Obtaining Diatomite Reinforced Epoxy Composite and Determination of Its Thermophysical Properties

Mustafa DAĞ

[1]Chemical Engineering, Engineering Faculty, Çankırı Karatekin University, Çankırı, Türkiye,

Abstract: In this research, a composite material was produced by adding diatomite soil to epoxy resin. The particle size of the diatomite used is in the range of 297 to 149 microns. It was dried at 378 K before being used as a filling material. By adding 0 kg, 0.001 kg, 0.002 kg, 0.004 kg, and 0.006 kg of diatomite to the epoxy matrix, the composite was produced under atmospheric conditions. To obtain a homogeneous structure, certain amounts of Epoxy A component and diatomite were mixed first. A selected amount of epoxy component B was then added to the mixture. After one day of curing in the laboratory, necessary tests and analyses were carried out. The surface morphology of the produced composite was examined by scanning electron microscopy (SEM). As a result of the analyses and tests, it was seen that the increase in the amount of diatomite increased the porosity in the composite. In addition, it was observed that the density decreased, and the thermal conductivity coefficient varied between 0.110 W /m.K and 0.095 W /m.K It was observed that the hardness was linearly in the range of 77-80 shore D. It has been determined that the addition of diatomite tends to increase the activation energy by modeling the thermal degradation experiments performed in the PID controlled system in nitrogen environment between 300 K and 900 K. Activation energy values are calculated according to the one-dimensional diffusion function with the highest correlation coefficient (R^2) according to Coats-Redfern method when the temperature rise is 10 K/min, and the conversion rate (α) is between 0.15 and 0.85.

Keywords: Diatomite, Epoxy Composite, Density, Hardness, Thermal Conductivity.

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***Corresponding author. E-mail:** mudag@karatekin.edu.tr.

1. INTRODUCTION

Due to the increase in the need for raw materials in proportion to today's technology, which is defined as the age of science, industry has led to the fact that it continues to search for resources at an increasing rate. The limited resources in the world and the lack of sustainable features of existing resources force researchers to develop renewable resources. In this context, the use of natural minerals and ores in nature as well as bio-resources is one of the driving forces of the solutions found for this resource generation. As another type of solution, it can also be said that the development of materials with different properties and the number of properties can be increased. It can be said that the development and production of composite materials with multiple properties is of vital importance to keep up with the speed of technology developing with innovation and to take maximum advantage of limited resources. Although the origin of composites dates back thousands of years and the first composites consisted of mud bricks mixed with straw, their importance has become more understandable in recent years. In addition, it is known from the literature that developed countries have devoted significant resources to the development and production of sustainable and renewable composites in recent years. The properties of composites are generally; low density, wear resistance, favorable fatigue and toughness properties, energy saving, low cost, resistance to oxidation, etc. can be mentioned. Its usage areas can be counted as military technologies, automotive industry, aviation, space technologies, clothing, packaging, medicine, and cleaning products. Such as the superior properties of composites, the cost of raw materials, the precision of production methods, and forming problems also have weak properties. In the production of composites, there are three basic parts, namely matrix, filler, and additive materials. The use of these parts with appropriate materials ensures the production of a composite with the desired properties. Especially at the production stage, a good selection of the matrix element helps to produce a quality composite. Different matrix materials can be used according to different industries (epoxy, polyurethane, polyester, etc.). The matrix element in question in this study is epoxy resin. The superior properties of epoxy resin can be said as the reasons for using it. The density of the epoxy resin is 1140 kg/m^3 at 298 K. The hardness of the epoxy resin takes a value between 70-90 in terms of shore D, depending on the condition of the material. In addition, the thermal conductivity of the epoxy resin can be between 0.15-0.2 W/m.K. These features can be said as excellent adhesiveness, chemical and thermal resistance, electrical insulation, thermoset resin property, chemical stability, low cost, curing shrinkage, and good mechanical properties. Due to these characteristics, it has a wide range of uses from aviation to the energy industry, from the construction sector to adhesives, and from marine vehicles to installation materials (Arat et al., 2022; Aydoğmuş, 2022; Aydoğmuş et al., 2022a, 2022b; Dağ et al., 2022; George & Bhattacharyya, 2021; İNal & Ataş, 2018, 2018; Kaya, 2016; Özdemir, 2019; Şahal & Aydoğmuş, 2021; Yalçın, 2010; Yanen et al., 2022). In the literature, it has been seen that inorganic or organic filling materials such as graphene, nano silica, nanoclay, polyimide, cellulose nanofiber, etc. are added to epoxy matrices to impart various properties. Apart from the outstanding properties of epoxy resins, there are also various weaknesses. Due to the three-dimensional network structure, high internal stress, high brittleness, poor fatigue resistance, microcrack formation, strength reduction during the high-temperature curing process, etc. can be counted. In this context, the study of eliminating weak properties by adding some additives and fillers continues to research. Epoxy resins are produced by the reaction of crosslinking monomers with curing agents during production. The epoxies used in the market are bisphenol A (BPA) based industrially produced petroleum-based resins. The use of petroleum-based ones in European and American continental countries is increasingly hindered since they cause health problems. Studies are continuing the elimination of harmful properties in composite production (Chen et al., 2019; Dahmen et al., 2020; Guo et al., 2021; Huang et al., 2012; Koo et al., 2016; Li et al., 2018; Ma et al., 2019; Mantecón et al., 1987; Pathak et al., 2016; Rad et al., 2019; Seachrist et al., 2016; Serra et al., 1986; Sogancioglu et al., 2017; Sun et al., 2014, 2015, 2020; Wongjaiyen et al., 2018; Xu et al., 2018; Yang et al., 2020). In recent years, research has been conducted on the use of diatomite in composite production. The most striking physical property of diatomite is its high porosity and low specific gravity. The thermal conductivity of diatomite is approximately 0.09 W/m^2 at low temperatures. This low thermal conductivity is explained by the porous structure and low density of diatomite. The specific gravity of the dry diatomite has a value between 150 and 400 kg/m^3. Diatomite also has properties such as being in the form of $SiO_2.nH_2O$, being abundant, has a high-water holding capacity, having a hardness of 1.5-6 Mohs, and is abrasive. It also suggests that due to its high chemical resistance, it will help to give the desired properties to composite material (Davis et al., 2016; Karaman et al., 2011; Qi et al., 2007; Qin et al., 2015; Taş & Çetin, 2012; Wang et al., 2015; Zhang et al., 2017) (33-39). In this study, it was aimed to produce diatomite added composite, which is a material of organic origin, by adding diatomite to epoxy resin. In addition, it is aimed to produce more effective composites by utilizing the superior properties of diatomite such as high porosity and low specific gravity.

2. MATERIAL AND METHOD

Epoxy A and Epoxy B components used in experimental studies were supplied by Turkuaz Polyester company. In addition, diatomite used as a filler was purchased from the Turkish diatom company. Diatomite is added to the epoxy resin A at different rates (0 wt.%, 1 wt.%, 2 wt.%, 4 wt.%, and 6 wt.%) and mixed at 1000 rpm for 5 min.

After adding epoxy resin B, respectively, at room temperature at a mixing speed of 1200 rpm for 90 seconds, they are poured into standard molds. After waiting 24 hours for the curing of the obtained samples, necessary tests and analyses have been carried out. In addition, mold release agents are applied to standard steel cylindrical molds, allowing the samples to come out easily. In Figure 1, the epoxy composite production scheme in experimental studies is expressed. Here, both the quantities of the ingredients and the order of use are very important. Also, the fillers should provide a homogeneous distribution to the synthesized composite.

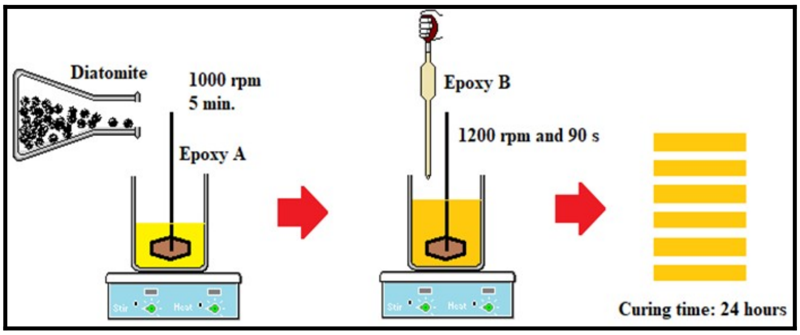

Figure 1: Production scheme of diatomite reinforced epoxy composite.

The amounts of each ingredient used are expressed in Table 1. The amounts of other components are kept constant, except for the filler (diatomite) used here.

Table 1: Production plan of diatomite-reinforced epoxy composite.

Epoxy A (kg)	Epoxy B (kg)	Diatomite (kg)
0.067	0.033	0
0.067	0.033	0.001
0.067	0.033	0.002
0.067	0.033	0.004
0.067	0.033	0.006

The devices and standards used for analyses in experimental studies are as follows: Shore D hardness tests have been carried out by the ISO 868 (ASTM D 2240) standard, and dielectric properties have been measured with the Novacontrol Alpha-A impedance analyzer at a temperature of 300 K in the frequency range of 10 Hz and 10 MHz.

3. RESULTS AND DISCUSSIONS

The variation of the density of the obtained epoxy composites is shown in Figure 2 depending on the diatomite ratio by mass. It is understood from the graph that diatomite reduces the density of the epoxy composite linearly. Obtaining a linear graphic can be interpreted as an indication that the mixture is homogeneous and well dispersed. When the distribution is not good, graphs with parabolic or different features are obtained in positive or negative directions. Based on the literature, it can be said that the addition of diatomite, which has a lower density than the epoxy resin, to the epoxy caused a tendency to decrease the density of the epoxy. For this reason, increasing the diatomite ratio played an active role in reducing the density of other samples.

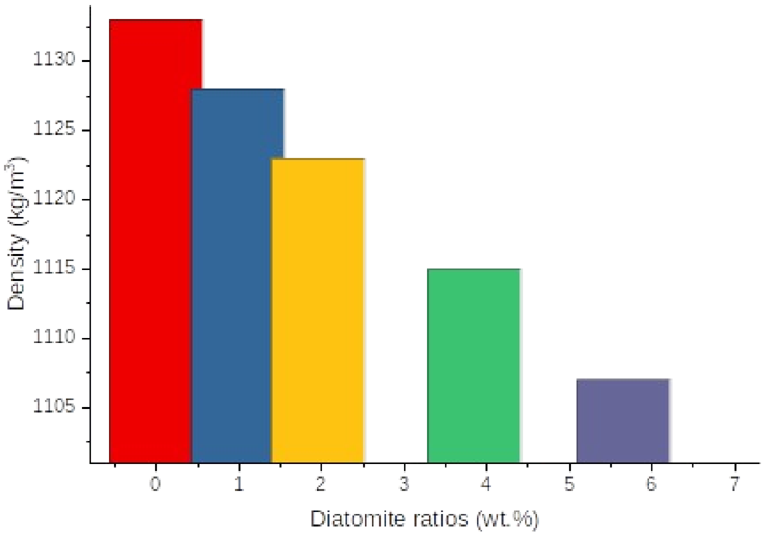

Figure 2: The effect of diatomite reinforcement on the density of epoxy composite.

In the graphs shown in Figure 3, it is seen that the hardness of the synthesized epoxy composites increased with diatomite reinforcement. Shore hardness unit is used to determine the hardness value of polymer or flexible materials, while Mohs hardness unit is used for the hardness value of ores or minerals. It is known in the literature that epoxy resins have a hardness between 70-90 as shore D. The hardness of diatomite is between 1.5 and 6 in Mohs. As can be seen in Figure 3, while pure epoxy Shore D is at the value of 77, it is seen that this value increases as the proportion of diatomite added to it raises. this suggests that this is since diatomite provides good physical adhesion during uniform dispersion in epoxy resin. Because Mohs hardness is based on the principle of measuring the hardness by drawing the sample. the deeper a sample is drawn, the less its hardness. The deep lines of the diatomite, which has a Mohs hardness of 1.5-6, formed good physical bonds with the epoxy and this can be interpreted as an increase in the hardness of the composite. As it is known, the more roughness there is in a material, the more adhesion and adhesion occur. Considering the adhesive properties of epoxy resins, this result has contributed to the accuracy of the interpretation.

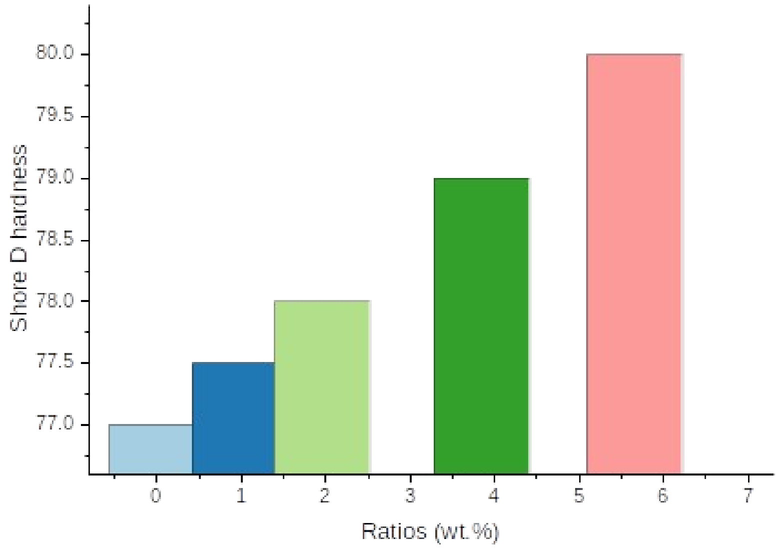

Figure 3: The effect of diatomite reinforcement on the hardness of the epoxy composite.

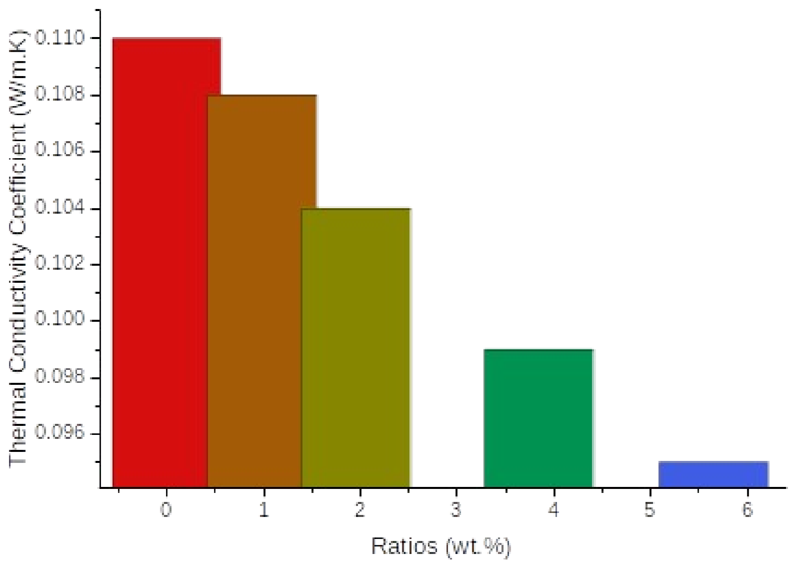

Figure 4: The effect of diatomite reinforcement on the thermal conductivity of the epoxy composite.

It is seen from the literature that the thermal conductivity of epoxy resin is between 0.15-0.2 W/m.K. The thermal conductivity of the diatomite added as an additive is approximately 0.09 W/m.K. When Graph 4 is examined, it is seen that while the thermal conductivity value of pure epoxy is 0.11 W/m.K, as the diatomite added to the composite increases, the thermal value gradually decreases, very close to the linear characteristic. The fact that the graph is close to linearity can be interpreted as a good mix and distribution, but not fully linear due to the effect of different reasons. As other reasons, it is thought that air gaps remain in the parts of the diatomite which have a porous structure in the inner regions during mixing and the thermal conductivity in the mortar affects the total thermal conductivity value. In addition, although the curing time is expected to be 24 hours, it is thought that there is no escape of air from the diatomites in the inner regions. It is also possible for the epoxy matrix to absorb air during mixing at 1000-1200 rpm.

Activation energy (*Ea*) values are calculated according to Coats-Redfern (Table 2). Activation energy (*Ea*), Arrhenius constant (*A*), and temperature rise rate (β) values were expressed. Coats-Redfern (Eq. 9) model are shown in the below equations.

$$\ln\left(\frac{g(\alpha)}{T^2}\right)=\ln\frac{AR}{Ea\beta}-\frac{Ea}{RT} \quad \text{(Eq. 1)}$$

Plotting *1/T* versus $\ln(g(\alpha)/T^2)$ according to Coats-Redfern method, the slope will give the value *-Ea/R*. Since the value of R = 8.314 J/mol·K here, the slope can be easily calculated. Coats-Redfern method has been preferred because it found the most compatible results with the n-order function. In this method, the highest correlation coefficient was obtained with three-dimensional diffusion equation. Activation energies of the pure epoxy and epoxy composites reinforced with fillers are calculated in thermal decomposition experiments carried out at a heating rate of about 10 K/min at 878 K.

Table 2: Activation energies of pure and diatomite reinforced epoxy composites.

Ratio (wt.%)	Activation Energy (kJ/mol)
0	185.26
0.99	189.65
1.96	194.70
3.85	199.86
5.66	207.43

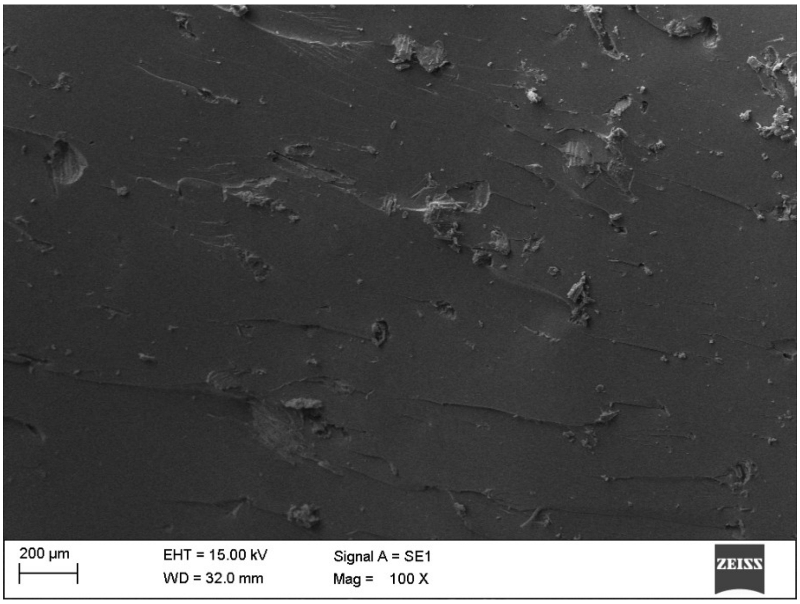

Figure 5: SEM image of diatomite (5.66 wt.%) reinforced epoxy composite.

In the SEM image, it was observed that the addition of diatomite increased the number of pores in the epoxy resin. Here, the addition of 5 percent diatomite led to the appearance of prominent pores. It is known from the literature that the SEM images of pure epoxy resin are in the form of a non-porous and flat surface. Here, it was observed that the pores started to appear in the diatomite additive at a low percentage. It is of no doubt that denser pores will be seen with the increase in the diatomite percentage. It has been observed in some experiments that the diatomite layer above a certain ratio causes irreversible breaks and cracks on the epoxy resin. The porosity of the matrix can provide significant advantages compared to the areas to be used (Conradi et al., 2020).

4 .CONCLUSIONS

In this study, a composite material was produced by adding diatomite to epoxy resin. Various tests were carried out on composite materials prepared by adding different amounts of diatomite. As a result of these tests, with the addition of diatomite, results were obtained in the form of a decrease in the density of the epoxy matrix, an increase in its thermal conductivity, and a low-slope linear increase in its hardness. When the SEM image is examined, it can be considered that the increase in porosity is the reason for the decrease in density. The increase in porosity also indirectly affected the thermal conductivity. It has been observed that it reduces the thermal conductivity by 13 percent. The increase in the activation energy can be interpreted as the addition of diatomite increases the thermal stability on the epoxy resin. As suggestions for future studies, the following points can be drawn: Lower diatomite sizes can be used in the matrix to reduce porosity, the effect of temperature can be examined by producing in different temperature environments, trials can be carried out with a second or third matrix partnership with epoxy resin.

5. ACKNOWLEDGMENT

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