EXPERIMENTAL STUDY OF EFFECT OF PONGOMIA POD POWDER AS FILLER ON SOME MECHANICAL, THERMAL AND FIRE RESISTANT PROPERTIES OF ARECA PHENOLIC RESIN COMPOSITES

Ravi Kumar B N[1], R. Shankara Reddy[2]
[1]*Assistant Professor, Mechanical Department, JNNCE, Shivamogga-577204*
[2]*Professor, Mechanical Department, RRCE, Bengaluru-560074*
[1]ravidhoddal@gmail.com,[2]prof.rsreddy@gmail.com

Abstract.

Polymeric Composites are the wonder materials becoming an essential part of today's life. When these composites are exposed to heat above the glass transition temperature of resin matrix, this leads to reduction in stiffness and strength of material and degrades the mechanical properties due to thermal degradation and combustion of the resin. This poor fire resistance of composites has been a major factor to limit their wide spread of applications. Composites require high flame retardancy which can be obtained by adding a filler material. Thus improving the thermal properties and fire retardant behaviour of polymers was a main challenge for extend their use to most of the applications. In fabrication of composite specimen process, the proportion of the Phenolic resin (primary phase) was fixed to 65% by volume. It is mixed with varying proportion of areca fibre (5%, 10%, 15% and 35%) and pongamia shell powder (30%, 25%, 20% and 0%) by volume to weight ratio and represented by APC05, APC10, APC15 and APC35 respectively. The prepared composite mixer was poured into mould cavity, kept it in an oven and heated to the temperature of 140°C for about 20 minutes. Finally it is cooled to the room temperature for about 24 hours. Mechanical, thermal and fire resistant properties are investigated. Thermal properties improved with increase in pongamia shell powder. Thermal stability, fire resistance properties and mechanical properties increases with increase in fibre proportion. It is expected optimum composition of the current work APC15 will be used in Aircraft industries, Aerospace applications, Chemical industries, Electrical s/m and Electronics, Automobiles, Ship building, Wind turbine blades, Thermal encapsulation, Flip chip applications, Thermal interface materials and even in the engine parts of modern automobiles.

Keywords:FireResistant,Filler,HeatDeflectionTemperature,LimitingOxygenIndex

INTRODUCTION

The enormous use of polymer materials in our daily life was due to their remarkable combination of properties like simple fabrication methods, low weight and their low cost. However, polymer materials are also recognized for their low thermal conductivity (K<0.5 W/mK), comparatively high flammability and mainly the production of corrosive or toxic gases and smoke of other materials during their combustion causes adverse effects on the environment. Thus the combined challenge consists in developing effective and environment friendly flame retardant system for polymer materials and also modern applications of polymers as heat sinks in electronic packaging needs new composites with comparatively high thermal conductivity. Thus improving the thermal properties and fire retardant behaviour of polymers was a main challenge for extend their use to most of the applications. Enhanced thermal conductivity in polymer materials may be achieved either by varying molecular orientation or by the addition of conductive fillers. Fire retardants are united with polymer resins to decrease their flammability, reduce production of volatiles and to reduce the pollutants. In the current work the new environment friendly polymeric matrix composite material was fabricated which having both good thermal and fire resistance properties by using phenolic resins as base matrix (primary phase), Areca fibres as reinforcement material and Pongamia seed shell as filler materials(secondary phase).

1.1. Material Selection

1.1.1 Phenolic Resin (Phenol Formaldehyde) as Matrix

Pure Phenolic resin can be obtained through the condensation reaction between phenol (C_6H_5OH) and formaldehyde (CH_2O). During reaction they producing methylene bridges between the phenol molecules, this is a condensation reaction as shown in the Figure 1.

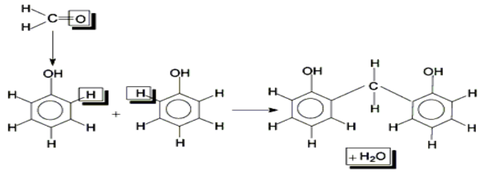


Fig 1. Condensation Reactions for the Formation Phenol Formaldehyde

Phenolic composites are mostly used for the production of responsible parts for various industries. Phenolic resins are known for their excellent thermal properties and chemical stability [10].Phenolic resin composites are currently used in many aircraft interiors. Along with the a variety of polymer resins, Phenolic resin maintain its spot in several industries a from a century after its introduction since of its better heat and flame resistance, mechanical strength and also shows good chemical resistance in opposition to various solvents like acids, water etc. The phenolic resin is shown in the Fig 2.

1.1.2 Reinforcement As Areca Fibre

Among all the natural fibre-reinforcing composite materials, Areca fibre appears to be a promising material because it is inexpensive; availability is abundant in South India and a very high potential perennial crop. Areca fibres belong to the class of Areca catechu L., under the family of palmecea and originate from the Malaya peninsular of East India. It is the Chief industrial cultivation is in South-East and South-West part of India. It was approximated that 6-7 Lakh tonnes of Areca fibre husk is present in south-west of India. The Areca fibre husk is a rigid fibrous portion which covers the endosperm. Also it constitutes 35–45% of the entire volume of the fruit. Areca fibre husk are largely composed of hemicelluloses and not of cellulose content [9].The Karnataka state is the first both in terms of area & production followed by Kerala & Assam states. Also the area under arecanut cultivation has risen more quickly in Shimoga district as compared to Dakshina Kannada & Uttara Kannada districts**.** Table 1 Shows Chemical compositions of few natural fibres. The extracted areca fibres are shown in fig 2 and Table 1 shows the Chemical compositions of few natural fibres.

Fig 2 Areca fibres

Table 1 Chemical composition of few natural fibres.

Natural Fibres	Lignin composition (%)	Hemicelluloses composition (%)	Cellulose composition (%)
Areca	13.2-24.6	36-64	--
Sisal	10-15	12	65-72
Maize stalk	10-14	20-23	37-42
Banana	5	19	62-60
Coir	40-46	0.14-.25	32-43

1.1.3 Pongamia Shell (Pod) Powder As Filler

To increase the thermal properties like thermal conductivity, specific heat, coefficient of thermal expansion and good thermal stability in properties like DSC, TGA, HDT and VSP, here Pongamia pod powder used as filler in this investigation. Pongamia pinnata is a fast growing medium sized ever green tree. The pongamia pinnata pod (shell) as shown in Fig 2 is a waste product, but when burnt, it has comparable energy to Brown coal, hence it can be use in either as a gasifier or just thrown back to the soil for fertilizer. Shell-approx 54% of the total seed in shell weight, can be use directly as a solid fuel for boilers; converted to sun diesel (biomass to liquid); or as an organic fertilizer.

Fig 3 Pongamia pinnata shell

2. LITERATURE SURVEY

Cedric sauder et al., [1] have designed high temperature fibre –testing apparatus and it is dedicated to determination of various properties at very high temperatures including electrical conductivity, young's modulus, thermal expansion coefficient strength. Two types of carbon fibres (a Pan-based and a Rayon-based fibre) have been investigated at temperatures up to 2000^0c.The measured properties are discussed with respect to micro structural features.

Suhreta Husic et al., [2] have described the thermal and mechanical properties of un treated E-glass fibre reinforcement composites prepared with soybean oil-based polyurethanes and petrochemical polyol. The results showed that mechanical properties such as tensile and flexural strength, tensile and flexural modules of the soypolyol based composites were comparable with those from composites based on petrochemical polyol. Since soya based polyurethanes offer better thermal, oxidative and hydrolytic stability than petrochemical based once.

James Giancaspro et al., [3] have investigated the fire resistance properties of bicomposite sandwich figs were manufactured by combining an inorganic potassium aluminosilicate matrix with waste sawdust. Based upon the test results obtained from the fire testing, increasing the inorganic blender content reduces the heat release rate of the biocomposite. The addition of fibre reinforcement facings decreases the amount of smoke released by the biocomposite sand witch.

3. FABRICATION OF THE COMPOSITES

3.1 Alkali Treatment

The obtained areca fibres have some acidic contents, so there PH value is more than 7. It may leads chemical reaction with phenolic resin and also from previous studies it is clear that chemically treated areca fibres shows better adhesion and gives a good mechanical strength in the composites. Thus extracted fibres have to be alkali treated before fabrication process. In the alkali treatment process first the areca fibres were treated in a solution of 10% KOH (potassium hydroxide), where the total volume of solution was 15 times the weight of areca fibres. The fibres were kept in this alkaline solution for about 36 hours at a temperature of 30° C. The Fig 4 shows the areca husk being soaked in 10% KOH solution. Fig 4 shows the alkali treatment of areca fibres

Fig 4 Alkali Treatment of Fibres

3.2 Details of Fabricated Specimens

After the proper selection of the materials and the required pre-treatment of the selected material the fabrication process was performed. Before the fabrication process the mould cavity has to be fabricated using MS square rod of thickness 3.2 mm, length 25 mm and breadth 14 mm suitable for the inserting in the electric oven. Initially the mould cavity was wrapped with aluminium foil from the inside with a tile at the bottom of the mould for a flat and good surface finish. Then the other tile was also wrapped by aluminium foil. Fig 5 shows the aluminium wrapped mould cavity and fig 6 shows Setup placed in Electric Oven.

Fig 5.Aluminium foil wrapped mould cavity

Fig 6. Setup placed in Electric Oven

Specimens were prepared for different proportion by using base material, reinforcement and the filler materials, whose details are given as shown in the Table 2. The materials are usually mixed according to % volume by calculating mass required to fill the mould cavity. The mixture was poured into the mould cavity and carefully rolled over the thickness of the mould using Hand Lay-Up technique. Then the other tile was also wrapped with aluminium foil and placed gently on the mould cavity. The set of the foil wrapped tiles and mould was placed over a MS plate, which has threaded holes at its corners. Then after proper aligning the other MS plate with free holes was placed over the mould setup and the whole set up was tightened using bolt and nuts. At the end the whole set up was placed over a stainless steel tray and gently placed in the oven. After the specimen reaches a temperature of 100-110°C the whole setup is reversed for further equal distribution of heat to both sides and again heated for a temperature of about 140°C of the mould, since re-crystallization of phenolic resin occurs at 140°C. Fig 7 shows the Fabricated Specimens.

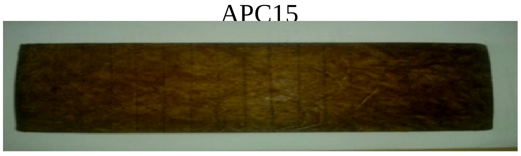
Table 2 Details of composite material fabricated

Sl No.	Designation	% of Areca Fibre	% of Pongamia Pod Powder	% of Phenolic Resin
1	APC05	05	30	65
2	APC10	10	25	65
3	APC15	15	20	65
4	APC35	35	0	65

APC05

APC10

APC15



APC35

Fig 7 Fabricated Specimens

4. EXPERIMENTATION

Experimentation is the significant part of this present work because the properties of newly fabricated composite materials were determined. After the completion of fabrication process the specimens were cut into the dimension according to the ASTM standards. Finally the specimens were tested for their thermal, fire resistance and mechanical properties.

4.1 Properties Tested

The fabricated slabs were prepared to specimens according to the fallowing ASTM standards whose details are shown in Table 3

Table 3 ASTM Standards

Properties	ASTM Standards
Thermal	Thermal Conductivity-ASTM E 1530 Heat Deflection Temperature-ASTM D 648 Coefficient of Thermal Expansion
Fire Resistance	Limiting Oxygen index-ASTM D2863-13
Mechanical	Tensile properties-ASTM D 638

5. RESULTS AND DISCUSSION

5.1 Thermal Properties

5.1.1 Thermal Conductivity

Table 4: Thermal Conductivity of Materials

Composite materials	**Temperature at bottom surface in K**	**Temperature at top surface in K**	**Thermal conductivity in W/m K**
APC05	40.4	28.9	0.80
APC10	41.3	29	0.65
APC15	44.7	31.1	0.51
APC35	44.0	29.1	0.49

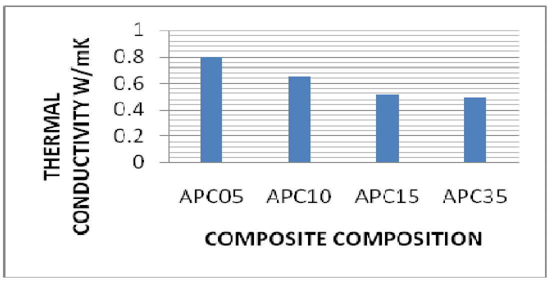


Fig 8 Thermal Conductivity v/s Composite Composition

The Fig 8 shows the graph of Thermal conductivity vs. Composite materials. From the Fig 8 results shows that the thermal conductivity of APC05 is greater than APC10, APC15 and APC35. Also it was observed that the thermal conductivity decreases with the increase in the fibre composition. This is because that the Pongamia pinnata shell powder has high heat carrying capacity, excellent heat conductivity, and good electron mobility as compare to the Areca fibres. As such if thermal conductivity is high, the material will more rapidly approach equilibrium with its surrounding, while if thermal conductivity is low, more energy will be stored and it will be harder to transmit heat through its material and a longer duration will be taken to reach its equilibrium state. But from above calculated thermal conductivity for composite material was much lower than metals. Because in metals the molecules are closely packed and when heat is applied there will be quick transfer of thermal energy takes place between the molecules. Also in metals the outer electrons are not closely bonded, when heat is applied these electron releases from atom structure and helps in heat transfer between the molecules.

5.1.2 Coefficient of Thermal Expansion:

Table 5: CTE

Composite materials	**Initial length in mm**	**Final length in mm**	**Change in length in mm**	**Coefficient of thermal expansion (10^{-5} / °C)**
APC05	128.8	128	0.8	14.90
APC10	126	125	1	18.90
APC15	126.2	124.2	2	37.80
APC35	128	126.3	1.7	31.52

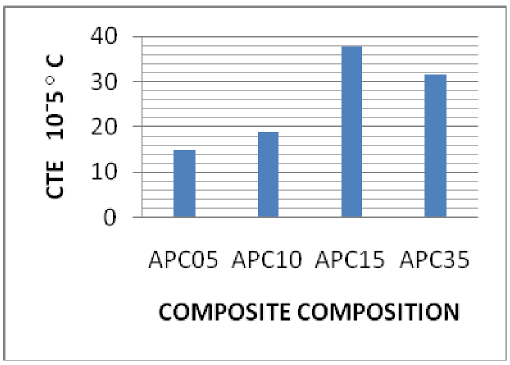


Fig 9 Coefficient of Thermal Expansion v/s Composite Composition

The Fig 9 shows graph of coefficient of thermal expansion vs. Composite materials composition. From the Fig 9 it was observed that thermal expansion coefficient is high for APC15 when compared to other composite materials like APC 5, APC 10 and APC 35. The lower CTE for the composite APC 5 and APC 10 is due to presence of more amount of Pongamia pinnata shell powder, since Pongamia pinnata shell powder have a lower CTE than areca fibres. Due to higher powder composition, more porosity and less density, so it experiences low CTE. But with increases in areca fibre it was found that there was a slight decrease in the CTE value. Because in APC35 the fiber concentration is slightly high, so fibers may not be properly wetted by resin.

5.1.3 Heat Deflection Temperature

Table 7: Heat Deflection Temperature

Composite materials	Heat Deflection Temperature in °C
APC05	85.3
APC10	152
APC15	177.6
APC35	178.8

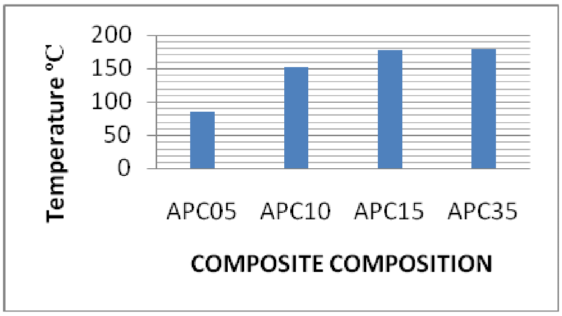


Fig 10 Heat Deflection Temperature v/s Composite Composition

The Fig 10 shows the graph of temperature vs composition of the composite. From the Fig 10 it can be observed that composition APC15 and APC35 have higher HDT as compared to APC05 and APC10. From the Fig 10, it can be stated that the HDT increases with decrease in the percentage addition of the Pongamia pinnata shell powder. It also shows that APC35 is better when compared to other composites APC05 and APC10. It was because as shell powder concentration increases voids between the molecules increases, so density also decreases. Hence when needle applies stress on the specimen it can easily penatrate in to the specimen and produces fixed deflection at lower temperature compared to the specimen with lesser concentration of Pongamia powder.

5.2 Fire Resistance Properties

5.2.1 Limiting Oxygen Index (Loi)

Table 8 Limiting Oxygen Index (LOI)

Composite materials	Final value of the oxygen concentration, CF %	Limiting Oxygen index(LOI), %
APC05	27	27.2
APC10	28	28.35
APC15	28	28.45
APC35	32	30.05

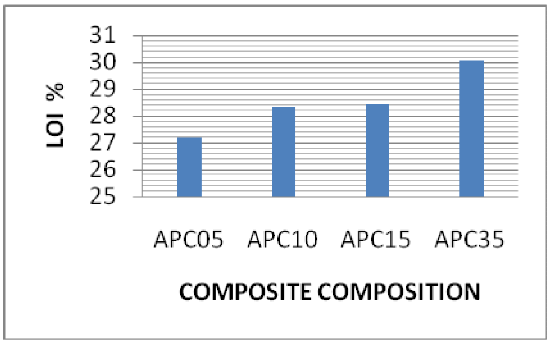


Fig 11 Limiting Oxygen Index (LOI) v/s Composite Composition

The Fig 11 shows the graph of Limiting Oxygen Index vs. composition of the composite. From the Fig 11, it can be observed that the limiting oxygen index decreases with increase in the percentage addition of the Pongamia pod shell powder. It shows that APC35 has comparatively highest limiting oxygen index than othe composites APC05, APC10 and APC15. It also shows that APC05 has the least limiting oxygen index has compared to other composites. It was because fibres has more flame retardent charcteristics than Pongamia pinnata shell powder as in order to ignite fibre material additional energy may required to vaporize the flammable volatiles. Also endothermic decomposition of fibres cools the condensed phase and the relesed water also cools and dilutes the flammable products in the vapour phase. Hence additional quantity of oxygen was required. From the Fig 11, it can be stated that the composition APC35 with highest LOI requires maximum oxygen concentration for burning of the composite material.

5.3. Mechanical Properties

5.3.1. Tensile Test

The tensile test was carried out using computerized universal testing machine. The Fig 13 shows the tensile modulus variation using bar chart.

Tensile Modulus

Table 9 Tensile Modulus

Composite materials	Tensile modulus, MPa
APC05	162
APC10	190
APC15	245
APC35	219

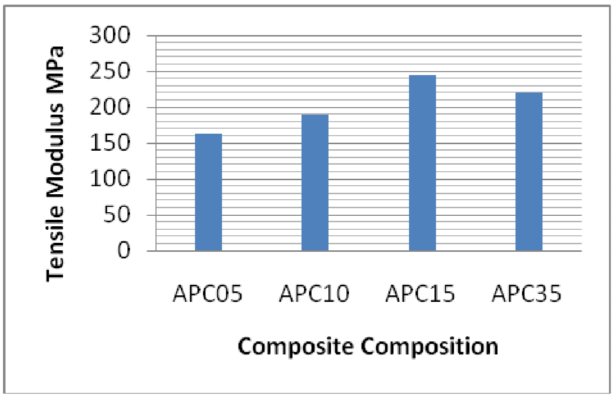


Fig12.Tensile Modulus v/s Composite Composition

The Fig 12 shows the graph of Tensile modulus vs composition of the composite. the obtained results show that APC15 has slightly greater than APC05, APC10 and APC35.The mechanical properties of composite materials depend primarily on the strength and modulus of the fibres, the strength and chemical stability of the matrix and the effectiveness of the bonding between matrix and fibres in transferring stress across the interface. Also fibres provide strength to the composite, but the fillers increses voids between the moleculse and decreses density and hense strenth. So the tensile strenth decreases with increase in powder concentration. Tensile strength of APC35 was slightly lesser than APC15 because in APC35 the fibre concentration slightly high, so fibres may not be properly wetted by resin. Hence bond between fibre and resin was not that much strong.

6. CONCLUSION

The following conclusions were drawn from the present work carried out.

- Thermal conductivity was less than one for all the composites. Also it was very less when compare to the metals. From the results APC05 shows maximum thermal conductivity of 0.8 W/mK. The thermal conductivity decreases with decrease in pongamia pod powder composition.
- From the coefficient of thermal expansion test the APC05 shows minimum CTE of 14.9×10^{-5} /°C and CTE value increases with increase in the fibre concentration.
- From graph it was observed that the APC15 and APC35 show the maximum heat deflection temperature of 177.6 and 178.8°C respectively. Also from the graphs both HDT increases with increase in fibre concentration.
- The thermal properties improved with the increase in the pongamia pod powder concentration in the composites. Thermal stability increases with increases in the fibre concentration.
- From the standard LOI test it was observed that APC35 shows maximum LOI of 30.05% and it was also observed that the LOI increases with increase in fibre concentration. Hence from the fire resistance tests it was found that the composite materials fire resistance behaviour increases with increase in fibre concentration.
- The results show that composite APC15 exhibits high tensile strength of 245 MPa. It was also found that tensile strength and ultimate load of composites were increases with increases in fibre concentration, but there was a slight decrease at higher fibre concentration.
- Thus, composite APC15 (Phenol formaldehyde=65%, Pongamia Pinnata Pod Shell Powder 20% and Areca husk fibre=15%) is a good composite material with satisfactory strength, thermal stability and fire resistant property when compared to other compositions like APC05, APC10 and APC35.
- Hence optimum composition of the current work APC15 will be used in Aircraft industries like Doors and Elevators, Aerospace applications, Chemical industries like Tanks, Pipes and Pressure vessels, Electrical s/m and Electronics, Automobiles, Ship building, Wind turbine blades, Thermal encapsulation, Flip chip applications, Thermal interface materials and even in the engine parts of modern automobile.

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