58
0 % CSP	13,68
25 % CSP	15,97
50 % CSP	16,72
75 % CSP	14,68
100 % CSP	15,20

Impact test

Izod impact test

Impact Tester used for both Izod and Charpy test is common. The apparatus contains a pendulum with a known dead-weight at the end of its arm swinging down and hitting the specimen while it is held tightly in a vertical position. The impact strength is found by the loss of energy of the pendulum. The prepared specimen has dimensions of 64 mm x 13 mm x 10 mm and 2 mm V-notch at an angle of 45° facing towards the pendulum with a striking distance of 32 mm from the upper tip of the specimen.

Charpy impact test

The apparatus contains a pendulum with a known dead-weight at the end of its arm swinging down and hitting the specimen while it is held tightly in a horizontal position. The impact strength is found by the loss of energy of the pendulum. The prepared specimen has dimensions of 55 mm x 10 mm x 10 mm and 2 mm V-notch at an angle of 45° facing away from the pendulum with a striking point at the middle of the specimen. The material impact strength results from the Izod and Charpy tests are shown in Table 4.

Table 4: Impact on materials.

Materials	Impact strength (MPa)	
	Izod	Charpy
Chipboard	4,2	3,2
0 % CSP	3,8	2,8
25 % CSP	4,0	2,8
50 % CSP	4,2	3,4
75 % CSP	3,8	3,2
100 % CSP	4,0	3,2

Microhardness measurement

The hardness of the prepared materials was studied by Vickers's microhardness test. The hardness of the material is defined as the resistance offered by it to the motion of dislocations, deformations, or damage under applied stress. To measure the hardness, prepared materials with a thickness of 10 mm were used and loads of different magnitudes were applied.

In these equations, which have the dimensions of stress, load F and diagonal length D are measured respectively. The variation of hardness with different loads for materials is shown in Table 5. Vickers hardness number (HV) was calculated for the materials by using the Equation 1.

$$HV = 1,85 \left(\frac{F}{D^2} \right) \quad (1)$$

HV- Vickers number in HV

F - Applied load in N

D² - Area of the indentation in m²

Table 5: Vickers Microhardness of the materials.

Materials	Vickers Microhardness (HV) at different loads in grams				
	25	50	100	200	500
Chipboard	15,12	23,46	39,31	39,30	71,40
0 % CSP	11,05	12,25	20,70	35,30	56,15
25 % CSP	13,50	21,65	32,60	40,35	75,55
50 % CSP	18,55	25,85	39,30	51,30	75,60
75 % CSP	13,20	16,85	18,00	24,10	51,15
100 % CSP	16,15	19,25	35,65	50,45	71,60

Water absorption test

A water absorption test was carried out for the five specimen materials of each 4 g weight is weighed with a precision of 0,001 g weighing machine and the sample materials were dried in an oven for 24 hours at 80 °C. The sample materials are immersed in both distilled water and sodium chlorinated water for 24 hours for 5 days at room temperature 24 °C.

At the end of the immersed periods, the specimens were removed from both the distilled water and salt water wet specimens weight values were determined. The water absorption was calculated from Equation 2 and the results are shown in Table 6.

$$\text{Water absorption (\%)} = \frac{m_0 - m_1}{m_1} \times 100 \quad (2)$$

Where

m₀- Initial mass of specimen material (g)

m₁- Mass of specimen material after drying (g)

Table 6: Water absorption of tested materials.

Materials	Water absorption (%)									
	Distilled water					Saltwater				
	Day 1	Day 2	Day 3	Day 4	Day 5	Day 1	Day 2	Day 3	Day 4	Day 5
Chipboard	8,54	19,9	35,9	48,12	63,42	5,81	14,68	28,3	36,34	43,85
0 % CSP	0	0	0	0	0	0	0	0	0	0
25 % CSP	0	0	0	0	0	0	0	0	0	0
50 % CSP	0	0	0	0	0	0	0	0	0	0
75 % CSP	0	0	0	0	0	0	0	0	0	0
100 % CSP	0	0	0	0	0	0	0	0	0	0

Time duration for drilling

The materials having dimensions of 210 mm x 40 mm x 10 mm are placed separately in a vice and clamped firmly, the HSS drill of 8 mm diameter is placed perpendicular to the material where the hole to be made and the time of a drill to make a through-hole is noted with a stopwatch is shown in Table 7.

Table 7: Duration period for drilling.

Materials	Time (s)
Chipboard	10,84
0 % CSP	11,18
25 % CSP	11,17
50 % CSP	11,24
75 % CSP	11,18
100 % CSP	11,06

Morphological studies

Scanning electron microscope (SEM)

The morphological studies of the samples were carried out by using a scanning electron microscope (SEM). SEM micrographs of the surfaces of the Materials were taken by using a ZEISS Evo® LS10 scanning electron microscope. The samples were first sputter-coated with a fine layer of gold under vacuum for 60 s. An accelerating voltage of 20 kV was used to get the SEM images.

Fourier transform infrared spectroscopy (FTIR) analysis

For finding the different functional groups in the materials were studied by FTIR in the ACIC Instrumentation center in St. Joseph's College, Tiruchirappalli. The FTIR spectra were recorded employing a Perkin Elmer make and Model Spectrum - II which was equipped with a germanium ATR crystal probe and allows direct recording of the spectra without sample preparation. The recorded spectra were averages of 32 scans over the often range 4000 cm⁻¹ to 400 cm⁻¹ at 4 cm⁻¹ resolutions.

Thermogravimetric analysis (TGA)

Thermal stability of the materials was determined by Thermogravimetric analysis realized in the temperature range of 30 °C to 900 °C at a heating rate of 10 J/s under nitrogen atmosphere, using a TG 209 F1 Libra apparatus (Erich NETZSCH GmbH & Co. Holding KG, Selb, Germany). 5 mg \pm 0,1 mg samples were placed on ceramic pans.

RESULTS AND DISCUSSION

The various mechanical properties such as tensile strength, hardness, impact strength, compressive stress, and physical behavior like percentage of water absorption and also the characterization study have been conducted for both the prepared composite material and chipboard with the help of FTIR, SEM, and TGA. The physical behavior study has been carried out in the CPS composite materials and wooden chipboard by soaked the samples in normal water and sodium chlorinated water for 5 days.

The increase of CSP filler from 0 % to 50 % the tensile stress and percentage of elongation of material has been increased and its maximum value of 23,56 MPa and 1,67 % for 50 % CSP composite material. And further increasing of CSP filler from 75 % to 100 % both the tensile and percentage of elongation is decreased due to the poor bonding of CSP and PKP filler materials (Shahzad 2015). The tensile stress and elongation of a percentage of chipboard material are 23,62 MPa and 1,78 % respectively. The results of both CSP composite materials and chipboard material are compared and shown in Figure 3. It shows that the deviation of tensile stress and percentage of elongation for 50 % CSP composite material and chipboard is 0,06 MPa and 0,11 % respectively. These variations are quite small as compared with the rest of the CSP composite materials and chipboard.

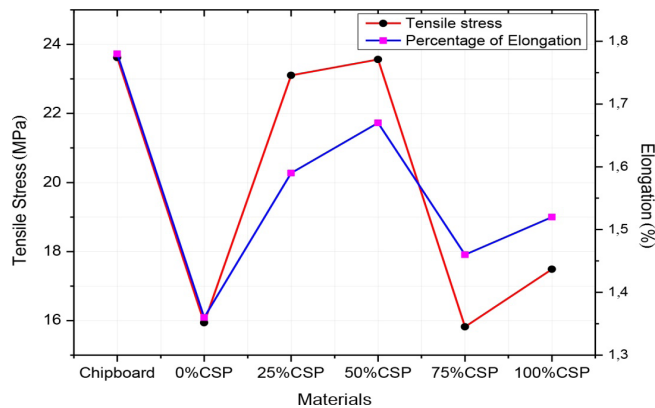


Figure 3: Tensile stress and Percentage of elongation of the materials.

With the increase of CSP filler from 0 % to 50 % it is observed that the compressive stress is increased and its maximum value of 16,72 MPa for 50 % CSP composite material. And further increasing the percentage from 75 % to 100 % it is identified that the crack formation has been occurred at 15 MPa due to the low bonding strength with increased PKP filler materials.

The percentage variation of compression stress value between 50 % CSP Material and the chipboard is 0,14 % and the results are shown in Figure 4. The compression stress variation is low down as compared with the rest of the CSP composite materials and chipboard.

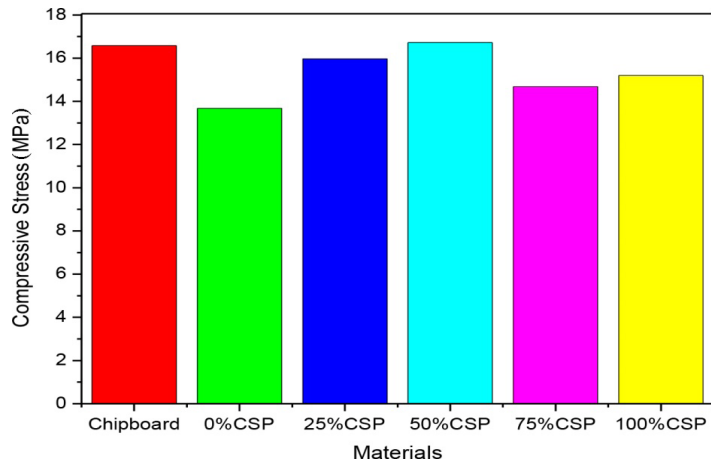


Figure 4: The compressive stress of the materials.

Figure 5 shows the impact values of CSP composite materials and chipboard. The impact tests of the CSP composite materials and wooden chipboard material are carried out in the Izod and Charpy tests. It is observed that the 50 % CSP composite material has taken up the maximum impact strength as compared with the rest of the percentage of CSP composite materials. It is identified that in the Izod impact test both 50 % CSP composite material and chipboard are take-up the same impact load. In the Charpy impact test, the minimum impact is 0,2 Mpa between 50 % CSP composite material and chipboard.

The different load values of 25 g, 50 g, 100 g, 200 g, and 500 g are applied to estimate the hardness in CSP composite material and chipboard. While applying the load of 25 g, 50 g, 100 g, 200 g, and 500 g the hardness values are increased in the 0 % to 50 % CSP composite materials and it's further decreasing in the 75 % and 100 % CSP composite materials.

It is observed that the 50 % CSP composite material having a high average hardness value than the chipboard shown in Figure 6.

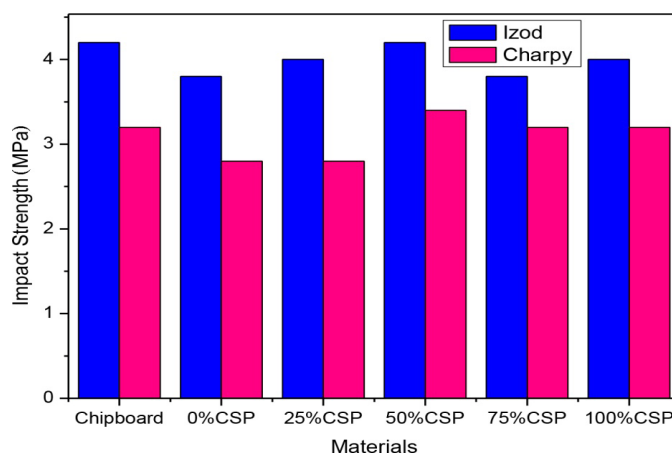


Figure 5: Impact of the materials.

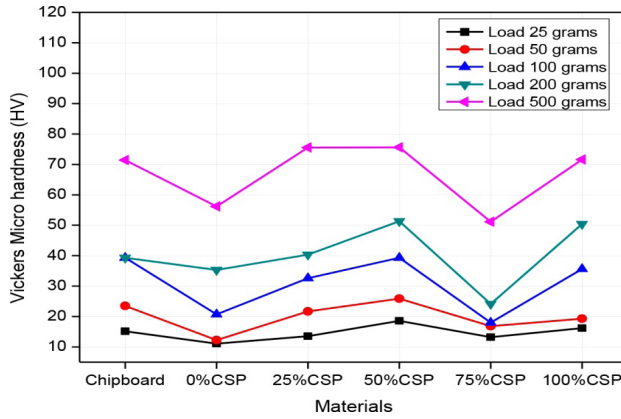


Figure 6: Hardness of the materials.

The percentage of distilled and saltwater absorption of the CSP composite materials that have been observed for 5 days is shown in Figures 7a and Figure 7b respectively. From the results, it is identified that the distilled and saltwater absorption percentages are zero (No water absorption) for the CSP composite materials, and for the chipboard, the distilled water absorption rate is increased between 8,54 % and 63,42 % and in the saltwater, the percentage of water absorption is increased between 5,81 % and 43,85 % is due to the layer of salt formation in the surface.

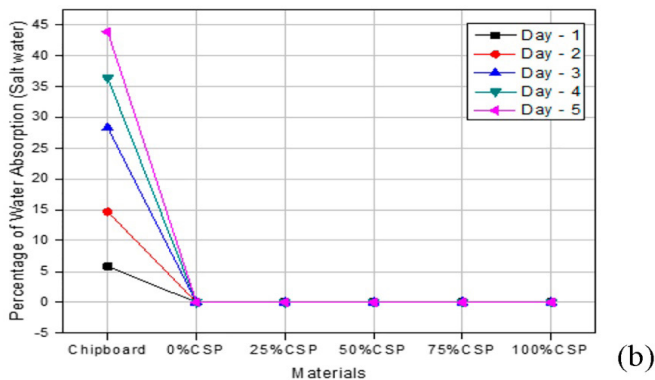
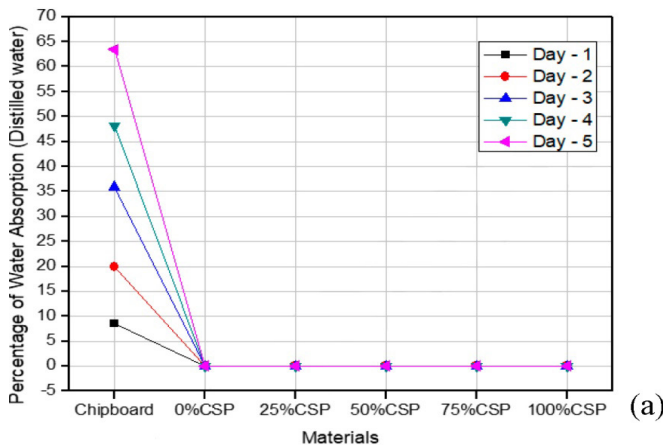


Figure 7: (a) Percentage of normal and (b) saltwater absorbed by the materials.

Figure 8 shows that the time taken for a drill is 10,84 seconds which is the minimum for chipboard as compared with CSP composite materials. But the interior surface of the drilled hole is smooth and even surfaces are formed in the CSP composite materials.

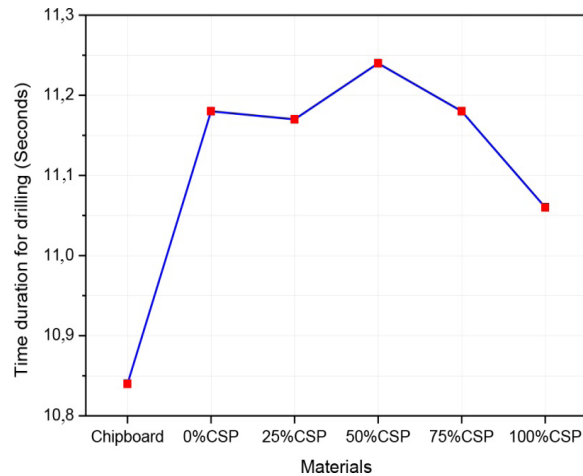


Figure 8: Time of drilling.

In chipboard (f), the drilled surface is not uniform due to low bonding strength. This causes damage to the material and the chips are stick out in the inner surface of the drilled portion as shown in Figure 9.

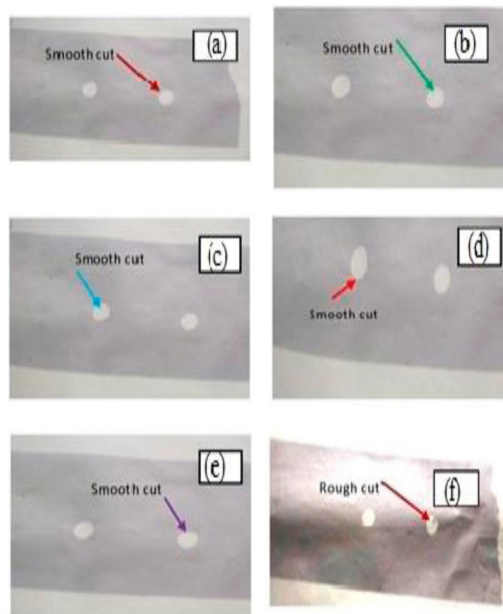


Figure 9: Drilled holes on composite materials.

Fourier-transform infrared spectroscopy (FTIR) analysis is carried out for both the CSP composite materials and the chipboard are carried out and the results are shown in Figure.10. The broad absorption band at 3500 cm^{-1} indicates bonded hydroxyl $-\text{OH}$ groups existing in the organic filler. Absorption peaks at wave numbers of around 2887 cm^{-1} , 1744 cm^{-1} , and 1054 cm^{-1} are assigned to $-\text{CH}$, $\text{C}=\text{O}$, and $\text{C}-\text{O}$ stretching's, respectively Moreover, the observed peak at 1623 cm^{-1} corresponds to $\text{C}=\text{C}$ stretching and confirms the presence of lignin and its aromatic group in the natural filler. At epoxy resin and composite sample spectra, the vibration band at 936 cm^{-1} , resulting from $\text{C}-\text{O}$ deformation of the oxirane group, was recorded. The sample containing 50 % of CSP composite materials shown a strong decrease in absorbance at this intensity therefore,

it can be stated that the curing process was modified by the presence of the organic filler.

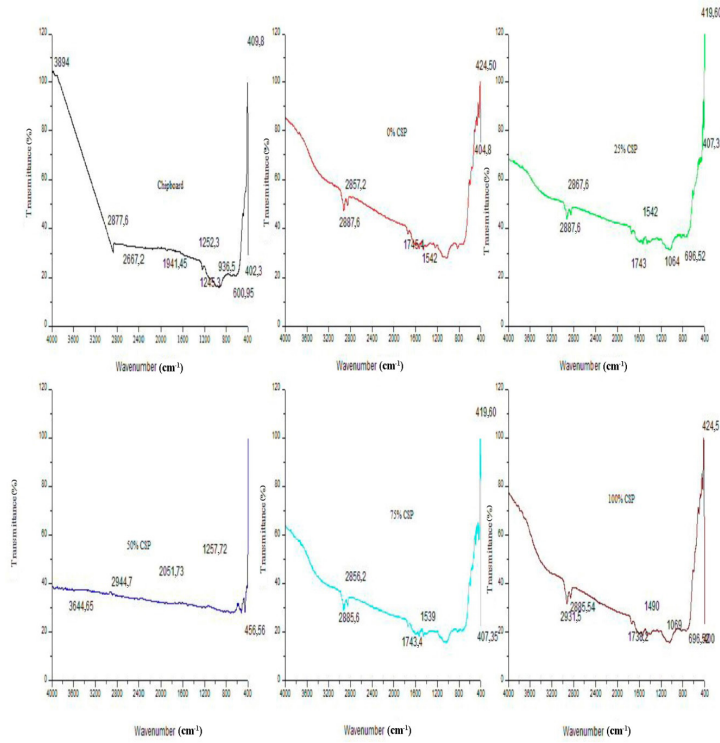


Figure 10: FTIR analysis of the materials.

In the Scanning electron microscope analysis, the images are taken at 500 X magnification are shown in Figure 11a, Figure 11b, Figure 11c, Figure 11d, Figure 11e and Figure 11f. It is observed that there is a uniform mixing of fillers and Epoxy in 50 % and 100 % CSP composite materials shown in Figure 11c and Figure 11e.

For the 25 % and 75 % CSP composite materials the filler particles are scattered and dispersed than 0 % CSP composite materials as shown in Figures 11b, Figure 11d, and Figure 11a and for the chipboard, there are small voids that are presented between the materials is shown in Figure 11f.

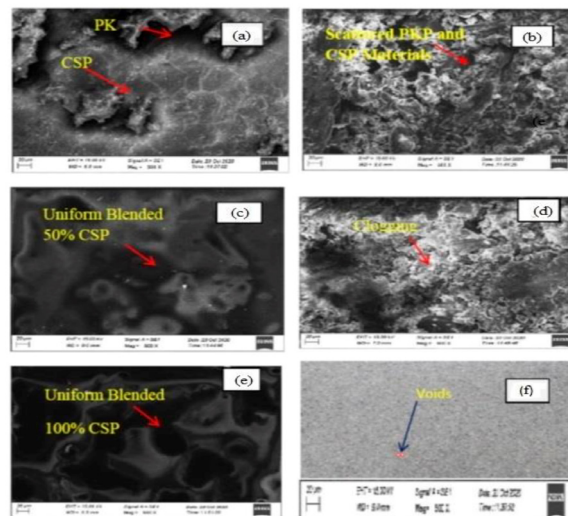


Figure 11: SEM images of composite materials.

The thermal stability of the materials is evaluated by Thermo-gravimetric analysis. The results are shown in Figure 12. It is noted that the CSP composite materials built-in with epoxy resin showed better stability and it is having a mass percentage more than the chipboard. The mass of the materials has been loosed between the temperatures 180 °C to 215 °C further it is constant between 215 °C and 900 °C.

It shows that the lowest improvement in thermal stability, measured the temperature for 5% mass loss at 17 °C, while the highest at 35 °C for samples containing 15 % and 25 % of the filler respectively. At 50 % mass loss, may be observed.

The maximum temperature for epoxy resin was found. It is measured at 350 °C, the material residual increased with increasing the number of organic fillers.

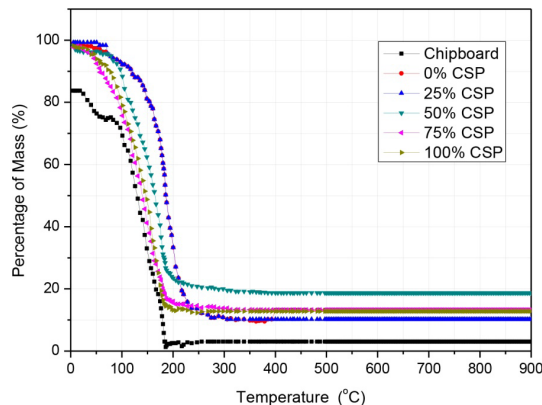


Figure 12: TG curve of materials.

CONCLUSIONS

The experimental studies are carried out on the coconut shell powder (CSP) is added with both palm kernel powder (PKP) with the epoxy resin (i.e., CSP composite materials) and chipboard. The various mechanical properties such as tensile strength, compressive strength, impact strength, and hardness are determined. The water absorption test, time taken for drilling, FTIR analysis, SEM analysis, and TGA analysis are carried out. The following are conclusions arrived at from the present studies.

It is observed that the 50 % CSP composite material is greatly influenced by the PKP filled volume fraction and it is good.

The water absorption tests are carried out in both the normal water and saltwater mediums, it is observed that the water absorption is zero percent in the CSP composite materials but in the chipboard, it is gradually increased in both the mediums.

It is identified that the time taken for drilling a hole in 50 % CSP composite material has taken more time than the other materials, but the drilled inner surfaces are smooth and even in CSP composite materials than a clipboard.

The FTIR analysis is carried out and found the chemical and its groups for all the materials.

SEM analysis shows that in the 50 % and 100 % CSP composite materials the fillers are uniformly mixed than the other materials.

From TGA results it is found that the maximum temperature was found for epoxy resin while increasing of Waste Shell powder caused a gradual decrease of 50 % mass loss temperature.

The composite prepared with 50 % CSP composite material having good tensile strength, less weight density, and good water resistance. It is suitable for various applications such as panel boards, an interior dash-board in automobiles, roof sheets, and doors.

Because of this, for more decades, the problem of plastic waste littering the earth and collecting in the

oceans will remain a problem in creating biodegradable polymers/blends/composites that can replace all other non-biodegradable polymers, governments have been able to give more attention and increase the usage of biocomposites in various applications.

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