Synthesis and Characterization of Non-Halogenic Fire Retardant Composite with Epoxy Resin and Additive Combination $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$

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Abstract. Epoxy has many advantages over other resin. However, in certain usage, additive is needed to raise the thermal resistance of the composites, while lowering its negative effect on health and environment. One of the most common additives used for thermal resistance is halogen. Halogen gives negative effect on health and environment because of the release of toxic gas following its combustion. An alternative for halogen substitution is using $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$. Therefore, in this research, the synthesis and characterization of non-halogenic fire retardant composite with epoxy resin and various concentration of additive $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$ was conducted. The characterization of this research is the fire retardancy, morphology dispersion, and mechanical properties of the synthesized composite, such as tensile strength and hardness. The result of this research is that epoxy resin 50% with 50% of additive $\text{Al}(\text{OH})_3$ gives the best flame retardancy behavior and mechanical properties. This composite gives flammability rating V-0 with T_{max} 364,3 °C, MLR 12,51 %/menit, total mass loss 57,26%, tensile strength 11,7 MPa, and hardness 79.

1 Introduction

Composite is a material that is widely applied in various fields. Epoxy resins, one of the components commonly used in the manufacture of composites has limitation in terms of fire resistance.

Epoxy is widely used in the manufacture of composites because of its advantages, such as its wide range of properties, relatively small shrinkage among other thermosets, resistance towards chemical, oil and water, and has excellent mechanical and electrical properties. Due to these advantages, epoxy resins are widely applied in various fields. However, due to its flaws that are easy to burn, it is necessary to improve the fire retardant capability of the material better, so it will be safer to use.

Composite materials that have such properties are called fire retardant composite. Currently, fire retardant composite is available and is used for applications of materials that are susceptible to temperature rise or even burning. Halogen additive can improve the composite's

fire retardancy but nowadays, the use of halogen additive has been minimized due to the release of halogen radical and halide acid which are harmful to the human health during burning^[1]. Alternative materials which are more environmental friendly and can be used as replacement for halogen additive are magnesium hydroxide ($\text{Mg}(\text{OH})_2$) and aluminium trihydroxide ($\text{Al}(\text{OH})_3$)^[2].

Both materials, aluminium trihydroxide and magnesium hydroxide, are known to be able to improve the fire retardancy of the composite material through decomposition mechanism that produce water vapour, absorb the heat, and form protective layer in the material surface.^[3] $\text{Al}(\text{OH})_3$ will begin to decompose at 200°C, whereas $\text{Mg}(\text{OH})_2$ will decompose at a higher temperature than $\text{Al}(\text{OH})_3$, at a temperature of 300°C^[4].

The effect of using $\text{Mg}(\text{OH})_2$ in the manufacture of fire retardant composite has been done by Tang et al. in 2013 using unsaturated polyester resin. Increasing the concentration of $\text{Mg}(\text{OH})_2$ to some extent provides a better fire retardancy effect on the composite material^[5]. In addition, the combination of $\text{Mg}(\text{OH})_2/\text{Al}(\text{OH})_3$ and

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microencapsulated red phosphor (MRP) has been researched by Liang et al. in 2014 using polypropylene resins. The results obtained show that the use of a combination of three is able to increase the capability of fire retardancy of the material [6]. Then, the combination of $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$ and the carbon black filler was investigated by Arfiana in 2016 using unsaturated polyester resins. The results obtained show that the addition of carbon black filler can improve the hardness and thermal stability [2].

Eventhough epoxy resin is one of the most common resin and applied in various filed, most research that has been done rather use unsaturated polyester resins, while there is currently little research on fire retardancy on composites that utilize epoxy resins. Therefore, through this research fire retardancy of epoxy resin can be studied and further improved. In addition, epoxy resins also have a wide range of properties and good mechanical and electrical properties. Moreover, epoxy resins have an advantage over unsaturated polyester resins, wherein epoxy resins have a lower shrinkage during curing and have faster curing time compared with unsaturated polyester resins.

Therefore, research on epoxy resin is required to improve its fire retardancy properties. In this study, this was conducted by using additives combination of $\text{Al}(\text{OH})_3$ and $\text{Mg}(\text{OH})_2$ on epoxy resins. The choice of these two additives is due to their properties that can produce water vapor when decomposed with fire and does not produce toxic compounds when caught on fire as occurs in halogen compounds.

2 Methodology

The composition of composites this study was varied as shown in Table 1.

Table 1. Varied Composition of Resin and Additive

Sample Code	% wt		
	Epoxy	$\text{Al}(\text{OH})_3$	$\text{Mg}(\text{OH})_2$
A0	50	0	0
A1	50	50	0
A2	50	35	15
A3	50	25	25
A4	50	15	35
A5	50	0	50

Composites with code A0 are neat resins, namely composites with epoxy content in the absence of additives. This neat resin is used as a standard in comparing the

synthesized composite characteristics with the addition of $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$ as additive. Meanwhile, the composite with additive $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$ comprises several variations of additive concentration to see and determine the epoxy resin composite with the best additive composition in terms of fire resistance, as well as to assess and determine which additive is more dominant in terms of improving fire retardancy in composite. Variations of the resin and additive compositions used can be seen in Table 1, where the column for epoxy consists of epoxy resin and hardener with 2:1 ratio.

Materials used in this study were epoxy resin and epoxy hardener (provided by PT Justus), aluminium hydroxide, and magnesium hydroxide. In the manufacture of these composites, stainless steel plate mould is used with a size that has been adjusted for testing flammability with UL94 standard and tensile strength with ASTM D638 type I standard. Composite with various additive concentrations which has been synthesized is then tested for fire resistance, thermal stability, mechanical strength, and its morphology is also observed.

The properties of fire retardancy were tested by flammability test and TGA-DSC, while the tensile strength and hardness were performed to see the mechanical properties of the composites, and SEM-EDX is used to observe the dispersion of additives within the composite.

Flammability test was performed based on the UL-94V standard procedure which resulted in four levels of flammability rating; V-0, V-1, V-2 and NR (non-rated) for the specimen that was completely burned. From this test, parameters obtained are time to ignition (t_i), time taken to retard the fire (t_r), and burning time (t_b).

For each concentration variation, testing required 5 samples. Each sample was burned for 10 seconds with a maximum repetition of 3 times if the sample had not burned or died after burning before the third repetition. From the test, the value of time to ignition (t_i), burning time (t_b) and time taken to retard the fire (t_r) are recorded. Burning material droplets are also observed to see if they are able to ignite the cotton located at the bottom of the test apparatus.

Flammability test UL-94V produces data that is used to see fire retardancy of the composite. Those data are time to ignition, time taken to retard the fire, burning time, and flammability rating. Time to ignition (t_i) is seen from the time required by the sample to be burned. Burning time value (t_b) is the sum of afterflame time ($t_1 + t_2$) and afterglow time (t_3). Afterflame time is the duration of the burnt sample for the first and second burning experiments, while the afterglow time is the length of time the sample burns on a third fuel trial. While time taken to retard the fire (t_r) is calculated when the sample begins to burn until the fire can be extinguished by itself. If the sample is burned completely then the comparable data for each resin/additive composite code is the value of t_i and t_b . Whereas if the fire can be extinguished after being burned then t_r data can be obtained.

Thermal stability test was performed by using Thermal Gravimetry Analysis (TGA). The result of this test was a graph that showed the temperature when the mass of material started to decrease (T_{onset}), the mass loss rate (%/min), and the maximum temperature (T_{max}) when the mass loss rate reached its highest point.

Mechanical properties observed in this study are tensile strength and hardness. The tensile strength was characterized based on ASTM-D638 type I. The hardness was based on ASTM D2240-15.

3 Results & Discussion

3.1 Flammability Analysis

Based on the parameters calculated in Table 2, which shown the tabulation data of resin/additive composite sample test results, code A1 has the greatest t_i value with the lowest t_r value and the best flammability rating (V-0) based on the UL-94V test scale. Thus, the addition of 50% $\text{Al}(\text{OH})_3$ additive to epoxy resin is capable of producing a fire retardant composite with best fire retardancy capability compared to composites with other concentration variation of $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$ additive.

Table 2. UL-94V Flammability Test Results

Sample Code	t_i (second)	t_r (second)	t_b (second)	Rating
A0	10	-	129.6	NR
A1	30	0	205	V-0
A2	22	25	153.6	V-0
A3	20	-	209.6	NR
A4	20	-	210.4	NR
A5	20	-	197.4	NR

From the flammability testing conducted, it is known that the sample neat resin with the code A0 burned with a value of t_i of 10 seconds. The neat resin data is the standard data that will be used as a benchmark for other composite code flammability tests. The time to ignition data for the resin/additive composite in Table 2 is made into graph form shown in Figure 1.

Figure 1. Comparison of Time to Ignition

Based on the time to ignition graph, figure 1, it can be seen that t_i of neat resin increases with the addition of additives, both $\text{Al}(\text{OH})_3$ and $\text{Mg}(\text{OH})_2$. In addition, this increase is most significant in code A1 with an additive content of $\text{Al}(\text{OH})_3$ 50%. In code A1 there is an increase in the value of t_i as much as 3-fold from t_i of neat resin. While in other run codes, t_i decreases with the decreasing of $\text{Al}(\text{OH})_3$ and increasing of $\text{Mg}(\text{OH})_2$. However, the A5 code with $\text{Mg}(\text{OH})_2$ 50% and doesn't contain any $\text{Al}(\text{OH})_3$ still has a higher t_i value than neat resin.

This suggests that the two additives, $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$, are capable of increasing time to ignition of epoxy composite neat resin. However, the additive $\text{Al}(\text{OH})_3$ is capable of significantly increasing the value of t_i rather than $\text{Mg}(\text{OH})_2$ additive. The $\text{Mg}(\text{OH})_2$ additive has a coagulation effect when added to the epoxy resin. The higher the concentration of $\text{Mg}(\text{OH})_2$, the thicker the texture of the resulting mixture. This condition causes stirring more difficult and causes the distribution of $\text{Mg}(\text{OH})_2$ in the mixture to be less evenly distributed, whereas the additive distribution may affect the flammability property of the material. In contrast to $\text{Mg}(\text{OH})_2$, the addition of $\text{Al}(\text{OH})_3$ results in a mixed texture that is sufficiently fluid, thus allowing the $\text{Al}(\text{OH})_3$ additive to spread more evenly in the mixture.

According to Hapuarachchi et al. (2009), the endothermic decomposition reaction due to the increase in temperature of the hydroxide group fire additive is capable of releasing moisture and absorbing heat in the material, which can slow down the process of material decomposition^[7]. In addition, the phenomenon also causes the material temperature to be under the ignition temperature for a certain time when the additive decomposition process occurs so that time to ignition material increases. Thus, the addition of $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$ additive is able to give a positive effect on composite fire resistance marked by the increase of time to ignition value.

Figure 2. Comparison of Time Taken to Retard the Fire

Time taken to retard the fire, t_r , is the time it takes material to be able to extinguish the flame. The

resin/additive composites in this study which demonstrate the ability of fire retardancy are composites with codes A1 and A2. In both of these codes, the results obtained are not uniform, where from the five test samples, two samples are able to extinguish the flame and some burn. This may be due to an uneven distribution of additives due to thickening, especially in code A2 when added with an $\text{Mg}(\text{OH})_2$ additive. The t_r value for the resin/additive composite can be seen in Figure 2.

The lower the t_r value indicates better fire retardancy capability because the flame can be extinguished faster. The data in Table 2 shows the t_r values for the composite codes A1 and A2 are 0 and 25 seconds respectively. The additive composition which gives the smallest t_r value is obtained in a combination of $\text{Al}(\text{OH})_3$ 50% additive (code A1), where for this composition, the composite sample is not burned by fire. As for neat resin (code A0), composite resin/additive with composition of $\text{Al}(\text{OH})_3$ 25%/ $\text{Mg}(\text{OH})_2$ 25% (code A3), $\text{Al}(\text{OH})_3$ 15%/ $\text{Mg}(\text{OH})_2$ 35% (code A4), and $\text{Mg}(\text{OH})_2$ 50% (code A5), the entire sample burns.

From the graph it can be seen that the use of $\text{Al}(\text{OH})_3$ 50% still gives better results when compared with the use of $\text{Mg}(\text{OH})_2$ with the same concentration. This occurs because of the thickening effect that occurs when the mixture is added with an $\text{Mg}(\text{OH})_2$ additive that reduces the homogeneity of the additive particle distribution in the mixture so that the fire retardancy is reduced.

Figure 3. Comparison of Burning Time

For burned composite samples, codes A0, A3, A4, and A5, the parameter which is compared is burning time or t_b values. The value of t_b indicates how long the sample burns after the source of the flame is removed. Thus, although the composite samples with the above codes have not demonstrated the capability of fire retardancy, the increase in t_b value can be an indication that there is an increase in the resistance of the material. The burning time graph for resin/additive composites can be seen in Figure 3.

From the burning time graph, it can be seen that for the four varied composite compositions burned, burning time increases with the increase of additive concentration of $\text{Al}(\text{OH})_3$ and the decrease of additive concentration $\text{Mg}(\text{OH})_2$. This shows that the influence of $\text{Al}(\text{OH})_3$ additive on the material's fire resistance is more dominant

than $\text{Mg}(\text{OH})_2$ additive. The effect is caused because the additive $\text{Mg}(\text{OH})_2$ tends to produce a thick mixture and difficult to stir. Therefore, when the $\text{Mg}(\text{OH})_2$ additive concentration is lower, the mixture becomes more homogeneous so that the fire retardancy of the material is better which is hinted by increased burning time for burned composite codes in the flammability test.

3.2 Thermal Stability Analysis

The thermal stability and decomposition pattern of the fire retardant composite material is known through thermal gravimetry analysis (TGA) with inert gas atmosphere conditions and heating rate of $10^\circ\text{C}/\text{min}$ to a temperature of 800°C . The inert gas atmosphere conditions were chosen to determine the effect of heat degradation on the decomposition of the material, because if using air atmosphere then the influence of heat degradation is difficult to observe due to the influence of thermal oxidation. The results of thermal stability testing are TGA, DTG, and DSC graph that can explain the phenomenon of decomposition that occurs in the material due to heat degradation.

Table 3. Thermal Stability (TGA-DTG-DSC) Test Results

Sample Code	T_{onset} ($^\circ\text{C}$)	T_{max} ($^\circ\text{C}$)	MLR (%/min)	Total Mass Loss (%)
A0	301.5	362.4	20.47	73.3
A1	260	364.3	12.51	57.26
A2	256.9	340.4	14.86	53.84
A3	231.4	362.5	21.89	59.44
A4	282.6	362	25.49	64.37
A5	365.2	352.8	14.68	61.39

From the TGA test temperature of onset, total mass loss, mass loss rate value, and maximum temperature is known. Further analysis between neat resin and other resin/additive composite compositions is easier to be done by comparing the value of decomposition temperature (T_{onset}), maximum temperature (T_{max}), mass loss rate (MLR), and total mass loss. The temperature of the onset is the temperature at which there is a phase change in the test material. From several peaks that appear on each DTG chart, the maximum temperature (T_{max}) taken is the temperature at which the mass loss rate (MLR) reaches the highest value. Total Mass Loss is the total mass lost at the end of the test. The details of the tabulation of the thermal stability test results for neat resins and resin/additive composites can be seen in Table 3.

From the result of TGA-DSC test for neat resin (A0), it can be seen that the decomposition process started to occur at temperature around 301.5°C (temperature onset) with maximum temperature at 362.4°C where mass loss rate (MLR) reached its highest value, $20.47\%/\text{min}$. In addition,

the total mass loss for neat resin reached 73.3%. This value indicates that most of the resin is lost due to temperature rise, where the sample mass reaches 26.06% at the end of the test at a temperature of about 800°C.

While the resin/additive composite composition with the best flammability rating (A1), generally exhibits better thermal stability properties than the neat composite resin (A0). This is indicated by the increase of the maximum temperature value (T_{max}), decrease of the MLR value, and also decrease of the total mass loss value. Thus, the best resin/additive composite with $Al(OH)_3$ 50% increased the thermal stability by increasing T_{max} to 364.3 °C, decreasing MLR to 12.51% /min and decreasing total mass loss to 57.26%.

For composite resin/additive codes A1 through A5 decomposition occurs gradually with different intermediates. The A1 to A5 resin/additive composite DTG graphs also show more than one peak due to the addition of $Al(OH)_3$ and or $Mg(OH)_2$ additives. The presence of more than one peak confirms the TGA graph that decomposition occurs in several stages. As for the DSC graph for the resin/additive composite there is a difference, where for codes A1 and A5 there is only one peak, whereas in the A2, A3, and A4 codes there are two peaks. The difference in the number of peaks is due to the presence of different additive content. In code A1 and A5 there is only one additive, $Al(OH)_3$ in A1 and $Mg(OH)_2$ in A5. While in code A2, A3, and A4 there is a combination of additive $Al(OH)_3/Mg(OH)_2$ which decomposes at different temperatures causing the existence of two different peaks.

The TGA test results for resin/additive composites in Table 3 shows that the onset temperature has a fairly varied trend. Compared to the T_{onset} of neat resin, the T_{onset} of resin/additive composite values for the A1, A2, A3, and A4 codes decreased. The largest decrease in T_{onset} occurred in A3 composite, which is 23%. While the resin/additive composite code A5 increased the value of T_{onset} by 21%. The same thing also occurs in the value of T_{max} where the trend is quite varied. When compared with neat resin (code A0), there is a decrease in T_{max} value in A2, A4, and A5 codes with the largest decrease in code A2 which is 6%. On the other hand, in composite A1 and A5, there is no significant increase of T_{max} value.

From the DTG graph and the TGA-DSC test results data in Table 3 above it can be seen that when compared with neat resin (A0), mass loss rate (MLR) for varied additive concentration A1, A2, and A5 has lower value. When compared with MLR of neat resin, MLR of the best flammability rating resin/additive composite (A1) experienced the greatest decrease, from 20.47% /min to 12.51% /min. This MLR value of A1 is the lowest value compared to other codes of resin/additive composite. In addition, when compared between the use of single additive $Al(OH)_3$ with $Mg(OH)_2$, MLR for composites with a single additive $Al(OH)_3$ has smaller value compared to composites with a single additive $Mg(OH)_2$. This

suggests that the additive $Al(OH)_3$ has a more dominant influence on MLR of composites. Addition of $Al(OH)_3$ and $Mg(OH)_2$ additives is also capable of decreasing the total mass loss of composite by about 8.93-19.46% by weight when there is an increase in temperature. The addition of $Al(OH)_3$ and $Mg(OH)_2$ additives tend to increase the thermal stability of the composite material especially when viewed from the decrease in total mass loss value.

3.3 Mechanical Strength Analysis

Tensile strength and hardness tests were performed on neat resin composite (A0) and resin/additive combination composite with $Al(OH)_3$ 50% which have the best flammability rating (A1). The graph of tensile strength test results can be seen in Figure 4.

Figure 4. Tensile Strength Comparison

From the graph above it can be seen that the addition of the additive lowers the resulting material tensile strength. Addition of $Al(OH)_3$ 50% additive caused a decrease in tensile strength from 31 MPa (neat resin) to 11.7 MPa, or a decreased by 62.4%.

According to Wang, et al. (2015), the polarity difference between the additive and the resin may cause the resulting composite compatibility to be low and the additive particles can not be dispersed evenly in the matrix, especially when the additive concentration is high [8]. Meanwhile, according to Devendra et al. (2013), the increase of additive concentration in composites can reduce the strength of interfacial bonds between additives and matrix [9]. These things tend to cause a decrease in the mechanical properties of the synthesized material. In addition, according to Reddy et al. (2015), the addition of additives up to 60% can lead to an increase in viscosity that triggers the formation of agglomerates which can then become a central point of stress and cracking [10].

As for the graph of hardness test results can be seen in Figure 5. From the graph of hardness, the effect of additive to the resulting composite hardness can be seen. The combined use of $Al(OH)_3$ additives by 50% can increase hardness by 21.22%, from 65.5 to 79. According to Flores et al. (2000), the increase in composite hardness with

additive additions may be caused by the additive particles that is evenly distributed resulting in a structure that can harden the material^[11].

Figure 5. Hardness Comparison

3.4 Morphology Analysis

The dispersion of additive particles as well as the interfacial interactions of particles with the matrix are two important factors affecting the resulting composite material^[12]. To see the dispersion of the additive particles, as well as the possible formation of aggregates and/or agglomerates in the composite, the tests performed using SEM-EDX tools include morphological tests and percentage analysis of elements.

The morphological and elemental percentage tests were performed for neat resin (A0) as well as resin/additive composite with the best flammability rating (A1). In addition, the test was also performed for composites with an additive of $\text{Mg}(\text{OH})_2$ 50% to see the homogeneity of the composite. The test is performed on the composite surface. The dispersion pattern of additive particles of $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$ in epoxy and morphological matrices for neat resin is shown in Figure 6.

Figure 6. Composite Surface (a) Neat Resin, (b) A1 ($\text{Al}(\text{OH})_3$ 50%), (c) A5 ($\text{Mg}(\text{OH})_2$ 50%)

Based on the image, it can be seen that on the surface of neat resin composite almost no air bubble exists. Bubble should be avoided in the manufacture of composites as they may negatively affect material characteristics. From general morphological testing it can be seen that adding additives tends to trigger agglomerates where additives accumulate in several places. In the resin/additive composite with concentration of additive $\text{Al}(\text{OH})_3$ 50% (A1) formed agglomerates of varying sizes but relatively smaller compared to agglomerates in resin/additive composite with concentration of additive $\text{Mg}(\text{OH})_2$ 50% (A5). This suggests that the additive $\text{Al}(\text{OH})_3$ is more easily dispersed in epoxy resins than with the $\text{Mg}(\text{OH})_2$ additive.

The morphological results show that the addition of $\text{Mg}(\text{OH})_2$ to the epoxy resin composite mixture tends to trigger the formation of larger size agglomerates which can lead to the formation of a material structure less resolved by the resin thus affecting the bond between the matrix (resin) and the additive particles. The more agglomerate formed with the addition of $\text{Mg}(\text{OH})_2$ will affect the composite fire resistance characteristics produced, where if the additive is not dispersed properly then the fire resistance properties obtained will also decrease. This is also supported by flammability test results that tend to decrease with the increasing concentration of $\text{Mg}(\text{OH})_2$. Therefore, it can be said that the morphological results show that $\text{Mg}(\text{OH})_2$ additive is difficult to dispersed well on the epoxy resin, so it tends to form agglomerates which can decrease the fire and mechanical resistance properties of the material.

4 Conclusions

This research showed that the best fire retardancy for epoxy resin with additive $\text{Al}(\text{OH})_3/\text{Mg}(\text{OH})_2$ was obtained from the composite with concentration of $\text{Al}(\text{OH})_3$ 50%. The addition of $\text{Al}(\text{OH})_3$ 50% to epoxy resin composite could also improve the thermal stability of composite by reducing MLR (mass loss rate) to 12.51% /min and total mass loss to 57.26%. On the other hand, the tensile strength of composite decreased to 11.7 MPa but the hardness improves to 79 based in hardness shore D scale. Overall, the addition of both $\text{Al}(\text{OH})_3$ and $\text{Mg}(\text{OH})_2$ to epoxy resin can improve the fire retardancy of composite even though $\text{Al}(\text{OH})_3$ have more dominant effect in increasing the fire retardancy of composite and both can be used as an alternative to synthesise fire retardant composite.

5 Acknowledgements

Great gratitude to PT Justus for providing materials needed in this research and PITTA Research Fund of Universitas Indonesia for the research financial support.

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