

Experimental Investigations on Thermal Properties of Coconut Shell Particles in DAP Solution for Use in Green Composite Applications

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Abstract

Frequent use of various types of green composite in different applications has led to the great development in material science technology. Coconut shell which is generally considered waste material is been analysed here to get its desired properties and characteristics. Basically composite particles are studied using various macroscopic and microscopic approach and with various advance techniques like Scanning electron microscopy (SEM), Water absorption and Thickness swelling test, Thermal analysis, Thermo Gravimetric analysis (TGA) and Differential thermal analysis (DTA). In our work we have focused on the thermal analysis of coconut shell particles using TGA and DTA technology and finally the conclusion is made regarding the feasibility of using Coconut Shell Particle in Application of green composites.

Keywords: Thermal analysis; DTA; TGA; Green composite

Introduction

Coconut shell as particulate filler material

Coconut shell is one of the most important natural fillers produced in tropical countries like Malaysia, Indonesia, Thailand, Sri Lanka and India. Many works have been devoted to use of other natural fillers in composite in recent past and coconut shell filler is a potential candidate for the development of new composites because of their high strength and modulus properties. The coconut particles also have remarkable interest in the automotive industry owing to its hard-wearing quality and high hardness (not fragile like glass fiber), good acoustic resistance, moth-proof, non-toxic, resistant to microbial and fungi degradation, and not easily combustible [1-3].

Reinforcing elements

Adding the reinforcement to the matrix phase improves the properties of the matrix material. Some of the reinforcement used is coconut shell particles, walnut shell particles and almond shell particles to improve the properties of the composite. Here we are concerned with Coconut Shell Particles [4].

Coconut shell particles: The coconut shell used in the present investigation was arranged from local market. In the present investigation coconut shell particles are formed by coconut shells that were previously washed and dried in convection oven at 100°C and then crushing them in Wiley mill and after that coconut shell particles were converted into powder form in grinder. After that coconut shell particles are treated with 5wt% of DAP solution and then mixed in resin and stirred mechanically by means of a high speed mechanical stirrer. Coconut shell and coconut shell particles are shown in the Figure 1 [5].

Matrix material

Matrix material is the material, which holds the relative position of the filler material, transfer stress from one dispersed phase to another dispersed phase and protects dispersed phase from environment. Here epoxy resin is used for the analysis.

Epoxy resin (CY-230): Polymer matrices are most commonly used because of cost efficiency, ease of fabricating complex parts with less tooling cost and they also have excellent room temperature properties when compared to metal and ceramic matrices. Polymer matrices

can be either thermoplastic or thermoset. The most commonly used thermoset resins are epoxy, polyester, vinyl ester and phenolics. Araldite CY-230 is a liquid solvent free epoxy resin. Curing takes place at atmospheric temperature and pressure ageing characteristics of the castings are good. Resin can be stored for at least a year if they are stored under cool, dry conditions in the original containers. In the present investigation CY-230 purchased from M/s CIBATUL Limited, India has been used as matrix material. The epoxy resins are being widely used for many advanced composites due to their excellent adhesion to wide variety of fibers, superior mechanical and electrical properties resistance to atmospheric attack of fully cured mixture are excellent, good performance at elevated temperatures. In addition to that they have low shrinkage upon curing and good chemical resistance. Due to several advantages over other thermoset polymers as mentioned above, epoxy resin has wide range of industrial applications because of their high strength and mechanical adhesiveness characteristic [6].

Thermal Analysis

Here in thermal analysis Thermo Gravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) are done. The analysis was carried out using TG analyzer (EXSTAR TG/DTA 6300) at IIT, Roorkee by heating the samples in still air (200 ml/min) at 10°C/min from 0°C to 1000°C. The plots are shown in Figure 3a-3f of this work [7].

Thermo gravimetric analysis (TGA)

Thermo gravimetry (TG) is the branch of thermal analysis which examines the mass change of a sample as a function of temperature in the scanning mode or as a function of time in the isothermal mode. Not all thermal events bring about a change in the mass of the sample (for example melting, crystallization or glass transition) but there are

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some very important exceptions which include desorption, absorption, sublimation, vaporization, oxidation, reduction and decomposition. TG is used to characterize the decomposition and thermal stability of materials under a variety of conditions and to examine the kinetics of the physiochemical processes occurring in the sample.

Differential thermal analysis (DTA)

In DTA, the temperature of a sample is compared with that of an inert reference material during a programmed change of temperature. The temperature should be the same until thermal event occurs, such as melting, decomposition or change in the crystal structure. If an endothermic event takes place within the sample, the temperature of the sample will lag behind that of the reference and a minimum will be observed on the curve. On the contrary, if an exothermic event takes place, then the temperature of the sample will exceed that of the reference and a maximum will be observed on the curve as shown in Figure 2. The peaks in graph shows the energy dissipation or energy absorption means whether the reaction in the material is exothermic (energy dissipation shown by EX) or endothermic (energy absorption shown by EN). The area under the endotherm or exotherm is related to the enthalpy of the thermal event, ΔH [8-10].

Discussions and Observations

Figure 3a shows thermogram of epoxy with 10 wt% of coconut shell particles and 10 wt% of hardener. Decomposition of this material has been accomplished under two stages ranging from 344°C to 399°C and 499°C to 527°C with corresponding rate of decomposition are 0.99 mg/min and 0.79 mg/min. Prior to 200°C, the weight loss of 8.13% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material. Maximum rate of decomposition is 99 mg/min and it occurs at 351°C. It can be seen that in first step, decomposition rate is higher than the second step. Such decomposition has been supported with the heat of fusion of -1.44 J/mg centered at the temperature 519°C with DTA signal of 100.5



Figure 1: Coconut shells.

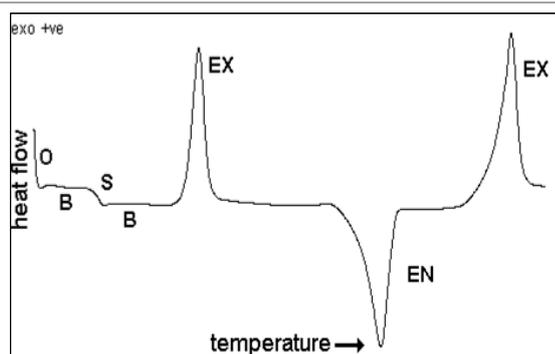


Figure 2: Example of DTA curve.

μV . The decomposition of the material leaved 21.20% residue of initial weight at 500°C.

Figure 3b shows thermogram of epoxy with 15 wt% of coconut shell particles and 10 wt% of hardener. Decomposition of this material has been accomplished under two and volatile matter associated with the material. The maximum rate of decomposition of 1.03 mg/min was observed at 524°C. Such decomposition has been supported with the heat of fusion of -1.82 J/mg centered at the temperature of 527°C with DTA signal of 139.9 μV . The decomposition of the material leaved 23.67% char residue of initial weight at 500°C. Stages ranging from 300°C to 400°C and 500°C to 531°C with corresponding rate of decomposition 0.89 mg/min and 0.75 mg/min. Prior to 200°C, the weight loss of 7.76% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material. The maximum rate of decomposition of 0.89 mg/min was observed in first step decomposition at 353°C. Such decomposition has been supported with the heat of fusion of -1.85 J/mg centered at the temperature of 526°C with DTA signal of 111.9 μV . The decomposition of the material leaved 22.29% residue of initial weight at 500°C.

Figure 3c shows thermogram of epoxy with 20 wt% of coconut shell particles and 10 wt% of hardener. Decomposition of this material has been accomplished under two stages ranging from 336°C to 400°C and 500°C to 518°C with corresponding rate of decomposition 0.83 mg/min and 0.84 mg/min. Prior to 200°C, the weight loss of 8.83% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material. The maximum rate of decomposition of 0.84 mg/min was observed at 506°C. Such decomposition has been supported with the heat of fusion of -1.96 J/mg centered at the temperature of 510°C to with DTA signal of 122.7 μV . The decomposition of the material leaved 20.29% residue of initial weight at 500°C.

Figure 3d shows thermogram of epoxy with 25 wt% of coconut shell particles and 10 wt% of hardener. Decomposition of this material has been accomplished under two stages ranging from 300°C to 400°C and 475 to 550 with corresponding rate of decomposition 0.72 mg/min and 1.03 mg/min. Prior to 200°C, the weight loss of 8.62% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material. The maximum rate of decomposition of 1.03 mg/min was observed at 506°C. Such decomposition has been supported with the heat of fusion of -1.81 J/mg centered at the temperature of 510°C with DTA signal of 136.2 μV . The decomposition of the material leaved 19.63% residue of initial weight at 500°C.

Figure 3e shows thermogram of neat epoxy with 30 wt% of coconut shell particles and 10 wt% of hardener. Decomposition of this material has been accomplished under two stages ranging from 339°C to 400°C and 500°C to 530°C with corresponding rate of decomposition 0.75 mg/min and 1.03 mg/min. Prior to 200°C, the weight loss of 8.46% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material. The maximum rate of decomposition of 1.03 mg/min was observed at 524°C. Such decomposition has been supported with the heat of fusion of -1.82 J/mg centered at the temperature of 527°C with DTA signal of 139.9 μV . The decomposition of the material leaved 23.67% residue of initial weight at 500°C.

Figure 3f shows thermogram of neat epoxy with 30 wt% of untreated coconut shell particles and 10 wt% of hardener. Decomposition of this material has been accomplished under two stages ranging from 300°C

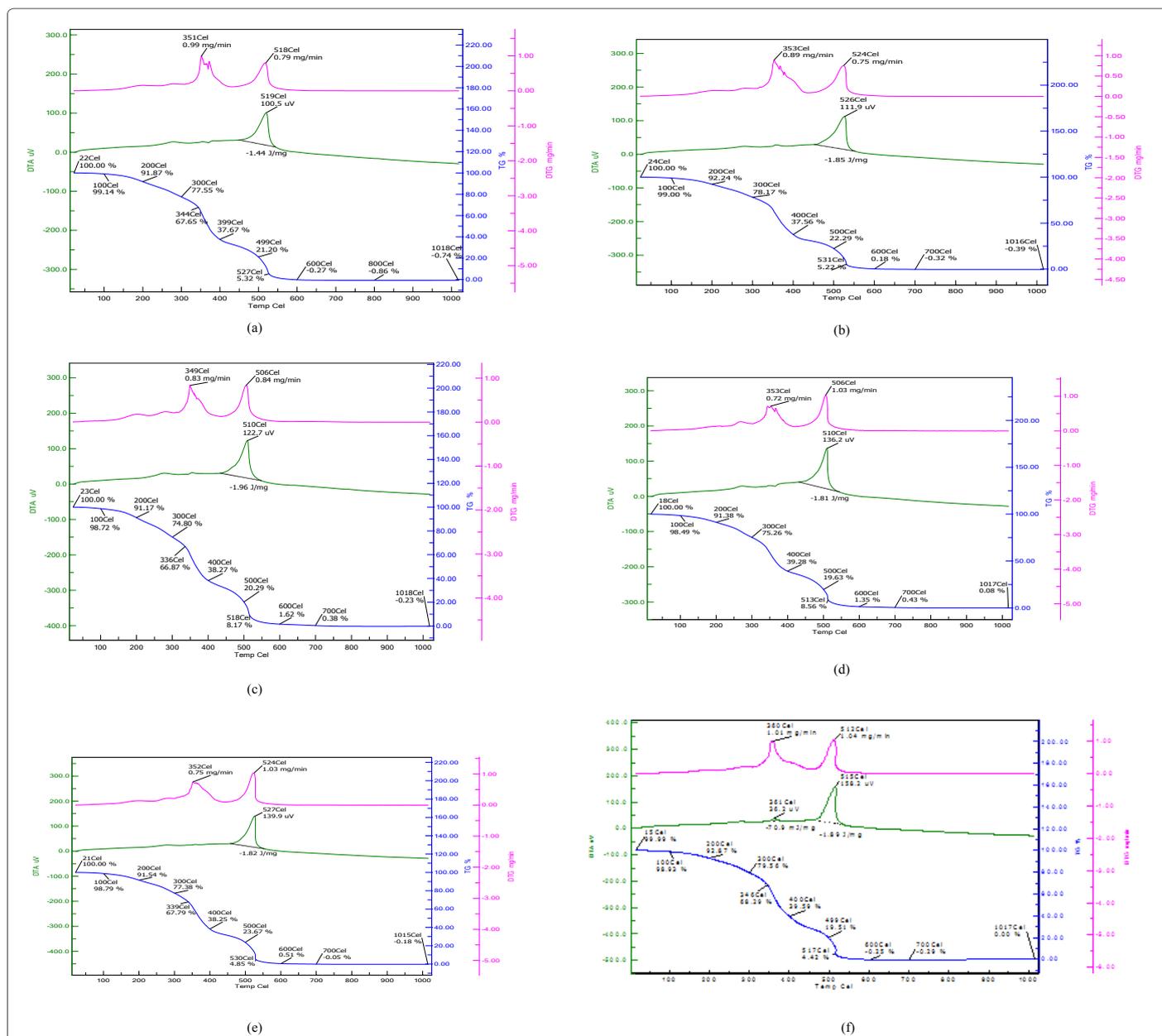


Figure 3: Thermal analysis of epoxy with DAP-treated (a): 10 wt% of csp (b): 15 wt% of csp (c): 20 wt% of csp (d): 25 wt% of csp (e): 30 wt% of csp (f): 30 wt% of csp (wt.).

to 400°C and 499°C to 517°C with corresponding rate of decomposition 1.01 mg/min and 1.04 mg/min. Prior to 200°C, the weight loss of 7.13% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material. The maximum rate of decomposition of 1.04 mg/min was observed at 512°C. Such decomposition has been supported with the heat of fusion of -1.89 J/mg centred at the temperature of 515°C with DTA signal of 158.2 μV. The decomposition of the material leaved 19.21% residue of initial weight at 500°C.

The thermal analysis data reveals that the 30 wt% of coconut shell particles had rendered a marginal increase in the thermal stability of composites. The comparison of untreated and DAP-treated 30 wt% of coconut shell particles composites shows that the presence of DAP increases the first and second decomposition temperature. This means

that the decomposition rates of DAP-treated coconut shell particles composites are lower than those of untreated coconut shell particles composites. TGA graph confirms that decompositions of DAP-treated coconut shell particles composites occur with a very low peak indicating a slow decomposition rate. At 500°C, DAP-treated 30 wt% of coconut shell particles composite give more char residue (23.67%) than the untreated 30 wt% of coconut shell particles composite (19.21%) which show that as the decomposition rate decreased, char residue increased.

Morphology

In the present investigation SEM was carried out for composite containing different weight percentage of DAP-treated coconut shell particles in the epoxy resin matrix. The state of dispersion of coconut shell particles into the resin matrix plays a significant role on the mechanical properties of the composite. Various methods such as SEM,

TEM etc. can be used to evaluate the particle dispersion in the material. Figures 4a-4g shows the SEM photograph of composite containing 0, 10, 15, 20, 25, 30 and 30 (without treated) wt% of coconut shell particles respectively. Figures 4a-4g are shown at 1000x magnification and Figure 4h is shown at 2500x magnification. Size of coconut shell particles is in the range of about 0.862 μm to 1.341 μm as seen in Figure 4h. Figures 4a shows the micrograph of neat epoxy. In this figure, fracture surface is plain and no shearing zone can be observed [11,12].

Figure 4b shows the micrograph of 10 wt% coconut shell particles filled in epoxy resin. In this figure shear bands can be seen which leads to shear yielding of the material. And the dispersion of coconut shell particles is good and no cavity can be seen. But at some places matrix cracking has taken place.

Figure 4c shows the micrograph of 15 wt% of coconut shell particles filled in epoxy resin. In this micrograph the cavity which has occurred due to increase in wt% of coconut shell particles can be seen. It can be seen from the Scanning Electron Microscope results that homogeneity between the coconut shell particles and the matrix decreases with increase in the coconut shell particles content, explains the decrease in strength with increased in the coconut shell particles content within the matrix of the composite. In the micrographs of 20, 25 and 30 wt% of coconut shell particles, some voids and cracks can also be seen signifying brittle cracking and brittle failure which leads to the poor mechanical properties and more water absorption rate and thickness swelling (Table 1) [13].

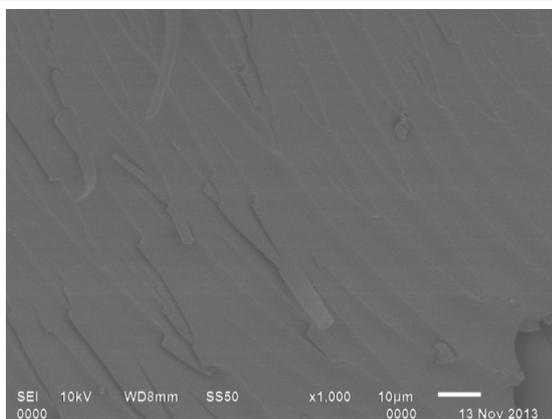


Figure 4a: Scanning electron micrograph for neat epoxy at magnification 1000x.

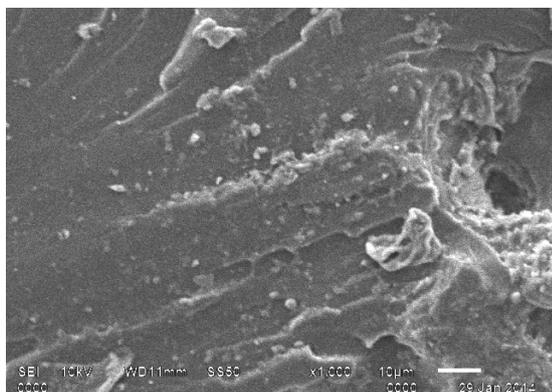


Figure 4b: Scanning electron micrograph for 10 wt% of csp in epoxy resin at magnification 1000x.

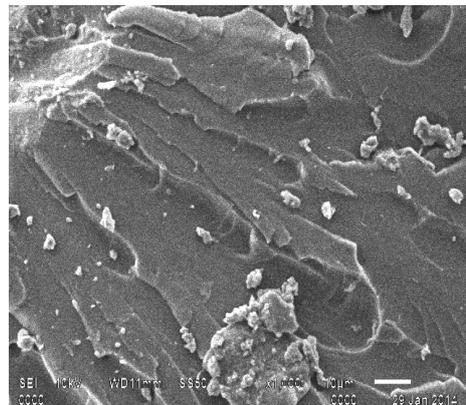


Figure 4c: Scanning electron micrograph for 15 wt% of csp in epoxy at magnification 1000x.

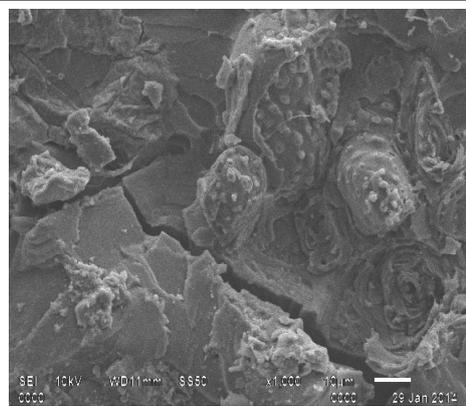


Figure 4d: Scanning electron micrograph for 20 wt% of csp in epoxy at magnification 1000x.

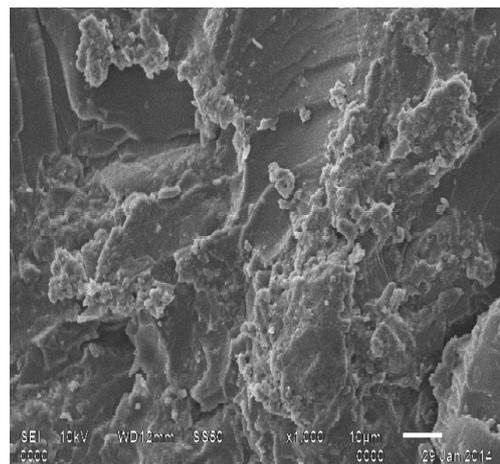


Figure 4e: Scanning electron micrograph for 25 wt% of csp in epoxy at magnification 1000x.

Conclusions

DAP-treated coconut shell particles effectively reduce the flammability of the filled bio-composite. The more the filler content, lower the linear burning rate and weight loss rate. 30 wt% of treated coconut shell particles composite reduce the flammability of the filled composite up to 30%. The thermal analysis data reveals that the 30

wt% of coconut shell particles had rendered a marginal increase in the thermal stability of composites. The comparison of untreated and DAP-treated 30 wt% of coconut shell particles composites shows that presence of DAP increases the second decomposition temperature. This means that the decomposition rates of DAP-treated coconut shell particles composites are lower than those of untreated coconut shell particles composites.

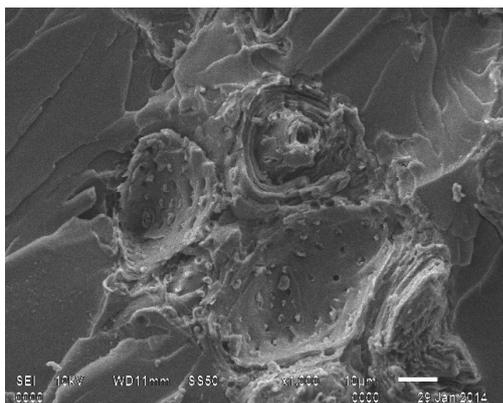


Figure 4f: Scanning electron micrograph for 30 wt% csp in epoxy at magnification 1000x.

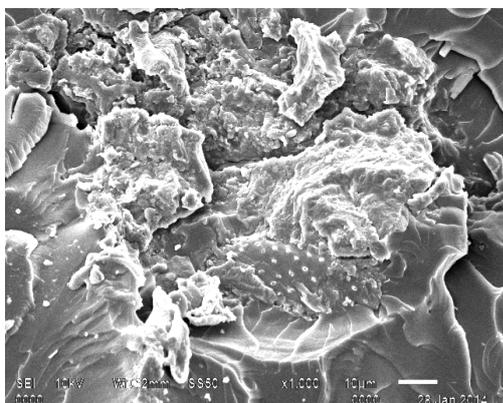


Figure 4g: Scanning electron micrograph for 30 wt% of csp (wt) in epoxy at magnification 1000x.

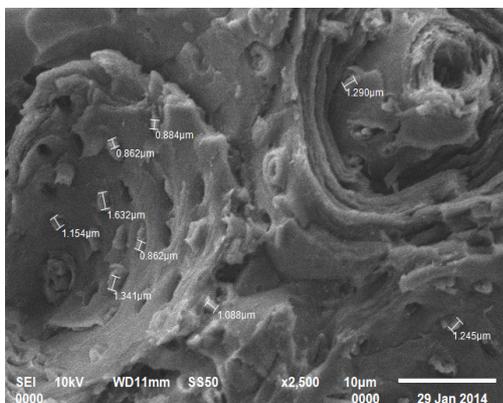


Figure 4h: Scanning electron micrograph for 30 wt% of csp in epoxy at magnification 2500x.

Amount of csp in %	10	15	20	25	30	30 (Untreated)
Amount of hardener in %	10	10	10	10	10	10
No. of decomposition stages	2	2	2	2	2	2
Temperature Range (a)	344-399	300-400	336-400	300-400	339-400	300-400
Decomposition Rate (mg/min)	0.99	0.89	0.83	0.72	0.75	1.01
Temperature Range (b)	499-527	500-531	500-518	475-550	500-530	499-517
Decomposition Rate (mg/min)	0.79	0.75	0.84	1.03	1.03	1.04
Weight loss before 200°C	8.13	7.76	8.83	8.62	8.46	7.13
Heat of fusion (J/mg)	-1.44	-1.82	-1.96	-1.81	-1.82	-1.89
Temperature at fusion	519	527	510	510	527	515
DTA reading in micro Volt	100.5	139.9	122.7	132.2	139.9	158.2
Residue left at 500°C	21.2	23.67	20.29	19.63	23.67	19.21
Max. Decomposition Rate (mg/min)	0.99	1.03	0.84	1.03	1.03	1.04
Temp. at Max. Decomposition °C	351	524	506	506	524	512

Table 1: Thermal analysis of coconut shell particles.

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