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Research Article

Study on the Synergies of Nanoclay and MWCNTs to the Flame Retardant and Mechanical Properties of Epoxy Nanocomposites

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Modern flame retardants are organic compounds containing halogen or phosphorus groups and are not always well dispersed in polymers. Thus, by using a small amount of nanoclay and multiwalled carbon nanotubes (MWCNTs), they can significantly reduce the number of conventional flame retardant additives, making the material with optimal flame retardant properties. Conventional flame retardants always have some negative effects on the mechanical properties of the polymer substrate, so by using nanoclay and MWCNTs, those adverse effects can be minimized and overcome. In this work, in order to improve the mechanical properties and flame retardant of nanocomposite materials, nanoclay I.30E and MWCNTs are mixed into epoxy, with the selected percentage of 2% and 0.02% by weight, respectively, stirring mechanically for 7, 8, and 9 hours at 3000 rpm at 80°C, then performing ultrasonic vibration for 6 hours at 65°C.

1. Introduction

Multiwalled carbon nanotubes tend to aggregate into bundles in solvents or in plastic if dispersion techniques are not reasonable. The uniform dispersion inside the polymer, improving wetting power and good adhesion, is an important issue in making nanocomposites [1–3]. Montazeri and Chitsazzadeh [4] studied the effect of ultrasonic parameters on the mechanical properties of composite MWCNTs/epoxy materials. In this study, the authors studied the effects of time and the power of ultrasonic stir to disperse 0.5% by weight of MWNTs in epoxy. To disperse MWNTs in polymer substrates, an ultrasonic stirrer was used with the capacity of 25, 50, and 100 W and the stirring time of 15, 45, and 135 minutes, respectively. The results showed that when the ultrasonic stirring time was increased, there was an increase in the initial value in tensile strength, and then there was a decrease in value at longer ultrasonic stirring time, despite using the ultrasonic stir method combined with mechanical stir to disperse MWCNTs into epoxy. Arash et al. have dispersed MWNTs with a content of 0.1, 0.5, 1, 1.5, and 2% by weight which is premixed with epoxy with a mechanical stirring method. Then, the mixture was stirred for 2.5 h at a capacity of 60 W with the ultrasonic stir. The dispersion results were tested by SEM and showed that the dispersion of MWCNTs was very good [5]. Kaynak et al. have studied the mechanical properties, flammability, and structural morphology of epoxy/clay nanocomposite resins. First, the nanoclay was dispersed in epoxy resin by mechanical stirring and then stirred in the ultrasound. Nanocomposite containing nanoclay was prepared at concentrations of 0.5, first, 2, and 3% by weight [6–8]. Recent research on the combined effect of organic clay minerals multiwalled carbon nanotubes into the system shows that the presence of organic clay minerals has increased the mechanical properties and flame retardant of polymers [9, 10]. Regarding the effect of MWCNTs and nanoclay on the mechanical properties of epoxy composite nanomaterials, the results showed that with 3% of nanoclay mass combined with 1% mass of MWCNTs, the mechanical properties were significantly improved [11]. Epoxy is a thermosetting polymer and engineering material for structural and composite applications. However, pure epoxy has the disadvantage of low hardness and toughness, which limits its use in practice. Epoxy has been

blended with polyamide (thermoplastic polymer) to enhance its toughness and mechanical properties. Combination of carbon nanoparticles in the epoxy/polyamide mixture has been used to improve the morphological and physical properties of the materials [12]. In addition to improving the brittleness of epoxy resins, Nguyen studied the modification of epoxy resins by epoxy oil and reinforced with MWCNTs [13]. The improvement of epoxy resin's flame retardants was also investigated by Guohua Chen et al., on the combination of MWCNTs with graphene nanoplatelet [14].

Studying the synergy between MWCNTs and nanoclay in the fabrication of nanocomposite materials to improve mechanical properties and flame retardation is still a problem that many scientists are concerned about [15–18]. There are many published works, and it is concluded that the combination of MWCNTs and nanoclay is effective in improving the mechanical properties and fire retardation [19–21]. The purpose of this project is to use ultrasonic vibration method with mechanical stirring to disperse MWCNTs and nanoclay and into epoxy resin substrate. Mechanical properties and flame retardants were studied. In this study, the specifications for fabricating nanocomposite materials are found on the basis of the synergies of MWCNTs and nanoclay. In terms of simple and effective techniques, materials with high flame retardant and improved mechanical properties are made.

2. Materials and Methods

2.1. Materials

(i) Epikote 240 epoxy (E 240) from bisphenol F, of Shell Chemicals (USA) with 24.6% epoxy content, equivalent of epoxy group 185-196, viscosity at 25°C: 0.7 ÷ 1.1 Pa s.

(ii) Diethylene triamin (DETA), Dow Chemicals (USA), chemical formula of DETA: $H_2N(CH_2)NH(CH_2)_2NH_2$, MW: 103 g mol^{-1}, specific gravity at 25°C: 0.95 g/cm^3.

(iii) Nanoclay I.30E of Nanocor (USA): ivory white powder, specific gravity 1.7 g/cm^3; denatured by octadecylamine.

(iv) Multiwall carbon nanotubes (MWCNTs) from Showa Denko (Japan). Synthesized by catalytic vapor deposition method. MWCNTs have an average diameter of 40-45 nm, average length 3, and a density of 0.08 g/cm^3.

2.2. Specimen Preparation. Nanoclay and epoxy E 240 resin were heated at 80°C for 1 hour to reduce viscosity. A percentage of mass nanoclay (2 wt.%) and MWCNTs (0.02 wt.%) was mixed into E 240 epoxy resin by the mechanical stirring method at a speed of 3000 rpm, keeping the temperature stable at 80°C for 8 hours then conducting ultrasonic vibrations in 6 h at 65°C. The homogeneous mixture was deaerated for 30 minutes, then added with a corresponding DETA curing agent, stirred with water mechanically (it is possible to add ice to water to reduce the temperature in the local curing reactor), and mixed so that the curing agent is at 60-80 rpm for 15 minutes. The mixture was poured into a stainless steel mold and allowed to solidify at room temperature for 24 hours, then stabilized 7 days to determine the properties, each property at least 5 samples.

2.3. Characterization and Testing

2.3.1. Mechanical Properties

(i) Tensile strength is determined according to ISO 527-1993 on INSTRON 5582-100 kN (United States) with a pulling speed of 5 mm/min, temperature 25°C and humidity 75%.

(ii) Flexural strength is determined according to ISO 178-1993 standard on INSTRON 5582-100 kN (United States) with bending speed of 5 mm/min, temperature 25°C and humidity 75%.

(iii) Compressive strength is determined according to ISO 604-1993 on INSTRON 5582-100 kN (United States), compression speed 5 mm/min, temperature 25°C.

(iv) Izod impact resistance is determined according to ASTM D265 on Tinius Olsen (USA). Measured at the Polymer Materials Research Center, Hanoi University of Science & Technology (HUST).

2.3.2. Flame Retardants Properties

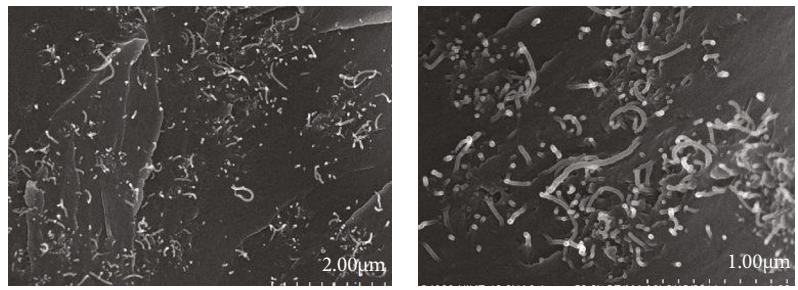
(i) Limit oxygen index (LOI) according to ASTM D2863-12 and JIS K720 (Japan): the bars used for the test are $150 \times 6.5 \times 3$ mm^3.

(ii) Horizontal combustion tests (UL-94HB): the test piece of the standard bar must be 125 ± 5 mm long, 13.0 ± 0.5 mm wide, and supplied in minimum thickness and thickness of 3.0 mm $(-0.0 + 0.2)$ (ASTM D635-12).

(iii) Fire resistance: equipment, specially designed for fire and flame retardant of thermoplastic, thermosetting, and hard and thin laminates. Designed and engineered to meet the following standards: ASTM D 757; specimen size $3.17 \times 12.7 \times 121$ mm^3. Maximum temperature 950°C. Measured at the Polymer Materials Research Center, Hanoi University of Science & Technology (HUST).

2.3.3. Method of Determining the Structural Morphology of Materials (FE-SEM). Morphological structure of fractured surface samples measuring tensile strength of composite materials surveyed by imaging on field emission scanning electron microscope (FE-SEM) (Evacseq error codes, S-4800 of Japan), performed at the Department of Superstructure Analysis, Department of Virus, Central Institute of Hygiene and Epidemiology. Scanning SEM electron microscope method on Jeol JSM-6360 LV device (Japan).

2.3.4. XRD. X-ray diffraction is performed on D8-Advance, Brucker (Germany) machine (CuKα radiation with a



(a) (b)

Figure 1: FE-SEM image of the fractured surface pulling the nanocomposite MWCNTs/epoxy E 240 with ultrasonic vibration dispersion mode 6 hours then 5-hour mechanical stirring. (a) Magnification ×20.0k. (b) Magnification ×50.0k.

scanning speed of 1°/min at 2θ range of 1-30°), and measured at the Laboratory of Materials Chemistry, Faculty Chemistry, Vietnam National University.

3. Results and Discussion

3.1. Study on the Method of Dispersing MWCNTs into Epoxy by Mechanical Stirring Technique Combined with Ultrasonic Vibration

3.1.1. FE-SEM Images of the Residua of MWCNTs/Nanoclay Epoxy Nanocomposites. Disperse 0.02% by weight of MWCNTs into E 240 epoxy resin by mechanical mixing method combined with ultrasonic vibration. In turn, mechanical stir of the samples was carried out at a rate of 3000 revolutions per minute for 5 hours, 6 hours, 7 hours, and 8 hours at 80°C; then, samples were supersonic vibrated for 6 hours at 65°C. Evaluation of the dispersion of MWCNTs through field emission scanning electron microscopy (FE-SEM) of fracture surfaces of the sample is shown in Figure 1.

This study evaluated dispersion level of MWCNTs in epoxy whether to have the orderly relationship between mechanical stir and ultrasonic vibration.

Figure 1 observes that if the dispersion in the following order is carried out, preultrasonic vibrations then stirring mechanically, it seems that the MWCNTs have a disturbance when they are stable after 6 hours of using the ultrasonic vibrator. Therefore, carbon nanotubes are distributed in epoxy resin with thick and irregular density. While using a mechanical mixer first, then conducting ultrasonic vibration, the color has a pure black color, and the MWCNTs are relatively better distributed (FE-SEM images in Figure 2).

Figures 2(a)–2(d) show that when stirring mechanically for 8 hours, with a speed of 3000 revolutions per minute, then using the ultrasonic vibrator for 6 hours, the MWCNTs dispersed and distributed with uniform density in epoxy.

Compared to other samples such as mechanical stiring for 5 hours, mechanical stirring for 6 hours, and mechanical stirring for 7 hours, the samples stirring mechanically for 8 hours is the best dispersed MWCNTs.

With mechanical stirring for 8 hours at a rate of 3000 rpm, no ultrasound is used; the multiwalled carbon nanotubes are not completely separated and are still wrapped into clusters like wrappers in which the flare consists of wrapped MWCNTs. Separated and uncoupled, these wraps of medium size are about 500 nm across (Figures 2(e) and 2(f)).

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Figures 2(a)–2(d) show that when stirring mechanically for 8 hours, with a speed of 3000 rpm, then using ultrasound for 6 hours, the MWCNTs dispersed and distributed evenly in epoxy. Compared with other samples such as mechanical stirring for 5 h, mechanical stirring for 6 h, and mechanical stirring for 7 h, the mechanical stirring of sample for 8 h carbon nanotubes multiwall dispersion is better.

3.1.2. Effect of Dispersion Mode MWCNTs on Mechanical and Flame Retardant Properties of Materials MWCNTs/Epoxy Nanocomposite. From Table 1, the sample with stirring mechanically within 8 hours and ultrasonic vibrator within 6 hours shows high mechanical strength. Specifically, the tensile strength, flexural strength, compression strength, and impact resistance increased by 34.83%, 26.97%, 26.31%, and 149.22%, respectively. The other samples (mechanically stirred within 5, 6, and 7 hours) are not enough in the time of stirring to support the ultrasonic vibrating. Therefore, the mechanical strength has increased but not as high as the sample stirred mechanically within 8 hours.

The effect of mechanical stirring time on flame retardant properties of nanocomposite materials MWCNTs/epoxy E 240 is shown in Table 2. As a result, in comparison to epoxy resin, all evaluation parameters of fire resistance increased due to good dispersion of MWCNTs and strong interaction with polymer chains. Nanocomposite (8/6) sample shows the best results.

3.2. Manufacturing Research about Epoxy-Based Composite Materials E 240 with Additional Nanoclay I.30E and MWCNTs

3.2.1. Disperse Nanoclay and MWCNTs into Epoxy. In order to improve the mechanical properties and flame retardant of nanocomposite materials, it is recommended to mix

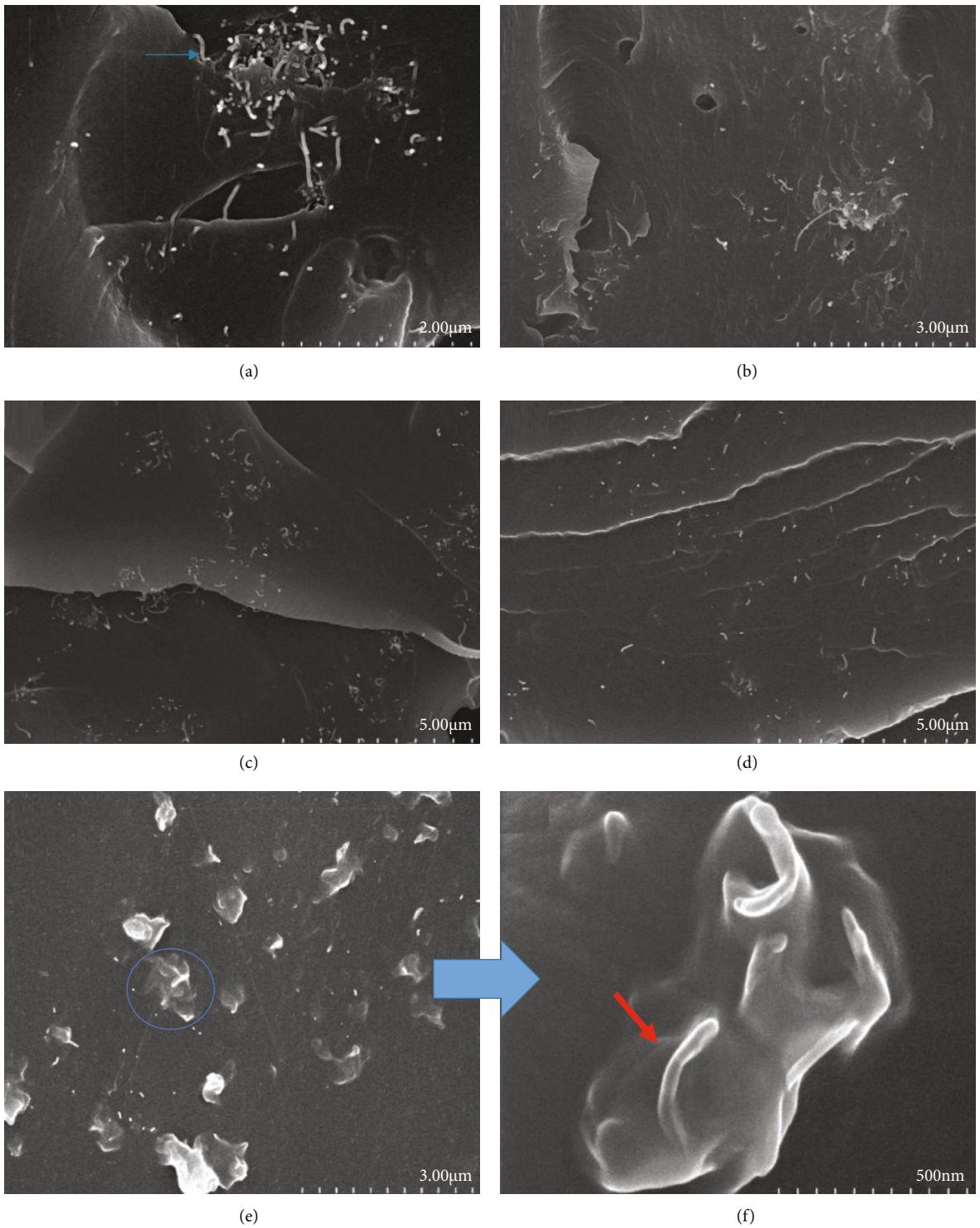

Figure 2: FE-SEM image of the surface of the fractured sample that dragged the nanocomposite material MWCNTs/epoxy E 240: (a) 5-hour mechanical stirring, ultrasonic vibration 6 hours; (b) agitator 6 hours, ultrasonic vibration 6 hours; (c) mechanical stirring for 7 hours, ultrasonic vibration for 6 hours; (d) stirring mechanical for 8 hours, ultrasonic vibration for 6 hours; (e, f) 8 mechanical stirring.

nanoclay I.30E and MWCNTs into epoxy, with the selected percentage of 2% and 0.02% by weight, respectively, stirring mechanically for 7, 8, and 9 hours at 3000 rpm at 80°C, then ultrasonic vibration for 6 hours at 65°C. FE-SEM image of fracture surfaces of the sample was used to evaluate dispersion. The results are presented in Figure 3.

From Figures 3(a)–3(d), MWCNTs can be dispersed uniformly in epoxy E 240 resin. In addition, in Figure 3(c), it can be seen that the MWCNTs are dispersed uniformly, besides the nanoclay layer I.30E dispersed in epoxy E 240 resin. With the surface capture not only observing the dispersion of MWCNTs but also including nanoclay, although not in any

Table 1: Effect of mechanical stirring time and ultrasonic vibration on the mechanical properties of MWCNTs/epoxy nanocomposite materials E 240.

Samples	Tensile strength, (MPa)	Flexural strength, (MPa)	Compressive strength, (MPa)	Impact strength, (kJ/m^2)
Neat epoxy resin	55.90	86.75	156.08	7.11
Nanocomposite (8/0)	53.02	91.15	183.71	8.17
Nanocomposite (5/6)	67.58	91.16	188.35	13.69
Nanocomposite (6/6)	68.28	91.90	187.26	14.25
Nanocomposite (7/6)	69.37	92.70	180.29	15.37
Nanocomposite (8/6)*	*75.37*	*110.15*	*197.15*	*17.72*

Note: *8-hour mechanical stirring, 6-hour ultrasound.

Table 2: Effect of mechanical stirring time and ultrasonic vibration on the flame retardant properties of nanocomposite materials MWCNTs/epoxy E 240.

Samples	LOI (%)	Combustion rate (mm/min)	UL94 HB (mm/min)
Neat epoxy resin	20.6	28.41	—
Nanocomposite (8/0)	22.1	24.90	26.80
Nanocomposite (5/6)	22.8	23.79	22.34
Nanocomposite (6/6)	22.8	23.45	22.06
Nanocomposite (7/6)	23.7	22.67	21.02
Nanocomposite (8/6)	23.7	22.15	20.08

strict order, the thermal conductivity of nanomaterials as well as the layers of coal formed after combustion can totally explain the possibility of circuit breaker fire of flame and heat transfer capability of the composite with additives MWCNTs/nanoclay I.30E.

The dispersion states of nanoclay I.30E in epoxy E 240 resin can be obtained from X-ray diffraction diagram of the sample MWCNTs/nanoclay I.30E/epoxy E 240 (Figure 4). For the nanoclay-I30E sample, there appears a peak at angle of $2\theta = 40$ corresponding to the basic distance $d = 22.128$ Å $= 2.2128$ nm. For nanocomposite samples with 2% mass of nanoclay I.30E and 0.02% mass of MWCNTs when stirring mechanically for 8 h and conducting ultrasonic vibration for 6 h, no clear peaks were observed on XRD spectra, and a silicate layer of nanoclay I.30E can be layered in epoxy E 240 substrate.

The thermal conductivity of nanomaterials as well as the layers of coal formed after combustion can explain the flame flammability and heat transfer of nanocomposite materials with nanoclay additives I.30E/MWCNTs/epoxy E 240.

About fire protection mechanism of nanoclay additives and multiwalled carbon nanotubes, with epoxy resin, oxidizing agents easily attack, and so, plastic samples will easily catch fire. MWCNTs have formed a thin film covering the outside of the material. Although it is thin, it is intricately interwoven by MWCNTs with the thermal conductivity along the tube axis as well as the thermal resistance between the pipe layers very well. The difference with the addition of MWCNTs and nanoclay made the nanoparticles/epoxy mixtures not easily liquefied as samples with only infinitive epoxy resin, even at high temperatures. Criteria for evaluating fire retardation are illustrated in Figure 5.

From Figure 5, it was found that the flame retardant ability of the E 240 epoxy composite material composite materials is present simultaneously with I.30E nanoclay and the multiwalled carbon tube through the limiting oxygen index (LOI) and was dominated by the dispersion method (the time of mechanical stirring combines ultrasonic vibration and no ultrasonic vibration). The effect of the combination of two nanoclay flame retardants I.30E and MWCNTs increased the limiting oxygen index and achieved an oxygen index of 24.1% for the MN (dispersed by ultrasonic vibration for 6 hours).

And the oxygen index is also improved when the method of changing dispersion is mechanical stirring and ultrasonic vibration, especially at the stirring time for 8 hours combined with ultrasonic vibration for 6 hours that got the highest LOI index of 25%. The presence of nanoclay I.30E and MWCNTs has increased the amount of oxygen needed for the ignition process, making it more difficult to burn materials. Class-structured networks formed by nanoclay I.30E and MWCNTs are an important role as a shield to prevent the formation of free oxygen radicals (O^O) from the surface of epoxy E 240 reducing.

The combustion rate during the UL 94HB test of nanocomposite materials is shown in Figure 6. Figure 6 shows that the combustion rate (the speed of fire) of the samples, determined by standard UL 94HB, reduced when nanoclay I.30E and MWCNTs are present simultaneously, compared to only nanoclay or MWCNTs samples. For samples with nanoclay and MWCNTs, the rate of combustion decreased the most—the lowest (18.60 mm/min) in the mechanical stirring mode for 8 hours combined with ultrasonic vibration for 6 hours.

Figure 6 shows SEM images of the residual char of MWCNTs/nanoclay epoxy nanocomposites (Figure 6) which was very dense and continuous; no holes could be found. This was determined by the dispersion states of nanocomposites in the residues.

Mechanical properties of nanocomposite MWCNTs/nanoclay I.30E/epoxy E 240 materials are presented in Table 3.

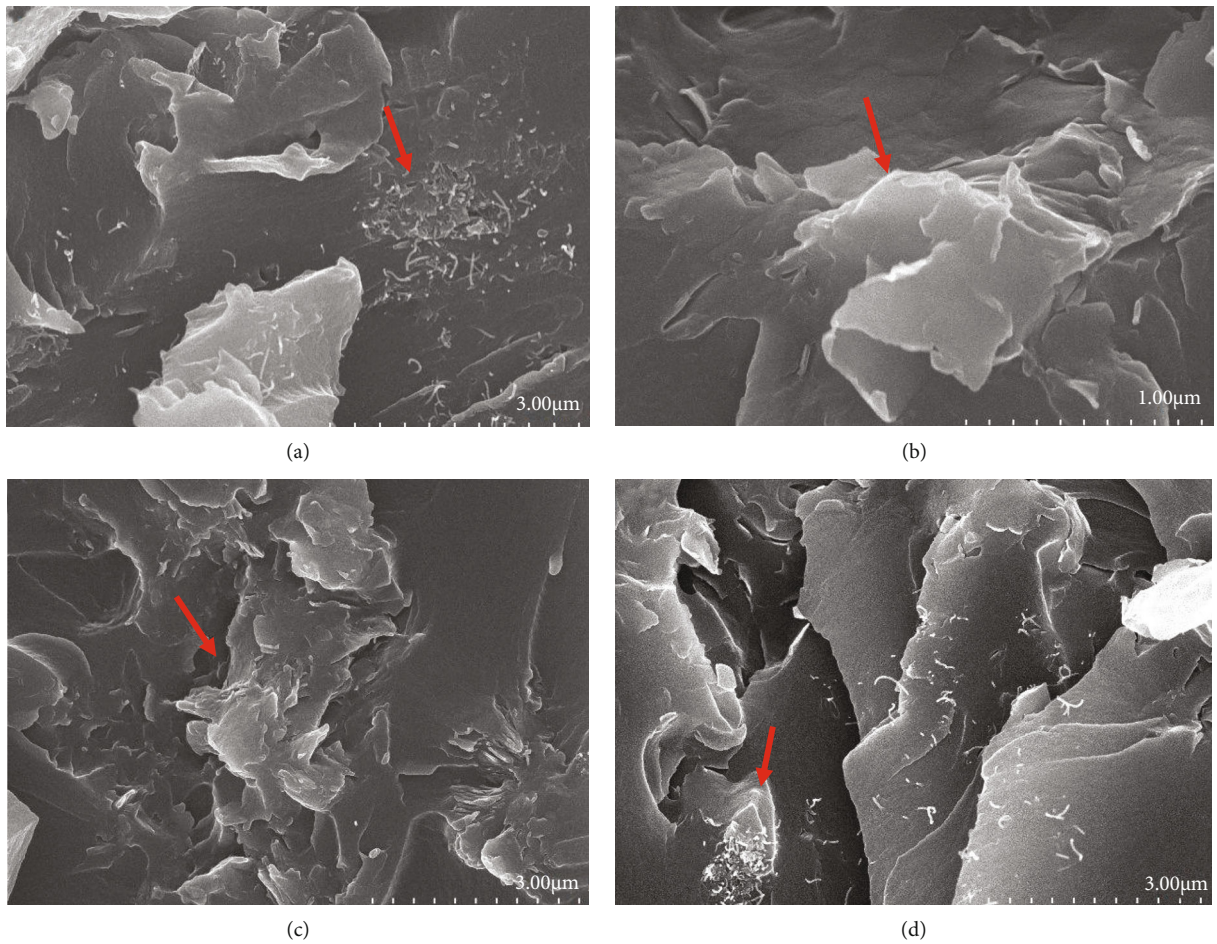

Figure 3: FE-SEM image of the fractured surface dragging the nanocomposite materials MWCNTs/epoxy E 240: (a) ultrasonic vibration for 6 hours; (b) stirring mechanically for 7 hours, ultrasonic vibration for 6 hours; (c) stirring mechanically for 8 hours, ultrasonic vibration for 6 hours; (d) stirring mechanically for 9 hours, vibrating ultrasound for 6 hours.

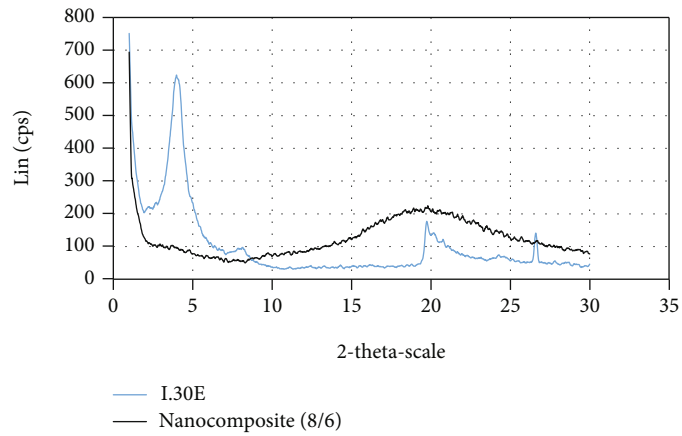

Figure 4: XRD diagram of MWCNTs/nanoclay I.30E/epoxy E 240 nanocomposite material when dispersed in an 8-hour mechanical stirring mode and 6-hour ultrasonic vibration: nanocomposite (8/6) and nanoclay (I.30 E).

From Table 3, the mechanical strength increases when mixing nanoclay I.30E and MWCNTs simultaneously into epoxy E 240. Especially when dispersing with mechanical stirring time for 8 hours, with ultrasonic vibration for 6 hours, the sample has high mechanical strength (95.5 MPa tensile strength, flexural strength 115.45 MPa, compressive strength 219.10 MPa, and impact resistance 22.30 kJ/m^2). Therefore, mechanical stirring technique with a speed of

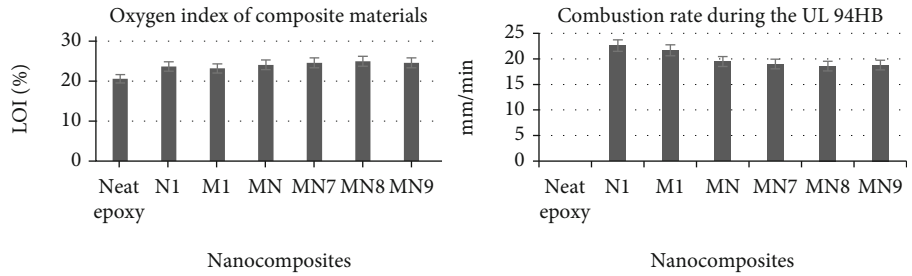

Figure 5: Oxygen index of composite materials: epoxy E 240/nanoclay I.30E (N1), epoxy E 240/MWCNTs (M1), MN (6-hour ultrasonic vibration), MN7 (stirring for 7 hours mechanically, 6-hour ultrasonic vibration), MN8 (8-hour mechanical stirring, 6-hour ultrasound), and MN9 (9-hour mechanical stirring, 6-hour ultrasound).

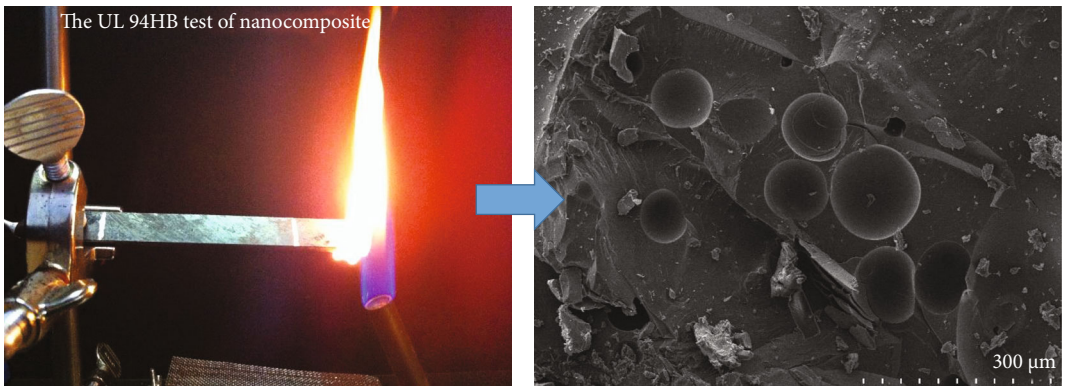

Figure 6: SEM images of the residua of MWCNTs/nanoclay epoxy nanocomposites.

Table 3: Mechanical properties of nanocomposite MWCNTs/nanoclay I.30E/epoxy E 240 when ultrasonic vibrating for 6 hours and mechanical stirring at 7, 8, and 9 hours combined with ultrasonic vibration for 6 hours.

Samples	Tensile strength (MPa)	Flexural strength (MPa)	Compressive strength (MPa)	Impact strength (kJ/m^2)
Neat epoxy resin	55.90	86.75	156.08	7.11
MWCNT/nanoclay (MN)	90.25	106.53	190.01	15.57
MWCNT/nanoclay (7) (MN7)	92.78	110.60	191.48	16.53
MWCNT/nanoclay (8) (MN8)*	*95.50*	*115.45*	*219.10*	*22.30*
MWCNT/nanoclay (9) (MN9)	93.12	112.10	200.56	20.63

Note: *MN8 (8-hour mechanical stirring, 6-hour ultrasound).

3000 rpm for 8 hours then conducting ultrasonic vibration for 6 hours was selected to continue the study in the following sections.

4. Conclusions

In this study, ultrasound as a conventional dispersion technique was used. The work studied the effects of the mechanical dispersion method combined with ultrasound on the mechanical properties, and fire resistance of epoxy nanocomposites was studied. Different mechanical stirring times (7, 8, and 9 hours) were conducted at the speed of 3000 rpm. SEM imaging and X-ray diffraction method were used to study the dispersion of MWCNTs and nanoclay I.30E. The results of the study of mechanical strength showed that stirring 3000 rpm for 8 hours at 80°C then ultrasonic vibrations for 6 hours at 65°C gave the highest learning strength (95.5 MPa tensile strength, flexural strength 115.45 MPa, compressive strength 219.10 MPa, and impact resistance 22.30 kJ/m^2). Fire resistance measurement results also showed high results in the fabrication condition of 8 hours mechanical stirring at a speed of 3000 rpm at 80°C, 6 hours ultrasonic vibration (LOI index of 25%; UL 94HB: 18.60 mm/min).

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The author declares that there is no conflict of interest regarding the publication of this article.

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