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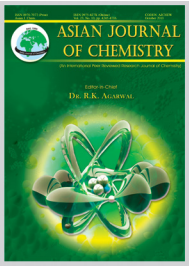

NOTE

Studies on Mechanical Properties of Schiffs Base Reinforced Epoxy Composites

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Composites obtained from filler reinforcement of natural sources shows improved mechanical properties and impart biodegradation to the polymer. In the present work Schiffs base obtained from the oxidized dialdehyde product of guar gum and 3,5,-dimethyl aniline was used as reinforcing material for epoxy resin. The mechanical properties of virgin polymer were compared with composite with different filler concentration. It shows improvement in tensile, flexural, impact properties up to 3 % concentration of fillers, further increase in concentration shows decrease in the values could be due to the agglomeration of particles in the polymer matrix.

Key Words: Guar gum, Periodate oxidation, Schiff's base, Composite, Mechanical properties.

Guar gum is a non-starch polysaccharides (NSPs), obtained from the endosperm of leguminaceae plant, *Cyamposis tetragonalobus*. It is a heteropolysaccharide consisting of a linear chain of (1→4)-linked β-D-mannopyranosyl residues to which are attached varying proportions of (1→6)-linked α-D-galactopyranosyl groups as single unit side chains[1,2]. Oxidation of polysaccharides results in aldehyde and acid, periodate oxidation characteristic for *vicinal* glycols it involves the formation of complex and yield dialdehyde derivative. It is characteristic for polysaccharides to know the linkage between the rings, it selectively cleaves the C_2-C_3 bond of glycosidic linkage to form dialdehyde derivative[3].

Schiffs base are formed from the reaction of aldehyde with primary amines. Schiffs base mainly used in anticorrosive coatings for its inhibiting activity of microbes[4]. It can be blended with resin to impart corrosive activity. Composites formed by reinforcement of fillers, fibres or nanoparticles to polymer resin for improved properties of polymer[5]. In the present work Schiffs base formed from dialdehyde galactomannan, oxidized product of guar gum and 3,5-dimethyl aniline used as reinforcement for epoxy resin. Various concentration of filler prepared by weight per cent of the filler to polymer, the following concentrations were prepared 1, 3 and 5 % *viz.*, the mechanical properties of the virgin polymer and filler reinforced composite were determined and compared in detail.

Guar gum was obtained from Sd fine chemicals, India. Sodium metaperiodate was obtained from Qualigens fine chemicals, Mumbai, India. 3,5-Dimethyl aniline was obtained from Fluka Chemie Switzerland. Epoxy resin GY250 representing diglycidyl ether of bisphenol-A (DGEBA) with epoxy equivalent weight (EEW) 180-190 was used for our study. HY951 (Triethyl tetra amine) curing agents were obtained from Huntsman (India). All the reagents used were of analytical grade.

Preparation of dialdehyde galactomannan: Dialdehyde galactomannan was synthesized according to the reported procedure[3]. Guar gum was dispersed in 500 mL of distilled water by stirring it with 1000 rpm for 0.5 h and the dispersion has been kept overnight for complete swelling of guar gum. Sodium metaperiodate was added to the dispersion while stirring with a magnetic stirrer. The reaction was performed in the dark at 35 ºC for 48 h. The reaction mixture was poured into three volumes of *t*-butyl alcohol, dialdehyde product was precipitated out, it was filtered dried and again redissolved in water to remove soluble impurities and precipitated the pure dialdehyde galactomannan with *t*-butyl alcohol.

Preparation of Schiffs base: Purified dialdehyde product was dissolved in 50 mL of methanol and stirred at room temperature for 1 h. Pre-dissolved 3,5,-dimethyl aniline in methanol was added in the ratio of 1:2 (v/v). This reaction was kept for overnight stirring, yellow colouration reveals the formation of imine. The product was concentrated in a rotatory evaporator under vacuum and washed with water to remove the unreacted dialdehyde galactomannan if any, filtered and dried at room temperature.

TABLE-1
MECHANICAL PROPERTIES OF PURE EPOXY AND SCHIFF'S BASE REINFORCED COMPOSITE

System	Tensile strength (Mpa)	Tensile modulus (Gpa)	Flexural strength (Mpa)	Flexural modulus (Gpa)	Impact strength
Epoxy	320	3.8	402	8.5	12.3
Epoxy + 1 % Schiffs base	394	4.3	456	9.1	13.3
Epoxy + 3 % Schiffs base	435	5.2	498	10.1	16.7
Epoxy + 5 % Schiffs base	423	4.9	487	9.7	15.9

Preparation of composite: Composite was prepared by dispersing various percentage of Schiffs base, the mixture was sonicated for 0.5 h, then curing agent was added in the ratio 2:1. The mixture was poured into preheated metal mold at room temperature for 24 h. The composites are further cut into required dimension to study the mechanical properties.

Mechanical properties: The tensile and three-point flexural tests for epoxy and composite samples were performed in Zwick-Z010 universal testing machine in accordance with ASTM D3039 and ASTM D790 standards, respectively. The cross-head speed for tensile testing was 2 mm/min. Flexural testing was conducted at 2.8 mm/min. The dimensions of the specimens for tensile and flexural testing were 250 mm × 25 mm × 6 mm and 120 mm × 13 mm × 6 mm (length × width × thickness), respectively. Span lengths for tensile and flexural specimens were 150 mm and 100 mm, respectively. Unnotched charpy impact tests were carried out using ALSA pendulum impact tester. The tests were conducted at impact velocity of 3.8 m/sec. The dimensions of impact specimens were 100 mm × 10 mm × 4 mm (length × width × thickness). The gage length was 80 mm. Four samples were tested for each sample group. The average values were reported including standard deviations. Edges of samples were trimmed to avoid stress concentration.

Dialdehyde galactomannan was characterized by FT-IR the introduction of new carbonyl stretching frequency at 1725 cm^{-1} and sharpening of hydroxyl peak indicates the formation of aldehydic functionality. The formation of imine is confirmed from the shift in λ_{max} and FT-IR of Schiff's base shows a strong absorption band at 1639 cm^{-1} attributed to the -CH=N- vibrations characteristic of imines which is not observed in dialdehyde galactomannan. The broad peak at 3405 cm^{-1} corresponds to the stretching vibration of -NH and -OH bonds shifted to higher frequency and changed from doublet band of $-NH_2$ to a single band for -NH indicates the formation of Schiffs base from dialdehyde galactomannan and 3,5-dimethyl aniline. The results obtained for mechanical properties of virgin epoxy, 1 %, 3 %, 5 % Schiffs base reinforced composite were summarized in Table-1, shows that reinforcement imparts considerable increase in the tensile, flexural and impact upto 3 % after that sudden decrease in values for 5 % and the same as seen in increased percentage this could be explained by the agglomeration of fillers in the polymer matrix and non-uniformity in dispersion at higher concentrations.

Conclusion

Schiffs base was synthesized and functional groups were endorsed by FT-IR spectroscopy. The results proves it can be used as reinforcing material in polymer matrix, the increased mechanical properties at lower percentage and decreases at higher concentrations shows it has similar property of natural fillers fibres. The biodegradability has to be studied to ensure biodegradation of the product.

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