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Research Paper

INTERACTING BLENDS OF COMMERCIAL UNSATURATED POLYESTER RESIN WITH ACRYLATED KETONE (ACETONE) RESIN

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The acetone-formaldehyde resin designated as Ketone Resin (KR) was prepared with more hydroxy functionalized groups. It was then acrylated by treatment with acryloyl chloride. The resultant acrylated KR designated as AKR was characterized by number of double bonds and IR spectral study. The blending of AKR with commercial unsaturated polyester (USPE) was carried out with various proportions. The curing of these USPE-AKR blends was carried out using Benzoyl peroxide (BPO) as a catalyst and N, N'-Dimethyl aniline (DMA) as a promoter. The glass fiber reinforced composites (i.e., laminates) of these USPE-AKR blends were fabricated. The mechanical and chemical resistance properties of the glass fiber composites have also been evaluated. The unreinforced cured samples of the USPE-AKR blends were also analyzed by thermogravimetry (TGA).

Keywords: Ketone resin, Acrylation, Unsaturated polyester resin, Glass fiber reinforced composites, Mechanical property, Thermo gravimetric analysis

INTRODUCTION

Polyesters (Bjorksten Research Laboratories, 1956; Karshak and Vinogradova, 1965; Goodman, 1988) are one of the most versatile synthetic copolymers. Polyesters are produced in high volume that exceeds 30 billion pounds a year worldwide (Goodman, 1988). They are widely used commercially as fibres, plastics, composites and for coatings applications too (Goodman and Rhys, 1965; Goodman, 1968; and

Morgan, 1965). They are heterochain macromolecules that possess carboxylate ester groups as an integral component of their polymer backbones. Polyesters have received a great deal of attention since the early work of Carothers, who initiated study on many step-growth polymerizations (Carothers, 1929). Carothers worked with aliphatic straight-chain polyesters, which were soluble in organic liquids, low melting and had poor resistance to hydrolysis. These

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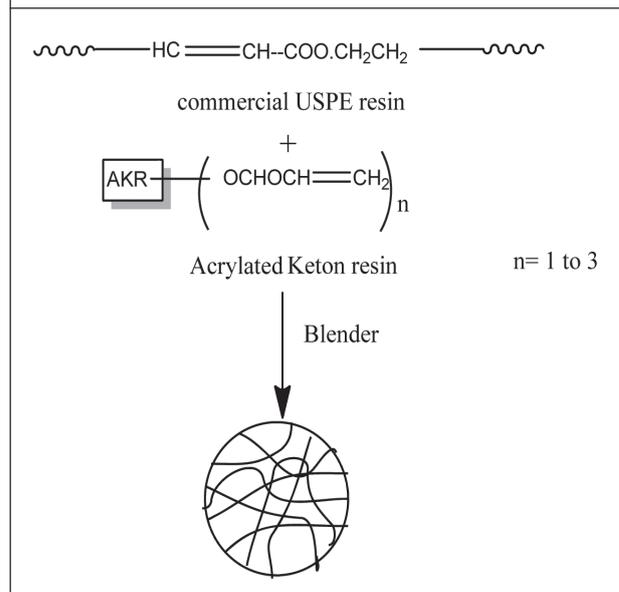
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polyesters were not used as textile fibers (Goodman, 1968;). The extension of these concepts later led to the discovery of nylon-6, 6 in 1935 and Whinfield and Dickson developed poly (ethylene terephthalate) (PET) in 1941 (Whinfield, 1946). A partially aromatic organic structure was necessary to increase melting temperature (T_m) above 250°C . A large number of polyester structures have found use in industry today which displays a wide variety of properties and applications. More detailed discussion may be found in a number of excellent books and reviews (Goodman, 1988; Goodman and Rhys, 1965; and Morgan, 1965).

Among the thermosetting resins such as, phenolic, amino, epoxy, and unsaturated polyester (UPE) (i.e., cross linkable) resins, the UPE resin are the most important because of their ease of formation, transparency, thermal stability and good thickness. They are synthesized in two steps. In first step, a low molecular weight polyester of about 700-4000, containing $\text{C}=\text{C}$ is synthesized and is then dissolved in vinyl monomer and is stabilized with inhibitor. They are based on several components that can be combined in different ratios and their choice is decided by the properties needed for particular application (Loewing and Weidmann, 1839; Crafts, 1862; Meyer V Ber. 1886) at normal temperature UPE resin can be cured with peroxides and accelerator. Cured UPE resin is thermosetting, infusible materials. They are mostly used as a matrix material for fabrication of glass fiber composite. The major application of UPE resin in automobile, marine and construction industries (Berenbaum *et al.*, 1967). Common applications are boat hulls, sports car bodies, truck cabs and public transport vehicle. They are also used for making decorative furniture

castings, ornamentals stone, buttons, tray, bowling balls, safety helmets, chemical processing equipment, missile shells and rocket motor cases. Aliphatic ketone both cyclic and acyclic and aryl alkyl ketones are reported to afford resinous product on condensation with formaldehyde mostly in presence of alkali. These resins are known as Keton Resins (KR). Aliphatic ketone like acetone, methyl ethyl ketone, cyclohexanon, α -methyl cyclohexanon and cyclopentanon are reported to afford such products. Aryl alkyl ketone like acetophenone are also reported to afford such resins. Like the other resins prepared using formaldehyde as a condensing monomer, the keton resins have complicated structures. There is some indirect evidence in favors of the major part of ketone resins. Although these resins have been known for long time, new type of resins and new area of application are always being found. In paint and coating, as in many other areas, these resins are used in combination with other binding agents, plasticizers, pigments, and process materials.

Scheme 1: Diagram for USPE-AKR Resins Blending



They are also used in printer and copier technology, adhesives, binding agent for corrugated cardboard, molding sand, laminateds (Iwamoto *et al.*, 2010). Looking to the properties of both USPE and KR resins, it was thought to blend both the resins to form new system. Hence, the present paper comprises the study of blends of USPE and acrylated KR resins. The work is given in Scheme 1.

Table 1: Charachtrisitc of USPE

Parameters	Unit	Test Result
Appearance	-	Clear Pale Yellow Liquid
Sp. Gravity @ 25°C	-	1014
Viscosity @ 25°C # 2, 30 RPM	Cp	450
Gel time @ 25°C	Min.	18
Peak Exotherm	°C	159
Volatile Content	%	36
Acid Value	mg KOH/gm	22

MATERIALS AND METHODS

Commercial unsaturated polyester resin (USPE) was obtained from Kemrock Industries and exports Ltd., Halol. The grade characteristics are as showing in Table 1. The continuous Glass fibers used for composites fabrication were obtained from Kemrock Industries and exports Ltd., Halol. All the other chemicals used were of laboratory grade.

SYNTHESIS ACETONE-FORMALDEHYDE RESIN

A mixture of acetone (1-mole), paraformaldehyde (0.66-mole) and 20 mL of 5% alcoholic KOH was heated at 40-45°C with continuous stirring for 15

min the resultant mixture were cooled at room temperature. The so called resin obtained was in the form of colorless thick liquid. It was neutralized by formic acid and dried azeotropically. The yield was 90% (Patel and Dixit, 1998a; and b).

SYNTHESIS OF ACRYLATED KETONE RESIN (AKR)

Acrylation of ketone resin has been carried out using acryloyl chloride as a reagent. The general procedure is as follows:

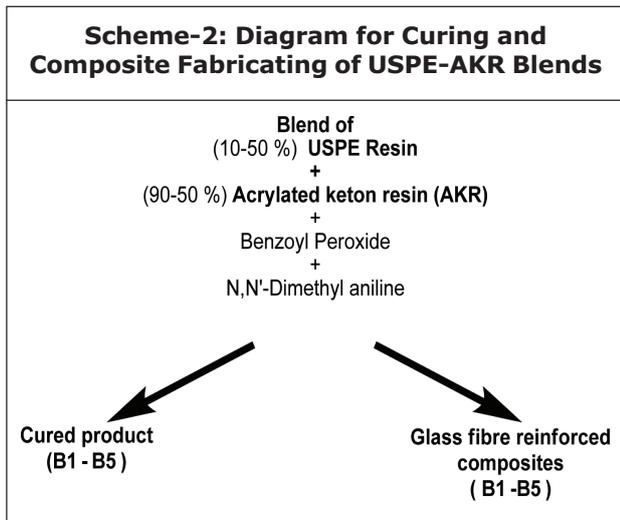
Ketone resin and acryloyl chloride both were taken in 1:2 mole ratios, 1.008 g of hydroquinone and 1.67 g imidazole were placed. The reaction content was heated in an oil bath at 100°C while stirring continuously. The reaction was carried out approximately for 7-8 h still the acid value was found to be between 2-5 (Patel and Patel, 2009a; and b).

BLENDING AND CURING OF USPE WITH AKR RESIN

The blending of USPE resin and AKR resin along with Benzoyl-peroxide (BPO) as a catalyst and N, N'-Dimethyl aniline (DMA) as promoter (Scheme 2) was carried out. The blending composition and shows in Table 2. The resulting solid hard materials (B1-5) were designated as unreinforced material.

Table 2: Blending Proportions of AKR and Commercial USPER

AKR	% of USPR	SAMPLE Designation
10%	90	B1
20%	80	B2
30%	70	B3
40%	60	B4
50%	50	B5



COMPOSITE FABRICATION

The composites were prepared using continuous glass fiber mat with a glass fiber: resin ratio of 60:40. 0.5% benzoyl peroxide as a catalyst based on the total weight of the blend Resins with DMA as promoter. This syrup was well stirred for about 5 min. The syrup was then applied to a 191 mm x 102 mm glass fiber ply. The prepregsplies were stacked on top of one another and pressed between steel plates coated with a Miler-film release sheet and compressed in a flat platen under 70 psi pressure. Keep it at room-temperature at their respective curing temperatures. The composites obtained were then machined to final dimensions.

MEASUREMENTS

Curing

A curing study of all the USPE- AKR blends Resin (B1-5) with without continuous glass fiber was carried out using Benzoyl peroxide as a catalyst, N, N'-Dimethyl aniline (DMA) as a promoter at room temperature.

THERMO GRAVIMETRIC ANALYSIS

Unreinforced cured blend samples were subjected to Thermo Gravimetric Analysis (TGA) on NETZSCH STA449F3 thermo gravimetric analyzer in nitrogen atmosphere at a heating rate of 20°C min⁻¹at GIRDA laboratory, Baroda. The TG thermo grams are show in Figure 1 and data are furnished in Table 3.

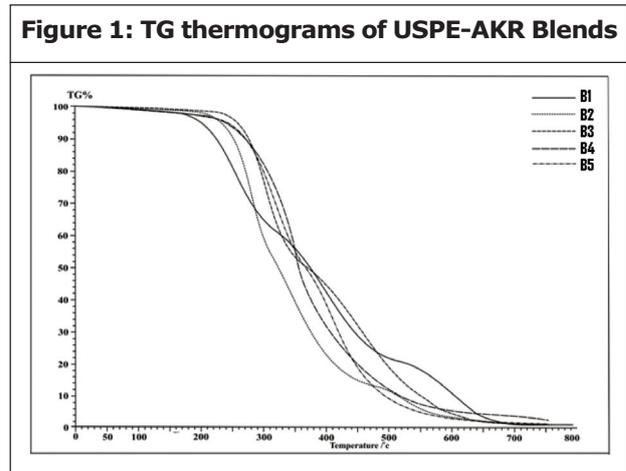


Table 3: TGA of Unreinforced Cured Sample of USPE-AKR Blends

SAMPLE Designation	% Weight loss at various Temp, °C From TGA						
	100°C	200°C	300°C	400°C	500°C	600°C	700°C
B1	0.956	5.096	35.032	57.325	78.344	89.172	98.727
B2	0.637	1.911	40.765	77.389	88.536	96.816	98.727
B3	0.637	1.911	22.930	55.415	80.892	95.542	98.727
B4	1.274	2.867	18.790	68.153	88.536	94.586	96.179
B5	0.956	2.867	20.383	61.784	90.765	97.134	98.408

COMPOSITE CHARACTERIZATION

Chemical Resistance Test

The resistances against chemicals of the composite were measured according to ASTM D 543. The specimen dimensions were 20 mm x 20 mm. The chemicals used for the study were H₂SO₄ (25% v/v), HCl (25% v/v), NaOH (25% w/v) ethanol, acetone, THF and DMF. The test was performed by dipping the composite sample in 100 mL each of the above reagents for 7 days at room temperature. After 7 days the specimens were taken out from the reagents and after drying, they were examined for the percentage change in thickness and weight their results are furnished in Table 5.

Mechanical Testing

All mechanical testing were performed using three test specimens and their average results are furnished in Table 5.

1. The Tensile strengths was measured on a UTM of Machine no. STS-165 at room temperature according to DIN EN 2747.
2. The Flexural Strengths was measured on a UTM of Machine no. STS-165 at room temperature according to DIN EN 2746.
3. The Inter Laminae shearing strengths were measured on a UTM of Machine no. STS-165 at room temperature according to ASTM D 3846.

RESULTS AND DISCUSSION

Having unsaturation in both the AKR resins and unsaturated polyester resin. These USPE resins (E1-5) are viscous resins which can be cured by adding benzoyl peroxide as a catalyst and N, N'-

Dimethyl aniline (DMA) as promoter at room temperature. The DSC study of all the blends did not give the proper thermo gram. The thermo grams show only steeper plot from the initial temperature. Thus may be due to fast curing reaction. Thus the DSC study has not been attempted. So all the blends were cured at room temperature for prolong time, while the curing of all the blends exists rapidly in the temperature range of 70-120°C. Thus, all the blends were cured manually below 120°C. This indicated that cross-linking took place rather than homo polymerization. The unreinforced cured samples of USPE-commercial UPR blends (B1-5) were analyzed by TGA. Examination of the TGA data in Table 4 reveals that the cured samples started their degradation at about 150°C and their initial weight loss was about 3%. This small weight loss might have been due to either insufficient curing of components used or due to the catalyst used. A weight loss of about average 20% was found at 300°C. However, the rate of decomposition increased very rapidly in the range of 300°C to 450°C and the products were lost completely beyond 800°C. The glass fiber reinforced composites of all USPE-commercial UPR blends (B1-B5) were also prepared curing at the room temperature. These composites were also analyzed for their chemical resistance and examination of results furnished in Table 4, reveals that the composites have good resistance to common solvents like ethanol, acetone and THF. The composites were also analyzed for their mechanical properties and their results are furnished in Table 5. Examination of the results reveals that the composites have good mechanical strength.

Table 4: Chemical Resistance Properties of Glass Fiber Reinforced Composites of USPE-AKR Blends

Resin System	Change in	Reagents						
		25% H ₂ SO ₄	25% HCl	25% NaOH	R-Spirit	Acetone	THF	DMF
B1	Thickness	5.21	3.15	3.79	0.80	0.86	2.60	5.26
	Weight	4.33	2.61	2.70	0.56	0.61	1.99	4.37
B2	Thickness	5.12	3.15	3.79	0.80	0.86	2.60	5.26
	Weight	4.33	2.61	2.70	0.56	0.61	1.99	4.37
B3	Thickness	5.13	3.14	3.80	0.81	0.85	2.61	5.25
	Weight	4.33	2.61	2.70	0.56	0.61	1.99	4.37
B4	Thickness	5.14	3.15	3.81	0.80	0.86	2.62	5.27
	Weight	4.33	2.61	2.70	0.56	0.61	1.99	4.37
B5	Thickness	5.12	3.15	3.79	0.80	0.86	2.60	5.26
	Weight	4.33	2.61	2.70	0.56	0.61	1.99	4.37

Table 5: Mechanical Properties of Glass Fiber Reinforced Composites of Styrenated UPEAs-Commercial UPR Blends

Sample Id	Tensile Strength Mpa	Flexural Strength Mpa	Inter Laminar Shearing Strength Mpa
B1	160.5	229.5	35.5
B2	168.5	231.5	35.5
B3	176.0	244.0	30.5
B4	149.5	225.0	32.5
B5	135.0	261.5	40.5

REFERENCES

- Berenbaum M B, Broderick E and Cheristina R C (1967), Thiokol Chemical Corp., US Pat. 3,317, 486, 1967.
- Bjorksten Research Laboratories (1956), *Polyesters and Their Applications*, Reinhold, New York, Unsaturated Polyester Resin for Specialty Applications 195.
- Carothers W H (1928), *J. Am. Chem. Soc.*, Vol. 51, No. 2548.
- Crafts J M (1862), *Ann.*, Vol. 124, No. 110.
- Goodman I (1968), 2nd Edition, Vol. 16, p. 159, Wiley, New York.
- Goodman I (1988), *Encyclopedia of Polymer Science and Engineering*, 2nd Edition, Vol. 12, Wiley, New York.
- Goodman I and Rhys J A (1965), *Polyesters, Saturated Polymers*, Iliffe Books, London, Vol. 1.
- Iwamoto T, Nagai T and Sawa T (2010), *International Journal of Solids and Structures*, Vol. 47, No. 2, pp. 175-185.
- Karshak V V and Vinogradova S V (1965), *Polyesters*, Pergamon, New York.
- Loewing C and Weidmann S (1839), *Ann.*, Vol. 46, No. 81.
- Meyer V., *Ber.* 19,325,1886.

12. Morgan P W (1965), *Condensation Polymers, By Interfacial and Solution Methods*, Inerscience Publishers, New York.
13. Patel H S and Dixit B C (1998), *International journal of polymeric materials*, Vol. 42, pp. 309-318.
14. Patel H S and Dixit B C (1998), *International journal of polymeric materials*, Vol. 40, pp 29-39.
15. Patel H S and Patel B K (2009), *International journal of polymeric materials*, Vol. 56, pp. 312-321.
16. Patel H S and Patel B K (2009), *International journal of polymeric materials*, Vol. 58, pp. 654-664.
17. Whinfield J R (1946), *Nature*, Vol. 158, No. 930.



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