Epoxy Resin/Polymer Blends: Improvement of Thermal and Mechanical Properties

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ABSTRACT: Fatty acid waste was recycled as raw material and utilized to synthesize epoxy (HP) or unsaturated ester group (OEE) containing polymers. The radicalic polymerization between the styrene and itaconic acid was carried out, too. Glycidyl ester of styrene-itaconic acid copolymer was obtained by esterification reaction with epichlorohydrin. The polymers were characterized by means of Fourier transform infrared spectroscopy and chemical analysis. The polymers were incorporated into diglycidyl ether of bisphenol A type commercial epoxy resin to prepare composites. The effects of polymer structure and amount on the physico-mechanical and thermal

properties of epoxy were investigated. Surface hardness, tensile strength, percentage elongation and stress at maximum load of the composites were obtained higher than pure epoxy resin. The composites reinforced with biobased polymers showed about 74.55–243% increase in elastic modulus over the pure epoxy matrix. Obtained Young's modulus values were higher for composites with styrene-based polymers. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 125: 38–45, 2012

Key words: waste; resins; composites; mechanical properties

INTRODUCTION

Thermosets such as epoxy and novolac resins are a class of important polymeric materials; they have widely been used as high performance materials such as adhesives, matrices of composites and electronic encapsulating materials.^{1,2} However, these thermosets are inherently of low impact resistance due to their high crosslinking density, which greatly restricts their application. A large number of studies have been conducted to improve the impact resistance of highly cross-linked epoxy resins (ERs) by modification. The effects of bio-based epoxy materials on the impact strengths of the diglycidyl ether of bisphenol A (DGEBA) epoxy system were investigated by Jin and Park. DGEBA was very brittle, exhibiting an impact-strength value of 13.9 J/m^2 , whereas the attained impact-strength value of the blends was 58% higher, 21.9 J/m at 60 wt % epoxidized soybean oil (ESO).³ Similar results have been reported by Miyagawa et al. using bio-based neat epoxy materials containing epoxidized linseed oil.4 Chen et al. used *n*-butyl glycidyl ether (BGE) to modify diglycidyl ether of bisphenol F epoxy system

The impact strength was appreciably enhanced by the introduction of BGE in the ER. An addition of 40 phr BGE resulted in the 77.6% increase in the impact strength compared to that for the unmodified epoxy.⁵ Two flexible polyoxypropylene diamines (D-230 and D-400) are selected to modify diethyl toluene diamine (DETD) cured DGEBA ERs by Yang et al. In general, the impact strength of ERs was increased with increasing the diamine content from 19.2 to 53.56 kJ/m².⁶ The polybutadiene particles were also adhered to the epoxy matrix and all modified epoxy networks exhibit higher impact resistance than nonmodified ER.⁷

During the past decades considerable efforts have been made to improve the toughness of epoxy thermosets.^{8–17} One of the successful routines of toughness improvement is to incorporate polymeric modifiers into thermosetting matrix to form fine morphological structures. Since the modifiers are some linear homopolymers or random copolymers, the modified thermosets would exhibit the phaseseparated morphology on the macroscopic scale. Recently many researchers have studied epoxy and polymer/epoxy-co matrix composites with inorganic fillers to improve the mechanical properties of the resin. Also, important investigations on ER-polymer composites have been known. Composites and nanocomposites from ESO were prepared by Liu and Erhan. The experiments were carried out for investigating influence of ESO/ER ratio (by weight) on mechanical properties of the composites. The results

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showed that flexural modulus increases as the ER amount increases.¹⁸ Form-stable polyethylene glycol (PEG)/epoxy resin (ER) composite as a novel phase change material was prepared using casting molding method. The heat mechanical deformation of PEG/ EP composites were very small.¹⁹ Amino-containing sulfonated poly(aryl ether ketone)/ER composites, prepared by Guo et al., were used as proton exchange membranes.²⁰ Corcion and Frigione investigated the rheological behavior of composites based on a trifunctional ER and a OH-terminated hyperbranched aliphatic polyester.²¹ A series of polycaprolactone-polydimethylsiloxane (PCS-2Si) modified ER composites were prepared and then cured by amino-glycidyl reaction and the sol-gel process. The morphology and thermal stability of the composite system were characterized as a function of PCS-2Si content. It was shown that the compatibility between epoxy and polymer increased with increasing content of PCS-2Si.²² The morphology and thermomechanical properties of the epoxy composites containing triblock copolymer which was synthesized via the ring-opening polymerization of é-caprolactone with dimethylsiloxane were investigated by means of AFM, TEM, DSC, and dynamic mechanical analy-sis.²³ Mülhaupt et al.^{24,25} reported the modification of ER with a branched poly(é-caprolactone)-block-polydimethylsiloxane block-poly(é-caprolactone) block copolymer, too. It is noted that the inclusion of a small amount of the copolymer (e.g., 5 wt % or more) leads to a significant increase in the toughness of materials whereas the strength of materials (e.g., strength at break and Young's modulus) was not obviously reduced.

In this work, we will report the synthesis of a novel bio- and styrene-based polymers. After that, the polymers were incorporated into epoxy to toughen the thermosets and investigated their physico-mechanical properties. To the best of our knowledge, no work has been reported on the mechanical properties of ERs modified by these types of polymers.

EXPERIMENTAL

Materials

The waste of sunflower oil as fatty acid (FA) was supplied from Zade Chemical Industry, Konya, Turkey. The thermosetting matrix used in this study was a commercially available bisphenol A-type ER (DER 321, Dow Chemical) modified with aromatic diluent and cured at high temperature with 30 wt % Polypox Hardener 043. The curing agent was a cycloaliphatic polyamine. The epoxy content of ER was 23.12%, as determined by titration. Synthesized two styrene copolymers: styrene-itaconic acid copoly-

mer (SIAC) and glycidyl ester of SIAC (SIAGE), and two bio-based polymers: oligo(ether-ester) (OEE) and homopolymer (HP) based on FA waste were used as modifiers in ER composites. Propylene oxide (PO), epichlorohydrin (ECH), styrene, itaconic acid, benzoyl peroxide (BPO), cationic catalyst $BF_3.O(C_2H_5)_2$ were purchased from Merck (Darmstadt, Germany).

Synthesis of glycidyl ester of fatty acid (FAGE)

Glycidyl ester of fatty acid (FAGE) was obtained by the esterification reaction of potassium salt of FA with epichlorohydrin in the alkaline medium. Ten gram FA waste and 10 mL benzene were taken to the flask for the reaction and 4 mL 40% KOH solution was added to this mixture. Three gram epichlorohydrin was supplemented to the mixed solution drop-by-drop at 40°C within 20–30 minutes. After this addition the temperature was increased to 70– 80°C and the mixture was boiled for 5 hours. The ester, that is obtained, was distilled under the vacuum.²⁶

Conjugated fatty acid glycidyl ester (KFAGE) was obtained with the same method from conjugated FA, which was synthesized from FA.²⁷

Synthesis of unsaturated oligo(ether-ester)s (OEE)

A 150 mL flask, equipped with a magnetic stirrer and thermometer, was charged with PO:FAGE in mole ratio 1 : 1. The mixture was cooled to 0°C with stirring and BF₃O(C₂H₅)₂ (1 wt %) was added to the mixture. After completion of the reaction, methanol (1 mL) was added to mixture to deactivate the catalyst. The starting materials and methanol were removed under reduced pressure (2 mmHg, 53°C). Yield was calculated from final weight.

Synthesis of homopolymer (HP) from konjugated FAGE

HP was obtained by homopolymerization of KFAGE in the presence of benzoyl peroxide (1 wt %) at 80°C for 5 h.

Synthesis of SIAC

A 150 mL flask, equipped with a magnetic stirrer, condenser and thermometer, was charged with styrene:acid in mole ratio 5 : 5. Then toluene and benzoyl peroxide (0.5 wt %) were added to the mixture. The mixture was heated to 80°C by stirring for 8 hour. After completion of the reaction, the copolymers were filtered and then dried under vacuum at 40° C. Yield was calculated from final weight.²⁸

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Eight milliliter of 40% KOH solution was added to 10 g dissolved dioxane SIAC and stirred for 30 min at 40°C. Then 10 g ECH was added gradually through dropping funnel and the mixture was heated to 80°C by stirring for 5 hour. After the organic phase was separated, the polymer was precipitated with methanol.²⁸

Analysis

The Fourier transform infrared (FTIR) spectra of synthesized polymers were recorded with UNICAM SP 1025 spectrometer.

Determination of epoxy group (\searrow)

To determine the percentage of the epoxy groups, 0.5 g sample was placed in a 250 mL flask and 25 mL solvent mixture (60 mL acetone + 1.5 mL concentrated HCl) was added to it. It was mixed for 2 h at room temperature. The epoxy groups in the sample was cleaved with excessive HCI and then the remaining HCI was titrated with 0.1N KOH. The epoxy group was calculated by the following equation:

Epoxy groups,
$$\% = (V_1 - V_2) \times 0.0043 \times F \times 100/m$$

where, V_1 is the 0.1*N* KOH used for the titration of control (mL), V_2 is the 0.1*N* KOH used for the titration of sample (mL), *F* is a titration factor for 0.1*N* KOH solution, *m* is the amount of the sample analysed (g), 0.0043 is the number of epoxy groups corresponding to 1 mL of 0.1*N* KOH.²⁹

Determination of ester group

Sample (2–3 g) was dissolved in a mixture of ethanol (25 mL) and benzene (50 mL). Twenty-five milliliter of an ethanolic solution of KOH (2*N*) was added. The mixture was refluxed for 1 hour. After cooling to room temperature, excess of KOH was titrated with HCl (1*N*) using phenolphthalein. Number of ester groups was calculated as mg KOH/g sample with following equation:

Ester group (mg KOH/g oligomer)

$$=\frac{56.1\times(V_1.N_1-V_2.N_2)}{m}$$

where, N_1 is the normality of the KOH solution, N_2 is the normality of the HCl solution, V_1 is the volume of the added KOH solution (mL), V_2 is the volume of the added HCl solution used for titration (mL), *m* is the amount of the sample analyzed (g), 56.1 is the molecular weight of the KOH.³⁰

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Determination of acid number (A.N.)

The amount of carboxyl groups was determined by functional group analysis. The A.N. was calculated as mg KOH/g sample. The sample was boiled under reflux with excessive KOH and then the remaining KOH was titrated with HCI solution.

A.N. (mg KOH/g polymer) = $56.1 (V_1N_1 - V_2N_2)/m$

where V_1 is the volume of the added 0.1N KOH (mL), N_1 is the normality of the KOH solution, V_2 is the volume of 0.1N HCl used for titration (mL), N_2 is the normality of the HCl solution, and *m* is the amount of the sample (g), 56.1 is the molecular weight of KOH.³¹

Determination of double bond

The number of double bonds was determined by titration method. The sample (0.2–0.4 g) in a stoppered-vial was dissolved in 15 mL of ethanol at 50– 60°C. Twenty-five milliliter of an iodine solution (2.57 g iodine in 100 mL of ethanol) and 200 mL of water at 30–35°C was added to the above-mixture. The mixture in stoppered-vial was sufficiently shaken and allowed to stand in the dark for 5 minutes. Then, the excess iodine was back titrated with a solution of 0.1N Na₂S₂O₃ using starch indicator. In the same conditions, a control titration was also performed without sample. Titration was disappeared. The iodine value (IV) was calculated as g $I_2/100$ g sample using the following equation:

$$\% IV = \frac{(V_1 - V_2) \times 0.012697}{m} \times 100$$

where, V_1 is the volume of 0.1N Na₂S₂O₃ used for control titration (mL), V_2 is the volume of 0.1N Na₂S₂O₃ used for titration with sample (mL), 0.012697 is the amount of iodine corresponding to 1 mL of 0.1N Na₂S₂O₃ (g), *m* is the amount of oligomer sample (g).³²

Forming of composites

The modifier in 10–25 wt % and ER matrix were mixed with mechanical stirring for 30 min. Afterwards, 30 wt % Polypox Hardener 043 was added and the mixture was degassed for 60 min at 40°C, then transferred into the mould. Composite specimens were prepared in stainless-steel moulds according to ASTMD 638 standard. The curing procedure was realized in an oven within 24 h while increasing temperature from 60 to 120°C.



Figure 1 Synthesis of styrene-based copolymers.

Mechanical testing

A Shore Durometer TH 210 tester is used for measuring hardness. The resistances to stretch properties were determined by Stretch and Pressing Equipment TST-Mares/TS-mxe.

Thermal analysis

The thermal analysis experiments were performed with a Seteram Thermogravimetric analyzer. Samples were heated under a nitrogen atmosphere from 50 to 600° C at a heating rate of 10° C min⁻¹.

RESULTS AND DISCUSSION

Characterization

SIAGE, bio-based HP and OEE were synthesized firstly by us. Synthesis reactions of styrene- and

FA-based polymers were shown in Figures 1 and 2.

Chemical structure of polymers was determined via FTIR spectrum and chemical analysis. Chemical analysis results were listed in Table I. As seen from Table I, the acid number of SIAC decreased after reaction with ECH. In SIAGE was determined epoxy and ester groups. On the other hand in the FTIR spectrums of styrene copolymers (Fig. 3), characteristic bands were appeared in the regions of 1700, 1580, and 3000 cm^{-1} for acid C=O group, aromatic C=C and acid OH group, respectively. But in the FTIR spectrum of SIAGE in distinct with spectrums of copolymers, characteristic band of C=O acid group did not appear. The most intense bands were observed at 1731 cm⁻¹ which have been assigned to ester C=O stretching and at 1246 cm⁻¹, which confirmed the presence of epoxide group in the copolymer. These results shows



R: CH₃(CH₂)₄CH=CH-CH₂-CH=CH(CH₂)₇

Figure 2 Synthesis of FA-based copolymers.

TABLE I Chemical Analysis Results							
Polymer	Ester number, mg KOH/g sample	Iodine value, g I ₂ /100 g sample	A.N., mg KOH/g sample	Epoxy group, %			
ER				23.12			
SIAC	-	_	260	_			
SIAGE	312.4	_	51.7	6.49			
HP	340.1	92.3	9.8	8.73			
OEE	340.6	156.8	10.1	7.97			

that reactions between SIAC and ECH were carried out (Fig. 1).

As shown in Figure 2, synthesized HP and OEE contains double bond on the main and side chains, respectively. In our previous study, by gas chromatography analysis it was determined that FA waste composed of about 55% of linoleic aicd.²⁶ Therefore the iodine value of OEE was higher than HP. Also by chemical analysis were determined ester groups for HP and OEE (Table I). In the FTIR spectrums of FAGE and OEE, characteristic bands were appeared in the regions of 1661 cm^{-1} for C=C, 1740–1746 cm^{-1} for C=O of ester, 722–724 cm⁻¹ for FA $-(CH_2)_4$ -units, and 1453–1455 cm⁻¹ for $-CH_2$ -C=O in acids (Fig. 4). A broad and intense absorption band at 1246 cm⁻¹ for epoxide group was seen in the spectrum of FAGE, which was not observed in OEE FTIR spectrum. Instead of this the appearance of band in the region of 1065 cm⁻¹ for ether confirmed the ringopening copolymerization reaction between FAGE and PO. In FTIR spectrum of HP characteristic bands were appeared in the regions of 1646 cm^{-1} for C=C, 1746 cm⁻¹ for C=O of ester, 718 cm⁻¹ for FA $-(CH_2)_4$ units, and 1453-1459 cm^{-1} for -CH2-C=O in acids. As seen from these results, synthesis of HP and OEE too were recognized.

Mechanical properties

The mechanical properties of polymers are influenced by molecular weight, crosslinking, branching,



Figure 3 FTIR spectrum of styrene-based copolymers.



Figure 4 FTIR spectrums of FAGE and FA-based polymers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

segmental motion, morphology, and external conditions such as temperature, pressure, loading rate, environmental condition, and extent of compound.^{33–37} The structure of the side-chain substituents on the polymer backbone is a major compositional factor impacts polymer functionality. Important aspects of substitution are the chemical structure of the substituents, the extent of backbone substitution, and the uniformity of substitution. The nature of the side chain substituent type also significantly impacts mechanical properties. Increasing the amount of highly polar, ionic side-chains tends to result in an increased tensile strength.³⁸ The effect of side group structure on the compressive strength of novel biodegradable polyphosphazene-based polymers was investigated by Sethuraman et al. Results of mechanical testing studies demonstrated that the nature and the ratio of the pendent groups attached to the polymer backbone play a significant role in determining the mechanical properties of the resulting polymer. The compressive strength of polymer with aliphatic alanine side group was significantly higher than polymers with aromatic alanine groups.³⁹



Figure 5 Dumb-bell shape of MPS specimens.

Modifier		Tensile strength (MPa)	Elongation at break			Hardness	
	Amount of modifier (%)		mm	%	e-modulus (GPa)	by shore durometer (HD)	
SIAC	10	116.69	0.385	0.260	23.47	67.50	
	20	327.52	0.622	0.420	37.33	73.50	
	25	316.73	0.529	0.352	39.64	74.50	
SIAGE	10	73.54	0.696	0.470	14.67	64.50	
	20	199.06	0.831	0.562	20.07	72.00	
	25	189.26	1.081	0.730	28.21	73.00	
HP	10	52.50	0.590	0.396	16.66	75.00	
	20	63.33	0.910	0.610	15.00	76.00	
	25	68.26	1.160	0.782	15.33	72.00	
OEE	10	62.76	1.010	0.681	12.33	65.00	
	20	214.75	1.054	0.712	18.33	63.00	
	25	183.37	1.068	0.721	16.07	58.00	
Pure epoxy resin	0	50.99	0.338	0.228	20.00	58.00	

 TABLE II

 Effect of Modifiers on Physico-Mechanical Properties of Epoxy Based Composites

Mechanical analysis was performed to study how different structures influence the mobility of the epoxy. To investigate physico-mechanical properties, such as tensile strength, elongation at break and hardness of the composites, mechanical testing was performed. The dumbbell shape of the specimens with proper dimension is shown in Figure 5.

The results are summarized in Table II. It was determined that physico-mechanical properties of composites changed depending on structures of polymers. The tensile test results determined for pure ER as follow: elongation percentage at break was 0.338% and tensile strength was 50.99 MPa. It can be seen that all composites show higher tensile strength and elongation at break than the pure epoxy matrix because of all used polymers have polar side chain groups. These properties of composites increase with an increase of polymer amount in all cases and the best results were obtained with OEE. As compared SIAC and SIAGE modified ERs, modification of SIAC with epoxy group to obtain SIAGE increased the elongation at break about twofold. The composites reinforced with bio-based polymers showed about 74.55–243% increase in elongation at break over the pure epoxy matrix. As seen from Table II, the appropriate polymer amount was 25 wt %. Bio-based polymers caused more elongation in composites than styrene-based polymers because of they are oligomers and well miscible with ER. Another reason may be related to the chemical structure. As shown in Figure 1, HP contains epoxy and OEE unsaturated ester groups on the side chain. Yang et al.⁴⁰ obtained that hyperbranched polyester Boltorn H30 enhanced mechanical properties of DGEBA. The results showed that addition of H30 of proper amounts can lead to simultaneous improvements in the tensile strength, failure strain and impact strength. The maximum impact strength is

obtained for the modified epoxy system with 10 wt % H30. The hydroxyls of hyperbranched polyester can react with the curing agent and hence the network structure of the cured epoxy systems can be tailored.^{41,42} Composites with SIAC showed a highest tensile strength values than others changed in the range of 116.69–327.52 MPa. This can be explained by the dicarboxylic acid polar groups of itaconic acid in the structure of SIAC. Tensile strength of composites with SIAGE, HP and OEE was higher than pure epoxy because of the polar groups in polymers structure, too.

As the Young's modulus (e-modulus) of materials was higher, they are resistant to strain deformation. Obtained Young's modulus values were higher for composites with styrene based polymers and comparatively lower for composites with bio-based polymers than ER. Young's modulus values are increased with amount of styrene-based copolymers. The best result was seen for composite with 25 wt % of SIAC as 39.64 GPa.

Hardness is not a reliable measure of stiffness. Hardness measurements derive from small deformations at the surface, whereas stiffness measurements such as measurement of tensile modulus derive from large deformations of entire mass. Unlike with metals, there is no correlation between hardness and tensile strength of elastomers. As the hardness increases, the tensile strength of an elastomer may increase to a maximum then decrease, or it may decrease from the beginning, depending on the formulation of the compound.43 From the hardness measurements, it was seen that all polymers increased the hardness of pure ER from 58 to 76 HD. Yang et al.⁴⁰ reported that flexible chains or groups were regarded to contribute to the increase in the impact strength. As seen from Table II, the hardness of HP modified ER was higher than OEE

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Figure 6 TGA curve of pure epoxy resin.

modified epoxy. This can be explained by the reacting of epoxy groups of HP with the curing agent.

Thermal stability

The principal characteristics and products of thermal degradation of a commercial ER prepared by 2,2-bis(4'-hydroxy phenyl)propane reaction of (bisphenol-A) with 1-chloro-2,3-epoxy propane (epichlorohydrin) have been studied by Grassie et al. The principal volatile products, acrolein, acetone, and allyl alcohol, are formed at 280°C and, although cross-linking is detectable at 220°C. Decomposition of the cross-linked resin occurs above 340°C when phenolic compounds appear.44 Erickson studied the thermal degradation of epoxy at 340°C. Evolution of phenol, 4-isopropylphenol, bisphenol A, and 4-tbutyl-o-cresol indicate that bond scission involving the bisphenol A moiety in the epoxy is a major decomposition mechanism.45 Neiman et al. reported that among the gaseous products formed in the thermal degradation in the temperature range 300-450°C



Figure 7 TGA curve of composite with 10 wt % HP.



Figure 8 TGA curve of composite with 20 wt % HP.

of the ER hardened by polyethylene polyamine were found H_2 , CO, CH₄, C_2H_4 , C_2H_6 , and C_2H_8 . Ethane, ethylene, propylene and propane are possibly produced by decomposition of the polyethylene polyamine radical.⁴⁶

During the last few years, there has been a growing interest in the use of polymers obtained from renewable resources because advantages of these polymers include their low production cost, in some cases, and their possible biodegradability. The importance of natural products for industrial applications also becomes very clear with increasing social emphasis on issues of the environment, waste disposal, and the depletion of nonrenewable resources. Therefore, for thermal analysis HP/ER composites were decided. Figures 6-8 shows TGA curves of the pure and HP modified ERs. The thermograms obtained during TGA scans were analyzed to give the percentage weight loss as a function of temperature (Table III). The thermal degradation of pure ER occurs in two-step. The first was in the range of 150–280°C and may be due to decomposition of end hydroxyl group of polyamine cured ER and olefin formation. The second degradation stage was observed at 335°C and showed decomposition of bisphenol-A group (Fig. 6). T_5 , T_{10} , and T_{50} (temperature of 5, 10, and 50% weight loss, respectively) are the main criteria indicating the thermal stability of the composites. The higher the values are, the higher is the thermal stability. Loss of weights in various

TABLE III Effect of HP Amount on Thermal Properties of Epoxy Based Composites

				•				
HP/ER	Weight loss (%)				T_5	T_{10}	T_{50}	
composite	200°C	300°C	350°C	400°C	450°C	(°Č)	(°C)	(°Č)
Pure ER	4	8	25	67	83	220	295	375
%10 HP	7	10	20	54	85	185	270	390
%20 HP	6	12.5	25	48	65	160	290	410

degradation temperatures and degradation temperatures at 5, 10, and 50% weight loss of composites are given in Table III, which indicate that the thermal stability of the pure epoxy was enhanced by the incorporation HP. Further, the incorporation of 10 and 20% HP in pure epoxy matrix increases the 50% decomposition temperature of pure epoxy by 15 and 35°C, respectively. As seen from Table III, the best thermal results were obtained for 20% HP above 350°C. This can be attributed to the crosslinking of double bounds of HP at higher temperatures.

CONCLUSION

Various styrene and FA-based polymers were used to prepare polymer/epoxy composites. Hardness, tensile and thermal properties of a bisphenol Abased ER modified with polymers and cured using polyamine were determined experimentally. All composites show higher elongation at break and tensile strength as compared with the pure epoxy matrix. The incorporation of the different polymers resulted in pronounced improvement in hardness, too. In the tensile test, the best elongation at break results were seen for composites with OEE. Composites consisting of HP have higher hardness and thermal resistance characterization.

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