



Enhanced impact strength of glyptal resin composites with red mud: A comparative study of incorporation methods

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Abstract. The purpose of this study was to investigate the use of red mud, as a sustainable filler for glyptic resin composites and injection methods on their impact strength, as well as to optimise the properties of the composites and promote environmentally friendly use of materials. Glyptal resin was synthesised from glycerol and phthalic anhydride. Two methods of red mud introduction were employed: mechanical mixing and *in situ* synthesis. The concentration of red mud ranged within 7-56 wt%. The impact strength of the resulting composites was evaluated using standard tests. The study showed that red mud significantly increases the impact strength of composites based on glyptal resins. The *in situ* synthesis method provided the best results, with a maximum impact strength of 22.05 N-m at a red mud content of 36 wt%, which is 12.5% greater than that of mechanically mixed composites. The optimum filler concentration was determined at 36 wt%, with a sharp decrease in strength at greater concentrations due to particle agglomeration. In addition, the impact strength increased with increasing synthesis time, reaching its peak at 480 min for composites with 7 wt% red mud. This study provided new insights into the integration of red mud as a reinforcing filler in glyptic resins and highlighted the advantages of *in situ* synthesis in achieving better dispersion and matrix interaction, which contributes to the development of stable polymer composites. The findings demonstrated the possibility of using red mud, a complex industrial waste, in the production of high-performance polymer composites. The study offered practical recommendations for optimising the concentration of fillers and synthesis methods, which would contribute to the development of environmentally friendly and cost-effective materials for industrial applications

Keywords: polymer composites; mechanical properties; eco-friendly materials; *in situ* method; fillers

Introduction

The growing demand for high-performance composite materials in various industries necessitates the development of innovative solutions that ensure superior mechanical properties, economic feasibility, and environmental sustainability. Polymer composite materials (PCMs) with fillers derived from industrial waste products have emerged as a promising area in materials science. Among these, red mud (RM), a byproduct of the alumina production process, presents strong potential as a cost-effective and sustainable filler for polymer matrices, including glyptal resins.

RM, with its high alkalinity, poses major challenges in terms of disposal and environmental impact due to its large-scale production and limited recycling options. According to B. Swain *et al.* (2020), over 150 million tonnes

of RM are generated annually worldwide, with the majority being still untreated, leading to risks of soil and water contamination. Its rich chemical composition, including oxides of iron, aluminium, and silicon, opens promising opportunities for reuse in value-added products such as fillers for polymer composites (Prasad *et al.*, 2022).

The use of RM as a filler has not only solved environmental problems, but also greatly improved the mechanical and thermal properties of composite materials. According to P. Wu *et al.* (2023), the incorporation of RMs into polymer composites improved tribological properties such as wear resistance and friction coefficient, while reducing production costs and increasing the thermal stability of materials.

Suggested Citation:

Melnyk, L. (2024). Enhanced impact strength of glyptal resin composites with red mud: A comparative study of incorporation methods. *Technologies and Engineering*, 25(6), 61-68. doi: 10.30857/2786-5371.2024.6.6.

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J. Chen *et al.* (2024) investigated the durability of high-performance composite materials based on RM, focusing on their resistance to chemical and physical factors. Their study demonstrated that modified RM considerably enhances the chemical resistance and mechanical wear resistance of composites, particularly in aggressive environments. However, the study did not address the effects of prolonged thermal cycling and humidity, which are critical for many practical applications. Future research should explore these aspects and improve RM modification methods to achieve more stable composite properties.

The modification of the RM played a crucial role in increasing its functionality. C. Ding *et al.* (2022) demonstrated that the use of chlorinated polyethylene as a coupling agent for RM surface treatment significantly improves the adhesion between the filler and the polymer matrix, thereby improving the mechanical properties of composites. The researchers attributed these findings to a reduction in interfacial defects caused by better adhesion and interaction between the composite components. Furthermore, the modification of RM with a titanate coupling agent and its subsequent incorporation into PVC to obtain composites showed a significant improvement in the thermal stability of the materials. The RM effectively neutralises HCl released during the thermal decomposition of PVC, delaying the onset of pyrolysis, and improving the overall properties of the composite. In addition, the high thermal resistance of RM helps to decelerate heat transfer, which further improves thermal stability.

P.K. Jena *et al.* (2022) synergistically modified epoxy resin with RM and vetiver fibres. Their findings suggested that increasing RM content in composites reduced the friction coefficient, specific wear rate, and wear loss, confirming that RM effectively enhances the wear resistance of epoxy resin-based composites.

Glyptal resins, known for their excellent adhesion, durability, and chemical resistance, are widely used in coatings, electrical insulation, and construction. Incorporating RM as a filler into glyptal resins improves their mechanical properties, particularly impact strength, while contributing to the recycling of industrial waste (Melnyk *et al.*, 2020).

Existing research on RM-based composites has predominantly focused on thermoplastics and thermosetting plastics, leaving glyptal resins understudied. J. Li *et al.* (2020) emphasised that the properties of such composites largely depend on the filler concentration and the method of its introduction. Chemical synthesis methods often provide better dispersion and interaction between the RM and the polymer matrix compared to mechanical mixing. Optimising these parameters is crucial to achieve a balance between mechanical properties and economic viability.

The purpose of this study was to investigate the effect of red mud concentration and incorporation methods on the impact strength of glyptic resin composites. Specifically, the study focused on determining the optimum

filler concentration and understanding how incorporation methods affect mechanical performance. To fulfil the purpose, the tasks were formulated as follows:

1. To evaluate the effects of the concentration of red mud (7-56 wt%) on the impact strength of composites based on glyptal resins.
2. To compare the effects of two incorporation methods – mechanical mixing and *in situ* synthesis – on the mechanical properties of composites.
3. To determine the optimum combination of filler concentration and synthesis method to maximise the impact strength of glyptal resin composites while ensuring economic and environmental feasibility.

Materials and Methods

The study used glyptal resin synthesised from glycerol and phthalic anhydride, following a standard procedure (Feng *et al.*, 2011). The initial weight ratio of glycerol to phthalic anhydride was maintained at 1:1.65, and the reaction was conducted at a constant temperature of 180°C in a reactor equipped with a mechanical stirrer and a thermometer to ensure uniform mixing and temperature control. The filler employed in the composites was red mud, sourced from the Zaporizhzhia Aluminium Plant (Ukraine), its characteristics were presented in the previous study (Melnyk *et al.*, 2020). To enhance its dispersion within the polymer matrix, the red mud was pretreated through ultrasonic grinding (Melnyk, 2023), reducing particle size, and improving its compatibility with the resin.

The composites were prepared using two different methods. The first method involved mechanical mixing, in which red mud at concentrations of 7, 15, 36, and 56 wt% was added to the pre-synthesised glyptal resin using a laboratory roll mill. This ensured uniform distribution of the filler in the resin matrix. The second method employed *in situ* synthesis, where red mud was pre-mixed with glycerol before adding phthalic anhydride. The polymerisation proceeded under the same conditions as described above. The *in situ* method was employed for red mud concentrations of 7, 15, 36, and 56 wt%. The synthesis process lasted 8 hours, during which aliquots were periodically taken for nuclear magnetic resonance spectroscopy (NMR) analysis to monitor the progression of the esterification reaction.

The NMR spectra obtained during the synthesis of glyptal resin, both in the absence and presence of red mud (7 wt%), provide valuable insights into the polymerisation reaction progress and the influence of the filler on the chemical transformation. The spectra were recorded at various time intervals: 30, 180, 305, and 480 min.

The prepared composites were applied as coatings on pre-cleaned glass, metal, or ceramic substrates using a brush. The coatings were left to cure at room temperature for 24 hours and subsequently subjected to thermal treatment at 60°C for 1-2 hours to ensure complete polymerisation and adhesion to the substrate.

The impact strength of the composite films was evaluated using the U-1A apparatus, manufactured by

NOVOTEST, Ukraine. The testing was conducted following the ASTM D2794-93 (2019) standard (Kholiavko & Vladymyrskiy, 2023). This method involved the instantaneous deformation of a coated metal plate under the free fall of a weighted striker. The apparatus included a base, stand, guide tube, pointer, stopper screw, release button, weight holding fixture, weight, striker with a ball, traverse, and anvil. During the test, the weight was raised to a predetermined height and secured with a stopper screw. The coated metal plate was placed on the anvil, ensuring that the impact area was at least 20 mm away from the edges or previously affected zones. By pressing the release button, the weight was allowed to fall freely, transferring the impact through the striker to the plate. The sample was inspected for cracks after each impact. If no cracks were observed, the height of the weight drop was increased incrementally by 5-10 cm, and the test was repeated until cracks appeared. The impact strength (W) was calculated as the product of the weight force (F) and the critical height (h), providing a quantitative measure of the material's resistance to impact deformation:

$$W = F \cdot h. \quad (1)$$

This methodology enabled a systematic evaluation of the impact resistance of glyptal resin composites, enabling a detailed comparison between the mechanical mixing and *in situ* synthesis methods.

Results

NMR Spectroscopic Analysis of the Research Objects' Synthesis

The study of the impact strength of composites based on glyptoplastics modified with red mud revealed major differences depending on the filler concentration and the

method of its introduction into the matrix. The study findings, visualised in Figures 1 and 2, provide a comprehensive view of the influence of these parameters on the mechanical characteristics of the composites, and their analysis helped to trace the esterification process and the role of red mud during synthesis.

For the synthesis without red mud (Fig. 1), the NMR spectra at 30 min revealed signals in the δ 12.75-14 region, corresponding to carboxylic acid protons. Signals at δ 5-5.7 (CHOOC) and δ 4-5 (CH₂OOC, CHOH) indicated the initial formation of ester bonds, reflecting the commencement of the acylation reaction between glycerol and phthalic anhydride. However, the persistence of carboxylic acid signals confirmed the incomplete nature of the esterification at this stage (Claridge, 2009). By 180 min, the intensity of the carboxylic acid signals decreased significantly, while the signals corresponding to ester groups (CH₂OOC, CHOH) became more prominent, suggesting that the esterification reaction was progressing. At 305 min, the carboxylic acid signals diminished further, and by 480 min, they were no longer detectable, indicating complete conversion of the starting materials into glyptal resin.

For the synthesis with 7 wt% red mud (Fig. 2), the spectra at 30 min showed comparable carboxylic acid signals; however, their intensity was slightly lower compared to the synthesis without red mud. This suggests that red mud, due to its basic properties, partially neutralised the carboxylic acid groups formed during the initial stages of the reaction. As the reaction progressed, the intensity of the carboxylic acid signals further declined, while the ester group signals became increasingly prominent. By 480 min, the spectra confirmed the complete disappearance of carboxylic acid protons and the dominance of ester signals, suggesting that the reaction had reached completion.

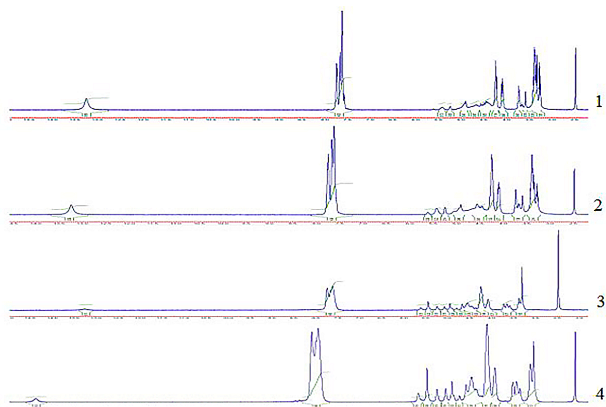


Figure 1. NMR spectra of aliquots taken during the synthesis of glyptal resin (without red mud) at various time intervals

Note: 1 – 30 min, 2 – 180 min, 3 – 305 min, 4 – 480 min. Signals indicate the progressive conversion of carboxylic acid groups (δ 12.75-14) into ester groups (δ 4-5 and δ 5-5.7)

Source: developed by the author of this study

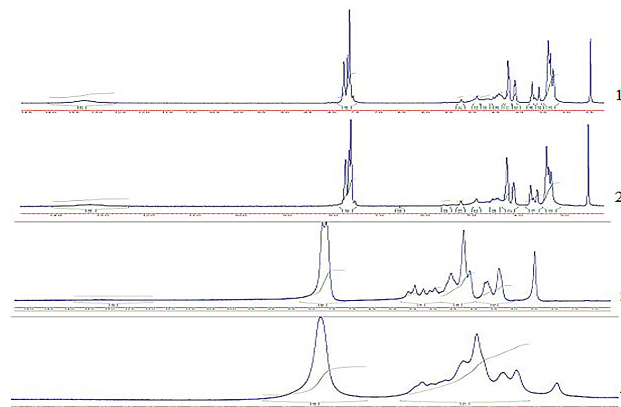


Figure 2. NMR spectra of aliquots taken during the synthesis of glyptal resin with 7 wt% red mud at various time intervals

Note: 1 – 30 min, 2 – 180 min, 3 – 305 min, 4 – 480 min. The reduced intensity of carboxylic acid signals (δ 12.75-14) at early stages reflects the interaction of red mud with the acidic intermediates

Source: developed by the author of this study

The presence of red mud was found to have a notable influence on the reaction kinetics. Specifically, the neutralising effect of red mud slightly reduced the intensity of carboxylic acid signals at earlier stages, suggesting an interaction between the filler and the acidic intermediates. Despite this, the overall progression and completion of the reaction were not hindered, as evidenced by the spectral similarity of the final products in both systems.

In conclusion, the analysis of NMR spectra demonstrated that the synthesis of glyptal resin proceeded effectively in the presence of red mud. While the filler interacted with the acidic intermediates, potentially altering the reaction pathway to a minor extent, it did not impede the overall reaction. These findings suggest that red mud not only serves as a filler but also may act as a mild catalyst or stabiliser during the polymerisation

process, contributing to the unique properties of the final composite materials.

Influence of synthesis time and incorporation method

Figure 3 presents the relationship between synthesis time and impact strength for glyptal resin composites with 7 wt% red mud. The composites prepared via *in situ* synthesis consistently demonstrated greater impact strength compared to those obtained through mechanical mixing. At 30 min, the impact strength of the *in situ* synthesised composite was 3.85% greater than that of the mechanically mixed composite, and this advantage increased to 9.13% by 305 min. This trend is attributed to the superior dispersion of the filler and stronger interfacial bonding achieved during chemical synthesis, which promotes the uniform distribution of red mud particles within the matrix.

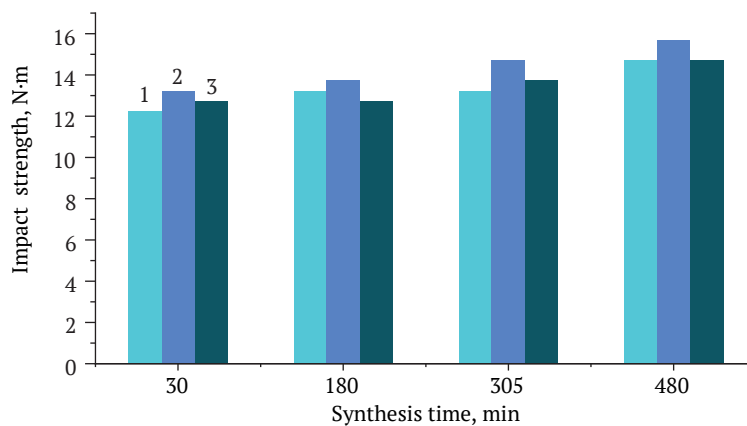


Figure 3. Impact strength of composites with 7 wt% red mud as a function of synthesis time

Note: 1 – pure glyptal resin, 2 – *in situ* synthesis (7 wt%), 3 – mechanical mixing (7 wt%)

Source: developed by the author of this study

Compared to pure glyptal resin, the composites synthesised *in situ* exhibited 6.67-10.78% higher impact strength across all synthesis times. This improvement highlighted the reinforcing effect of red mud as a filler, which enhanced the load distribution and impact resistance of the composite.

Influence of filler concentration

Figure 4 presents the impact strength of composites with varying concentrations of red mud, prepared by both methods.

For composites produced via *in situ* synthesis, the impact strength increased with filler concentration, reaching a maximum value of 22.05 N·m at 36 wt%. Beyond this concentration, a sharp decline in strength was observed, with the value dropping to 11.27 N·m at 56 wt%. An analogous trend was observed for mechanically mixed composites, with maximum impact strength of 19.6 N·m at 36 wt% and a decrease to 10.78 N·m at 56 wt%.

The decrease in impact strength at high filler concentrations can be attributed to agglomeration of red mud particles, which creates stress concentration points and weakens the material. This issue was more pronounced in

mechanically mixed composites, where the filler-matrix interaction was less effective, resulting in lower overall impact strength compared to the *in situ* synthesised composites.

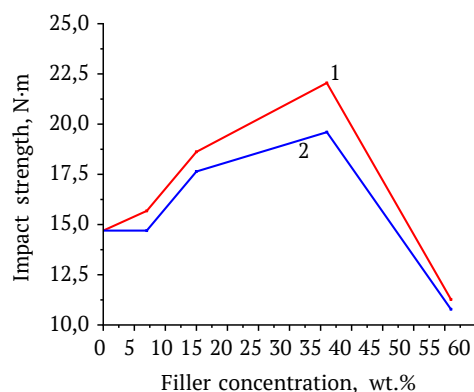


Figure 4. Impact strength of composites as a function of red mud concentration for *in situ* synthesis and mechanical mixing

Note: 1 – *in situ* synthesis, 2 – mechanical mixing

Source: developed by the author of this study

The *in situ* synthesis method consistently outperformed mechanical mixing in terms of impact strength, regardless of filler concentration. At the optimum concentration of 36 wt%, the impact strength of the *in situ* synthesised composite was 12.5% greater than that of the mechanically mixed composite. This superior performance is explained by the chemical incorporation of red mud into the polymer matrix during synthesis, which enhances filler dispersion and bonding at the molecular level.

These findings suggest that the inclusion of red mud as a filler not only improved the mechanical properties of the glyptic resin but also provided a sustainable solution for the disposal of industrial waste. The optimum filler concentration of 36 wt% ensured maximum reinforcement without compromising the integrity of the composite.

Discussion

The integration of red mud as a filler in glyptic resin composites was systematically studied, and its impact on mechanical properties, particularly impact strength, was analysed. The findings demonstrated a considerable enhancement in impact strength when red mud was incorporated, with a maximum value observed at 36 wt% filler concentration using the *in situ* synthesis method. This discussion contextualises these findings within the existing body of research and examines their implications for industrial applications.

A.C. Mârşolea (Cristea) *et al.* (2023) studied the development of polyurethane composites using industrial plastic waste, pyrite, and RM, finding that these fillers can increase the mechanical strength and thermal stability of composites by up to 25%, especially with preliminary chemical treatment. At the same time, the researchers noted that the long-term stability of such composites in aggressive environments is still unexplored, and the tests were limited to static loads without analysing dynamic properties, suggesting the need for further research to improve technologies. The greater impact strength values obtained by the *in situ* synthesis method (12.5% greater than mechanical mixing) confirmed the significance of chemical incorporation for effective filler dispersion and matrix interaction, consistent with previous studies.

The chemical bonds formed during the *in situ* synthesis improved the compatibility between filler and resin matrix, which was confirmed by both J. Chen *et al.* (2024) and the findings of the present study on the potential of red mud. Red mud can act not only as a filler but also as a reinforcing agent, improving mechanical properties through integration at the molecular level. J. Chen *et al.* (2024) additionally proposed combining RM with paper residues to create composites, which increased the crystallinity of polymers and their mechanical properties due to the effects of RM as a heterogeneous nucleator.

Additionally, the trend of declining impact strength beyond 36 wt% filler concentration caused by particle agglomeration mirrors observations by R. Prasad *et al.* (2022), who highlighted analogous challenges in achieving

optimum dispersion in red mud-based composites. This phenomenon underscores the critical role of processing techniques and the necessity of fine-tuning filler concentrations to maximise composite performance. The ultrasonic pretreatment of red mud employed in this study proved effective in mitigating, albeit not entirely eliminating, the agglomeration effects.

J. Liu *et al.* (2018) developed ethylene-vinyl acetate-based composites (ELDHS-SDS-KH) using HCl-modified RM as a flame-retardant filler. Their findings revealed that these modified composites not only emitted fewer harmful substances during combustion but also exhibited improved thermal stability compared to unmodified samples.

L. Qiu *et al.* (2020) proposed another innovative approach, working with RM/phenolic foam composites. The researchers investigated the effects of silane coupling agents and RM on the thermal properties of these materials. Their findings revealed that adding an optimised amount of modified RM increases the limiting oxygen index and greatly enhances the thermal stability of the composites. Y. Wang *et al.* (2021) highlighted the synergistic effects arising from combining RM with other industrial wastes, such as fly ash. Their study confirmed that such combinations improve the interfacial properties of composites, enhancing their overall mechanical strength.

Another group of researchers focused on developing hybrid polymer materials using RM and fibres. A.K. Sinha *et al.* (2021) synthesised an epoxy composite with RM and abaca fibres. By employing the response surface methodology, the researchers demonstrated that optimising RM particle size and fibre concentration considerably improves the wear resistance and mechanical properties of the composite.

S. Nayak & A. Satapathy (2020) examined the effects of RM on bamboo fibres modified for use in polymer composites. Their findings showed that these materials exhibited enhanced tensile strength, flexural strength, and impact resistance, demonstrating their potential for applications in construction and industry. S. Vigneshwaran *et al.* (2020) also explored the potential of RM combined with coconut sheath fibres. The researchers found that silane-treated fibres combined with 20% RM greatly improved the mechanical properties of composites, such as tensile strength, flexural strength, and impact resistance. However, increasing RM content to 30% led to particle agglomeration, negatively affecting material properties.

O. Çimen & H.I. Günaydın (2024) focused their efforts on developing geosynthetic barrier materials using RM and polysulfone (PSU). Through the phase inversion method, the researchers successfully integrated RM into the polysulfone matrix, greatly improving the material's tensile strength. Using SEM, AFM, XRD, and TGA techniques, the researchers confirmed the effective dispersion of RM within the matrix and enhancements in its mechanical and thermal properties. However, the researchers also noted that the long-term stability of these materials under variable environmental conditions was still

unexplored, paving the way for further research. This underscored the significance of RM in creating sustainable construction materials.

J. Liu *et al.* (2020) synthesised a composite material based on RM and polyacrylic acid (RM/PAA) using a graft polymerisation method in a reverse-phase suspension system. This material demonstrated excellent adsorption properties, achieving a maximum adsorption capacity for Cd(II) ions at 96.15 mg/g.

The increase in impact strength with synthesis time, peaking at 480 min for composites with 7 wt% red mud, further supports the hypothesis that extended reaction times enhance the uniformity of filler dispersion and promote stronger matrix-filler interactions. This observation is consistent with findings of L. Qiu *et al.* (2020), who reported improved mechanical properties in composites subjected to prolonged synthesis durations. Notably, the basic properties of red mud appeared to neutralise acidic intermediates during polymerisation, possibly acting as a mild catalyst, as suggested by the NMR spectral analysis.

Comparing these findings with studies on other polymer matrices, such as the research by S. Vigneshwaran *et al.* (2020) on coconut fibre-reinforced composites, revealed both similarities and distinctions. While both studies emphasised the reinforcing effects of fillers, the chemical composition and surface characteristics of red mud uniquely influence its interaction with glyptal resin, resulting in distinct mechanical property enhancements.

From a practical perspective, the optimum filler concentration of 36 wt% identified in the present study provides a valuable guideline for industrial applications, balancing mechanical performance with material integrity. Being a challenging industrial waste, the use of red mud in glyptal resin composites not only addresses environmental concerns but also offers an economically viable alternative to conventional fillers.

However, some limitations persist. The long-term stability of these composites under environmental stressors, such as temperature fluctuations and moisture exposure, warrants further investigation. Additionally, upscaling the *in situ* synthesis method for industrial production poses challenges that must be addressed.

In conclusion, the present study contributed to the growing body of knowledge on sustainable composite materials by demonstrating the feasibility and advantages of red mud as a filler in glyptal resins. The findings highlighted the need for continued research into optimising synthesis methods and exploring other applications of these eco-friendly composites.

Conclusions

The study demonstrated a significant influence of red mud concentration and methods of its incorporation on the impact strength of glyptal resin composites. The obtained findings confirmed that the set objectives were successfully achieved, and the purpose of this study was fulfilled. Evaluating the effects of filler concentration on the mechanical properties of the developed composites revealed that the addition of red mud greatly improves impact strength. Its maximum values were achieved at a filler concentration of 36 wt% and amounted to 22.05 N-m. Increasing the red mud concentration beyond this value led to a decrease in strength due to particle agglomeration, which created localised stresses in the matrix.

Comparing the methods of red mud incorporation into glyptal resin revealed that resins filled during *in situ* synthesis demonstrated superior mechanical properties compared to those obtained through mechanical mixing, providing a 12.5% increase in impact strength. This suggests a more uniform distribution of filler particles in the matrix and better interaction at the molecular level.

The duration of synthesis also significantly affected the completion of the process and, consequently, the improvement of the mechanical properties of the resulting coatings. It was established that maximum impact strength values were achieved at a synthesis duration of 480 min, particularly for composites with a low filler concentration (7 wt%). The obtained findings confirmed the prospects of using red mud as a reinforcing filler for polymer composites. The optimum combination of filler concentration (36 wt%) and the method of incorporation ensure the creation of materials with enhanced impact strength, opening new opportunities for their application in construction, electrical engineering, and other industrial sectors.

The practical significance of this study lies in addressing the environmental problem of red mud disposal, allowing for a reduction in industrial waste volumes and the creation of cost-effective materials with improved mechanical properties. Further research may focus on expanding the use of other industrial wastes as fillers, optimising the synthesis process to improve energy efficiency, and exploring the application possibilities of the developed materials in various industrial fields.

Acknowledgements

None.

Conflict of Interest

None.

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Підвищена ударна міцність композитів із гліфталевої смоли з червоним шламом: порівняльне дослідження методів введення

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Анотація. Метою цього дослідження було вивчити використання червоного шламу як екологічно безпечного наповнювача для композитів на основі гліфталевої смоли та методів його введення, а також оптимізувати властивості композитів і сприяти екологічно раціональному використанню матеріалів. Гліфталева смола була синтезована з гліцерину та фталевого ангідриду. Було застосовано два методи введення червоного шламу: механічне змішування та синтез *in situ*. Концентрація червоного шламу варіювалася від 7 до 56 мас. %. Ударну міцність отриманих композитів оцінювали за допомогою стандартизованих тестів. Дослідження показало, що червоний шлам значно підвищує ударну міцність композитів на основі гліфталевої смоли. Метод синтезу *in situ* забезпечив найкращі результати, досягаючи максимальної ударної міцності 22,05 Н·м за концентрації червоного шламу 36 мас. %, що на 12,5 % перевищувало значення для композитів, отриманих механічним змішуванням. Оптимальна концентрація наповнювача була визначена як 36 мас. %, тоді як при вищих концентраціях спостерігалось різке зниження міцності через агломерацію частинок. Крім того, ударна міцність збільшувалась зі зростанням тривалості синтезу, досягаючи свого максимуму на 480 хв для композитів із 7 мас. % червоного шламу. Це дослідження надало нову інформацію про інтеграцію червоного шламу як армуючого наповнювача в гліфталеві смоли та підкреслило переваги синтезу *in situ* у досягненні кращої дисперсії та взаємодії з матрицею, що сприяє розробці стабільних полімерних композитів. Результати демонструють можливість використання червоного шламу, складного промислового відходу, у виробництві високопродуктивних полімерних композитів. Дослідження пропонує практичні рекомендації щодо оптимізації концентрації наповнювачів і методів синтезу, що сприятиме розробці екологічно чистих і економічно вигідних матеріалів для промислового використання.

Ключові слова: полімерні композити; механічні властивості; екологічні матеріали; метод *in situ*; наповнювачі