

Weathering-Exposure Impact on the Thermal Stability and Fire Resistance of GFRP Composites with Pumice-Based Nanosilica Filler

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ABSTRACT

This study examined the impact of 0, 30, 60, and 90 days of weathering exposure on the thermal and fire performance of Glass Fiber Reinforced Polymer (GFRP) composites filled with Nano-Silica (NS). Characterization was carried out through visual observation, macro photography, Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), Ignition Time (IT), and Burning Rate (BR), following the ASTM standards. The results demonstrated a progressive degradation due to weathering, evidenced by the reduced initial decomposition temperatures, lower exothermic peak intensity, surface cracking, and delamination. Specifically, a noticeable color shift from yellowish to dark brown indicated surface oxidation. TGA revealed a decline in thermal stability, with the final residue dropping to about 45% after 90 days, while the DSC results showed a decrease in transition temperatures and exothermic response. The IT gradually decreased from 66 s (unexposed) to a minimum of 39 s after 90 days of weathering, while the BR increased from 0.237 mm/s to 0.33 mm/s over the same period. Nevertheless, NS continued to provide partial protection, despite the reduction in its effectiveness, remaining an effective material in improving thermal stability and fire resistance.

Keywords-weathering; GFRP composites; nano-silica ;thermal stability; fire resistance

I. INTRODUCTION

GFRP composites are widely utilized in the industrial sector due to their high mechanical strength and low density [1]. Their application in railway infrastructure has increased from 60% to 85%, while in aircraft structures has reached approximately 50%, improving fuel efficiency and service life. The inclusion of glass fibers reinforces polymer matrices, particularly the Unsaturated Polyester Resin (UPR), which are highly flammable due to their molecular structure, composed of aromatic rings and long aliphatic chains [2].

To mitigate this flammability, researchers have incorporated flame-retardant fillers into the polymer composites. These additives suppress combustion by releasing cooling vapors, forming char layers, and limiting oxygen diffusion [3]. Among these, NS is preferred due to its high melting point (> 1700 °C), thermal barrier properties, and its ability to enhance interfacial bonding through nanoscale dispersion [4-7].

The effectiveness of flame-retardant fillers in improving fire and thermal performance has been demonstrated. For instance, authors in [8] incorporated NS into a UPR matrix, resulting in a higher TGA residue, and a slower weight loss rate. The DSC findings also revealed a decomposition peak shift to 508 °C. In [9], the combination of NS with Ammonium Polyphosphate (APP) improved the Limiting Oxygen Index (LOI) and reduced the heat release rates. Similarly, authors in [10] found that using nano-Active Fillers of Pumice Particles (nAFPP) with Aluminum Trihydrate (ATH) and boric acid, allowed for a reduced ATH content without compromising the thermal performance.

Despite the advantages of the NS fillers, maintaining long-term fire resistance under weather exposure remains a significant challenge [11]. Weathering, driven by UV radiation, moisture, and thermal cycling initiates photo-oxidative and hydrolytic degradation, leading to chain scission, carbonyl formation, and weakened fiber-matrix bonding [12, 13]. These mechanisms result in surface cracking, delamination, and porosity, which compromise structural integrity and fire

resistance [14]. Although early-stage oxidation can temporarily form a protective surface layer, this barrier worsens over time, reducing thermal stability and fire performance [15, 16]. The impact of environmental exposure on the thermal and fire properties of composites has been examined. Specifically, authors in [17] indicated a reduced weight loss in hybrid UPR/GF/kenaf composites with an increasing exposure time over a 3-month period. The influence of filler addition on weathering resistance was examined in [15, 18] and the results revealed that ZnO and Fe₂O₃ nanoparticles reduced UV-induced damage while enhancing LOI values. Additionally, authors in [19] demonstrated that Carbon Nanoparticles (CNPs) dispersed in UPR formed a rigid network under UV exposure, which inhibited heat diffusion and delayed decomposition.

Although the effectiveness of NS as a filler in thermal properties has been extensively studied, limited research has addressed its performance under direct outdoor weathering. This study investigates the impact of natural outdoor weathering for 30, 60, and 90 days on the thermal stability and fire performance of GFRP composites containing NS.

II. MATERIALS AND METHODS

A. Materials Preparation

The UPR used in this study was 268 BQTN, supplied by Singapore Highpolymer Chemical Products (SHCP). Methyl Ethyl Ketone Peroxide (MEKP), marketed as MEPOXE and provided by PT. Kawaguchi Kimia Indonesia, served as the curing agent. Glass Fiber (GF) reinforcements included Woven Roving (WR) and Chopped Strand Mat (CSM) of type EMC200, obtained from PT. Makmur Fantawijaya Chemical Industries. The NS filler was synthesized from pumice rock collected from Mount Rinjani, located on Lombok Island, Indonesia.

B. Composite Preparation

The GFRP composites were fabricated using the hand lay-up method in a wax-coated glass mold (300 × 200 × 3 mm). These composites were composed of 70 wt.% UPR, 20 wt.% GF, and 10 wt.% NS. The UPR and NS were mixed together at 3000 rpm for 5 min and then allowed to degas for more 5 min.

MEKP (1 wt.%) was then added. Following the CSM-WR-CSM lay-up, the laminate was cured at room temperature for 24 h and post-cured at 100 °C for 60 min to ensure cross-linking and moisture removal. Finally, the specimens were cut according to the ASTM standards for further testing.

C. Weathering Treatment

To evaluate the environmental degradation, the GFRP composites were subjected to natural atmospheric exposure for 0, 30, 60, and 90 days. In order to simulate natural weathering, the samples were mounted facing the equator at a 45° angle. The sampling period was approximately three months at the Research and Development Center of Sebelas Maret University, Karanganyar, with data obtained on a weekly basis. The treatment codes for each composite condition are listed in Table I.

TABLE I. COMPOSITE TREATMENT CODES

No	Name	Duration of weather exposure
1	C	0 days
2	CNS30	30 days
3	CNS60	60 days
4	CNS90	90 days

D. Characterization and Testing

Standard rod-shaped specimens (125 ± 5 mm \times 13.0 ± 0.5 mm \times 3.0 mm, -0.0 to $+0.2$ mm tolerance) were utilized in order to assess their IT and BR through a burning test (ASTM D635-03). The thermal degradation was analyzed using both TGA and DSC with a PerkinElmer STA 6000. The key parameters, such as heating rate and temperature range, were recorded. Additionally, the surface morphology after combustion at each weathering stage was examined macroscopically using an Olympus SZ1145TR lens.

III. RESULTS AND DISCUSSION

A. Visual Observations

The visual examination of the exposed specimens revealed a continuing color change that correlated with the duration of the environmental exposure (Figure 1). Specifically, the CNS30 specimen exhibited a yellowish hue, which deepened to brown in CNS60 and became significantly darker in CNS90. This observation in surface coloration can be attributed to environmental factors, including UV radiation, moisture, and thermal cycling. Among these, the UV radiation had the greatest impact by initiating photo-oxidative reactions that induce chain scission at the unsaturated sites within the polyester matrix [20]. These reactions resulted in the formation of new chromophoric groups that absorb visible light, thereby producing a darker appearance on the composite surface. As a result, the observed discoloration can serve as a qualitative indicator of an increasing surface deterioration with extended exposure duration [13].

The prolonged UV-induced oxidation can lead to surface photodegradation [21]. This mechanism typically begins with the absorption of UV photons by catalytic residues, hydroperoxides, carbonyl groups, or unsaturated molecular

structures present in the resin. The excited molecular states subsequently trigger macromolecular reactions that compromise the composite's physical and thermal properties [14]. Authors in [22] demonstrated that significant color change can occur after 300 h of environmental exposure, while in [23], it was observed that an elevated nano-filler content may further accelerate the discoloration due to filler agglomeration or refractive index mismatch between the filler and the surrounding polyester matrix.

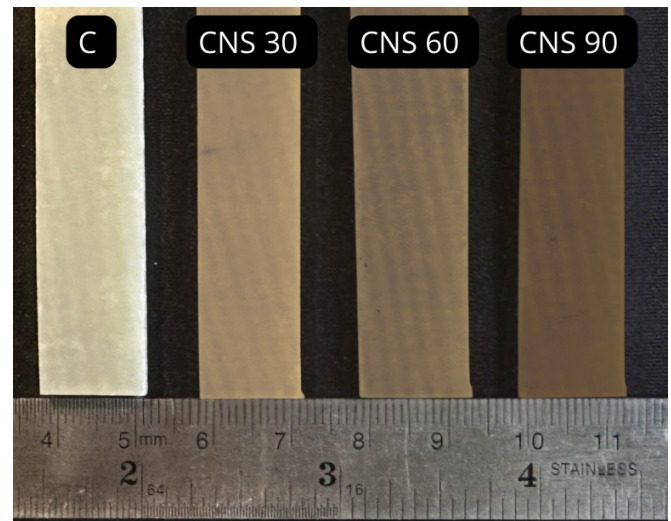


Fig. 1. Color change of specimens.

B. Macro Photographic Observations

A macro-photographic analysis was conducted to investigate the morphological changes in the GFRP composites after 90 days of weathering and subsequent combustion. Figure 2 illustrates the macro-images of specimen surfaces after 30 and 90 days of weather exposure.

The specimen subjected to 30-days of environmental exposure (Figure 2(a)) retained a quite stable surface integrity after combustion. This was evidenced by the formation of a uniformly distributed char layer across the surface, which served as a thermal and oxygen barrier that reduces the combustion reaction rate and limits the heat transfer to the interior of the composite. The presence of the NS was indicated by white residues that were homogeneously dispersed on the surface. These residues were formed when NS was exposed to elevated temperatures (800 – 1000 °C), undergoing a partial decomposition or reduction into amorphous silica. Concurrently, the polyester matrix underwent thermal decomposition (pyrolysis), producing CO and elemental carbon. The resulting reaction products, including lower silicon oxides (SiO) and amorphous silicon (Si), contributed to the formation of a visible white layer on the surface of the charred composite [10].

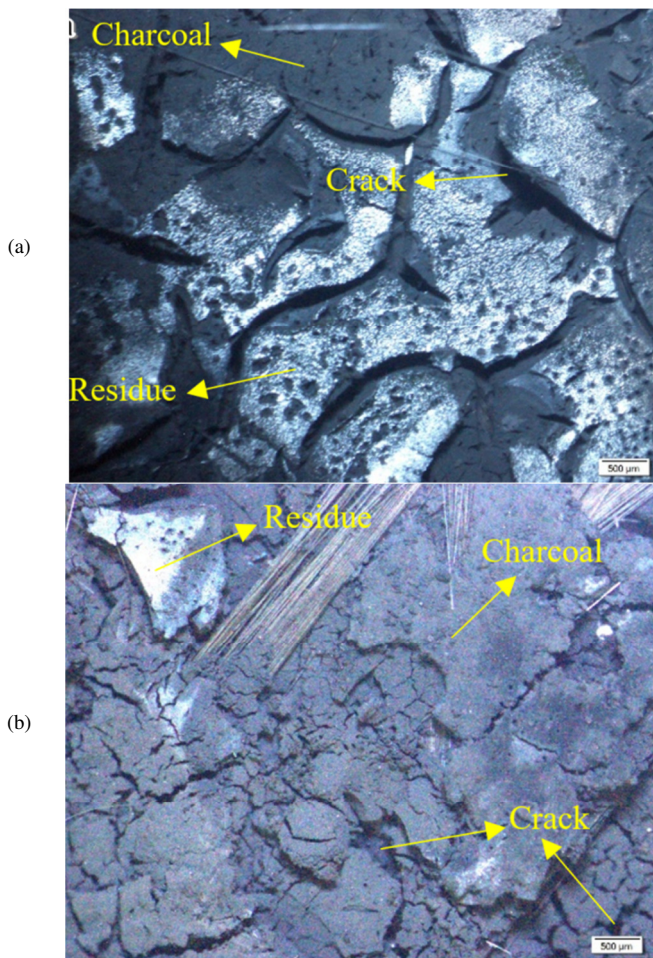


Fig. 2. Macro photographs of composite surface post-combustion: (a) 30-day weathering, (b) 90-day weathering.

In contrast, Figure 2(b) revealed a more severe surface degradation, including prominent surface cracks and signs of delamination. This deterioration was attributed to the cumulative effects of the photo-oxidation and thermal cycling, which weakened the interfacial adhesion between the fiber and the matrix. The presence of cracks facilitated the diffusion of oxygen and heat into the composite interior, leading to accelerated and uncontrolled combustion reactions [24]. These findings indicated a significant reduction in fire resistance with increasing durations of environmental exposure, underscoring the detrimental impact of the long-term weathering on composite performance.

C. TGA and DSC Analysis

The TGA and DSC results, presented in Figures 3 and 4, respectively, indicated a progressive decline in the thermal stability of the GFRP composites with increasing exposure duration. The TGA provided quantitative information about structural integrity by measuring the mass loss as a function of temperature. At the same time, DSC captured the changes in the material's heat flow behavior, reflecting its reactive thermal capacity.

The control specimen (C) exhibited the highest thermal stability, with a gradual mass loss and a final residue exceeding 90% at 400 °C. This observation was different from those in [8], where mass loss began at 327 °C despite the incorporation of 10 wt.% NS into the UPR matrix. In contrast, the specimens exposed for 30 and 60 days (CNS30 and CNS60) exhibited a sharper mass loss starting at approximately 250 °C, with final residue levels between 50-55%. The most severe degradation occurred in the CNS90 specimen, which demonstrated an earlier onset of decomposition and retained only 45% residue [25, 26]. These findings are consistent with those of [17], where oxidation and hydrolysis in a three-month period of environmental exposure accelerated the mass loss in GFRP composites by up to 25.6%.

The structural deterioration identified through TGA was associated with photo-oxidative and hydrolytic degradation mechanisms activated by a long-term exposure to UV radiation, elevated humidity, and thermal fluctuations [27]. These degradation pathways, also observed by the surface discoloration depicted in Figure 1, included the cleavage of ester linkages within the polyester matrix and degradation of the fiber-matrix interface, leading to the formation of low-molecular-weight volatile fragments with poor thermal stability [28]. This degradation mechanism was also reported in [15], where it was observed that UV irradiation promoted the accumulation of carbonyl groups in the UPR matrix filled with ZnO, leading to a reduction in molecular weight and an earlier onset of thermal decomposition.

The DSC results, presented in Figure 4, supported the TGA findings, revealing significant alterations in the heat flow characteristics of the composites following weathering. The control sample (C) exhibited two sharp and high-intensity exothermic peaks at approximately 400 °C and 510 °C, corresponding to the decomposition of polyester functional groups and the complete combustion of organic components, respectively. In contrast, the weathered samples (CNS30, CNS60, and CNS90) showed broader and less intense exothermic responses, along with shifts toward lower transition temperatures. These changes were indicative of a reduction in the composites' reactive energy content, attributable to molecular and structural changes induced by the environmental degradation [29].

The reduction in exothermic intensity suggested a diminished concentration of thermally active functional groups. This is attributed to chain scission events, elevated carbonyl content, and molecular weight reduction, all of which contribute to a reduction in thermal energy release [15]. Furthermore, the uncontrolled crosslinking, initiated by free radicals from photo-oxidative processes, increased the rigidity of the polymer matrix, thereby decreasing the chain mobility and suppressing the intensity of the thermal transitions [30]. These findings were supported in [8], where it was noted that although NS could enhance the initial thermal stability, their effectiveness diminished over time under prolonged oxidative exposure, which compromises the composite's intermolecular network.

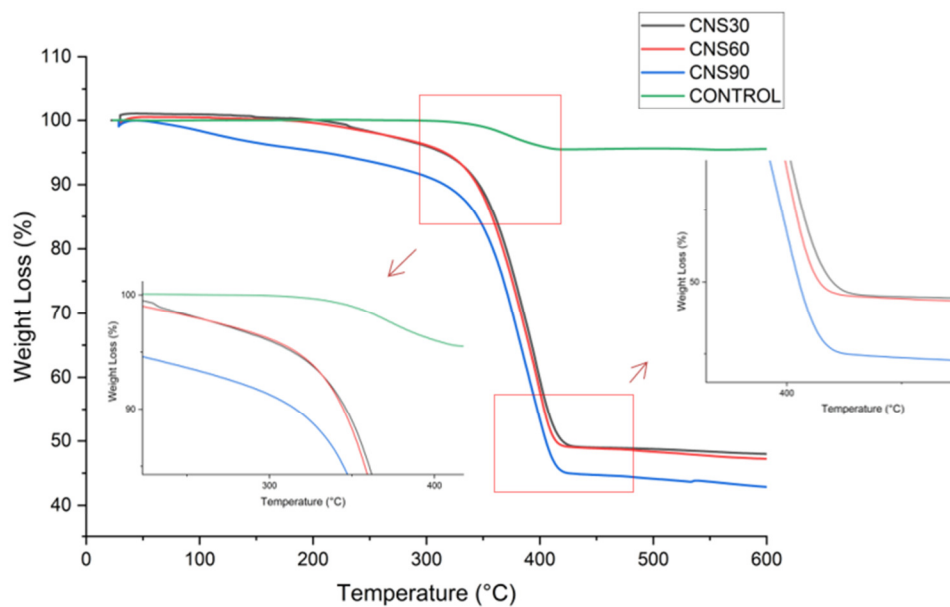


Fig. 3. TGA curves of NS-filled GFRP composites under weathering exposure.

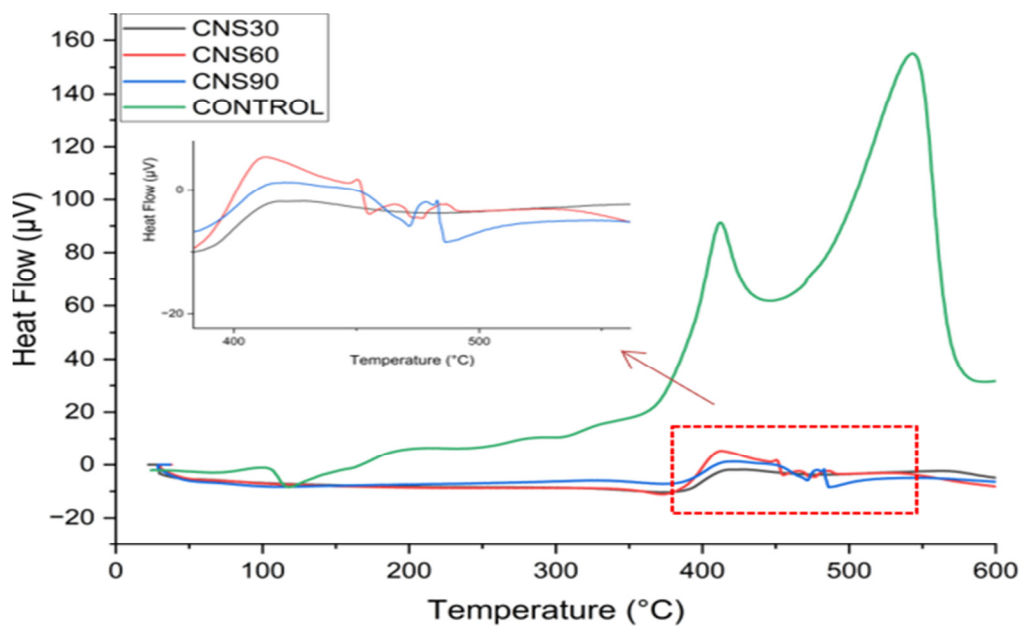


Fig. 4. DSC curves of NS-filled GFRP composites under weathering exposure.

D. Burning Properties

IT and BR are key parameters for evaluating the fire resistance of the composites. As presented in Figures 5 and 6, the control sample (C) exhibited the best performance, with an IT of 66.2 s and a BR of 0.237 mm/s. After 30 days of weathering (CNS30), the IT slightly decreased to 66 s, while BR increased to 0.298 mm/s. A further degradation was observed after 60 days (CNS60), with an IT of 63.4 s and a BR of 0.330 mm/s. The most significant change occurred after 90 days (CNS90), where IT dropped to 38.8 s and BR reached 0.331 mm/s. This trend confirmed that a longer weathering

exposure accelerated ignition and flame propagation, thereby reducing the fire resistance.

This trend aligned with the TGA and DSC results, which indicated a progressive decline in the thermal stability over time. As the thermal mass loss increased and the DSC exothermic peak decreased, ignition occurred more rapidly, and the BR increased [31, 32]. The SEM observations further revealed microcracks and delamination, which facilitated the heat and oxygen diffusion and diminished the barrier effect of NS [33]. After 90 days, the silica residue layer became fragmented, resulting in a 41% reduction in IT and a 40% increase in BR compared to the control specimen (C). These

findings were consistent with those of [34], where photodegradation weakened the protective role of NS in thermoset matrices.

The degradation of the UPR matrix and NS after 90 days of environmental exposure significantly reduced IT and increased BR in the CNS90 specimen compared to CNS30 and CNS60. Despite the decline in fire performance, CNS90 outperformed UPR/GF composites without fillers, as reported in [3], with an IT of 32 s and a BR of 0.49 mm/s. Additionally, CNS90 showed a better fire resistance than the GFRP composites filled with 4 wt.% nano-clay, as reported in [35], where the BR reached 0.383 mm/s.

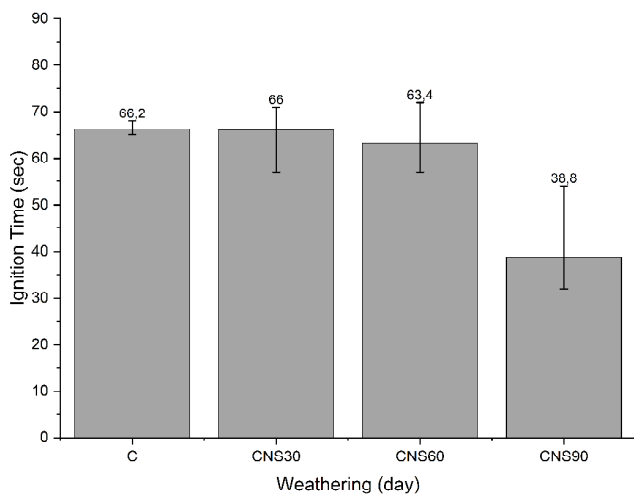


Fig. 5. IT of NS-filled GFRP composites after weathering exposure.

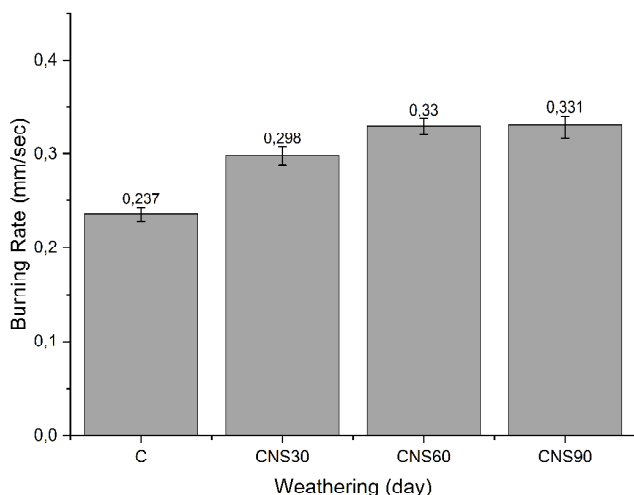


Fig. 6. BR of NS-filled GFRP composites after weathering exposure.

These results further support the findings of [19], where it was emphasized that the effectiveness of nano-fillers depends on the structural integrity of the polymer matrix. Although thermal degradation occurred due to environmental exposure, the addition of NS continued to provide partial protection after three months of aging. This highlighted its role as a flame-

retardant filler in GFRP composites, demonstrating that NS not only functions as a mechanical additive, but also contributes functionally to fire resistance under weathered conditions.

IV. CONCLUSION

This study evaluated the effects of 0, 30, 60, and 90 days of weathering on the thermal and fire performance of Glass Fiber Reinforced Polymer (GFRP) composites filled with Nano-Silica (NS) fillers. The key findings are summarized as follows:

- The visual observations revealed a gradual darkening of the composite surface, transitioning from yellow (30 days) to brown (60 days), and dark brown (90 days).
- The macro photographs presented a uniform char layer on the CNS30 sample, while CNS90 exhibited several cracking and delamination.
- The TGA analysis confirmed a gradual reduction in the thermal stability. The control specimen began to degrade above 400 °C and retained more than 90% residue, while the weathered samples exposed for 30-60 days started to lose mass around 250 °C, and the 90-day sample showed degradation below 240 °C.
- The exothermic DSC peaks, initially observed at 400 °C and 510 °C, declined in intensity.
- The Ignition Time (IT) gradually decreased from 66 s (control) to 63 s (60 days) and reached a minimum of 39 s after 90 days.
- The Burning Rate (BR) increased from 0.237 mm/s to 0.33 mm/s after 90 days of exposure.

These findings indicate that the addition of NS continued enhancing the thermal stability and fire resistance, even when the composite was exposed to outdoor environmental conditions for three months.

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