Uncured and Cured State Properties of Fly Ash Filled Unsaturated Polyester Composites

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ABSTRACT: Physical properties of fly ash filled unsaturated polyester composites in both uncured and cured states have been studied with special reference to the effect of degree of loading, nature of filler surface, and surface treatment of filler. The effect of filler surface on curing and oil absorption characteristics of filler were also examined. In the uncured state, sedimentation rate test and viscosity measurement for fly ash reinforced composites were performed. For cured fly ash filled unsaturated polyester composites, tensile properties decreased with the addition of fly ash particles whereas surface treatment led to improved mechanical properties and resistance to swelling. In terms of dynamic mechanical thermal analysis, effects of both filler and surface treatment on loss factor (tan δ) were discussed. Tan δ value and damping temperature range increased to the 15% fly ash addition. The composite having 15% unsilanized fly ash was found to have the highest tan δ and damping temperature range together with maximum performance in terms of tensile properties and swelling behavior. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 77: 1128–1136, 2000

Key words: unsaturated polyester composites; fly ash; surface treatment; static–dynamic mechanical properties; swelling behavior

INTRODUCTION

Fossil fuels are used in modern power plants throughout the world to produce electrical energy. The inorganic residue that remains after coal is burned is known as fly ash. These by-products rapidly accumulate and cause enormous problems of disposal unless a way can be found to utilize them through resource recovery programs. Fly ash is the finely divided coal combustion by-product collected by electrostatic precipitators from the flue gases.[1]

Fly ash has been used as a source of spherical fillers for some years mainly in civil engineering studies especially for the production of lightweight and high strength concrete.[2] It has also been used in some resin systems. Srıvastaka and Shembekar[3] have evaluated tensile and flexural properties of epoxy resin filled with fly ash particles and observed that the tensile strength of epoxy resin filled with fly ash decreases, whereas the fracture properties and the modulus of elasticity increase with increasing percentage of fly ash. Therefore, it is advisable to use fly ash when the void formation cannot be controlled. Banko et al.[4] have performed a study to investigate the effect of fillers on the temperature relaxation behavior of unsaturated polyester based on recycled poly(ethylene terephthalate). It has been reported that fly ash particles increased tensile modulus of the resin at all temperatures whereas they reduced the tensile peak strength of the composite via disrupting the material homogeneity, which then resulted in contribution to the creation of stress concentrations in the vicinity of the filler

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particles. Scanning electron microscope observation of fractured fly ash–polyester composites has been reported with some results indicating that the impact strength decreases with increase in volume fraction of fly ash and this was explained by poor resistance of added filler to impact.[5] In another study, the influence of variation of load and sliding distance on the abrasive behavior of fly ash glass fiber-reinforced polyester composites were reported by Chand and Gautam.[6] It was found that minimum wear loss was obtained with lower load on material in the case of the highest fly ash-loaded sample, then wear loss increased with increasing the load on the sample further for the same sample.

This article reports the detailed uncured and cured state property optimization in fly ash filled unsaturated polyester resins in terms of easy processibility, maximum vibrational damping, optimum mechanical properties, and minimum swelling in hydrocarbon media. To enhance the mechanical properties of resultant composites, the surface of fly ash was treated with a functional coupling agent so that "coupling action" between the surface of the filler and polymer matrix could take place. The physical properties such as dynamic, static-mechanical, thermal, and swelling behaviors of the composites in the cured state were all examined as a function of degree of loading, nature of filler surface, and surface treatment. Special attention was given also to the investigation of viscosity and sedimentation rate of composites in the uncured state.

EXPERIMENTAL

Materials

Unsaturated polyester resin including 30% styrene was the product of Cam Elyaf A. Ş., Istanbul, Turkey, with a brand name of Neoxil-NX188. Fly ash (ca. 0.1 μ) which has almost 17% hollow spheres by weight was supplied from Ferro-Chrome Production Plant of Antalya, Turkey. Methyl ethyl ketone peroxide, which was in the form of 50% solution, as initiator and cobalt naphthanate (Co-naphthanate), which contains 6% cobalt, as promoter were purchased from Aldrich and used as received. The silane coupling agent, A-174, 3-methacryloxy propyl trimethoxy silane was supplied from Union Carbide, UK. Acetone and ethanol were the product of Merck (Darmstadt, Germany) and were used as received.

Preparation of the Composites

Application of the coupling agent was done as follows: the silane coupling agent (4% of filler, by weight) was added to the solution of distilled water and ethanol (20% of distilled water, by weight) previously adjusted to pH 5.5. The filler was mixed with the coupling agent solution and stirred with a high shear mixer for 1 h. The mixture was filtered on a Buncher filter. The treated filler was then dried at 120°C for 24 h.

All composites were prepared by adding different weight fractions of unsilanized and silanized fly ash to unsaturated polyester resin which has 0.25%, by weight of resin, Co-naphtenate promoter and 30%, by weight, of resin, containing styrene. After stirring the mixture with a high shear mixer, 2%, by weight of resin, methyl ethyl ketone peroxide initiator, which decomposes into free radicals at room temperature, was added just before molding and mixed. Then the mixture was directly poured into glass molds having necessary dimensions for physical tests, and left for 24 h to complete curing reaction and give composites.

The compounding of unsaturated polyester resin was performed by the use of both unsilanized and silanized fly ash fillers in 10, 15, 20, 25,and 40 weight percentages. PEC15FA and PEC15FAS indicate polyester composites filled with 15%, by weight of resin, unsilanized and silanized fly ash fillers, respectively. Trial mixes of the fly ash with the polyester resin determined that a practical limit for the fly ash content was no more than 25% by weight. Beyond that limit, the workability of the fresh mix was decreased.

Physical Testing of Composites

Oil absorption value of fly ash filler was determined by the standard method of the Institute for Turkish Standards, TS 2583.[7] This standard determines the absorption of refined linseed oil by the pigments. A glass plate with dimensions of 30 $\times$ 40 cm in which a definite amount of filler was put and a 10 mL buret divided by 0.1 mL and filled with refined linseed oil were used. Then the oil in buret was dropped onto the filler, until a smooth and soft paste was formed. The oil absorption value was obtained as the ratio of the volume of absorbed oil to the weight of filler (*V*mL/100 g filler).

To determine how fast fly ash filler, both in unsilanized and silanized forms, is settled down in well-mixed unsaturated polyester resin solution, a 25-mL graduated cylinder was used to follow the sedimentation rate of filler in unsaturated polyester resin solution before curing reaction. For this purpose, a certain amount of the selected liquid composite was placed in the abovementioned graduated cylinder and the volume of supernatant solution was measured at different time intervals. The slope of the volume versus time graph was accepted as the sedimentation rate.

Viscosity measurements of unsaturated polyester resin solutions filled with fly ash were conducted with a Haake Viscotester at 28°C. These measurements were performed with filled composites as well as with unfilled matrix itself (neat polyester) after mixing the solution at high shear for 15 min.

Both cure kinetic of unsaturated polyesters and the effect of fly ash surface on curing rate were examined with Rheometric Scientific Differential Scanning Calorimeter (DSC). Samples of 25–35 mg were scanned isothermally at 40°C for 50 min. Analysis was conducted under nitrogen gas with a gas flow rate of 10 mL/min.

The swelling behavior of the composites was followed with a traveling microscope (Geartner 7109-46). The samples' dimensions of 2 × 5 × 2 mm were placed in acetone at room temperature for 34 h. The swelling ratio, q, was defined as the ratio of the volume of the swollen to that of unswollen composites. The swollen lengths were measured at definite times with the help of the traveling microscope and 0.001-mm sensitivity. Two samples for each composite were studied to obtain average swollen length. The swelling ratio was obtained by the following equation[8]:

$$q = V/V_o = (L/L_o)^3$$

where V_o and V are the volumes of unswollen and swollen composites, respectively. L_o and L are the lengths of unswollen and swollen composites, respectively.

Tensile stress–strain data were determined using a Zwick Universal Testing Machine (Model 1446) at room temperature with strips having approximate dimensions of 15 × 80 × 2 mm and at 50 mm/min drawing rate.

The dynamic mechanical thermal analysis of the samples was performed using a Polymer Laboratories Dynamic Mechanical Thermal Analyzer (DMTA). Sample dimensions of 20 × 10 × 2 mm were scanned at the frequency of 1 Hz and heating rate of 5°C/min under nitrogen gas atmosphere. Dynamic moduli (E', E'') and tan δ values were determined in a temperature range varying between 20 to 120°C.

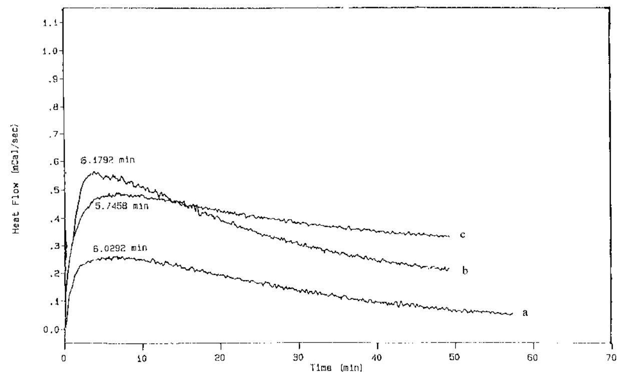

Figure 1 DSC thermograms of (a) neat polyester, (b) 20% unsilanized PEC20FA, and (c) 20% silanized PEC20FAS, fly ash filled composites.

RESULTS AND DISCUSSION

Characterization of Fly Ash Surface

It has long been recognized that the surface characteristics of particulates and fibrous reinforcement greatly influence the curing behavior of thermosetting resins. It is reported that free radicals coming from radicalic initiator are able to form a charge transfer complex with inorganic oxides on the surface of glass beads without surface treatment, inhibiting the curing reaction.[9] This causes a decrease in both the conversion and reaction rates of polymerization while cure time increases.

In this study, effect of fly ash filler on cure time of the selected polyester composites, which is recorded as the time required for the mass to attain a rubbery consistency after initiator addition, were examined by using DSC technique. Cure time of neat polyester, 20% unsilanized and 20% silanized fly ash filled composites are given in Figure 1.

As can be seen from Figure 1, the addition of 20% unsilanized fly ash to the polyester resin causes a somewhat lower curing reaction rate than the neat polyester which is probably due to diffusion dominated curing process resulting from high viscosity filled polymeric medium[10] and inhibition effect of charge transfer complexes occurring between free radicals and inorganic oxides on the filler surface.[9] But mainly it can be safely stated that fly ash addition does not greatly alter the cure time of the polyester composites.

On the other hand, silanized fly ash filler in the same composition (PEC20FAS) causes little decrease in cure time compared with unsilanized counterpart (PEC20FA). This indicates that the use of silane coupling agent to modify the surface of the fly ash has enhanced the curing reaction of the resin. It was also speculated that the inhibition of the curing reaction of unsaturated polyester resin would be less, and thus the curing reaction of the resin would be enhanced, if the surface of the glass beads is treated with a coupling agent. Thereby, the formation of charge transfer complex is inhibited by the surface coverage that means a decrease in cure time.[11,12] Similar observations, increase in the rate of vulcanization of mica reinforced polybutadiene composites and glass bead filled polyester composites with surface silanization, were also reported by Nugay et al.,[13] Lee and Han,[11] and Shieh and Hsu,[12] respectively.

To obtain high conversion in the cure reaction, the unsaturated polyester/styrene mol ratio should be 1 : 2.2. If filler used has a high oil absorption value, it will absorb some styrene around it and cause a weak styrene region, which does not participate in the curing reaction. Therefore, fly ash filler was performed on a standard test. Results indicated that for 100 g filler amount, fly ash absorbed 75 mL. When this number is compared with that of precipitated silicate particulate whose commercial name is Cab-o-sil and oil absorption value is 320 mL, which is undesirable for curing,[7] fly ash was found to have an optimum value.

Finally, it can be safely concluded that fly ash has no negative effect on the curing kinetics of the polyester resin which could have a profound influence on the mechanical properties of the resultant polyester composites.

Characterization of Fly Ash Filled Polyester Composites in the Uncured State

Sedimentation Behavior of Uncured Fly Ash Filled Composites

Sedimentation tests were conducted to determine how fast the filler of liquid moulding resins settle down in mould. It is generally desirable to have low sedimentation rate for homogeneous filler distrubution throughout the final article to have good physical properties.

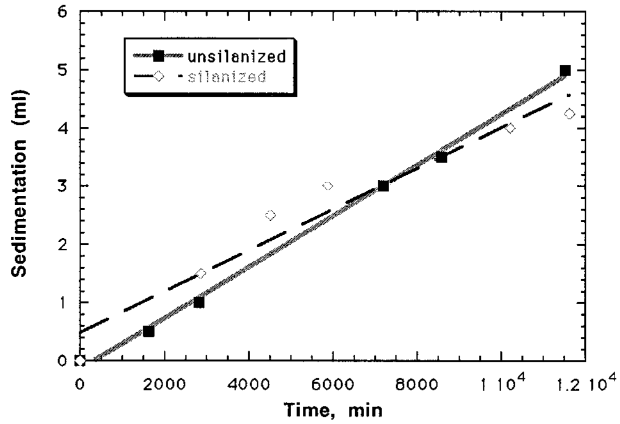

Figure 2 Sedimentation rate of composites having 10% unsilanized fly ash, PEC10FA and 10% silanized fly ash, PEC10FAS.

Sedimentation rate of fly ash filler in unsaturated polyester resin was examined in the uncured state. Figure 2 shows the variation of sedimentation of fly ash particles in polyester with time in the composites having 10% both unsilanized and silanized fly ash, PEC10FA and PEC10FAS, respectively.

It is clear from the Figure 2 that sedimentation rate decreases when fly ash particles are silanized. The more gradual settlement of the silanized fly ashes in the composite may be attributed to both the distribution of the filler particles throughout the polyester matrix and suspension in it for a longer time by the help of silane coupling agent. This is, of course, a major benefit in terms of practical applications.

Viscosity Behavior of Uncured Fly Ash Filled Composites

Fillers strongly influence the flow characteristics of molding compounds. Filler type and amount have a tremendous effect on viscosity which is critical while working with molding compounds.

The variation of viscosity with both unsilanized and silanized fly ash reinforcing are given in Figure 3. As the amount of fly ash in uncured composite increases, viscosity increases. These observations are in agreement with those reported by Han and Lem.[14] In their study, it was found that the viscosity of suspension was increased as either the particulate concentration was increased or the size of particulates was decreased.

As seen from Figure 3, since the viscosity reaches to very high values, especially after 25%

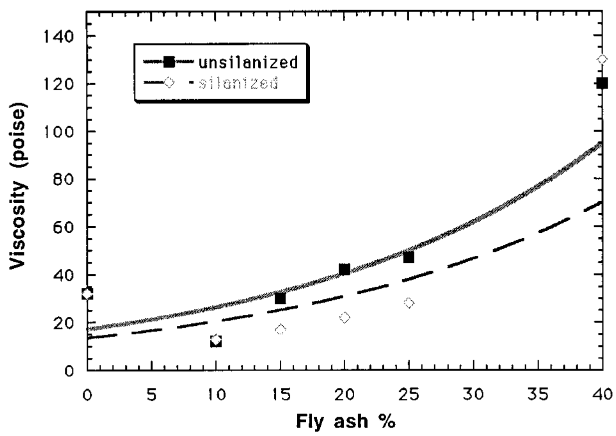

Figure 3 Variation of viscosity of uncured polyester composites with unsilanized and silanized fly ash loading.

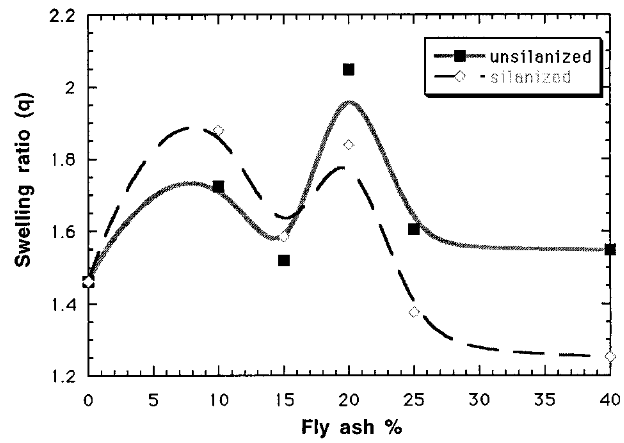

Figure 4 Variation of swelling ratio with unsilanized and silanized fly ash loading in polyester composites.

fly ash loading, the workability of fresh mix decreases for 40% fly ash loaded composition, causing molding of the resin to be difficult. It is well known that too high viscosity results in a short-shot (incomplete filling of the mold cavity), upsetting the balance between the speeds of the flow process and curing process.[14]

On the other hand, silanization of fly ash surfaces causes a decrease in viscosity for 15, 20, and 25% fly ash compositions. It is obvious here that surface treatment inhibits the agglomeration of filler, particle-to-particle contact, and provides perfect dispersion in the system, causing higher loading with no viscosity increase. Similar results were observed also for both unsilanized and silanized $CaCO_3$ particulates in unsaturated polyester resin.[14]

Characterization of Fly Ash Filled Composites in the Cured State

All physical properties of fly ash reinforced polyester composites are summarized in Table I.

Swelling Behavior of Fly Ash Filled Composites

Swelling behavior of a crosslinked polymer in a solvent was investigated to understand the dimensional stability of the crosslinked polymers in solvent medium. Generally, if the solid polymer has a high crosslink density, it will absorb less solvent and then swell in small amounts.

In Figure 4, equilibrium swelling ratio values of composites, indicated in Table I for both unsilanized and silanized fly ash polyester composites were plotted as a function of fly ash loading.

Table I Physical Properties of Fly Ash Filled Polyester Composites

Samples	Stress at Break (MPa)	Elongation at Break (%)	Work Up to Fracture (Nm)	Elastic Modulus (MPa)	Swelling Ratio (q)
Neat	26.35	9.83	1.40	45.00	1.462
PEC10FA	17.50	5.3	0.55	107.04	1.723
PEC15FA	16.25	7.07	0.55	49.01	1.520
PEC20FA	12.00	5.60	0.35	55.48	2.048
PEC25FA	17.05	6.45	0.70	48.03	1.605
PEC40FA	8.00	4.22	0.25	56.22	1.548
PEC10FAS	17.95	5.18	0.50	117.02	1.879
PEC15FAS	21.40	6.82	0.80	90.68	1.585
PEC20FAS	19.00	5.35	0.60	101.23	1.838
PEC25FAS	18.90	4.46	0.45	99.12	1.375
PEC40FAS	12.90	4.445	0.45	106.49	1.252

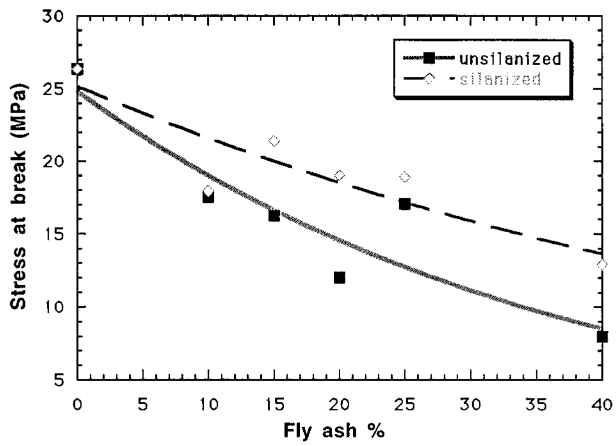

Figure 5 Variation of stress at break values with a concentration of unsilanized and silanized fly ash for polyester composites.

As illustrated in Figure 4, there is a little increase in swelling at small amounts of filler, followed by a sudden decrease up to the composite of PEC15FA. The swelling ratio increases for PEC20FA and then again decreases and remains constant until 40% fly ash loading. It is well known that a polymer network filled with a filler swells to a much lesser extent than the unfilled network.[15] Because fillers have higher modulus than the matrix, they increase the stiffness and rigidity of the polymer and enhance the resistance of the material to solvent by means of preventing the diffusion of the solvent molecules into the polymer.

Surface treatment also affects the swelling behavior. Silanization of fly ash surfaces, as can be seen in Figure 4, leads to a decrease in swelling, especially after 10% fly ash loading. This may be due to both improved dispersion of fillers by the surface coverage and enhanced polymer filler interaction as also reported for mica reinforced polybutadiene composites,[13] mica/silica fume hybrid reinforced polydimethylsiloxane composites[16] and mica/carbon black hybrid reinforced nitrile rubber composites.[15] In this study, although after 25% unsilanized and silanized filler loading, swelling values are considerably low but these compositions exhibit also very high viscosities leading to some difficulties in both mixing and moulding processes.

Static Mechanical Properties of Fly Ash Filled Composites

Figure 5 shows the variation of stress at break or tensile strength of composites reinforced with unsilanized and silanized fly ash.

It is also seen that the addition of unsilanized fly ash generally causes a decrease in tensile strength which may be due to the fact that addition of rigid fine particles to polymers decreases tensile strength of the composite like other fillers such as rice husk ash and ipomoea powder.[17] Similar behavior was observed for tensile strength of epoxy resin filled with fly ash composites,[3] and the decrease in tensile strength was attributed to the presence of voids and formation of air bubbles which not only reduce the stress bearing area but also act as stress raisers, which initiate the cracks.

Effect of silanization on fly ash reinforcement is observed by an increase in tensile strength for each composition as can be seen in Figure 5.

It is well known that if there is adhesion between polymer and filler, tensile strength of the composite increases. If there is no or weak adhesion, tensile strength decreases.[18] Silanization was found to have most beneficial effect especially on PEC15FAS composition via minimizing the effect of voids which then led to maximum tensile strength. Figure 6 presents elongation at break values of fly ash filled polyester composites.

The addition of fly ash makes the composites brittle as is evident from the lower percentage of elongation at break at high fly ash loading compared with neat polymer. In terms of silanized fly ash, little decrease is observed in elongation at break which may be attributed to less elasticity due to the higher crosslinking resulting from methacrylic group of silane coupling agent.

Modulus of elasticity of composites as a function of fly ash loading is given in Figure 7. Elastic

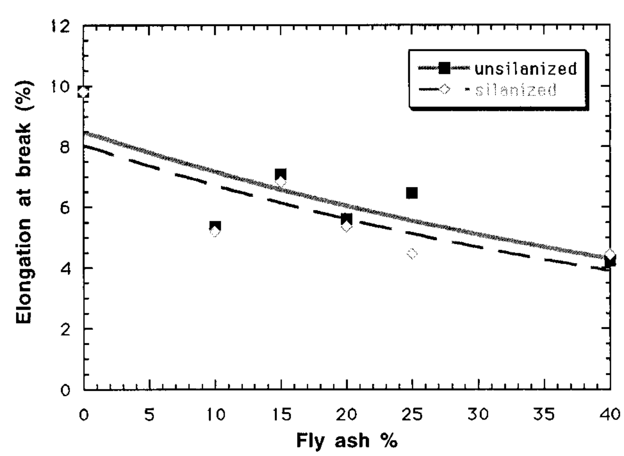

Figure 6 Variation of elongation at break with a concentration of unsilanized and silanized fly ash for polyester composites.

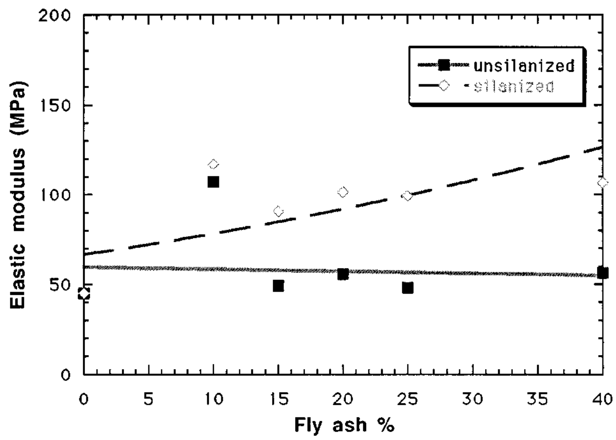

Figure 7 Variation of elastic modulus with a concentration of unsilanized and silanized fly ash for polyester composites.

modulus increases with 10% fly ash loading and then decreases and then levels off. The probable reason for the low modulus of elasticity is the presence of voids in terms of higher fly ash loading. On the other hand, surface treatment enhanced the stiffness of all composites which may be due to increased unsaturation from the methacryl group of silane and so participation in curing reaction.

It is well known that the area under stress–strain curves (work up to fracture) is a measure of the toughness and gives an idea about energy absorbed by the specimen to fracture or in short how the matrix dissipates energy in the case of damage or fracture of the composite. Work up to fracture values as a function of unsilanized and silanized fly ash loading are presented in Figure 8.

It is obvious from the Figure 8 that toughness of fly ash polyester composites decreases with the fly ash addition but the compositions having 15 and 25% unsilanized fly ash exhibit the maximum toughness values among the others. The composition, on the other hand, having 15% silanized fly ash, exhibits the maximum toughness in agreement with its high tensile strength and high elongation at break (Figs. 5 and 6).

Dynamic Mechanical Properties of Fly Ash Filled Composites

Because polymers are viscoelastic in nature, their mechanical properties exhibit a pronounced dependence on temperature and rate of deformation. In a number of engineering applications, the material may be subjected to dynamic stress conditions. The mechanical properties of the material under static loading may not fully describe its performance in such applications. Therefore, the cured samples of the resins were also investigated in terms of dynamic mechanical behavior. The viscoelastic behavior of unsaturated polyester resins was studied in a temperature range of 20–120°C at a fixed frequency of 1 Hz.

The loss factor (tan δ) is one of the damping parameters of interest because it is a measure of ability of the polymer to convert mechanical energy into heat at a temperature or frequency of interest. Tan δ and damping temperature range (width of the tan δ peak) values measured at 1 Hz for the composites are tabulated in Table II.

Figure 9 shows tan δ versus fly ash loading in polyester composites.

These values reach to a maximum value with the addition of unsilanized fly ash to 15% and then decrease slightly to a certain value which is still quite higher than neat polyester which is also clear from Figure 10.

This increase may have resulted when the material was applied by external force in optimum filler state, the internal friction among macromolecule segments and fillers increases much more to convert the mechanical energy into heat. Then, the increment of the internal friction increases the mechanical dissipation and enhances the vibration damping.[13]

Silanization was found to be effective especially after 15% loading and caused a slight decrease in damping. Here, it is quite clear that since silanization acts as additional crosslink points, which is also evident from low degree of

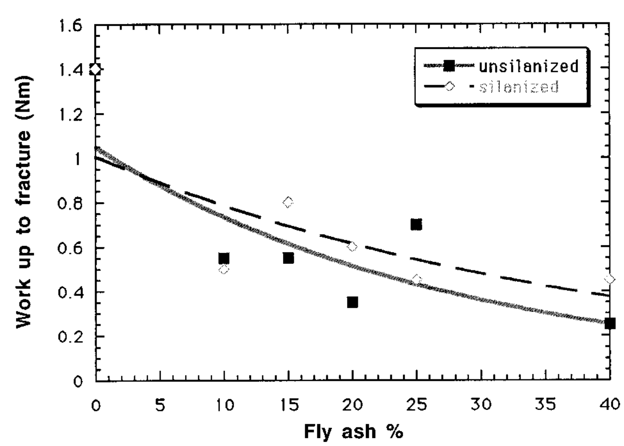

Figure 8 Variation of work up to fracture values with concentration of unsilanized and silanized fly ash for polyester composites.

Table II Tan δ and Damping Temperature Range of Polyester Composites

Samples	Tan δ	Damping Temperature Range (°C)
Neat	0.294	20
PEC10FA	0.701	60
PEC15FA	0.737	69
PEC20FA	0.616	67
PEC25FA	0.590	51
PEC40FA	0.564	68
PEC10FAS	0.702	63
PEC15FAS	0.728	62
PEC20FAS	0.640	65
PEC25FAS	0.567	60
PEC40FAS	0.494	59

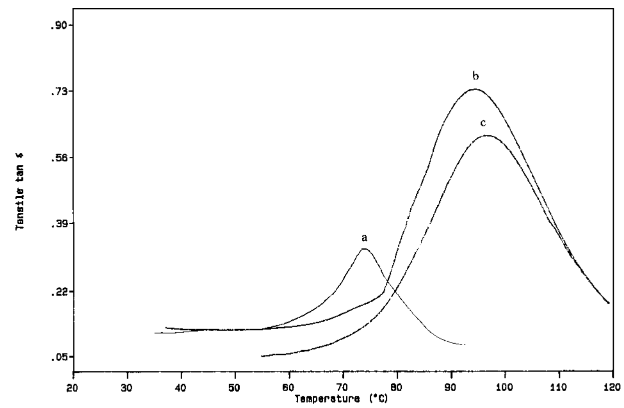

Figure 10 DMTA spectra of (a) neat polyester, (b) 15% unsilanized, PEC15FA, and (c) 20% unsilanized, PEC20FA, fly ash filled composites.

swelling, high tensile strength, and high modulus values (Table I), internal friction will be less likely to lead to lower damping values.

All these results indicate that the composite having 15% fly ash, PEC15FA, seems the optimum filler loaded composite in terms of dynamic mechanical response with highest damping and largest damping temperature range in which a composite can act as an effective damper.

CONCLUSIONS

Curing characteristics of fly ash filled polyester composites showed that the addition of unsilanized fly ash to the polyester resin causes somewhat higher cure time than the neat polyester composites. Silanized fly ash filler in the same composition causes little decrease in cure time, indicating enhanced curing reaction of the resin. But mainly it can be said that fly ash addition does not greatly change the cure time of the polyester. On the other hand, fly ash is found to have a quite low and acceptable oil absorption value which does not greatly affect polyester/styrene mol ratio.

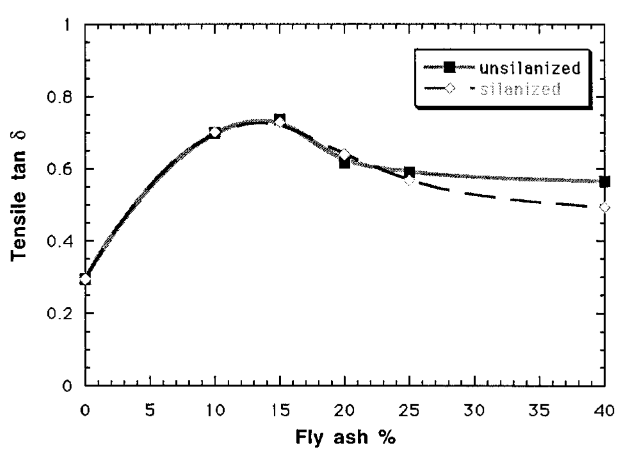

Figure 9 Variation of dynamic tan δ values with concentration of unsilanized and silanized fly ash for polyester composites.

Both unsilanized and silanized fly ash composites in the uncured state exhibited low sedimentation rate values. Compared with unsilanized fly ash, silanized fly ash settled down more gradually resulting from improved dispersion of filler throughout the final article, which shows promising good physical properties.

Viscosity behavior of uncured fly ash filled polyester resins indicated that an increase in unsilanized fly ash increases viscosity and too high viscosity especially after 25% loading caused molding process to be difficult. It is possible to use silanized fly ash for further decreases in viscosity to avoid this drawback even at higher loading degrees.

It was found that cured fly ash filled polyester composites swell in very small amounts especially the composite having up to 15% fly ash. A decrease in swelling is much more pronounced in the case of silane-treated fly ash composites.

In unsilanized fly ash polyester composites in cured state, the addition of unsilanized fly ash decreases tensile strength. Silanization of fillers causes a significant increase in tensile strength for each composite. It seems that most beneficial effect can be obtained with 15% silanized fly ash addition to polyester matrix in terms of maximum tensile strength.

The addition of fly ash makes the composites stiffer due to lower percentage of elongation at break at high fly ash loading compared with neat polymer. On the other hand, surface treatment enhanced the stiffness of all composites with a certain degree of decrease in elongation at break values.

Polyester composites cause a decrease in toughness with the fly ash addition but the compositions having 15 and 25% unsilanized fly ash exhibit a synergistic effect via maximum toughness values among the others. The maximum toughness value observed for the composition, on the other hand, having 15% silanized fly ash was found to be in good agreement with its high tensile strength and high elongation at break values.

Dynamic mechanical data showed that although the tan δ peak intensity increases with the addition of unsilanized fly ash filler in general, the intensity increase in composition having 15% fly ash was found to be remarkable. Silanization, on the other hand, caused a slight decrease in damping factor especially after 15% loading

All these results indicate that among the fly ash filled polyester composites, the one having 15% fly ash seems to be the most beneficial composite in terms of dynamic mechanical response with highest damping and largest damping temperature range in which composite can act as an effective damper.

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