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Synthesis, Characterization and Mechanical Evaluation of the Phenol-Formaldehyde Composites

B.S.KAITH and AASHISH CHAUHAN*

Dr. B. R. Ambedkar National Institute of Technology
(Deemed University), Jalandhar (Pb.) 144 011, India.

*aashishchauhan16@yahoo.co.in

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Abstract: Phenol: formaldehyde ratio was varied in the synthesis of phenol-formaldehyde resin and used to prepare the composites. These composites were then evaluated for their mechanical strength on the basis of tensile strength, compressive strength and wear resistance. Composite with better strength was characterized by IR, SEM, XRD, TGA/DTA and further studies were carried out for its physico-chemical and mechanical properties like viscosity, modulus of rupture (MOR), modulus of elasticity (MOE) and stress at the limit of proportionality (SP) *etc.*

Keywords: Mechanical properties, Strength, Modulus.

Introduction

Phenoplasts or the phenolic (PF) resins are produced by the condensation of phenol and formaldehyde. These were the first synthetic thermosets. They have a wide range of applications as lacquers and varnishes, molding compounds and laminates for wall panels and table tops *etc.* About 41% of the PF resins are used in plywood manufacture and about 14% in insulation¹

Composites are the wonder materials, which after meeting the challenges of aerospace have cascaded down for catering to the domestic and industrial avenues. In the recent times many successful efforts have been made to prepare phenol-formaldehyde composites and to increase their mechanical potential by using different reinforcements like wood particle, hemp scotch,² banana fiber³, cellulose fiber⁴ *etc.* Kaith *et al*⁵⁻⁷ reported the mechanical studies of the polymer composites reinforced with flax-g-copolymers.

The versatility of the phenol: formaldehyde resin based composites inspired to prepare its composites by altering the phenol: formaldehyde ratio and screening them on the basis of tensile strength, compressive strength and wear resistance. The best results thus obtained would then be further evaluated for their physico-chemical, mechanical & thermal behavior.

Experimental

Phenol (S.D. Fine-Chem Ltd., India) and formaldehyde (CDH, India) were used as received. Composites were prepared in Compression Molding Machine (SANTECH India Ltd.). Libror AEG-220 (Shimadzu) electronic balance was used for weighing.

Wear resistance testing of the composites was carried out on the Wear & Friction Monitor (DUCOM-TR-20L), Compressive/Tensile strength, MOR, MOE and SP of the samples were tested on the Universal Testing Machine (HOUNSFIELD, H25KS). Curing and degasification were done on Compression Molding Machine (Santech India Ltd.).

Viscosity was tested on viscometer (Julabo ED). IR (Perkin Elmer) was used by forming KBr (Aldrich) pellets. XRD was done on Bruker D8 Advance on Zero background, PMMA holder. SEM was (LEO SERIES 435-25-20). TGA/DTA was done on thermal analyzer (Linseis, L81 11).

Preparation of the resin

Phenol-formaldehyde resin was prepared using the procedure as reported earlier⁵. Using, formaldehyde: phenol (1:0.25; 1.0: 0.5; 1.0: 0.75; 1.0: 1.0; 1.0: 1.25) in variable ratios, of their molecular weights, respectively. However, formation of the resin and composite thereafter with F: P in 1: 0.25 ratio was not feasible.

Preparation of the composites

The resin was degassed and poured into molds. The molds were coated inside with oleic acid to avoid adhesion of the mixture and for the ease in removal of the composites. The whole assembly was then placed inside the hot press and cured at 100°C and a pressure of 100 Kg/cm² for 6-7 hours was applied. Mean of three specimen samples were used as a result to determine the mechanical properties. The mechanical tests were conducted in ambient conditions.

Wear test, modulus of rupture, modulus of elasticity and stress at limit of proportionality wear resistance was calculated as per ASTM D 3702 and MOR, MOE and SP were tested on the basis of ASTM D 790 as per the method reported earlier⁸.

Results and Discussion

Reaction mechanism

In phenolic resin the major reactions involved are the addition to give methylol phenols, condensation of a methylol phenol and a phenol to give a methylene bridge, condensation of two methylol phenol and a phenol to give a methylene bridge, condensation to two methylol groups to give an ether bridges and decomposition of the ether bridges to methylene bridge and formaldehyde⁹. The mechanism used for the formation of the resole type PF resin involves the protonation of the carbonyl group of the formaldehyde followed by the electrophilic aromatic substitution at the *ortho* and *para* positions of the phenol. Phenol reacts with excess formaldehyde under basic conditions to form mono, di and tri methylolphenols. Phenol is present as resonance stabilized anion and substitution is exclusively at *ortho*, *para*. On heating the methylolphenols condense to give resole, which on further heating under basic or neutral or slightly acidic condition gives resite^{9,1}.

Resole

Mechanical screening of the P-F composite

Wear resistance

As is evident from the Figure 1, that the phenol-formaldehyde resin formed in the ratio of 1: 0.75 of formaldehyde: phenol, had better wear resistance of $0.09, 0.15$ and 0.2×10^{-4} g/m at all loads of 1, 2 and 3 Kg, respectively. On the other hand, formaldehyde: phenol in 1: 0.5 ratio showed $0.21, 0.22$ and 0.25×10^{-4} g/m; while 1:1 ratio showed $0.3, 0.32, 0.35 \times 10^{-4}$ g/m and 1: 1.25 ratio had $0.42, 0.44$ and 0.45×10^{-4} g/m wear resistance at 1, 2 and 3 Kg, respectively. The weight loss in these composites mainly takes place due to abrasion and frictional heat generated due to sliding.

Figure 1. Effect of change in formaldehyde: phenol ratio on the wear rate of the phenol-formaldehyde composites

Tensile and Compressive strength

As it is evident from the Figure 2, that the composite prepared by phenol-formaldehyde resin formed by formaldehyde: phenol in ratio of 1: 0.75 had better compressive and tensile strength of 780 N and 100 N with extension of 0.09 and 0.09 mm, followed by the ratio 1: 0.5 that had 725 and 90 N compressive and tensile strength with an extension of 0.8 and 0.7 mm, F: P::1:1.5 had 500 and 85 N force at an extension of 0.5 and 0.4mm and F: P with ratio 1: 1 had strength of 300 and 80 N with an extension of 0.7 and 0.3 mm, respectively.

Figure 2. Effect of change in formaldehyde: phenol ratio on the tensile and compressive strength test of the phenol- formaldehyde composites

Characterization

From the above discussion, it is clear that the composites formed in the ratio 1.0: 0.75 of formaldehyde: phenol of their molecular weight had better mechanical properties. So, these composites were subjected to characterization.

Infra red analysis

The IR spectrophotometer shows significant peaks at 3422 cm^{-1} due to -OH , 2920 cm^{-1} of CH_2 stretch, $\text{C}=\text{C}$ stretch at 1609.9 & 1508.6 cm^{-1} , 1475.5 cm^{-1} due to CH_2 bend, $\text{C}=\text{C}$ bend at 1374.9 cm^{-1} and 1235 cm^{-1} due to C-O stretch.

X-ray powder analysis

The X-ray powder diffractometer studies depict the d-values, continuous scan were taken between 3 to 40 at 2θ scale at 3 sec scan speed and increment rate of 0.05. Copper was the source of X-ray, Ni filter and scintillation counter mechanism. PMMA holder with zero background was used. Corundum was used to calibrate the system. The small crystal form of sample was subjected to analysis

Figure 3. The XRD pattern of the P-F composite

Scanning electron microscopy

Since the composite had non conducting behavior so, they it was gold plated to have an impact. The Figure 4 clearly shows the well packed smooth surface of the composite.

Figure 4. The P-F resin

Physico-thermal, mechanical evaluation

The formaldehyde: phenol in 1: 0.75 after being characterized were subjected to study their physical, thermal and mechanical behavior.

Viscosity and the pH

The viscosity of the resin was tested at different temperatures as it varies with the change in temperature as shown in Table 1.

Table 1. The Viscosity of the P-F resin at different temperatures.

Sr. No.	Temperature °C	Viscosity, Centipoise
1	40	120
2	50	100
3	60	90
4	70	110
5	80	150

The pH of the resin was found to be 8.5.

Thermal study

The each sample of weight 6.0 mg was taken into the silica crucible for TGA / DTA analysis in air with temperature ranging from 50 to 1000°C. The heating rate of the sample was 10°C per min and scanned for 60mins. Air was taken as the reference for zero and the results, Delta-M (mg) *vs.* temperature (°C) in case of TGA and DTA signal (μV) *vs.* temperature (°C) in case of DTA, were obtained.

The degradation occurs in various forms as deacetylation, dehydration, decarboxylation and chain scissions *etc.* The TGA /DTA results as shown in Table 2 depicts the high initial and final decomposition temperature *i.e.* IDT and FDT which signifies the higher thermal stability. The major weight loss is between 500 to 551°C. TGA results have been supported by the DTA results. In DTA, there are exothermic peaks, the first transition hump at 527°C revealed the dehydration, adsorption and oxidation from the semi-crystalline host and the sharp major peak at 551°C signifies the irreversible dissociation of the crystallites whereas the small peak at 560°C may be due to fusion.

Table 2. The thermograms results of the TGA/DTA studies of the P-F resin.

Sample	IDT	Wt. loss	FDT	Wt. loss	% Residue	DTA peaks °C (μV)
PF resin	500	26.61	600	72.41	0.16	527(7.3), 551(14.4), 560(10.5)

Mechanical studies

The MOR, MOE and SP have been shown in the Table 3 which clearly show the increase in the mechanical strength 8.40. When the P-F composite was tested for its hardness it was found to be brittle.

Table 3. The mechanical evaluation of the P-F composite

Sample	MOR N/mm ²	MOE N/mm ²	SP N/mm ²
P-F composite	43.20	496.12	38.40

Conclusion

The phenol-formaldehyde resin has immense applications in the development of latest technologies and one of the best uses is to prepare the composites. These composites not only have better physico-chemical, mechanical and thermal properties for domestic and industrial application avenues but can further be improved by reinforcing them with natural fibers to incorporate high strength and avail better use of waste biomass

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