EXPERIMENTAL STUDIES ON THERMAL BEHAVIOR OF ALKALI TREATED GROUNDNUT SHELL PARTICLE REINFORCED POLYMER COMPOSITES PRASANNA C. KATTIMANI^{a1}, NIRAJ G. PAI BHALE^b, P. K. SRIVIDHYA^c AND SAISH N. RIVANKAR^d

abdDepartment of Mechanical Engineering, Agnel Institute of Technology & Design, Mapusa, Goa, India cDepartment of Mechanical Engineering, Periyar Maniammai University, Vallam, Thanjavur, India

ABSTRACT

Polymers reinforced with synthetic fibers such as glass, carbon and aramid yield advantages of high stiffness and strength to weight ratio as compared to conventional construction materials, i.e. wood, concrete and steel. In spite of these advantages, the predominant use of synthetic fiber-reinforced polymer composite has a tendency to decline because of its high-initial costs, use in non-efficient structural forms and most importantly adverse environmental impact. On the other hand, the increased interest in using natural fibers as reinforcement in plastics to replace traditional synthetic fibers in some structural applications has become one of the main concerns to study the potential of using natural fibers as reinforcement for polymers. In view of this, researchers are concentrating their attention on natural fiber composites (i.e. bio-composites), which are composed of natural or synthetic resins, reinforced with natural fibers. Subsequently, manufacturing of high-performance engineering materials using renewable resources has been pursued by researchers across the world owning to renewable raw materials are environmentally sound and do not cause health problem. The present work includes the preparation of bio composites reinforced with alkali (NaOH) treated groundnut shell particle in vinyl ester resin matrix. Experimentally the thermal properties are tested and evaluated under varying parameters such as particle size, % filler weight and resin type. The different composite specimens have been prepared as per the full factorial design of experiments (FFD). The statistical analysis using analysis of variance (ANOVA) has been carried out to identify the significances of the parameters affecting the thermal behavior of prepared composites.

KEYWORDS: Natural Fiber, Groundnut Shell, Composite Polymer, Orthogonal Array, Design of Experiments, Factorial Design, ANOVA, Mechanical Properties.

The composites industry has begun to recognize that the commercial applications of composites promise to offer much larger business opportunities than the aerospace sector due to the sheer size of transportation industry. Thus the shift of composite applications from aircraft to other commercial uses has become prominent in recent years. Increasingly enabled by the introduction of newer polymer resin matrix materials and high performance reinforcement fibers of glass, carbon and aramid, the penetration of these advanced materials has witnessed a steady expansion in uses and volume. The increased volume has resulted in an expected reduction in costs. High performance FRP can now be found in such diverse applications as composite armoring designed to resist explosive impacts, fuel cylinders for natural gas vehicles, windmill blades, industrial drive shafts, support beams of highway bridges and even paper making rollers. For certain applications, the use of composites rather than metals has in fact resulted in savings of both cost and weight. Some examples are cascades for engines, curved fairing and fillets, replacements for welded metallic parts, cylinders, tubes, ducts, blade containment bands etc.

The need of composite for lighter construction materials and more seismic resistant structures has

placed high emphasis on the use of new and advanced materials that not only decreases dead weight but also absorbs the shock and vibration through tailored microstructures. Composites are now extensively being used for rehabilitation/strengthening of pre-existing structures that have to be retrofitted to make them seismic resistant, or to repair damage caused by seismic activity. Unlike conventional materials (e.g., steel), the properties of the composite material can be designed considering the structural aspects. The design of a structural component using composites involves both material and structural design. Composite properties (e.g. stiffness, thermal expansion etc.) can be varied continuously over a broad range of values under the control of the designer. Careful selection of reinforcement type enables finished product characteristics to be tailored to almost any specific engineering requirement.

SPECIMEN PREPARATION

Specimens were prepared by using groundnut shell particles and vinyl ester polymer resin by hand layup technique in a wooden mould box of dimensions 100mm diameter and 10mm thickness at room temperature. First the groundnut shells were washed with water to remove the sand, oily and greasy substances and other impurities, and then the groundnut shells were treated with NaOH solution followed by washing with distilled water. Subsequently, the groundnut shells were dried at room temperature, by using different techniques, the required particle sizes are maintained as per the experimental requirements. The prepared groundnut shells is grinded into various grain sizes (0.5mm, 1mm and 1.5mm) and is thoroughly spread with the matrix material (vinyl ester) to which curing additives are added in the proportion of Cobalt Octane 1.5% by the weight of vinyl ester, which acts as the accelerator, Di Methyl Aniline 1.5% by the weight of vinyl ester, which is a promoter, and Methyl Ethyl Ketone Peroxide 1.5% by the weight of vinyl ester, which acts as catalyst and pressed with load and allowed to cure for 2 hours, so as to achieve uniform thickness.

INVESTIGATION STUDY

The current proposed work relates to the preparation of composites with groundnut shell particles as a reinforcement and vinyl ester resin as a matrix material aiming at investigating the thermal properties by varying the parameters such as particle size, % NaOH and % filler weight using Taguchi's factorial design. 3 levels are defined for each of the factors identified and are summarized as shown in Table 1.

		Test levels		
Parameters	Units	Ι	II	III
Particle size	mm	0.5	1.0	1.5
% NaOH	%	0	10	20
% Filler weight	%	20	40	60

Table 1: Variables and Levels

	Table	2:	L_{27}	(3 ³)	Orthogonal	array
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Specimen No.	Particle Size (mm)	NaOH (%)	Filler Weight (%)
1	1	1	1
2	1	2	1
3	1	3	1
4	2	1	1
5	2	2	1
6	2	3	1
7	3	1	1
8	3	2	1
9	3	3	1
10	1	1	2
11	1	2	2

12	1	3	2
13	2	1	2
14	2	2	2
15	2	3	2
16	3	1	2
17	3	2	2
18	3	3	2
19	1	1	3
20	1	2	3
21	1	3	3
22	2	1	3
23	2	2	3
24	2	3	3
25	3	1	3
26	3	2	3
27	3	3	3

Thermal Conductivity Test

Test Specimen: According to ASTM E 1530-99, the test specimens for the thermal conductivity test were prepared as shown in Figure 1.



Figure 1: Specimen for thermal conductivity test.

Test Procedure: The thermal conductivity test is carried out as per the ASTM E 1530-99 standard. A circular disc shaped specimen 100 mm diameter and 10 mm thickness is prepared. The test setup consists of heating element, aluminium plates and thermocouples. The heating element is connected to a conducting material (aluminium plates) of the same size as that of the specimen. Two thermocouples are connected to two specimens over their surfaces and one thermocouple is connected to the heating element. The entire setup is packed with glass wool in order to prevent any loss of heat from the specimen.

Thermal Expansion Test

Test Specimen: The specimen shall be of the full thickness of the material and shall be 150 mm length and 40 mm width as shown in the Figure 2.

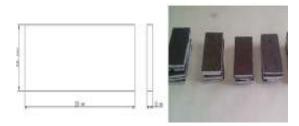


Figure 2: Specimens for thermal expansion test

Test Procedure: According to ASTM D 696 testing is been carried out. The setup consists of a heater above which aluminium plate is placed. The specimen is placed over the aluminium plate. Two dial gauges of range 0.001-1 mm are placed at both the ends of the specimen. A thermocouple is placed at the centre of the specimen which is connected to the temperature measuring instrument. Reading "0" is taken as initial reading. The linear expansion values are noted at various temperatures and then the coefficient of linear thermal expansion is calculated.

Specific Heat Test

Test specimen: The specimen shall be of the full thickness of the material and shall be a sphere of 30 mm diameter as specified in the drawing shown in the Figure 3.

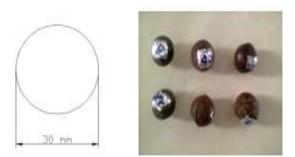


Figure 3: Specimens for specific heat test

Test Procedure: Testing is been carried out according to ASTM E 1269. The specimen of groundnut shell and resin is cast in the form of sphere using a wooden mould. Two thermocouples are placed at different depths and different positions of the specimen. Connect the thermocouples to the temperature measuring instrument and place the specimen in a thermally evacuate chamber. Inside the chamber a heater coil of size of the specimen is placed and power supply is given through the dimmer stat, voltmeter and ammeter. Heat supplied is noted down for 5 degree rise in temperature of the specimen, also note down the time required for that. Measure the weight of the specimen

and repeat the procedure for different specimens and calculations are done.

Thermal Diffusivity Test

The physical significance of thermal diffusing is associated with the propagation of heat into the medium, during changes of temperature with time. Higher the thermal diffusivity faster the propagation of heat into the medium. The temperature in the interior of the solid will vary continuously with position and time. The larger the thermal diffusivity, the less time is required for heat penetration into the solid. It can be represented by

$$D = \frac{K}{\rho \times C_p} \quad \text{in m/s}^2$$

where,

D

o

= Thermal diffusivity in m^2/s

K = Thermal conductivity in $W/m.^{0}C$

= Density of the specimen in Kg/m^3

 C_p = Specific heat of the specimen in J/Kg

From Table 4, one can observe from the ANOVA that % filler weight has greater influence (P_F =33.6589 %) on thermal conductivity. It can also be observed that particle size (P_P =31.57%) and % NaOH treatment (28.36%) have considerable influence on the property of the composites. The error is found to be (P_E =1.3524 %) which indicates every parameter has an individual significant influence on the property of the composites.

From the Table 5, one can observe from ANOVA that, % NaOH treatment has the greater influence (P_N =49.57%) and Particle size has considerable (P_P =35.46%) contributions on the thermal expansion property of composites. It is observed that filler weight has 6.5% influence, error is found to be (P_E =0.4688%) which indicates that the interactions among each parameters is most significant on the property of the composite.

From Table 6, one can observe from the ANOVA that, particle size has the greater influence (P_p =62.75%) and % NaOH (P_N =16.42%) & % filler weight (P_F =12.42%) have considerable influence on the specific heat property of the composites. It can be observed that the interaction between particle size and % NaOH has a considerable influence. It is also found that there is a minimum error (P_E =1.95%) which

indicates that there is a maximum interaction of each

parameters on the property of the composite.

TEST RESULTS

Trail no	Thermal conductivity	Thermal expansion	Specific heat	Thermal diffusivity
11 11 110	(W/m ⁰ C)	(10 ⁻⁵ / ⁰ C)	(J/Kg ⁰ C)	(m^2/s)
1	0.242486	5.665	1535.545545	1.66473E-07
2	0.214123	5.551	1709.702479	1.31443E-07
3	0.209837	5.424	1870.293052	1.13572E-07
4	0.233822	5.643	1389.900092	1.88978E-07
5	0.189056	5.162	1652.865197	1.18879E-07
6	0.173550	5.086	1663.321118	9.7205E-08
7	0.211554	4.753	1313.719355	1.60184E-07
8	0.189451	4.378	1345.674574	1.31601E-07
9	0.178903	4.356	1556.850473	1.08185E-07
10	0.218439	5.51	1325.716011	1.84469E-07
11	0.193575	5.21	1481.538473	1.62341E-07
12	0.191969	5.075	1514.554691	1.4496E-07
13	0.196925	4.872	1203.194736	1.27011E-07
14	0.173550	4.637	1214.835047	1.64529E-07
15	0.165541	4.41	1237.854667	1.56855E-07
16	0.189746	4.245	1143.029732	1.77866E-07
17	0.173434	4.047	1202.444122	1.46995E-07
18	0.170466	3.853	1243.161639	1.35704E-07
19	0.206365	5.22	1046.434928	2.36304E-07
20	0.193518	5.1	1131.096286	2.13369E-07
21	0.173615	4.7	1377.600557	1.39618E-07
22	0.183679	3.915	1145.753012	2.03924E-07
23	0.167961	3.664	1148.081232	1.85146E-07
24	0.150342	3.581	1241.939587	1.4094E-07
25	0.170066	3.943	1011.608809	2.04098E-07
26	0.162233	3.46	1068.927011	1.55686E-07
27	0.160086	3.338	1117.177005	1.93193E-07

Table 3: Measured values of thermal properties of composites as per FFD

Table 4: ANOVA for thermal conductivity property of composites

Source	DOF	SS	Variance	% Contribution
A (Particle size)	2	8.7108	4.3554	31.5751
B (% NaOH)	2	7.8256	3.9128	28.3662
C (% Filler weight)	2	9.2857	4.6429	33.6589
AB	4	0.0522	0.0261	0.1892
BC	4	0.5755	0.2878	2.0863
AC	4	0.7647	0.3823	2.7718
Error	8	0.3731	0.0466	1.3524
Total	26	27.587	1.0611	100.00

SS- Sum of Squares, DOF-Degree of Freedom

Source	DOF	SS	Variance	% Contribution
A (Particle size)	2	17.8841	0.5146	35.4640
B (% NaOH)	2	24.9998	3.0267	49.5742
C (% Filler weight)	2	3.2936	0.0896	6.5311
AB	4	3.6989	0.9247	7.3348
BC	4	0.1338	0.0335	0.2653
AC	4	0.1825	0.0456	0.3618
Error	8	0.2364	0.0296	0.4688
Total	26	50.4290	1.9396	100.00

Table 5: ANOVA for thermal expansion property of composites

Table 0. ANOVA for specific near property of composites						
Source	DOF	SS	Variance	% Contribution		
A (Particle size)	2	32.9258	16.4629	62.7588		
B (% NaOH)	2	8.6148	4.3074	16.4204		
C (% Filler weight)	2	6.5182	3.2591	12.4240		
AB	4	1.8183	0.4546	3.4659		
BC	4	0.7975	0.1994	1.5202		
AC	4	0.7687	0.1922	1.4653		
Error	8	1.0207	0.1276	1.9455		
Total	26	52.4641	2.0178	100.00		

Table 6: ANOVA for specific heat property of composites

Table 7: ANOVA for thermal diffusivity property of composites

Source	DOF	SS	Variance	% Contribution
A (Particle size)	2	35.7785	17.8893	37.5124
B (% NaOH)	2	2.0345	1.0172	2.1331
C (% Filler weight)	2	30.3836	15.1918	31.8560
AB	4	0.2285	0.0571	0.0596
BC	4	8.8421	2.2105	9.2707
AC	4	3.1342	0.7835	3.2861
Error	8	14.9764	1.8720	15.7021
Total	26	95.3778	3.6684	100.00

From Table 7, one can observe from the ANOVA that, particle size has the greater influence (P_R =37.51%) and % filler weight (P_F =31.85%), this clearly indicates that the thermal diffusivity of composite is greatly influenced by the particle size and % filler weight. It can also be observed that the interaction between % filler weight and % NaOH has more influence 2.21% on thermal diffusivity in comparison with other interactions. The error is found to be 15.70% which indicates that reduction in the % error will results in the improvement of the property of composites.

Main effects plot for thermal conductivity & Expansion

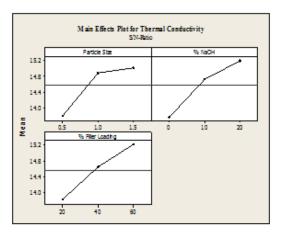


Figure 4: Main effects plot for thermal conductivity

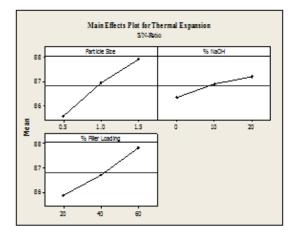


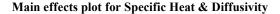
Figure 5: Main effects plot for linear thermal expansion

Inference for Thermal Conductivity: The analysis of the influence of each control factor on thermal conductivity property of groundnut shell reinforced polymer composites is performed with sgnal-to-noise ratio response table using Minitab 16 statistical software is shown in Figure 4. The optimal testing condition of these control factors are easily determined from graph. The response graph shows the change of mean when the settings of control factors was changed from one level to another level.

From the main effects plot of Figure 4, it is observed that, the S/N ratio for 0.5 mm particle size is minimum which indicates high thermal conductivity and as the particle size increases from levl 1 to level 2 the conductivity is found to decrease. Further, from level 2 to level 3 there is a marginal increment in S/N ratio value which indicates lower thermal conductivity. The filler loading for level 1 shows greater value of thermal conductivity at level 1 and further increase in filler loading, reduction in the property is observed. % NaOH parameter showed higher thermal conductivity in level 1 and reduction in the property is found in the levels 2 to 3.

Inference for Thermal Expansion: The main effects plot of Figure 5 for thermal expansion showed that, the signal-to-noise ratio is lower for the particle size with 0.5 mm which indicates high thermal expansion. Further, it is observed that the S/N ratio linearly increases from level 2 to level 3 which indicates reduction in the expansion coefficient. It can also be observed that, as the filler loading increases the signal-to-noise ratio increases which illustrates that expansion coefficient decreases with increase in the filler content. % NaOH also clearly indicates that there is a linear

increment in the S/N ratio which is analogous to lower thermal expansion from level 1 to level 3.



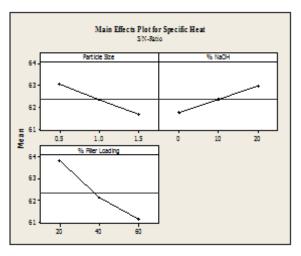
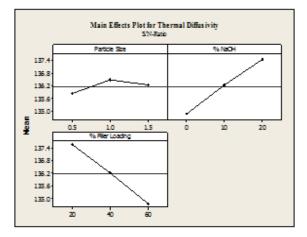


Figure 6: Main effects plot for specific heat





Inference for Specific Heat: The main effects plot of Figure 6, for speicific heat showed that, particle size changing from level 1 to level 3 the specific heat will reduces below the mean value of S/N ratio, this is because of increased filler content. The filler loading shows linearly decreasing in specific heat property of the composite specimen from level 1 to 3. It can be observed that the untreated composite specimen showed lower specific heat as compared to the specimens with 10% and 20% NaOH treatment [Sherely and Abderrahim, 2008]. Specific heat is maximum for the composite specimen with 20% NaOH treatment.

Inference for Thermal Diffusivity: The main effects plot of Figure 7, for thermal diffusivity showed increment in duffusivity from particle size from level 1

to level 2 and further it decreses from level 2 to level 3: however it is observed that the diffusivity is linearly increasing for % NaOH treatment from level 1 to level 3. The increase in filler loading showed linearly decrease in thermal diffusivity from level 1 to 3. This indicates that the present composite materials require a longer period of time either for heating up or cooling [Rafah, 2010].

Interaction Plot for Thermal Conductivity S'N-Batio 5ec 05 10 133 10 ž ż ÷. . ús. 10 13

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Interaction effects plot for Thermal Conductivity &

Figure 8: Interaction effect plots for thermal conductivity

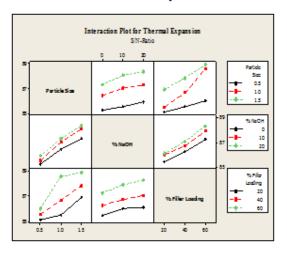


Figure 9: Interaction effect plots for thermal expansion

Inference for Thermal Conductivity: The interaction of partcle size, % NaOH treatment and % filler weight on the thermal conductivity property of groundnut shell particle reinforced polymer matrix composites is shown in Figure 8. It is found that there is a strong relation between particle size and % NaOH treatment from level 1 to 2, and the interaction among partcile size with % filler loading has significant effect on thermal

conductivity, i.e. the linearly increasing in the S/N ratio value is observed from level 1 to 3. Also there is a strong interactions found between % filler loading and % NaOH treatment at level 1 to level 3 showed higher values of S/N ratio which indicates lower thermal conductivity. Thus from above interactions it is concluded that interaction among partcle size and % NaOH and also % filler weight are found with equal importances.

Inference for Thermal Expansion: The interaction effect plots of Figure 9 for thermal expansion, the interaction of particle size and % NaOH treatment has a significant effect on thermal expansion. It is also observed that strong interaction between particle size and % filler loading. It can also be observed that there is positive improvements in the signal to noise ratio values of thermal expansion for 10% and 20% NaOH treatment composites. The interactions between all the three parameters (particle size, % NaOH and % filler weight) are considered to be of greater significance.

Interaction effects plot for Specific Heat & Thermal Diffusivity

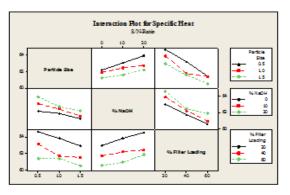


Figure 10: Interaction effect plots for specific heat

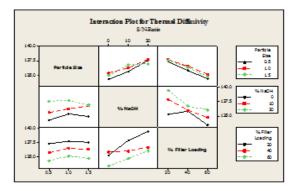


Figure 11: Interaction effect plots for thermal diffusivity

Inference for Specific Heat: The interaction plot of Figure 10 for specific heat, it is observed that interaction between particle size and % filler loading is

strong than compared to interaction between particle size and % NaOH treatment. However there is a synergestic interaction of % filler loading and % NaOH treatment which showed greater S/N ratio at level 1 to level 3. However, the interaction between particle size and % NaOH treatment showed least significance. On the other side the interaction between particle size and % filler loading has less significant effect on specific heat.

Inference for Thermal Diffusivity: The interaction plot of Figure 11 for thermal diffusivity displays that the interaction between particle size and % NaOH treatment has stronger influence. It can be observed that the S/N ratio linearly increase from level 1 to level 3. It can also be observed that the interaction between particle size and % filler weight showed decrement in the thermal diffusivity values with increase in the filler content from level 1 to 3. On the other side the interaction between % NaOH and % filler weight has greater influence on the thermal diffusivity property of the composite.

CONCLUSION

The current investigation highlights the preparation and characterization of bio composites with natural fiber (groundnut shell) based polymer composites consisting of groundnut shell particle as reinforcement and vinyl ester resin as a matrix material. The developed composites are characterized with respect to thermal characteristics. The experiments have been planned as per full factorial design (FFD) of experiments to prepare the different composite specimens for studying the effects of particle size, percentage NaOH treatment and percentage filler loading on the thermal behavior of polymer composites. The analysis of variance (ANOVA) has been performed to identify the significance of each individual control factors and their interaction effects on the proposed properties of composites.

The following conclusions are drawn from the experimental results:

- The hand lay-up technique can be used successfully for the fabrication of a groundnut particle reinforced with vinyl ester matrix material.
- The thermal properties can be successfully analyzed using the principles of design of experiments (DOE).
- The fibers treated with solution of NaOH alkali treatment show less thermal conductivity than other chemically modified fibers. Such understanding of structure property relationship will not only help

open up new avenues for these fibers, but also highlight the importance of this agricultural material, which form one of the abundantly available renewable resources in the world.

- The experimental results indicate that particle size, percentage NaOH treatment and percentage filler weight, along with two factor interactions are also important on the proposed properties of composites prepared.
- From the experimental investigation, it is found that, the most of the groundnut shell particle reinforced composites prepared will satisfy the minimum requirement thermal conductivity and thermal expansion as specified by Standard E 1530-99 and, 0.1503 W/m⁰C and 3.338 X 10^{-5/0}C, which is used for the general application as an insulating material. The composite boards also satisfy the thermal properties in comparison with wood. Hence the prepared composites can be used as an alternate for wood and wooden products.
- Thermal conductivity and thermal expansion found to be minimum for the composites with 20% NaOH treatment, particle size of 1.0 mm and 60% filler loading.
- The composite with particle size of 1.0 mm and 20% NaOH treatment and 60% filler loading has shown reliable results for thermal properties such as thermal conductivity, linear thermal expansion, specific heat and thermal diffusivity.

SCOPE FOR FUTURE WORK

- The current work is focused on thermal properties like thermal conductivity, thermal expansion, specific heat and thermal diffusivity. A detailed study on the other thermal properties such as dynamic scanning calorimetry (DSC), differential thermal analysis (DTA), thermal mechanical analysis (TMA), thermal stability, thermal resistance, U value, etc. can be carried using the developed composites.
- The ranges of the control variables like particle size (mm), % NaOH treatment and filler content (%) can be still varied depending upon the practical considerations.
- The groundnut shell particle could be used along with artificial fibers as reinforcement and can be analyzed further for better properties.
- The similar analysis can also be carried for determining the mechanical and acoustical behavior of the proposed composites.

• This work could be extended for thermoplastic materials which can be used as matrix materials and groundnut shell particles as reinforcement to identify the performances of composites.

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