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Effects of pumice additions on thermal and mechanical behaviors of epoxy resin

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Abstract. In this study, thermal conductivity, mechanical properties, and thermal degradation of pumice-added epoxy materials were investigated. 2%, 4%, 6%, 8%, and 10% of pumice was added to the epoxy resin (EP) % by weight. Various types of analyses and tests were conducted to determine the thermal conductivity, mechanical properties, and thermal degradation of these epoxy materials. The tests and analyses proved that the addition of pumice leads to a decrease in the thermal conductivity coefficient and density of the pure EP material. It also increases the degree of hardness. The addition of pumice had a positive effect on mechanical properties. Compared to pure EP, it increased the tensile strength, Young's modulus, bending strength, and flexural modulus. As a result of TGA analysis it was determined that with the incorporation of pumice into the EP, its decomposition rate progressed more slowly. At 800°C, the carbon residue improved as a result of the addition of pumice.

Key words: epoxy resin; pumice; thermal degradation; thermal conductivity coefficient; mechanical properties.

1. INTRODUCTION

Epoxy resins (EP), due to their good mechanical properties, are widely used in electrical and electronic fields, as adhesives, coating materials, and aerospace fields, as well as in structural areas [1, 2]. The EPs obtained as a result of the mixture of the two components pass from the liquid state to the solid state by the reaction of the three-dimensional cross-linked thermoset hardener and linear epoxy resin (This process is generally called curing). They reach their final hardness after the required curing process. At this point, epoxy resins cured with diamines are brittle [3]. As brittleness properties are added to EPs, which are known for their weak thermal conductivity and weak flammability properties, their fields of application are restricted [4]. Various fillers such as fiber, glass fiber, clay, woven graphite, glass graphite, urea-formaldehyde, and boron were used in studies on the elimination of these negative properties [5–9].

While improving the thermal conductivity and combustion resistance of materials, changes in their mechanical properties should be monitored [10]. In addition, the chosen filler should not increase the cost of the main material. Thermal and electrical conductivity, flame resistance (thermal degradation), and mechanical properties can be improved by adding a filler material to the epoxy matrix [11–17].

If we examine the one-dimensional heat transfer on the EP material, it occurs by conduction through the structure of solid polymers. A second transmission mechanism takes place in the form of cell gas conduction. Researchers and leading companies in the sector aim to reduce the amount of heat transfer that occurs through conduction. For this, they tried to deter-

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mine additive materials that would prevent the inflating gas in the EP structure from leaving the cell and act as a diffusion barrier. At the same time, similar studies are carried out to reduce the cell size of the resin structure [18]. The large grain size of the selected additive has negative effects [19]. Therefore, smallsized materials should be chosen as additives as much as possible [20, 21].

Pumice is an aggregate of volcanic origin with a vitreous structure rich in silica. It is the most popular type of natural porous lightweight aggregate known as industrial raw material [22, 23]. Thanks to the voids in its structure, it has high heat and sound insulation properties [24]. Pumice has a porous structure, lightness, high insulation effects, extraordinary resistance to atmospheric conditions, and high pozzolanic activity. For this reason, it has very common usage areas in the world, especially as a building material [25–28]. It is an inexpensive material that is easily available in the market.

The microparticle size of the pumice used in this study may result in strong interfacial interactions between the fillers and the polymer matrix [29]. In addition, it is thought to be a welladditive material by the reason of its low thermal conductivity and non-flammable properties, and its uniform distribution in the epoxy material. Alongside obtaining high mechanical and thermal properties, the addition of pumice as a filler causes less usage of the resin and reduces the direct cost.

Ahmetli *et al.*, mixed the precipitate obtained from marble processing plant wastewater using pumice coagulant with commercial epoxy resin at a rate of 20% by weight. They found that the inclusion of marble processing waste particles increased the degradation temperature of pure epoxy by $5-50^{\circ}$ C. Composites reinforced with pumice, which is a waste of marble processing, showed an increase of approximately 10% in elastic modulus, while surface hardness, tensile strength, percent elongation, and stress at maximum load also improved compared to pure resin [30]. In another study, Ahmetli *et al.*, synthesized

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epoxy toluene oligomer as a comatrix at a rate of 50% by weight with commercial epoxy resin. They used 20% zeolite, sepiolite, or pumice as a filling material. They determined that thermal stability was significantly improved in all samples, with an increase in thermal degradation temperatures and coal residue rates. They reported that surface hardness and tensile strength also showed better results than pure EP [31]. Another study on the evaluation of natural wastes resulting from the operation of quarries was reported by Cunha *et al*. In the study, using various formulations of epoxy and polyester matrices, Cunha *et al.* added 50% and 70% pumice into the structure. Mechanical properties with a tensile strength of 91.63 MPa and Young's modulus of 13.65 GPa were determined in the sample with 70% additive content. They reported that the addition of pumice with these properties was promising [32].

In addition, as a result of the literature review conducted by the author, it was evident that there was not enough study in the literature on the determination of the technical properties of pumice-added EPs. The studies conducted are given above. In this study, the thermal and mechanical properties, and combustion behaviors of pumice-added EPs were investigated. 2%, 4%, 6%, 8% and 10% of pumice was added to the EP in proportion to weight. X-ray diffraction (XRD), thermal gravimetric analysis (TGA), FT-IR, mechanical tests, thermal conductivity, hardness, limit oxygen test (LOI), and vertical combustion tests were performed and compared with pure EP.

One of the main purposes of this study is to reduce the thermal conductivity of EP as a result of the addition of pumice. Then, its usability as a thermal insulation material is investigated. In addition, the changes in mechanical and thermal properties after the addition of pumice were investigated.

2. MATERIALS AND METHODS

2.1. Materials

Epoxy samples were produced of epoxy resin, consisting of two components – I:1000gr (Bisphenol: the epoxy value of 0.51 mol/100 g) and II:500gr (Hardener; polyamine). Epoxy resin was obtained from MARKER KIMYA, Turkey. It is transparent in color, does not contain solvents and volatile substances, and has an approximate density of 1.20 g/cm^3 . The mixing ratio is 2:1 and has a gelling time of 100-120 minutes. They harden after 24 hours, which is sufficient curing time, and reach their final hardness after 2 weeks.



Fig. 1. Pumice powder

Pumice used as additive material was supplied as a 5-micron thick powder from MINER MADENCILIK, Turkey. It is off-white in color and its density is between 400–600 kg/m³. The basic technical characteristics of the micronized pumice used are given in Table 1. The image of the pumice powder is given in Fig. 1.

Properties	Pumice				
Color	Off-white				
Hardness (MOHS Scale)	6				
рН	6.5–7				
Moisture (%)	1				
Density (kg/m ³)	400-600				
Heat resistance (°C)	1240				
Thermal conductivity (W/mK)	0.11-0.21				
Non-flammability class	A1				
Sound insulation (dB)	45–55				
Chemical content					
Major elements	%				
SiO ₂	65.00				
Al ₂ O ₃	11.67				
CaO	0.55				
MgO	0.15				
Na ₂ O	4.12				
K ₂ O	4.10				
Fe ₂ O ₃	1.75				
TiO ₂	0.03				

Table 1Technical properties of pumice

2.2. Sample preparation

In the preparation of the samples, the fluid obtained which the component II is half as much as the component I and the additive material (pumice) determined in % ratio, was then poured into a silicon mold. The details of this process are as follows. In order to ensure homogeneous distribution of the additive material in the composition during the production, component I and with 2% of pumice the total mass was added to the agitation vessel. It was then mixed at low speed for 3 minutes. After adding the component II, it was mixed at low speed for 5 minutes and later rest time starting of 5 minutes. Rest time at finished come round to casting into a silicone mold was carried out. The sample was unmould after 24 hours (The 24 hour curing time was obtained from the technical data table). Likewise, they were produced for the other additive ratios of 4%, 6%, 8%, and 10%. In order to clarify and easy interpret the obtained results, the samples were named PURE, PUM02, PUM04, PUM06, PUM08 and PUM10, respectively, according to their additive ratios (Fig. 2).



Effects of pumice additions on thermal and mechanical behaviors of epoxy resin

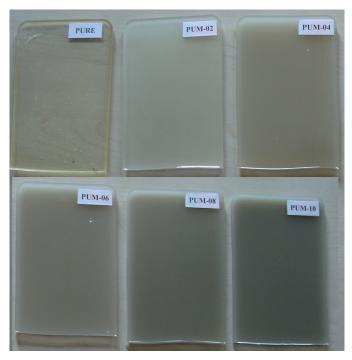


Fig. 2. Pure and pumice-added EPs samples

- 2.3. Characterization
- XRD patterns were obtained from a Bruker XRD system AXS D8 Advance.
- A Perkin Elmer Diamond spectrometer was used for the FT-IR spectra.
- TG/DTA analysis was thanks to Perkin Elmer Diamond. The analyses were conducted by increasing the heating rate by 10°C per minute, up to 800°C.
- The thermal conductivity was determined by Kyoto KEM QTM 500. accordingly, with the standards of ASTM C 1113-90.
- Hardness values were determined by Tronic PD801 as Shore D.
- The vertical burning (UL 94) tests were performed with ASTM D3801 and TS EN ISO 11925-2 standards. Test samples were the dimension of $130 \times 13 \times 5$ mm³. The limiting oxygen index (LOI) was obtained according to ASTM D2863.
- The mechanical properties of EP samples were determined with Shimadzu AG-XD. EPs were tested tensile that according to ASTM E4 and ASTM D638 standards were tested for 5 mm thickness samples and at a speed of 1 mm/min. Three-point bending tests were used for flexural properties. These tests were made within the limits of the standard of ASTM D790. Each sample was tested three times, and the averaged results were used.
- The size of the specimen was $150 \times 100 \times 5 \text{ mm}^3$. After the production processes were carried out, the weights of the EPs were determined with a 0.01 g scale and their dimensions were determined with a 0.02 mm precision caliper. The densities of EPs were calculated with these data.

3. RESULTS AND DISCUSSION

3.1. Structure characterization

Figure 3 shows the XRD patterns of pure EP and added EPs. When the graphs are viewed, it is seen that the XRD peaks for pumice are of lower intensity than pure EP. Intensities of XRD peaks decreased with the amount of additive increased in pumice-added EPs. Its lower intensity can be considered proof of good dispersion of the filler in the polymer matrix [33]. $2\theta = 13.0$ to 29.0° and the main reflection peak is $2\theta = 18.0$ to 20.0° for pure EP. The second group of peaks is the domeshaped curve between $2\theta = 40-50^{\circ}$. These represent broad amorphous peaks of typical EP characteristics [34]. Pumiceadded EPs exhibit similar peaks to pure EP. In addition, weak crystallization peaks ($2\theta = 19.6^{\circ}$, 20.0° and 20.7°) are observed in the structure, they represent pumice arise quartz and albite minerals [22, 35]. Also, we can say that adding pumice into EP does not affect or decrease the crystallinity. As a result, XRD patterns of all pumice samples confirm that the main structure of pumice is amorphous, which complies with the standard types for natural pozzolans (ASTM C 618) [26].

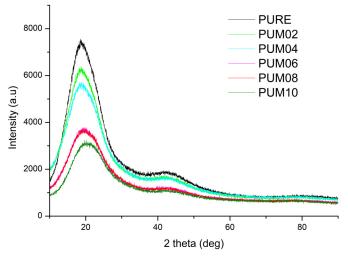


Fig. 3. XRD diffraction patterns of pure and pumice-added EPs

Epoxy resins are low molecular weight polymers containing more than one epoxide group and turn into thermoset material after curing. It is formed by the polymerization of bisphenol and epichlorohydrin. FTIR was used to characterize the structure of EPs. FTIR spectra of pure and pumice-added EPs are shown in Fig. 4. The characteristic absorption peaks formed by the crosslinking of pure epoxy resin can be seen in the spectrum in Fig. 3. These correspond to 2922 cm⁻¹, -CH₂-, -CH₃asymmetrical stretching, 2855 cm^{-1} and 2872 cm^{-1} -CH₂-, -CH₃- symmetrical stretching, 1608 cm^{-1} , 1582 cm^{-1} , and 1508 cm⁻¹ Ar -C=C-H stretching, 1456 cm⁻¹, C-H stretching vibrations of -CH₂-, -CH₃- groups [33], 1297 cm⁻¹ tensioning vibration band of C-O-C in the oxirane ring [36], 1236 cm⁻¹ and 1178 cm⁻¹ -C-C-O-C- stresses, the band of 1116 cm⁻¹ C–N stretching vibrations, 1008 cm⁻¹ vibrations of C-O-C bonds [37], 824 cm⁻¹ Ar =C-H, C-H, and 557 cm⁻¹ -C-H, -N-H bending [38].



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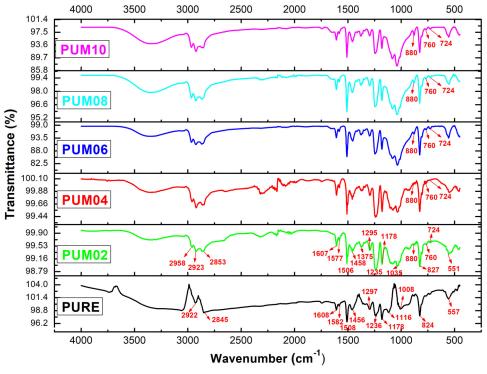


Fig. 4. FTIR spectra of pure and added EPs

The FT-IR spectrum of added pumice displays a powerful large band in the 3600 cm⁻¹-3200 cm⁻¹ region assigned to O-H stretching vibrations. 2958 cm⁻¹, 2923 cm⁻¹ -CH₂-, -CH₃indicate asymmetrical stretching vibrations, 2853 cm⁻¹ -CH₂-, -CH3- symmetrical stretching vibrations. Although the vibrations at 1607 cm^{-1} , 1577 cm^{-1} , and 1506 cm^{-1} assigned to the C=C and C-C bonds of the benzene rings of the resin increase with the resin ratio, they do not show significant changes during polymerization [39]. 1590 cm⁻¹ C=C stretching vibrations occurring in aromatic rings [33], 1458 cm⁻¹ -CH₂-, -CH₃- is the bending stretching [38]. The newly formed peaks correspond to 1375 cm⁻¹ C-N stretching [2] 1079 cm⁻¹ and 1035 cm⁻¹ peaks indicate -C-O-C- stretching. As a result of pumice doping, it is seen that the intensity of the C-H stretching bands at 880 cm^{-1} , 760 cm⁻¹, and 724 cm⁻¹ in the FT-IR spectra increased compared to pure EP.

3.2. Thermal stability

TGA measurements are important analyses used to summarize the thermal stability and the combustion behavior of polymerbased materials [40]. TG and DTG graphs of pure EP and pumice-added EP materials are given in Figs. 5 and 6. It is seen from the TG and DTG curves that pure and pumice-added EP materials show similar three-step decomposition behavior. However, with the addition of pumice, there is no improvement in the maximum decomposition temperatures of the EP material, but decomposition rates decrease depending on the amount of pumice.

In the first decomposition step, a small number of volatile compounds formed by the breaking of the moisture and weak

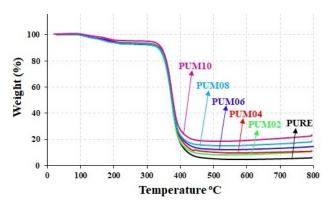


Fig. 5. TGA diagram of pure and added EPs

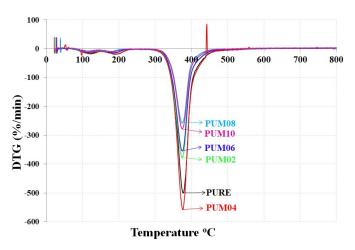


Fig. 6. DTG diagram of pure and added EPs



bonds in the material are separated. The maximum temperature of the first decomposition point $(T_{1 \text{ max}})$ is 119.8°C for pure. This value increased in PUM04 and reached 124.8°C. In the second decomposition step, it was determined that the softer segment was decomposition. Maximum temperature $(T_{2 \max})$ was 188.9°C for pure. $T_{2 \text{ max}}$ 5.3°C increase was determined as 194.2°C in PUM04. The main decomposition occurred in the third step. In particular, the hard segments with the highest decomposition rate were degraded at the third step, the main degraded step. In the third decomposition step, the maximum decomposition temperature $(T_{3 \max})$ of the pure material was determined as 376.9°C. $T_{3 \text{ max}}$ was determined as 374.5°C, 376.4°C, 375.0°C, 375.3°C, and 374.9°C for the addition of 2%, 4%, 6%, 8%, and 10% pumice, respectively. It was previously reported that the decomposition of cross-linked resin in phenolic compounds occurs above 340°C [30,41]. The addition of pumice did not have a positive effect on the maximum decomposition temperature. However, when the maximum degradation rate values were examined, it was determined that EPs with pumice added decomposed more slowly than pure EP [42].

T5_{*wt*}, T10_{*wt*}, and T50_{*wt*} temperature increases were parallel to the added pumice ratio. T5_{*wt*}, T10_{*wt*}, and T50_{*wt*} temperatures for Pure were determined as 186.9°C, 333.7°C, and 376.4°C, respectively. The same temperatures were obtained for PUM10 as 297.6°C, 343.2°C, and 378.3°C, respectively. As a result of the addition of pumice, an increase was achieved at temperatures where a mass loss was observed. Pumice was chosen as an additive material because it has high thermal stability. In addition to these, as expected, there is an increase in the amount of carbon residues at 800°C depending on the pumice addition rate in the material. The carbon residues at 800°C were 6.15% and 23.54% for Pure and PUM10, respectively.

Table 2TGA-DTG data for pure and added EPs

	<i>T</i> _{5wt%} (°C)	<i>T</i> _{10wt} % (°C)	<i>T</i> _{50wt} % (°C)	$T_{1 \max}$ (°C)	$T_{2\max}$ (°C)	$T_{3 \max}$ (°C)	800°C residue (%)
PURE	186.9	333.7	376.4	119.8	188.9	376.9	6.15
PUM02	186.8	332.3	374.3	120.1	183.2	374.5	11.59
PUM04	196.2	338.5	377.1	124.8	194.2	376.4	11.14
PUM06	190.6	337.0	375.6	113.1	186.3	375.0	17.04
PUM08	182.1	336.2	375.7	113.9	177.2	375.3	18.75
PUM10	297.6	343.2	378.3	116.5	177.2	374.9	23.54

 $T_{wt\%}$; thermal decomposition temperature at 5%, 10% and 50% weight loss.

 T_{max} ; the temperature of the peak maximum at the first step, second step, and third step of degradation (°C).

3.3. Flame retardancy and combustion behavior

The limiting oxygen index and vertical burning values of added and pure EP resins are given in Table 3. Pure EP showed a value of 26.5% as LOI, while the UL-94 test showed no results. Pure EP showed no result in the UL-94 test but showed an LOI of 26.5%. It was previously reported that thermosets are flammable and have poor flame retardancy [43]. The addition of pumice to EP prevented dripping and gained flame-retardant properties. When the pumice-added samples are examined, it is seen that the LOI value increases as the additive ratio increases. UL-94 value was determined as V-0 for pumice-added samples. In general, it can be deduced from the data that the addition of pumice can impart good anti-drip properties and flame retardancy to the EP, even at low additive ratios.

 Table 3

 Vertical burning (UL-94) and Limiting oxygen index (LOI) test results

Sample	PURE	PUM02	PUM04	PUM06	PUM08	PUM10
UL-94	No scaling	V-0	V-0	V-0	V-0	V-0
Dripping	YES	NO	NO	NO	NO	NO
Cotton ignited	YES	NO	NO	NO	NO	NO
LOI (%)	26.5	29.4	30.2	31.8	32.1	33.4

3.4. Mechanical properties

Tensile test results of pure EP and EPs with additives are given in Figs. 7 and 8, bending test results are given in Figs. 9 and 10,

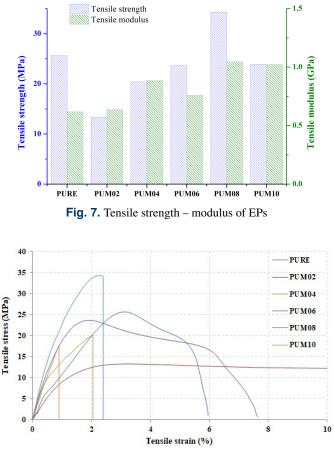


Fig. 8. Tensile stress-strain curves of EPs

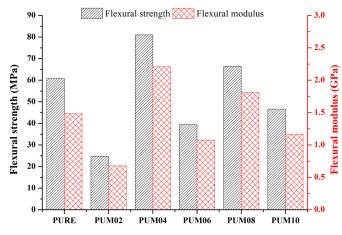


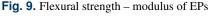


Tensile Flexural Tensile Flexural Tensile Young's Elongation Flexural Flexural fracture fracture break break Sample strength modulus at break energy strength modulus energy strain strain (MPa) (GPa) (%) G_F (MPa) (GPa) G_F (mm)(mm)(N/mm) (N/mm) PURE 25.65 0.6183 8.02 0.0389 0.077 60.79 1.4820 0.0022 0.267 **PUM02** 13.31 0.6371 6.79 0.0751 0.080 24.84 0.6775 0.0049 0.733 PUM04 20.39 0.8854 4.94 0.0490 0.077 81.00 2.2091 0.0016 0.164 PUM06 23.65 0.7591 5.44 0.0422 0.047 39.43 1.0754 0.0032 0.287 PUM08 34.30 1.0451 6.02 0.0291 0.056 66.46 1.8125 0.0020 0.200 PUM10 23.88 0.8609 6.90 0.0418 0.029 46.65 1.1664 0.0025 0.230

Table 4Mechanical properties of samples

and data on mechanical properties are given in Table 4. The mechanical properties of pumice-added EPs were examined, and lower tensile strength values were obtained compared to pure EP (except for PUM08). An increase in Young's modulus values was determined in all samples compared to pure EP. The tensile strength, which was 25.65 MPa for Pure, increased to 34.30 MPa for PUM08, and Young's modulus increased from 0.6183 GPa to 1.0451 GPa (with a 95% confidence interval).





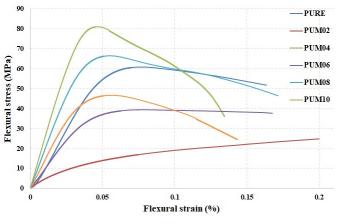


Fig. 10. Flexural stress-strain curves of EPs

Flexural strength increased in some of the added EPs compared to pure EP. Flexural strengths of 60.79 MPa and 81.0 MPa were obtained for Pure and PUM04, respectively. Flexural modulus increased from 1.4820 GPa to 2.2091 GPa (with a 95% confidence interval). Yawas *et. al.* kaolin-reinforced epoxy composite the optimum tensile strength and flexural strength obtained 64.66 MPa, and 74.84 MPa, respectively [44]. As a result, it was determined that the addition of pumice had positive effects on the mechanical properties of EPs.

3.5. Thermal conductivity, density and hardness

The most basic parameter in determining the thermal insulation properties of a material is the thermal conductivity coefficient. Thermal conductivity depends on the cell size of the material, its density, the number of closed cells, and if any, the thermal conductivity of the additive material [45]. For this reason, it is a frequently used method to reduce the thermal conductivity of the main material by adding filler with low thermal conductivity of 0.11–0.21 W/mK was added to reduce the thermal conductivity value of the epoxy resin.

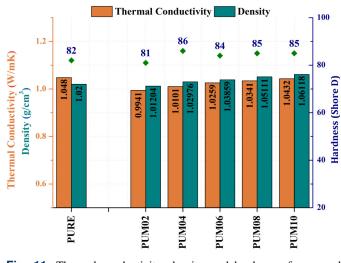


Fig. 11. Thermal conductivity, density, and hardness of pure and added EPs



Effects of pumice additions on thermal and mechanical behaviors of epoxy resin

The effects of adding pumice to EPs on thermal conductivity, hardness, and density variation are shown in Fig. 11. The thermal conductivity of EP (PUM02) with 2% added was reduced by 5.14%, and then it was observed that the k value increased with increasing pumice percentage, and it was close to the thermal conductivity value of pure EP. This situation is directly proportional to the increase in density. Although the thermal conductivity coefficient of the used filling materials is low, when it forms a structure with EP, it helped to form a heat conductive chain that can effectively improve its thermal conductivity and started to increase the conductivity value of EP. Jasim et al. obtained similar results in their study, using micro aluminum as a filler in epoxy, and Sever et al. in their study on pumice-added polypropylene [47, 48]. In addition, Canbolat et al., say that more generally, the thermal conductivity coefficient of materials depends not only on the humidity, density, and temperature of the material but also on the chain structure, macromolecular and atomic, as well as on the interaction with the molecular chain or fiber [49]. The density of pumice decreases at low adding rates compared to pure EP. However, we can say that adding more than 4% affects the density value increasingly. Hardness (Shore D) values obtained a general improvement with the addition of pumice compared to pure EP.

4. CONCLUSIONS

Effects of pumice additives on thermal decomposition, combustion, mechanical properties, and thermal conductivity of epoxy resin were investigated. It was determined that the typical amorphous structure of EP was formed from the XRD peaks, and the main structure preserved its amorphous feature with the addition of pumice. As a result of the addition of pumice from the FT-IR spectra, it was observed that a peak was lost due to the opening of the epoxy rings in the samples. As a result of TG and DTA analysis, the addition of pumice did not affect the maximum decomposition temperature. However, the rate of degradation decreased depending on the amount of pumice. The increase in the amount of carbon residue at 800°C was from 6.95% to 23.54%. UL 94 value was determined as V-0 for all pumice-added samples. The LOI value increased with the increasing amount of pumice. And no dripping occurred. Its mechanical properties improved in tensile strength and Young's modulus, also flexural strength and flexural modulus. The thermal conductivity value decreased by 5.14% with the addition of pumice at a low rate. The hardness value (Shore D) has increased in general.

In conclusion, better results were obtained in thermal properties, mechanical properties, and combustion properties of pumice-added epoxy resins compared to pure EP.

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