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Hybrid Coatings for Wood Protection Against Fire Action

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ABSTRACT

This study looks into the impact of composite coating of epoxy/aluminum hydroxide Al $(OH)_3$ / sodium borate (Borax) on the wooden surface. This work aimed to ascertain the connection between these substances' ignition characteristics and wood surface to study a fire behavior of epoxy composite. The intumescent coating that protects wood surfaces from wildfire is often utilized in places of employment and residence where safety is necessary. The aluminum hydroxide and sodium borate were combined and added to the epoxy resin as a hybrid coating (EAB)wt.% 20%, and subsequently implemented by dipping method of wood specimens. GO added to the hybrid coating to improve the scratch resistance of the coating in addition to improving the mechanical properties. This work investigated and discussed the morphology, flammability and mechanical properties of the hybrid coating, hybrid coating's morphology, flammability and mechanical properties. The results demonstrated that the wood surface coated with hybrid coating has a good fire-retardant quality, where a clear improvement in fire retardant, a burning rate lowered by 23%, and the hardness increased by 72%, while there was decrease in contact angle with the addition of GO because GO is a hydrophilic.

1. INTRODUCTION

Materials known as polymers are used in many different aspects of daily life. based on their application in a number of technological, industrial, and research domains. Polymeric materials have a substantially bigger influence on society. Additionally, due to the high strength polymeric materials' great mechanical properties, low processing temperatures, high performance, affordability, and remarkable environmental stability, there is a growing need for low-weight composites composed of these materials [1]. Because of its useful qualities, including its complexion, low mass, great strength, and low level of insulation, wood has been used as the main raw material for structures. For safety reasons, wood application is limited in particular fields due to its high ignition [2]. Epoxy resin (EP) is a thermosetting polymer material that is widely used in the construction, automotive, coatings, electronics, and aerospace fields because of its numerous benefits, including superior adhesion, chemical resistance, conductive properties, and excellent mechanical strength [3]. Flammability is one of the main demands that restricts the use of polymer materials. The polymer's ignition is the first step of the material's combustion and a crucial one for the progression of combustion [4]. When polymers are heated enough, they break down gradually and release flammable gasses that combine with the oxygen in the surrounding air to create an ignitable source. Either impulsively or at the flash point, ignition happens when the temperature is high enough for autoignition. Heat is created during combustion, and some of that heat is passed to the substrate to encourage more breakdown. A self-sustaining combustion cycle will occur if there is sufficient heat to continue the rate of polymer breakdown while keeping the volatile concentration within the flammability limitations [5], as in Figure 1.

EP has a relatively low index of limiting oxygen (LOI) of 19.8%. Huge numbers of people could be killed and significant property damage could result from the emission of toxic smoke and heat during the combustion process [6]. By forcing the material to expand and froth into a thick, highly resistant to heat, as well as highly porous char layer, the endothermic break down reaction phase at high temperatures provides intumescent coating with flame protection. Structural elements can be coated with intumescent substances by spraying or painting a liquid compound over them. The compounds solidify as an intumescent covering in the air after a specific amount of time [7]. One way to combat flammability is with flame retardant coating, which is primarily classified into two types: (1) intumescent and (2) non-intumescent. Because intumescent paints create a foam-like structure on the material surface that inhibits the flow of heat and oxygen, they are preferred over non-intumescent coatings. Thus, it has been proven that using intumescent flame retardant (IFR) coating techniques in fact is better appropriate for minimizing wood flame damage [8]. High resistance to low combustibility and toxicity of combustion gases, low rate of ignition and flame propagation, low rate and amount of smoke generation, reduced flammability during use, acceptability in appearance and attributes for certain end-uses, and little to no economic penalty are the characteristics of the ideal flame-retardant polymer system. Thus, a flame-retardant treatment needs to be economical, effective, and suitable for use in commercial equipment [9]. Generally speaking, physical flame retardant addition and chemical interaction modification are the two main methods used of enhancing the flame retardancy of polymer materials [10]. Adding flame retardants to polymer materials can enhance their flame-retardant properties and increase their flame survival rate by three times [11]. In order to shield the wood substrate from igniting, the IFR coating process can create a layer of foam that is charred and protective [12]. Flame retardant molecules can cross-link and cyclize to generate a charred layer that restricts the flow between the substrate and the environment in terms of heat and oxygen, flame retardants (FRs) are chemical substances that can withstand direct flame. They work by increasing ignition resistance and reducing the rate at which flames spread [13, 14]. These substances are either integrated into the polymer structure as internal flame retardants or added as ineffective chemical additives known as external flame retardants that are mixed in with polymeric materials without experiencing any chemical reactions [15]. There are two known ways to put out a flame: the first involves creating non-combustible gases that keeping oxygen from getting to the flame zone, this results in free radicals to poison the flame and put it out. The subsequent technique, relies in the warmth energy flame hypothesis, which holds those chemical fire retardants require thermal energy to Break down, which lowers the temperatures of the material with a heat surface to below the temperature of ignition, which puts out the flame [16]. There are four main materials that all function differently to slow down flames. These substances include inorganic and halogen-containing flame retardants, phosphorus, and nitrogen [17]. Due of its many benefits, including little smoke and low toxicity upon combustion, it has been utilized widely [18]. Figure 2 demonstrates the strategy for the creation of intumescent char layers on the polymer surface that acts as an insulating layer to keep out gas and heat transfer to the substrate characteristics [19].

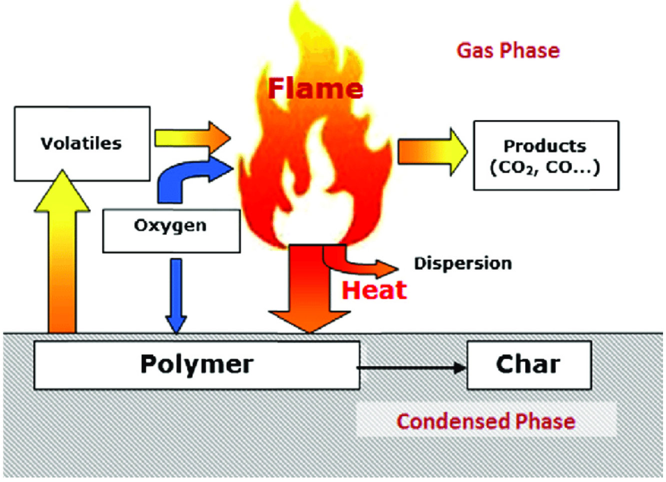


Figure 1. The combustion cycle: coupling of energy feedback from flame to combustible degradation products [5]

When polymers are heated enough, they break down gradually and release flammable gasses that combine with the oxygen in the surrounding air to create an ignitable source. Either impulsively or at the flash point, ignition happens when the temperature is high enough for autoignition. Heat is created during combustion, and some of that heat is passed to the substrate to encourage more breakdown. A self-sustaining combustion cycle will occur if there is sufficient heat to continue the rate of polymer breakdown while keeping the volatile concentration within the flammability limitations.

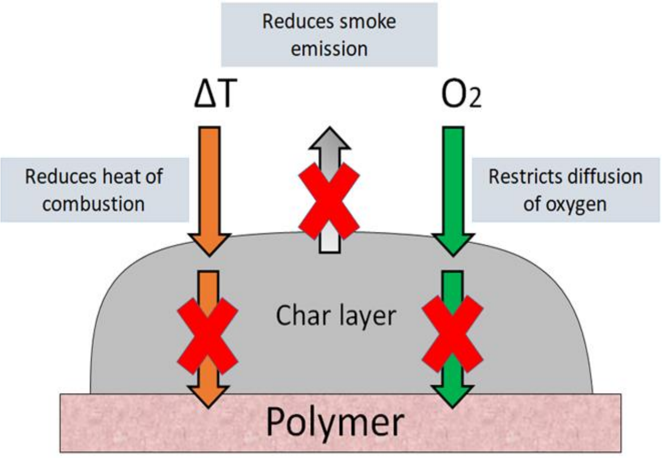


Figure 2. Scheme for intumescent char layer [19]

Heat, pyrolysis, ignition, and combustion are the four steps that typically make to a polymer's combustion process. The process starts with heating the polymer to a particular temperature with a heating device. This causes the polymer's backbone to break and decompose, which can result in the formation of the following products: (1) gasses that burn easily, such as CO, methane, and ethane; (2) molten liquid created by the thermal breakdown of polymers; (3) gasses that are not combustible like CO_2; (4) tiny solid particles made of soot or pieces of polymers; and (5) carbonaceous, as in Figure 3 [20].

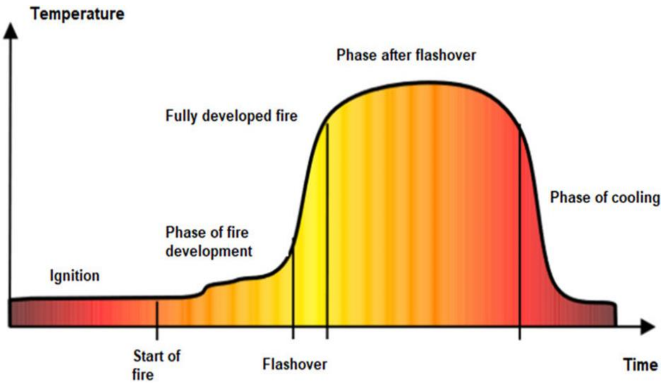


Figure 3. Stages of fire development: From ignition to decay [20]

Borax compounds have been added to different thermoset polymers as a flame-retardant ingredient. The majority of research focuses on epoxy resin; sodium borate (Na20•2B2 O3•10H2O) is used in this study because it provides various quantities of water into the environment, acting as an insulation and low-melting solid. Their viscous glassy melts can either cover the surface of the pyrolyzing polymer or char, mending fissures and acting as a barrier to heat and decomposition products, or they can generate intumescence by evolved breakdown gasses, primarily water [21].

As a flame-retardant filler, aluminum trihydride ($Al (OH)_3$) is the most often utilized inorganic hydroxide. $Al (OH)_3$ is treated at temperatures between 190 and 230 C, which is below its breakdown point, depending on the particle size. In thermoplastics, thermosetting resins, and elastomers that are treated at temperatures lower than 200℃ and with particle sizes more than 50 m, Al $(OH)_3$ is typically utilized as a flame-retardant filler [22, 23]. At approximately 180℃, the material decomposes by an endothermic dehydration reaction, producing water vapor. Water vapor absorbs heat and dilutes and cools the flame zone environment. In particular, the involved decomposition reactions are the following:

$$2Al(OH)_3 \rightarrow Al_2O_3+3H_2O \quad (1)$$

Nano graphene oxide (NGO) was utilized to increase the coating mixture's mechanical qualities and flame efficiency. Numerous studies have demonstrated that the analogs of graphene can have a major impact on the burning, of polymers because of their strong barrier function, big particular surfaces capacity for adsorption, and excellent thermal resistance, all of which are advantageous for reducing heat and mass transfer [24].

Wu et al. [25] created an intumescent flame-retardant glue and manufactured a type of flame-retardant ornamented plywood. When the ratio of IFR was less than 0.60, the shear strength of FDP was greater than 0.97 MPa, meeting the China National Standard's (GB/T 9846, 0.7 MPa) requirements for plywood. The thermal stability and intumescent behaviors of adhesives were taken into consideration when optimizing the composition of the flame-retardant glue. FTIR was employed to describe the chemical changes in the adhesives throughout the charring operation. To sum up, the new technique employed to create the FDP turned out to be workable. The use of this technology eliminates the drawbacks of impregnation, physical mixing, and flame-retardant coatings more effectively and more easily than traditional procedures.

Zinc borate (ZB) was employed as a synergistic a flame-re, aluminum hydroxide ($Al(OH)_3$) and magnesium hydroxide (Mg $(OH)_2$) as flame-resistant additives, and polypropylene (PP) as the matrix resin [26]. Twin-screw extruders were used to produce the flame-retardant composite materials PP/Al $(OH)_3$/Mg $(OH)_2$ and PP/Al $(OH)_3$/Mg $(OH)_2$/ZB. Measurements were made of the composites' oxygen indicator (OI), burn velocity, and smoke concentration in order to determine their flame-retardant characteristics. The findings demonstrated that OI rose as filler concentration in order expanded and dropped as filler particle diameter grew.

In the current work coating are designed from two fillers (Al $(OH)_3$, BX separately based on epoxy resin matrix and choose these fillers because it is economical in terms of cost and application on woods to study its effect individually on prevent or slow down the spread of flames during a fire incident. Epoxy-based coatings are commonly used due to their excellent adhesion properties, chemical resistance, and durability. Borax and Aluminum hydroxide are incorporated together into epoxy-based as hybrid coating (EAB), to knowing the improvement in extinguish ability and to enhance the fire resistance of the coating system. Borax provides intumescent properties by forming a protective char layer upon exposure to heat, while aluminum hydroxide contributes to cooling effects and further suppression of flames. This combination improves the overall fire performance of the coating by reducing flammability. GO has been added to the hybrid coating to improve scratch resistance.

2. METHOD AND MATERIALS

2.1 Materials

The Epoxy and resin hardener used in this study were obtained from (Renksan – Ren floor HT 2000), Turkish with a density of 1050 Kg/m^3, hardener to it with a ratio of (2:1) for curing, aluminum hydroxide $Al(OH)_3$, is supplied from Redial De Hen Germany, and sodium borate (Borax) (99.5% pure) were obtained from LOBA CHEMIE PVT.LTD. and nano graphene oxide NGO from the local market. All materials were used as received.

2.2 Measurements and characterization

The fire performance of these chemicals combined was evaluated in a Flame horizontal test which is generally applied to evaluate the fire performance of a material according to ASTM-D 638. The samples' hardness was measured using a German-made Shore D hardness device model (TH 210 FJ)., to measure the resistance of a material to scratches and abrasion use scratch hardness ISO 1518 standard as in Figure 4, UV-Visible– CECIL 2700 computerized spectrophotometer used to assess the sample's optical characteristics, the sliding test carried out on the pin-on-disc wear machine (ASTM G99) under dry circumstances, the wear samples were evaluated, and the hydrophobicity was assessed by measuring a water contact angle using "SL200B Optical Dynamic / Static Contact Angle Meter". The surface morphology and microstructure of the epoxy composite were characterized by scanning electron microscope (SEM) this experiment was carried out according to (ASTM 986-04).

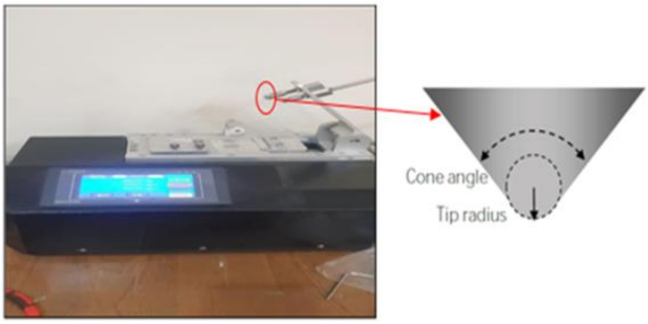


Figure 4. Illustration of the scratch hardness device

2.3 Preparation strategies of hybrid flame retardants and application on the wood surfaces

Flame retardant (FR) coating system were prepared using different concentrations of two retardant materials (Aluminum Hydroxide $Al(OH)_3$ and Sodume borate): wt. % 0,2,4,6,8,10, and 20 separately with Epoxy. The dispersion of borax and Aluminum Hydroxide in resin was carried out by using a mechanical stirrer for 15 minutes. Then the hardener (with a ratio of 2:1) was added and was mixed for 5 minutes. The prepared mixture was put in the vacuum for 20 minutes to eliminate bubbles, and the resultant solution was used for coating the wood substrate using the dipping method and left for 160 hours at room temperature. For hybrid coating(EAB) a variety weight percentages of hybrid filler (5Wt.% of $Al(OH)_3$ and 1 5 wt.% Borax), then the hardener (with a ratio of 2:1) was added and was mixed for 5 minutes. The prepared mixture was put in the vacuum for 20 minutes to eliminate bubbles, and then used for coating by dipping method. For hybrid- nano coating EABG 20Wt.% was chosen of the hybrid filler, where graphene oxide was added with a variety weight (0.1,0.2,0.4, 0.8) wt.%, respectively. Table 1 shows the sample details.

Table 1. Details of the sample

Sample No.	Sample Details
EP/BX	EP+(0,2%,4%,6%,8%,10%,20%) BX
EP/Al (OH)$_3$	EP+(0,2%,4%,6%,8%,10%,20%) Al(0H)3
EAB	EP/ [5%Al (OH)$_3$+15%BX]
EABG1	EP/20% [5%Al (OH)$_3$+15%BX]/0.1GO
EABG2	EP/20% [5%Al (OH)$_3$+15%BX]/0.2GO
EABG3	EP/20% [5%Al (OH)$_3$+15%BX]/0.4GO
EABG4	EP/20% [5%Al (OH)$_3$+15%BX]/0.8GO

The nanographene oxide was dispersed for 15 minutes at room temperature using ultrasonic (SJIA-1200WMTI) with epoxy resin separately after that, the fillers were added. The prepared coating was applied on the wood substrate using the dipping method. Wood samples were cut according to the flame test (ASTM D 635-03), with standard dimensions (125 mm,13 mm, 3 mm) length, width and thickness respectively. Grasping the sample at the extremity that is most distant from the reference mark of 25 mm, after applying a flame at (45°) for 30 seconds without moving, see Figure 5, note the amount of time that has passed (t) in seconds and calculate the length of burned material in millimeters. Figure 6 illustrates the engineering dimensions of the sample and flame device. Finally Calculating the burning rate (v)mm/sec according to Eq. (2).

$$V=60L/t \qquad (2)$$

where, L the burned length, in(mm) millimeters, and the time, in(s) seconds.

Figure 5. Sample during a flame test

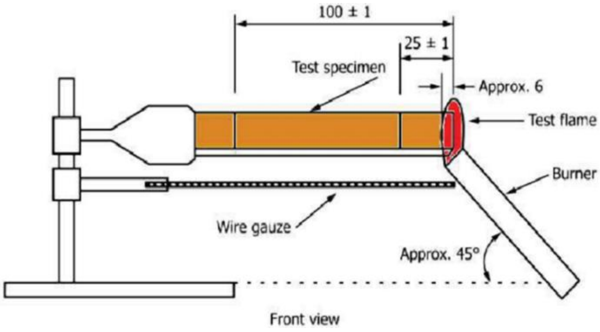


Figure 6. Flame test

3. RESULTS AND DISCUSSION

3.1 Flame test analyses

Burning rates were calculated using the flame test (ASTM D 635-03). In this test, the effect of adding the flame retardant agents [Al(OH)$_3$, BX] on the fire resistance behavior of the coating, and the results showed a significant improvement in the fire resistance of resultant composites coatings compared with pure coating. This test indicated that the epoxy coting's rate of burning has been continuously decreasing as the percentage weight of additives increases, see Figure 7, Pure epoxy coating can simply catch flame in the atmosphere, when addition (2,4,6,8,10,20 wt.%) of each filler to the epoxy separately can leads to decreasing the burning rate and this behavior due to gas phase suppression, which entails releasing non-flammable gases like H_2O, SO_2 and CO_2. Thermal decomposition of borax can produce H_2O, which dilutes and reduces flammable gases and disrupts chemical reactions during the burning of borax. Meanwhile, aluminum hydroxide acts by absorbing heat at high temperatures. The burning rate decreases from 1.25 mm/min for pure epoxy to 0.18 mm/min with the addition of borax, and from 1.25 mm/min to 0.15 mm/min for aluminum hydroxide.

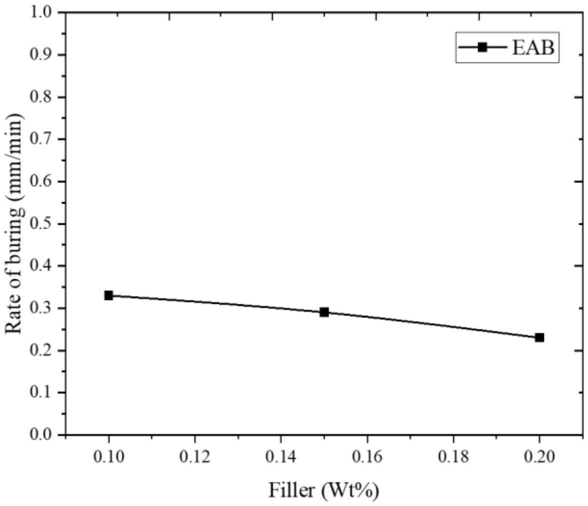


Figure 7. Illustration of the effect of graphene oxide addition on fire behavior

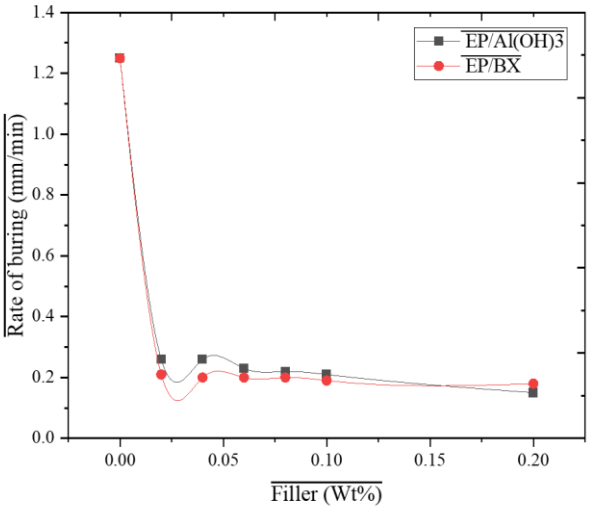


Figure 8. Illustration of the burning rate of flame retardants on epoxy

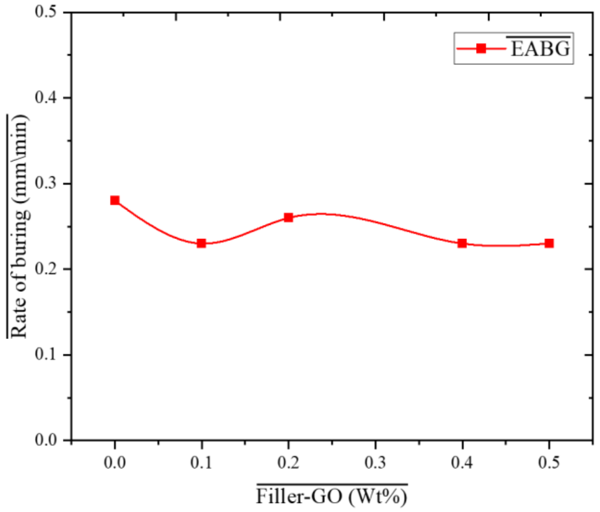


Figure 9. Illustration of the burning rate of hybrid coating ($Al(OH)_3$ and Borax)

Consequently, their combined action as a hybrid coating results in a significant improvement in flame retardancy, as shown in Figure 8 [27, 28]. Figure 8 illustrates the effect of adding graphene oxide (GO) to the hybrid coating on the burning rate. The results showed a slight effect of adding 0.1 and 0.2 wt.% GO to the hybrid coating, which contains 20 wt.% (5% $Al(OH)_3$ and 15% Borax). Figure 9 shows the burning rate of the hybrid coating (5% $Al(OH)_3$ and 15% Borax).

3.2 Ultraviolet-visible spectrophotometer analysis

The absorption can occur when light passes through a thin film sample. The difference in the incident light and the transmitted light is used to determine the actual absorbance. The Beer-Lambert law, is the principle behind absorbance spectroscopy:

$$A=\mathrm{Log}\,(I_O/I) \qquad (3)$$

where, A is the measured absorbance, I_O is the intensity of incident light at a given wavelength, I the transmitted intensity. The intensity of the incident light I_O is the sum of the intensities of transmitted, absorbed and reflected light.

$$I=I_T+I_A+I_R \qquad (4)$$

accordingly,

$$I_T/I_O+I_A/I_O+I_R/I_O=T+A+R=1 \qquad (5)$$

Using this equation, we have calculated the absorption as:

$$A=1-T-R \qquad (6)$$

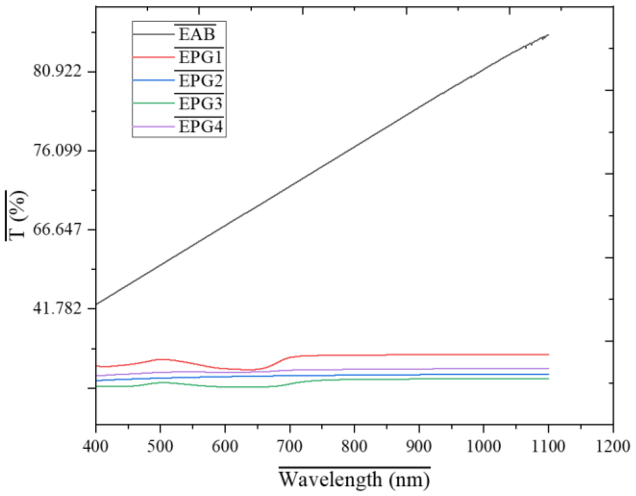
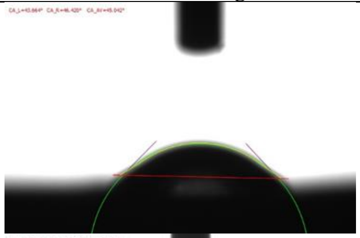
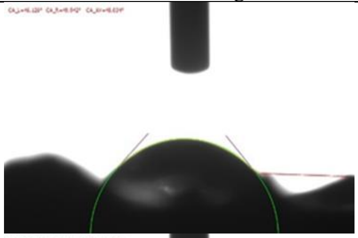
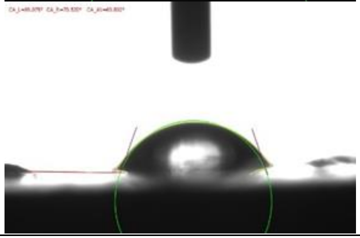
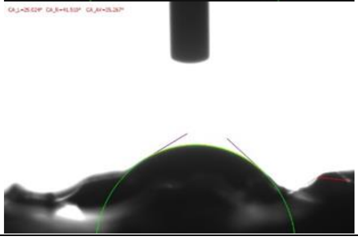


Figure 10. Ultraviolet-visible spectrophotometry of hybrid composite coating

The output data of wavelength, transmittance, and absorbance from a computer program are used to deduce the optical energy band gap, fundamental optical edge, and all optical constants [29]. The transmittance as a function of wavelength for (name of material) with different constructions is measured at room temperature. The transmittance spectra for (name of material), which vary with construction, show that the maximum transmittance occurs in the range of 350-850 nm. Small variations in transmittance have been observed with changes in construction within the same range. Transmittance decreases significantly as the construction increases; this behavior is attributed to the growth of grain size and an increase in defect states near the bands, as shown in Figure 10.

Table 2. Contact angel measurement

No.	Contact Angle	Contact Image	No.	Contact Angle	Contact Image
EAB	75°		EABG2	50°	
EABG1	69°		EABG3	35°	

3.3 Water contact angle

Table 2 shows the contact angles measured by using an optical contact angle instrument (CAM110, Tainan, Taiwan), CA measurements were used to determine the hydrophilic nature of the hybrid coating. The contact angle was then measured by placing five individual 5 µL drops of deionized water at different points on the wood surface. For every sample, at least five measurements were collected, and the resulting values were then averaged. Measuring the contact angle of the finished coating the wettability of the coating was assessed. This test was carried out with distilled water at (30°C), where the contact angle was measured after 5s. Images representative the contact angles on the hybrid coating EAB and compare it with hybrid nanocoating sample [EABG1, EABG2, EABG3]. The water droplets were rapidly absorbed on the hybrid coating surface data which was taken every ten seconds, while there is decreasing with the addition of GO and these results may be due to the GO has a hydrophilic behavior [30].

3.4 Hardness analysis

The hardness results were obtained from the average of three different locations on the coated surface. It is well-known that hardness depends on the resistance to penetration at the outer surface.

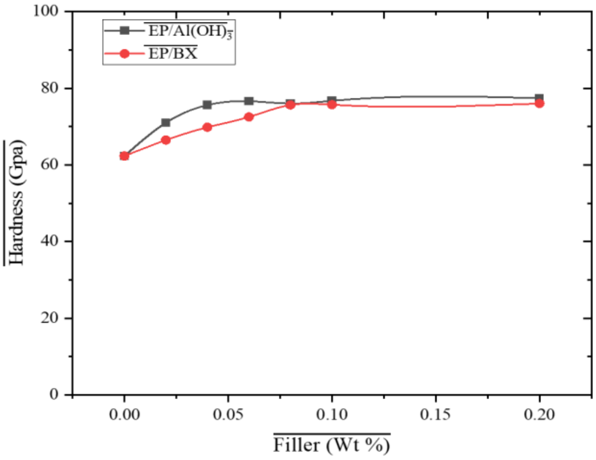


Figure 11. Effect of fillers on the hardness of epoxy coatings

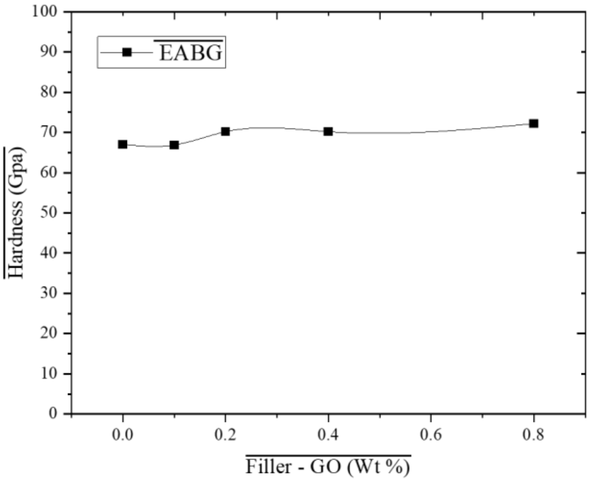


Figure 12. Impact of graphene oxide on the hardness of epoxy coatings

In this study, Shore Hardness (D) was used to measure hardness. The hardness of the epoxy increased with higher weight percentages of fillers ($Al(OH)_3$, Borax), as shown in Figure 11. This increase is attributed to the good compatibility and interaction between the components and the matrix, in addition to the homogeneity. Figure 12 illustrates the effects of graphene oxide (GO) on the hardness; referring to the hybrid coating (EAB), hardness increased as the percentages of GO increased [31].

3.5 Wear rate analysis

Figure 13 shows the effect of adding graphene oxide (GO) on the wear rate of hybrid coatings subjected to a 15 N load. Initially, the specific wear of the hybrid coating is high with increased $Al(OH)_3$ and borax fillers because the fillers can act as abrasive agents. Abrasive particles can lead to increased wear rates by causing more significant material removal from the surfaces in contact. This abrasiveness could be contributing to the higher wear rates observed in the test. Additionally, aluminum tends to form oxide layers on its surface when exposed to air. The aluminum particles in the epoxy composite can wear away, exposing fresh aluminum surfaces that rapidly oxidize and form transfer films on the disc. These oxide films can increase wear rates due to their abrasive nature and poor lubricating properties. However, when adding GO, the wear rates show a completely distinct shift, initially decreasing and then nearly stabilizing at 0.8% GO. It is important to note that the larger initial variations in wear rate are associated with topographic variance before testing. As the test progresses, wear tracks form on the contacting surfaces, which cause the wear rate to decrease [32].

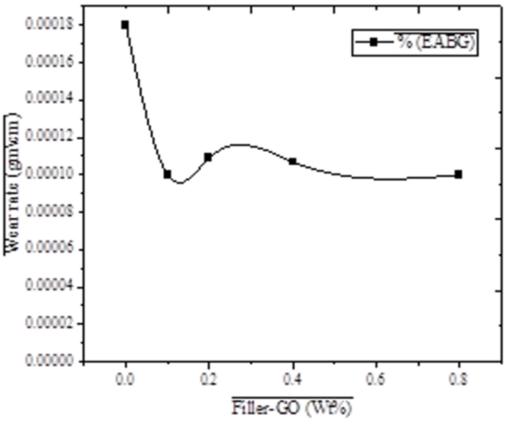


Figure 13. Effect of graphene oxide addition on the wear rate of hybrid coatings

3.6 Scratch hardness

Examination of the scratch hardness, as shown in Figure 14, revealed a significant increase in scratch hardness with an increase in the percentage of graphene oxide (GO) in the coating. This confirms the enhanced scratch resistance of the prepared coatings. The scratch hardness of the hybrid coating EAB increased from 121.753% to 236.26% for the hybrid nanocoating EABG under a 5 N probe load. The scratch hardness is calculated according to Eq. (7).

$$Hs = \frac{4qp}{\pi W2} \tag{7}$$

where, Hs is the scratch hardness, P is the normal load applied, W is the scratch width after the test and q is a factor that varies from 1, for elastic materials, 2 for plastic materials (q is taken to be a mean value of 1.5 in this study as most polymers are viscoelastic.

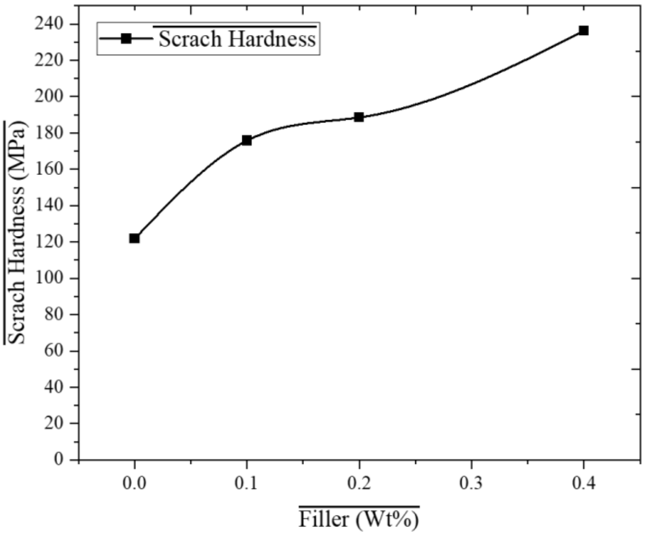
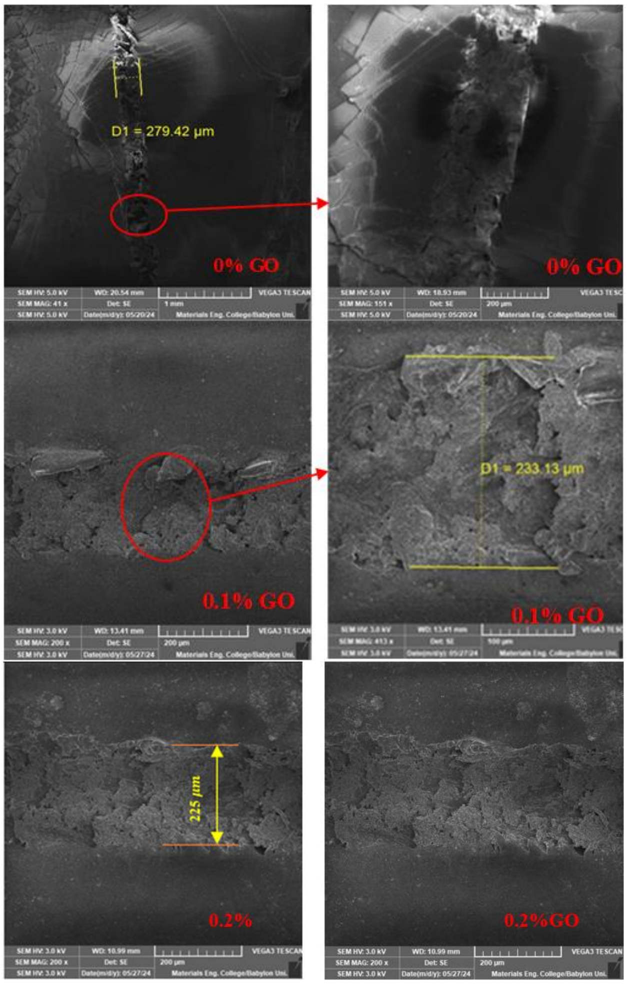


Figure 14. Illustration of scratch hardness on hybrid coating

3.7 Surface morphology

SEM images, as illustrated in Figure 15, show the morphology of the surface along the scratch path, where the crack width was measured.



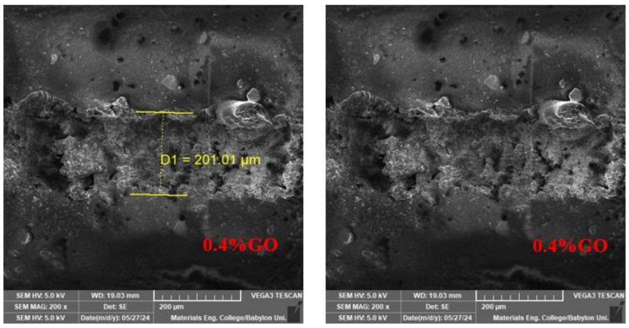


Figure 15. Morphology of the coating

The images also reveal the failure behavior against scratching and the ductile behavior of the epoxy before the addition of GO. Subsequently, the failure behavior gradually changed to brittle failure, demonstrating the effect of adding GO on the mechanical properties of the resulting coating.

4. CONCLUSION

Enhancing fire safety in a variety of applications, such as electronics, textiles, and building materials, requires the use of flame-retardant coatings. These coatings function by either preventing materials from igniting or by reducing the rate at which fire spreads. A range of chemical compounds, including those based on phosphorus, minerals, or halogenation, can be used to synthesize them; each has a unique mechanism of action and effect on the environment. In this work, epoxy was used as a matrix material, and aluminum hydroxide and borax were used as flame retardant fillers, these fillers are efficient in retardant the flame and keeping epoxy coating from burning, Where the burning rate decreased by about 83.33%. When GO was introduced, it had a direct effect on increasing the hardness of the resulting coating by 25%, and adding GO also can enhance the scratch resistance by 48.46%, The wear rate decreased by 44.45% due to present of GO. The current work opens horizons in preparing flame-resistant hybrid composite coatings.

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