ISSN: 2181-3337 | SCIENTISTS.UZ VOLUME 5 ISSUE 9 | SEPTEMBER 2026

STUDY OF THE MECHANICAL STABILITY AND FIRE RESISTANCE OF FLAME-RETARDANT-MODIFIED POLYESTER–MINERAL COMPOSITES

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DOI: 10.67620/sai.59a0010

ABSTRACT

This study investigated the composition, preparation, mechanical behavior, and fire performance of decorative polymer–mineral composites based on polyester resin. $CaCO_3$, SiO_2, and marble granules were used as mineral fillers, while ammonium phosphate and $Al(OH)_3$ were incorporated as flame-retardant additives. Poliefir-1–Poliefir-4 and an Analog sample were compared in terms of maximum force, maximum stress, elongation, and strain. Poliefir-2 showed the highest strength, whereas Poliefir-3 showed greater deformability. Among the flame-retardant formulations, FR-1 combined high load-bearing capacity with deformation stability, while FR-9 exhibited the highest maximum stress. Limiting oxygen index (LOI) results indicated that increasing the flame-retardant phase improved resistance to combustion; FR-9 and FR-10 reached the highest LOI ranges, supporting the effectiveness of the combined ammonium phosphate and $Al(OH)_3$ system. Overall, the results demonstrate that the proportions and types of mineral fillers and flame-retardant additives strongly influence both mechanical response and fire resistance. Optimizing these components can provide a practical balance among strength, deformation capacity, and fire safety for decorative composites used in construction and interior finishing applications.

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Keywords: *polyester composites, mineral fillers, flame retardants, mechanical properties, limiting oxygen index (LOI).*

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Introduction

Study of the Mechanical Stability and Fire Resistance of Flame-Retardant-Modified Polyester–Mineral Composites

Unsaturated polyester resins (UPRs) are widely used thermosetting matrices because of their processability, cost effectiveness, and broad mechanical and chemical performance, including in construction-related applications (Malik et al., 2000; Seraji et al., 2022). Their inherent flammability, however, has motivated extensive research on additive and reactive flame-retardant systems (Chu, Hu, et al., 2024).

Ammonium polyphosphate (APP) is among the most common phosphorus-containing flame retardants for UPR, but its loading level and interaction with the polymer matrix can affect both fire performance and mechanical properties (Chu, Lu, et al., 2024). Prior work has examined APP in

combination with phosphinate salts (Reuter et al., 2020), triphenyl phosphate (Pan et al., 2012), aluminum hydroxide (Chen et al., 2017), metallic oxides and alumina-based fillers (Tibiletti et al., 2011), and magnesium hydroxide (Tang et al., 2013), demonstrating that flame-retardant efficiency depends strongly on the broader formulation.

Recent studies have also explored surface-modified APP and hybrid architectures, including chitosan/APP-coated diatomite (Chen et al., 2020), ionic-liquid-functionalized imogolite nanotubes with APP (Zhu et al., 2022), Schiff-base-coated APP (Zhang et al., 2023), and other synergistic systems designed to preserve multiple functional properties (Liu et al., 2020). Mineral reinforcement is also relevant: marble-containing polyester composites can provide useful combinations of mechanical properties and fire resistance (Abenojar et al., 2021). The limiting oxygen index (LOI) is a widely used indicator of combustion resistance, with ambient-temperature testing standardized in ISO 4589-2 (International Organization for Standardization, 2017).

Against this background, the present study examined decorative polymer–mineral composites based on polyester resin and modified with $CaCO_3$, SiO_2, marble granules, ammonium phosphate, and $Al(OH)_3$. Poliefir-series and FR-series formulations were compared to determine how mineral-filler type and flame-retardant loading affected maximum force, maximum stress, elongation, strain at break, and LOI. The objective was to identify formulations that provide an appropriate balance between mechanical stability, deformation capacity, and fire resistance.

Materials and Methods

Materials and Formulations

The study investigated decorative polymer–mineral composite materials based on polyester resin, including Poliefir-1, Poliefir-2, Poliefir-3, Poliefir-4, and Analog samples, as well as fire-resistant formulations designated FR-10, FR-9, FR-1, FR-4, and FR-3. In all compositions, polyester resin served as the binder matrix. Finely dispersed $CaCO_3$ and SiO_2 were used to form the mineral phase, while marble granules with a particle size of 0.5–3 mm were incorporated into selected formulations. To improve the fire resistance of the composites, ammonium phosphate (AP) and $Al(OH)_3$ were introduced as flame-retardant additives. Accelerator and curing-agent components were used to control the curing process of the polyester resin.

The compositions of the Poliefir-series samples differed in the type and amount of mineral fillers. Poliefir-1 contained 29.31 wt.% polyester resin, 54.63 wt.% $CaCO_3$, 5.86 wt.% SiO_2, and 10.00 wt.% ammonium phosphate, with no marble granules or $Al(OH)_3$ added. Poliefir-2 consisted of 36.84 wt.% polyester resin, 18.87 wt.% SiO_2, 34.14 wt.% marble granules, and 10.00 wt.% ammonium phosphate. $CaCO_3$ and $Al(OH)_3$ were not used in this formulation. Poliefir-3 contained 27.68 wt.% polyester resin, 51.60 wt.% $CaCO_3$, 5.54 wt.% SiO_2, 10.00 wt.% ammonium phosphate, and 5.00 wt.% $Al(OH)_3$. Poliefir-4 consisted of 34.79 wt.% polyester resin, 17.82 wt.% SiO_2, 32.25 wt.% marble granules, 10.00 wt.% ammonium phosphate, and 5.00 wt.% $Al(OH)_3$. In all Poliefir-series compositions, small amounts of accelerator and curing-agent components were added to bring the total formulation to 100 wt.%. The Analog sample was used as a reference specimen for comparative evaluation of the mechanical properties of the experimental composites and was prepared under the same technological conditions as the other samples.

In the FR-series composites, the main compositional variables were a decrease in polyester resin content and an increase in the proportion of flame-retardant components. The FR-1 formulation selected for mechanical testing contained 30.00 wt.% polyester resin, 27.00 wt.% $CaCO_3$, 5.00 wt.% SiO_2, 20.00 wt.% marble granules, 8.00 wt.% ammonium phosphate, and 9.80 wt.% $Al(OH)_3$. In FR-3, the polyester resin content was reduced to 29.00 wt.%, while the formulation contained 25.00 wt.% $CaCO_3$, 5.00 wt.% SiO_2, 20.00 wt.% marble granules, 10.00 wt.% ammonium phosphate, and 10.80 wt.% $Al(OH)_3$. FR-4

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consisted of 29.00 wt.% polyester resin, 18.00 wt.% $CaCO_3$, 6.00 wt.% SiO_2, 27.00 wt.% marble granules, 10.00 wt.% ammonium phosphate, and 9.80 wt.% $Al(OH)_3$.

In the FR-9 formulation, which contained a higher proportion of flame-retardant additives, polyester resin accounted for 26.00 wt.%, $CaCO_3$ for 21.00 wt.%, SiO_2 for 5.00 wt.%, marble granules for 16.00 wt.%, ammonium phosphate for 16.00 wt.%, and $Al(OH)_3$ for 15.83 wt.%. In the FR-10 composition, the polyester resin content was likewise maintained at 26.00 wt.%, while the $CaCO_3$ content was reduced to 14.00 wt.%, the SiO_2 content was increased to 6.00 wt.%, and the amount of marble granules was increased to 23.00 wt.%. This formulation contained 16.00 wt.% ammonium phosphate and 14.82 wt.% $Al(OH)_3$. In all FR-series compositions, the amounts of accelerator and curing-agent components were maintained at approximately 0.02 wt.% and 0.14–0.18 wt.%, respectively.

Specimen Preparation and Testing

The prepared mixtures were initially cured at 20–25 °C for at least 6 h. After completion of the curing process, the specimens were removed from the molds and stored under stable conditions prior to testing. In particular, the results obtained for Poliefir-1–Poliefir-4 and the Analog sample made it possible to evaluate the effects of mineral filler type and $Al(OH)_3$ addition on the mechanical properties. The FR-1, FR-3, FR-4, FR-9, and FR-10 formulations enabled a comparative assessment of changes in tensile behavior and strain-at-break characteristics associated with increasing amounts of ammonium phosphate and $Al(OH)_3$.

Fire performance was evaluated using the limiting oxygen index (LOI), and the resulting LOI values were compared across the Poliefir and FR formulations.

Results and Discussion

It was found that the tensile mechanical properties of polyester-based decorative polymer–mineral composites were significantly dependent on their composition. The results presented in Table 1 show that the maximum load, stress, elongation, and relative strain values of the Poliefir-1–Poliefir-4 and Analog samples did not change according to the same trend. This indicates that the load-bearing capacity of the composites is determined not only by the proportion of the polyester matrix but also by the type, particle dispersion, amount, and degree of distribution of the mineral fillers within the matrix.

Table 1

Tensile Mechanical Properties of Poliefir-1–Poliefir-4 and Analog Decorative Polymer–Mineral Composites

Sample name	Maximum force, calculated over all regions (N)	Maximum stress, calculated over all regions (MPa)	Maximum elongation, calculated over all regions (mm)	Maximum strain, calculated over all regions (%)	Force at break, sensitivity: 10 (N)	Stress at break, sensitivity: 10 (MPa)	Elongation at break, sensitivity: 10 (mm)	Strain at break, sensitivity: 10 (%)
Poliefir-1	1851.56	8.05025	1.90920	6.36400	1851.56	8.05025	1.90920	6.36400
Poliefir-2	2280.93	8.14617	3.64787	12.1596	2280.93	8.14617	3.64787	12.1596
Poliefir-3	1812.79	4.31617	4.45417	14.8472	1804.45	4.29630	4.46670	14.8890
Analog	1802.23	6.00744	1.51787	5.05956	1802.23	6.00744	1.51787	5.05956
Poliefir-4	1935.86	5.69370	2.51957	8.39856	1935.86	5.69370	2.51957	8.39856

Mechanical Properties of Poliefir-Series Composites

According to the data in Table 1, the Poliefir-2 sample exhibited the highest maximum force, reaching 2280.93 N. Its maximum stress was also 8.14617 MPa, the highest value among the Poliefir formulations investigated. The use of 36.84 wt.% polyester resin, 18.87 wt.% SiO_2, and 34.14 wt.% marble granules with a particle size of 0.5–3 mm in this formulation may have promoted effective load distribution between the polymer phase and the rigid mineral particles. As a result, the material's resistance to external loading increased. The maximum force of Poliefir-2 was approximately 26.6% higher than that of the

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Analog sample, indicating that the selected mineral composition was effective in enhancing mechanical strength.

For the Poliefir-1 sample, the maximum force was 1851.56 N and the maximum stress was 8.05025 MPa. Although these values were slightly lower than those of Poliefir-2, the stress value was substantially higher than that of the Analog formulation. The $CaCO_3$ content in Poliefir-1 reached 54.63 wt.%, providing a high proportion of the mineral phase. Finely dispersed $CaCO_3$ particles may fill voids in the polymer matrix and contribute to densification of the composite. At the same time, the absence of marble granules limited the deformation behavior of this sample: the maximum elongation was 1.90920 mm and the relative strain was 6.364%.

A different trend in mechanical properties was observed for the Poliefir-3 sample. Although this formulation was not the leading one in terms of maximum force and stress, the maximum elongation reached 4.45417 mm and the strain reached 14.8472%. At break, these values were 4.46670 mm and 14.8890%, respectively. Thus, Poliefir-3 demonstrated relatively high deformability. This behavior may be associated with the incorporation of 5 wt.% $Al(OH)_3$, the reduced polyester-matrix content, and the presence of a $CaCO_3$-based mineral phase. The combined effect of the mineral and flame-retardant components altered not only the stiffness of the material but also its deformation mechanism.

In Poliefir-3, the maximum force was 1812.79 N and the force at break was 1804.45 N, with only a very small difference between them. This indicates that the sample did not undergo a sharp loss of load after reaching the maximum load. At the same time, the close values of maximum strain and strain at break indicate that the deformation process continued consistently up to failure. Such behavior may be important for decorative materials because the ability of a material to undergo a certain degree of deformation under external loading rather than immediately transition to brittle failure can improve service reliability.

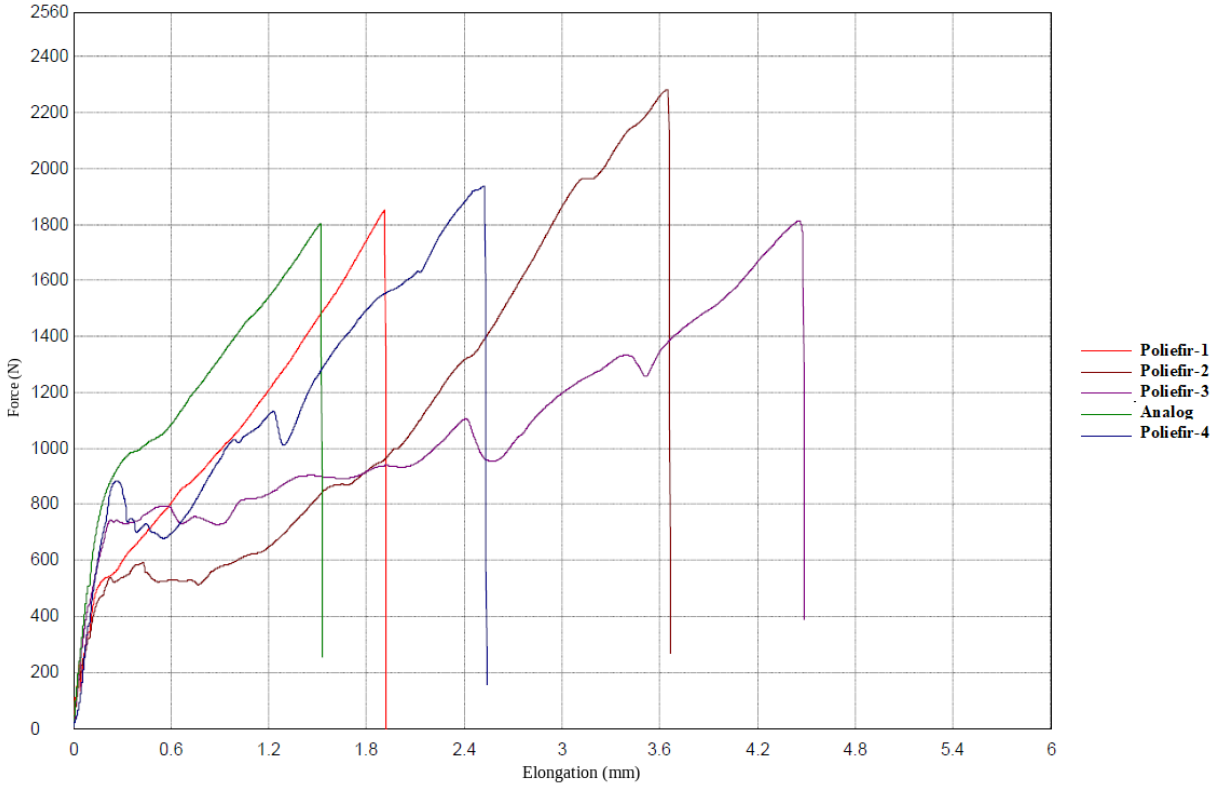
The Poliefir-4 sample exhibited a maximum load of 1935.86 N, which was higher than that of the Analog sample. However, the maximum stress was 5.69370 MPa, lower than the values obtained for Poliefir-1 and Poliefir-2. The maximum strain was 8.39856%, while the elongation was 2.51957 mm. The simultaneous presence of marble granules and $Al(OH)_3$ in this formulation produced intermediate mechanical behavior: the material deformed more than Poliefir-1 but did not reach the extensibility observed for Poliefir-3.

The Analog sample, used as the reference, exhibited a maximum force of 1802.23 N and a stress of 6.00744 MPa. Its maximum elongation was limited to 1.51787 mm and its strain to 5.05956%. Thus, most of the experimental formulations showed an increase in either load-bearing capacity or deformability relative to the Analog sample. In particular, the pronounced increase in strength for Poliefir-2 and in deformation capacity for Poliefir-3 confirms that decorative materials with different performance characteristics can be obtained through targeted modification of the formulation parameters.

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Figure 1

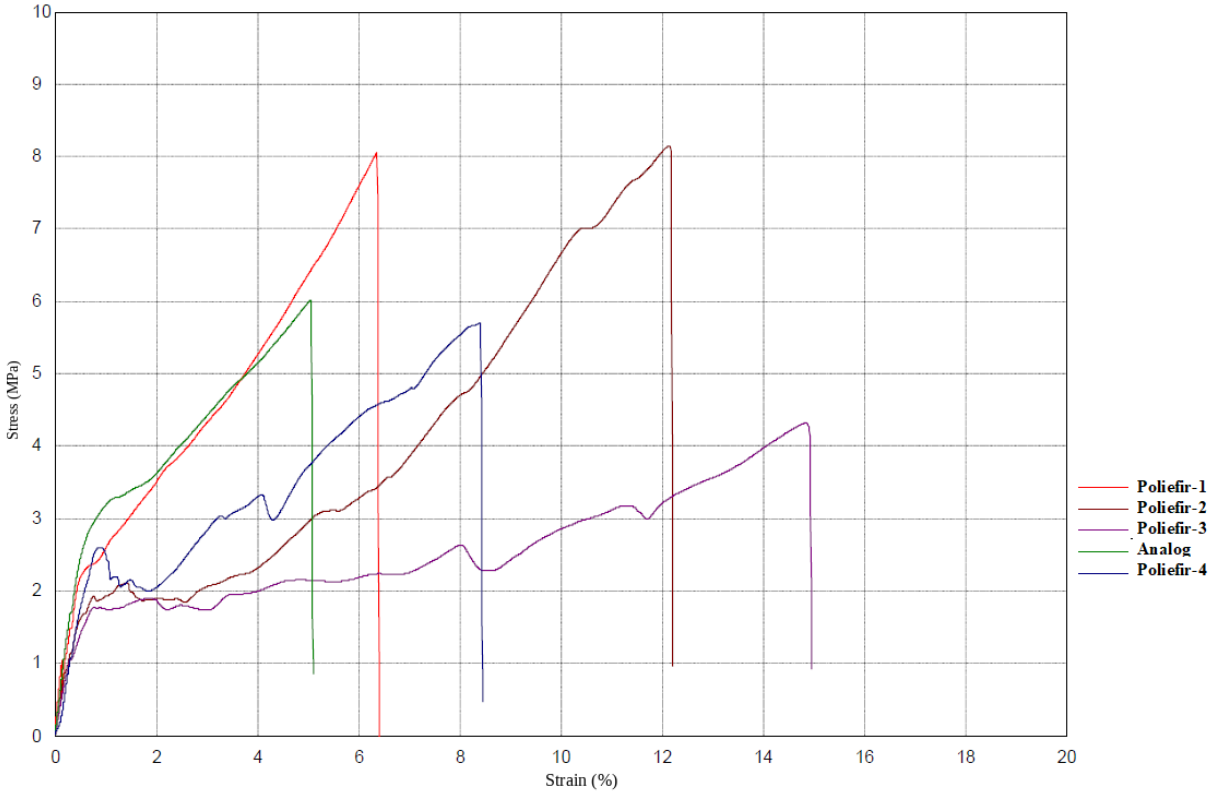
Maximum Force Values of Poliefir-1–Poliefir-4 and Analog Samples



The results presented in Figures 1–2 complement the data in Table 1 and more clearly illustrate the differences between the samples in their ability to reach maximum values and in their deformation ranges. Joint evaluation of the tabular and graphical data shows that Poliefir-2 is distinguished by its strength characteristics, whereas Poliefir-3 exhibits relatively greater deformability. Poliefir-1 and Poliefir-4 demonstrate mechanical behavior intermediate between these two cases.

Figure 2

Maximum Strain Values of Poliefir-1–Poliefir-4 and Analog Samples



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Tensile Properties of FR-Series Composites

In the FR formulations with increased amounts of flame-retardant components, the changes in mechanical characteristics were more pronounced. According to the data in Table 2, FR-1 exhibited the highest maximum load in this group, reaching 2285.74 N. Its maximum elongation was 5.02377 mm and its strain was 16.7459%, among the highest values in the group. At break, the force was 2281.93 N, the stress 7.13102 MPa, the elongation 5.03127 mm, and the strain 16.7709%. The near coincidence of the maximum and break values indicates that the material maintained stable resistance until the end of loading.

The FR-1 formulation contained 30 wt.% polyester resin, 27 wt.% $CaCO_3$, 5 wt.% SiO_2, 20 wt.% marble granules, 8 wt.% ammonium phosphate, and 9.80 wt.% $Al(OH)_3$. At this ratio, a relatively balanced structural system may have formed among the organic matrix, finely dispersed particles, and coarse mineral filler. Consequently, the composite was able to withstand a high load while retaining substantial elongation.

The FR-9 sample exhibited the highest maximum stress, reaching 9.32198 MPa. The maximum force was 2237.27 N, the elongation 4.28413 mm, and the strain 14.2804%. The maximum and break values for this sample were observed to coincide completely. This indicates that failure of the FR-9 sample occurred very close to the final loading point. Despite increasing the ammonium phosphate content to 16 wt.% and $Al(OH)_3$ to 15.83 wt.%, the material retained a high level of mechanical stress. This result indicates that increasing the flame-retardant content does not necessarily lead to a sharp reduction in mechanical strength; rather, the effect develops in conjunction with the overall mineral structure of the composite.

Table 2

Tensile Mechanical Properties of FR-1, FR-3, FR-4, FR-9, and FR-10 Decorative Polymer–Mineral Composites

Sample name	Maximum force, calculated over all regions (N)	Maximum stress, calculated over all regions (MPa)	Maximum elongation, calculated over all regions (mm)	Maximum strain, calculated over all regions (%)	Force at break, sensitivity: 10 (N)	Stress at break, sensitivity: 10 (MPa)	Elongation at break, sensitivity: 10 (mm)	Strain at break, sensitivity: 10 (%)
FR-10	159.814	0.98650	0.18517	0.61722	—	—	—	—
FR-9	2237.27	9.32198	4.28413	14.2804	2237.27	9.32198	4.28413	14.2804
FR-1	2285.74	7.14295	5.02377	16.7459	2281.93	7.13102	5.03127	16.7709
FR-4	1816.80	5.67751	4.94193	16.4731	1816.14	5.67543	4.95687	16.5229
FR-3	1824.96	6.51770	2.59800	8.66000	1773.95	6.33553	2.62303	8.74344

Note. Dashes indicate break values that were not reported for FR-10.

For FR-4, the maximum load was 1816.80 N, the maximum elongation 4.94193 mm, and the strain 16.4731%. These values indicate that although its load-bearing capacity was lower than that of FR-1, a high level of deformability was retained. The 27 wt.% marble granules and 10 wt.% ammonium phosphate in the FR-4 formulation had a significant effect on the structural response of the composite. The force at break was 1816.14 N, almost equal to the maximum value, indicating that the material maintained a stable state under load for a longer period.

For FR-3, a maximum force of 1824.96 N and a maximum stress of 6.51770 MPa were recorded. The maximum strain of this formulation was 8.660%, while the strain at break was 8.74344%. Compared with FR-1 and FR-4, FR-3 exhibited lower elongation capacity. In addition, a decrease from 1824.96 N to 1773.95 N was observed from the maximum force to the force at break. This indicates a certain degree of structural weakening after the sample reached the maximum load. Nevertheless, the force at break retained approximately 97.2% of the maximum value, meaning that most of the load-bearing capacity was preserved immediately before failure.

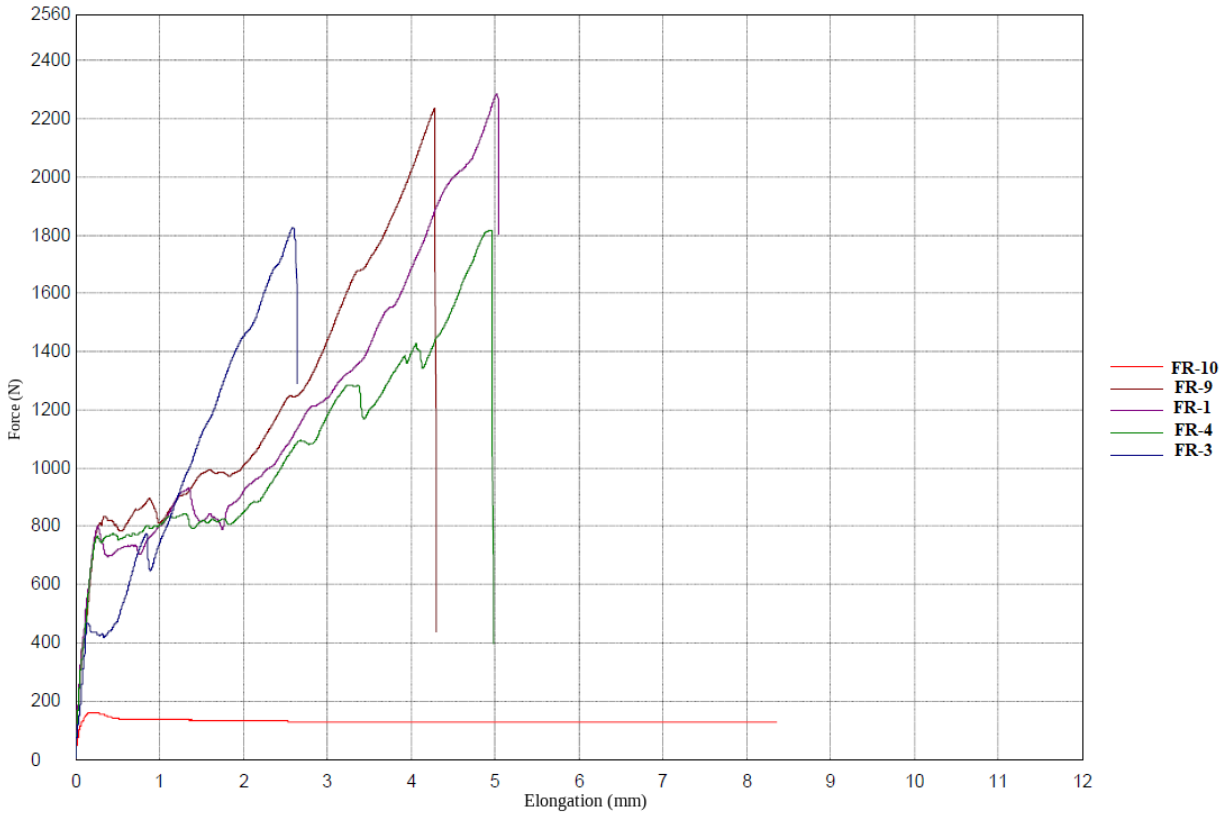
The most pronounced difference was recorded for the FR-10 sample. Its maximum force was only 159.814 N, the maximum stress 0.98650 MPa, the elongation 0.18517 mm, and the strain 0.61722%. In addition, separate values at break were not recorded. These results show that FR-10 achieved substantially

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lower mechanical performance than the other FR samples. In the FR-10 formulation, the reduction of $CaCO_3$ to 14 wt.%, the use of 16 wt.% ammonium phosphate and 14.82 wt.% $Al(OH)_3$, and the resulting change in the overall mineral-to-flame-retardant ratio may have impaired the load-transfer mechanism within the polymer matrix. However, attributing such a sharp decrease solely to compositional factors requires caution. Sample preparation quality, internal voids, defects, or individual variations during testing may also have influenced the result.

Figure 3

Maximum Force Values of FR-1, FR-3, FR-4, FR-9, and FR-10 Samples



The graphical results presented in Figures 3–4 illustrate the non-uniform mechanical behavior of the FR formulations. FR-1 retained a wide deformation range together with a high load-bearing capacity, while FR-9 was distinguished by the highest stress value. FR-4 exhibited high elongation at a relatively lower load level. FR-3 showed moderate mechanical characteristics, whereas FR-10 differed markedly from the other samples by its substantially lower values.

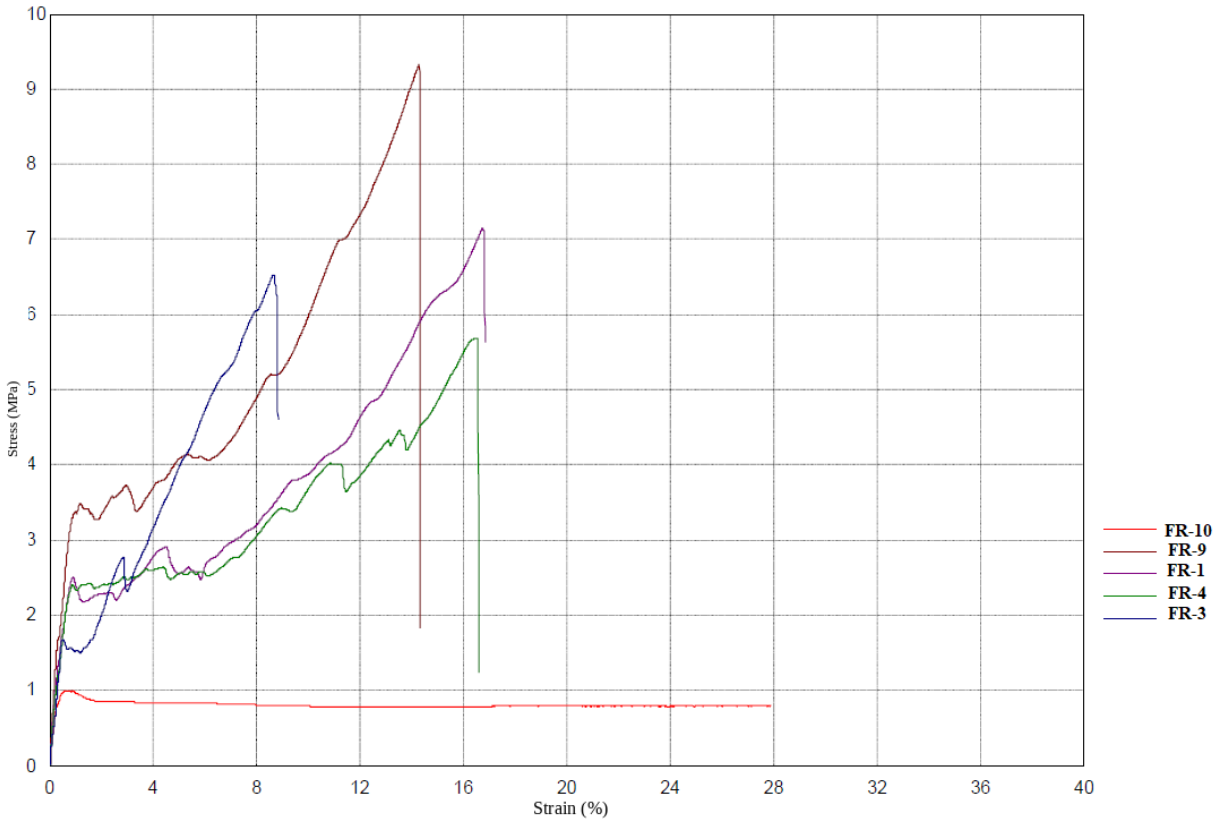
Comparative Evaluation of the Poliefir and FR Series

Comparison of the results for the two groups showed that the effects of mineral fillers and flame-retardant additives on mechanical properties depend on the overall composition of the formulation. In the Poliefir series, Poliefir-2 was distinguished by maximum strength, whereas Poliefir-3 exhibited high deformability. In the FR group, FR-1 simultaneously demonstrated a high load and large relative strain, while FR-9 reached the highest stress.

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Figure 4

Maximum Strain Values of FR-1, FR-3, FR-4, FR-9, and FR-10 Samples



In addition, the maximum loads of Poliefir-2 and FR-1 were within nearly the same range, amounting to 2280.93 and 2285.74 N, respectively. However, the maximum strain of FR-1 was 16.7459%, higher than the 12.1596% recorded for Poliefir-2. This indicates that, in certain formulations, the introduction of flame-retardant additives can preserve or even increase deformation capacity.

The obtained results indicate that, when designing decorative polymer–mineral composites, it is insufficient to focus on maximizing only a single parameter. A balance must be established among high load-bearing capacity, adequate deformation reserve, and fire resistance. From this perspective, the Poliefir-2, Poliefir-3, FR-1, and FR-9 formulations demonstrated promising results for different functional applications. FR-1 was particularly distinguished by the simultaneous combination of high maximum load, elongation, and strain, whereas FR-9 was characterized by its resistance to high mechanical stress.

Overall, the experimental data confirm that the ratio among mineral fillers, the polyester matrix, and flame-retardant components is one of the principal factors determining the mechanical properties of the composite. The formation of a dispersed mineral structure by $CaCO_3$ and SiO_2, the role of marble granules as a structural load-bearing phase, and the influence of ammonium phosphate and $Al(OH)_3$ on the polymer–filler interface while simultaneously improving fire resistance lead to the complex development of the final properties.

Fire Resistance by Limiting Oxygen Index

The fire resistance of polyester-based decorative polymer–mineral composite materials was evaluated using the limiting oxygen index (LOI). According to the experimental results, the LOI values of the Poliefir-1, Poliefir-2, Poliefir-3, and Poliefir-4 samples were 27.8, 29.8, 32.5, and 35.4%, respectively. These values indicate that resistance to combustion increased with increasing amounts of flame-retardant and mineral components. A similar trend was observed in the FR-series composites. The LOI was 27–29% for FR-1, 28–31% for FR-3 and FR-4, and 34–38% for FR-9 and FR-10. In particular, increasing the amount of the flame-retardant phase resulted in a substantial rise in the oxygen index of the FR-9 and FR-10 samples. The results show that the combined use of ammonium phosphate and $Al(OH)_3$ reduces the tendency of the composites to ignite and improves their fire safety.

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Conclusion

The conducted studies showed that the mechanical and fire-performance properties of decorative polymer–mineral composites based on polyester resin are directly dependent on the proportions of their constituent components. The Poliefir-2 sample was distinguished by high maximum force and stress values, whereas Poliefir-3 exhibited greater elongation and deformation capacity. In the FR series, FR-1 simultaneously retained high load-bearing capacity and deformation stability, while FR-9 was characterized by the highest stress value. The oxygen index results showed improved resistance to combustion in formulations containing increased amounts of ammonium phosphate and $Al(OH)_3$; in particular, the LOI values of the FR-9 and FR-10 samples increased to a higher range. Overall, a coordinated ratio of $CaCO_3$, SiO_2, marble granules, and flame-retardant additives makes it possible to achieve an appropriate balance among strength, deformation capacity, and fire safety in decorative composites.

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