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Thermal and Flame Retardant Performance of Glass Fiber-Reinforced UPRs Composites Modified with Boric Acid and Sodium Silicate

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***Abstract** -- This study investigates the effect of sodium silicate (Na_2SiO_3) and boric acid (BA) as flame retardant additives in unsaturated polyester resin (UPRs) reinforced with E-glass fibres. The fire performance of the composites was evaluated based on varying additive concentrations by % volume fraction, using standardised testing methods in burning rate (BR) with ASTM D 635-22. Results indicate that Na_2SiO_3 contributes to fire resistance by forming a thermally stable, glass-like barrier, while BA proves more effective by interrupting combustion reactions and generating protective boron oxide residue. Notably, the combination of Na_2SiO_3 and BA produced a synergistic effect, resulting in a significant reduction in burning rate. Thermogravimetric analysis (TGA) further confirmed the enhanced thermal stability of the composite, particularly in the NB10 formulation with 2% Na_2SiO_3 and 8% BA, which demonstrated the slowest degradation at elevated temperatures. These findings suggest that optimising the ratio of Na_2SiO_3 and BA yields high performance, fire retardant composites suitable for applications demanding superior fire resistance and thermal durability, such as electric vehicle safety components.*

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1. INTRODUCTION

Composite materials made with a polymer matrix, commonly known as polymer matrix composites (PMCs), have experienced significant growth and are now considered a breakthrough in various industries. These materials combine a polymer base such as epoxy, vinyl ester, or polyester, with reinforcing agents like glass, carbon, or aramid fibres. PMCs are valued for their lightweight nature, high strength, affordability, ease of manufacturing, and adaptability across various applications. These properties make them a viable alternative to conventional materials such as steel and aluminium.

One of the most widely used polymer matrices is polyester resin, particularly in combination with glass fibres. This composite system is popular due to its favourable balance of mechanical strength, corrosion resistance, low cost, and lightweight nature. However, despite these advantages, polyester/glass fibre composites exhibit relatively low fire resistance, which poses a challenge in applications requiring high thermal safety, such as in the automotive sector [1], [2].

Fire resistance is becoming increasingly crucial in material selection, especially in the development of electric vehicles (EV), where safety regulations demand materials that can withstand thermal hazards. Composite materials used in battery casings must be lightweight and durable and exhibit strong fire retardant properties. One strategy to improve fire resistance is the incorporation of flame retardant additives into the polymer matrix. Compounds such as sodium silicate (Na_2SiO_3) and BA have gained attention as effective, non toxic, halogen free flame retardants [3].

Sodium silicate, derived from quartz sand, enhances fire resistance by forming a heat-insulating protective

layer on the composite surface during combustion. BA, obtained from boron, is an environmentally friendly additive that reduces the spread of flames by forming a stable char layer. Its relatively low melting (170 °C) and boiling point (300 °C) allow it to act effectively at moderate fire temperatures [4], [5].

In addition to flame retardancy, thermal stability plays a critical role in electric vehicle battery protection. Highrate charging and discharging can trigger thermal runaway in lithium-ion batteries, making external thermal shielding essential. Battery casings must provide mechanical and thermal protection, especially under fire exposure [6].

Based on this issue, this study focuses on the addition of Na_2SiO_3 and BA to improve the fire resistance of polyester and glass fibre composites. The key variables examined include time to ignition, burning rate, and thermal stability, and they were evaluated using Thermogravimetric Analysis (TGA). The main objective is to determine whether these additives significantly enhance the fire performance of the composites, thus contributing to safer materials for automotive applications.

Previous research has demonstrated the effectiveness of Na_2SiO_3 and BA as flame retardants in different polymer systems. For instance, Tsuyumoto, a Japanese researcher, reported an increase in fire resistance in EVA composites using Na_2SiO_3. The result was then supported by Wibawa, who studied GFRP composites and found that BA and Na_2SiO_3 increased fire resistance and mechanical strength. However, this study did not focus on polyester-based composites or comprehensively assess the synergistic effects of the two additives using TGA-based thermal metrics [4], [7].

Therefore, this study aims to fill that gap by investigating the combined use of Na_2SiO_3 and BA in polyester/glass fibre composites and evaluating their fire performance through ignition time, burning rate, and thermal stability. The novelty of this research lies in its specific application to polyester composites for battery casings in electric vehicles, a field where fire safety is paramount.

2. METHODOLOGY

Material Preparation

In this study, the matrix material used was Unsaturated Polyester Resin (UPRs) from the 268 BQTN series, which has a density of 1.113 g/cm³. This particular resin was selected due to its widespread use in industrial composite applications, especially in automotive and construction sectors, owing to its good mechanical properties, ease of processing, and cost-effectiveness. Compared to other types of thermosetting resins, such as epoxy or vinyl ester, 268 BQTN polyester resin offers a more economical alternative while still providing adequate strength and stiffness. Additionally, its relatively low viscosity facilitates better mixing and impregnation of additives and reinforcements, making it suitable for flame retardant modifications in composite materials.

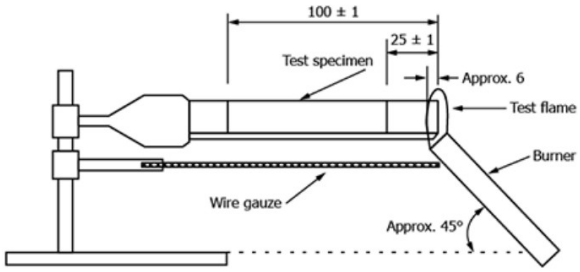
The curing process was supported by Methyl Ethyl Ketone Peroxide (MEKP) as the catalyst, supplied by PT. Kawaguchi Kimia Indonesia. As reinforcement, random E-glass fiber from Fantatex was used, with a density of 0.02 g/cm². The flame retardant additives applied were BA in powder form (≤74 µm) from Heansa Kimia and Na_2SiO_3 gel from SHI SAM MAS. The specimen fabrication process began with mixing the polyester resin, Na_2SiO_3, and BA based on the volume fraction compositions detailed in Table 1. The additive proportions were carefully varied to investigate their individual and combined effects on the fire resistant characteristics of the resulting composites.

Table 1. Sample code

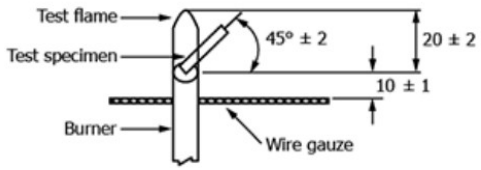
Sample Code	UPRs (% Vol)	Glass Filler (% Vol)	Sodium Silicate (% Vol)	Boric Acid (% Vol)
UPRs	90	10	0	0
A1	87.5	10	2.5	0
A2	85	10	5	0
A3	82.5	10	7.5	0
A4	80	10	10	0
B1	87.5	10	0	2.5
B2	85	10	0	5
B3	82.5	10	0	7.5
B4	80	10	0	10
NB01	85	10	4	1
NB02	85	10	3	2
NB03	85	10	2	3
NB04	85	10	1	4
NB05	85	10	5	0
NB06	85	10	0	5
NB07	80	10	8	2
NB08	80	10	6	4
NB09	80	10	4	6
NB10	80	10	2	8

The resin and additive mixture were mixed using a mixer at 3000 rpm for 5 minutes, followed by a 5 minute rest period to remove air bubbles. After that, the MEKP catalyst was added at 1% of the resin weight and stirred for 2 minutes.
The glass fiber cut according to the mold was inserted into a 25×25 cm glass mold with a thickness of 3.2 mm. The resin and additive mixture was poured over the fiber in the order of resin-fiber-resin. The mold was first coated with astralon plastic and wax, then the composite material was added. After 24 hours, the composite specimen was removed and cut according to the standard used. Finally, a post-curing process was carried out for 1 hour at a temperature of 100ºC.

Burning Test Method
The testing standard used was ASTM 635-22. The composite specimen was placed horizontally and one end was burned for 30 seconds, then the burning time and rate were measured at 100 mm from the exposed end. The final result was taken from the average of the burning data [8].



Front view



Side View
Figure 1. Burn Test Preparation

Thermogravimetric analysis (TGA)
TGA testing can reveal the thermal stability and decomposition behavior of composite specimens. The test is conducted in a nitrogen atmosphere to prevent oxidation. Each specimen is placed in a platinum crucible and heated up to a maximum temperature of 600°C at a heating rate of 10°C/min.
The mass loss of the composite specimens is analyzed as a response to heat exposure, which indicates the fire resistance of each sample. This study uses four composite specimen variations: pure polyester resin (UPRs), UPRs + 10% Na_2SiO_3 (A4), UPRs + 10% BA (B4), and one combined formulation expected to show the best performance in fire resistance tests. The TGA curves will be analyzed to identify key factors affecting fire resistance, such as degradation, mass reduction, and the residual mass of the composite specimens.

3. RESULT AND DISCUSSION

Burning Test
The fire resistance test was conducted to analyze the ability of the composite specimen to reduce the burning rate. This test provides insight into how effective the additives are in enhancing fire resistance. All composite samples, made using the hand lay-up process with various compositions, were tested, as illustrated in Figure 2.


Figure 2 Burn Test Process

The study results on UPRs with a single additive revealed that incorporating Na_2SiO_3 and BA significantly lowered the material's burning rate. For the control sample of pure UPRs without any additives, the highest burning rate recorded was 0.353 mm/s, which indicates that pure polyester resin is highly flammable. Adding Na_2SiO_3 to samples A1 to A4 reduced the burning rate gradually, with the lowest value of 0.192 mm/s at a concentration of 10%. This occurs because Na_2SiO_3 plays a role in forming a protective layer that slows down heat transfer during the burning process[9].

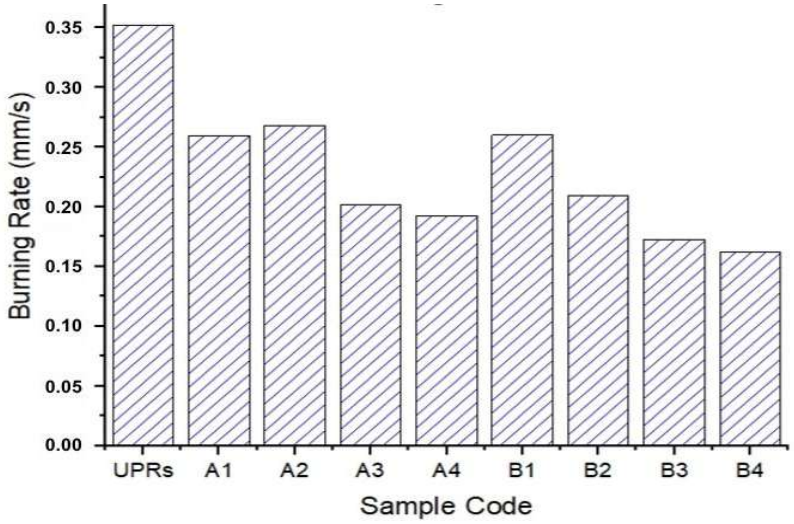


Figure 3 Burning Test Results of Samples with single additive

Furthermore, adding BA to samples B1 to B4 showed higher efficiency in suppressing the burning rate, with the lowest value of 0.162 mm/s at a concentration of 10%. The increase in BA concentration is directly proportional to the decrease in the burning rate. BA acts as a fire retardant that stops the combustion reaction, producing a protective residue inhibiting fire spread. Compared to Na_2SiO_3, BA is more effective in increasing fire resistance at the same concentration. Overall, using each additive Na_2SiO_3 and BA provides fire retardant properties to polyester resins.

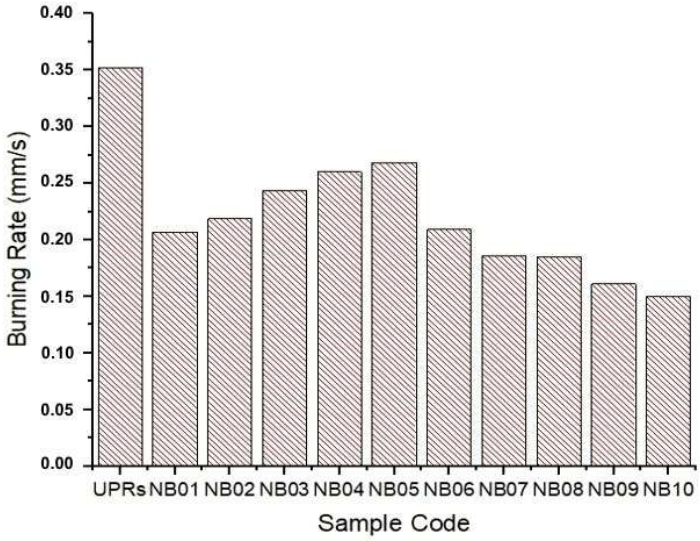


Figure 4 Samples with combination additive

On the other hand, based on the data and graphs of the combination of the concentrations of the two additives, the combination of Na_2SiO_3 and BA in UPRs significantly affects the burning rate of the material. The addition of additives in various combinations of Na_2SiO_3 and BA in samples NB01 to NB10 as a whole can reduce the burning rate, with the lowest value of 0.150 mm/s in sample NB10 (2% Na_2SiO_3 and 8% BA).

Samples with a higher proportion of BA than Na_2SiO_3 tend to show a more significant decrease in the burning rate. For example, NB09 (4% Na_2SiO_3 and 6% BA) has a burning rate of 0.161 mm/s, lower than A04 (10% Na_2SiO_3 and 0% BA), which reaches 0.192 mm/s or NB07 (8% Na_2SiO_3 and 2% BA) which reaches 0.186 mm/s. The phenomena show that BA is more effective as a fire retardant than Na_2SiO_3.

Overall, using Na_2SiO_3 and BA additives in certain combinations can improve the fire resistance of

polyester resin. The optimal combination is found in the ratio with BA dominance so that the resulting material is safer and suitable for applications requiring high combustion resistance. With the role of Na_2SiO_3 forming a protective layer that slows down heat transfer during the combustion process, and at the same time, BA releases water vapor that acts as a heat absorber and dilutes oxygen/flammable gas in an endothermic reaction that produces metaboric acid and boron oxide glass that strengthens the charcoal layer making the combustion rate process increasingly inhibited and successively also reduces the level of smoke production in the combustion process [10].

Thermal Retardant (TGA)

Based on the results of thermogravimetric analysis (TGA), it can be seen that the thermal performance of each sample is greatly influenced by the type and proportion of additives used [2]. The UPRs sample without additives showed the most significant weight loss after the temperature reached 300°C, indicating that pure polyester resin has low thermal stability due to its chemical structure, which is easily degraded at high temperatures[11]. The addition of Na_2SiO_3 (A4) moderately increased thermal resistance by forming a protective silica network, although weight loss occurred quite rapidly. On the other hand, the addition of BA (B4) led to a greater improvement in thermal resistance. This is due to its ability to form a protective boron oxide layer, which helps to block heat transfer and slow down the rate of decomposition.

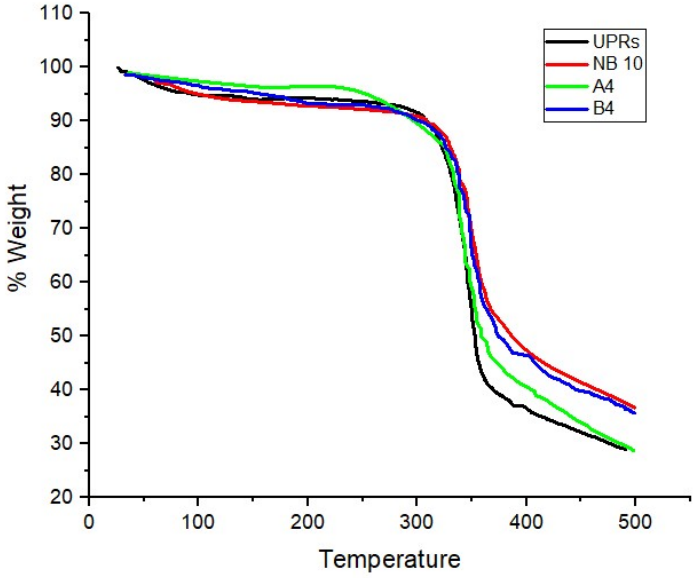


Figure 5 Thermal characterization of composite

The sample NB10, which combines 2% Na_2SiO_3 and 8% BA, delivered the best results in the TGA test. Its thermogravimetric curve exhibited the slowest rate of mass loss compared to all other samples, indicating superior thermal stability. The synergistic effect of these two additives is thought to enhance the material's internal structure while offering chemical protection against high temperatures. Na_2SiO_3 creates a protective silica network that slows the spread of heat, while BA forms a boron oxide layer that absorbs heat and prevents further combustion.

Overall, the TGA results corroborate previous findings from combustion tests that combining Na_2SiO_3 and BA is the most effective approach to improving the thermal performance and fire resistance of UPRs composites. The NB10 formulation provided the most optimal protection against thermal degradation. It is a superior material candidate for applications in high temperature environments, such as protective components in electric vehicles or transportation systems requiring high thermal safety standards[12].

4. CONCLUSION

In conclusion, this study shows that the addition of Na_2SiO_3 and BA as additives to UPRs reinforced with E-glass fibre significantly enhances fire resistance. The composite without additives exhibited the highest burning rate at 0.353 mm/s. In comparison, the addition of 10% Na_2SiO_3 reduced the burning

rate to 0.192 mm/s an improvement of approximately 45% due to the formation of a protective silica-based layer during combustion. BA demonstrated even greater effectiveness, with a 10% concentration lowering the burning rate by 54%, resulting in a value of 0.162 mm/s. The most notable result was observed in the NB10 sample (2% Na_2SiO_3 and 8% BA), which achieved the lowest burning rate of 0.150 mm/s a 58% reduction compared to the unmodified composite. This indicates a synergistic effect between Na_2SiO_3 and BA in enhancing fire resistance. The success of this combination highlights its potential as a safer and more reliable composite material for applications requiring high fire resistance, particularly in electric vehicle battery protection components. Beyond EV battery casings, the improved fire-retardant performance of these modified composites also makes them suitable for use in building materials, transportation interiors, electrical enclosures, and other structural components exposed to fire risk.

Future research should explore the long term thermal and mechanical stability of the modified composites under real operational conditions, including high humidity or prolonged heat exposure. Investigating the effects of other environmentally friendly flame retardants and optimizing additive dispersion techniques could further improve performance. Additionally, scaling up the manufacturing process and conducting cost-benefit analyses will help assess the feasibility of industrial applications.

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