



Performance of Metakaolin-Reinforced Unsaturated Polyester Resin in Metal Recovery from Wastewater and Potential of Wear Resistance

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Abstract

Polymer composites reinforced with clay have garnered substantial academic and industrial interest owing to their environmental and mechanical benefits. This research focuses on the effectiveness of metakaolin-unsaturated polyester composites for sequestering Ni²⁺ ions from Ni(NO₃)₂ aqueous solutions and on their tribological performance. The composite materials were synthesized with varying metakaolin concentrations (1, 1.5, 3, and 5 wt.%) to evaluate the influence of clay content on nickel ion recovery and wear resistance. The experimental results show a direct correlation between the reinforcement percentage and the functional properties of the composites. The highest value (61.415%) of Ni²⁺ recovery efficiency was achieved at a metakaolin concentration of 5 wt.%, which is due to the increased density of active adsorption sites on the composite surface. Conversely, composites with lower metakaolin content exhibited diminished recovery rates due to a limited number of available sites for ion adsorption. Wear resistance results show the same trend, wherein the composite with 5 wt.% metakaolin exhibited maximum wear resistance, characterized by minimal material loss at the applied loads (5, 10, 15, and 20 N). Furthermore, Shore-D hardness measurements indicated a gradual enhancement in the material's hardness with increasing metakaolin content, which confirms the reinforcing role of the clay in enhancing the mechanical properties. Scanning Electron Microscopy (SEM) micrographs showed the formation of wear grooves aligned with the sliding direction during wear testing. In addition, agglomerates and surface deposits were formed that are correlated with the adsorption of metal ions onto the composite surface. These results prove that the incorporation of metakaolin as a reinforcing agent confers dual advantages: it significantly enhances the adsorptive capacity for Ni²⁺ ion recovery and improves the tribological and mechanical properties of the composite. This positions the material for use in environmental remediation, especially wastewater treatment, and in engineering applications requiring enhanced durability.

Keywords: *Metakaolin Reinforcement, Ni²⁺ Ion Recovery, Polymer Composite, Shore-D Hardness, Wear Rate.*

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1. Introduction

Unsaturated polyester resins are utilized across various industrial applications due to their cost-effectiveness and advantageous mechanical and chemical properties. In addition, fillers and reinforcing agents are important to enhance specific characteristics of these resins, such as wear resistance, water absorption, and other mechanical behaviors [1-4]. Metakaolin, produced by calcining kaolin, is used as a filler in polymer composites due to its fine particle size makes it an effective additive for improving the overall properties of the composite material [5]. Wear is a characteristic that occurs on the surface of materials, particularly in applications involving motion between surfaces [6]. In a polymeric composite material, the wear mechanism is influenced by operational factors and environmental conditions [7-8]. Wear property was correlated with material recovery, where materials showing higher elastic recovery usually show higher wear resistance and result in a reduced amount of material loss during the wear process [9]. The recovery property of a material is a key factor in determining its wear behavior [10]. Recovery refers to a material's ability to regain its original shape and dimensions after the mechanical stress applied to it is removed. This ability stems from the material's capacity to store elastic energy temporarily during

deformation, which results from friction [11-13]. Recently, studies have been conducted on the wear behavior of polymer-metakaolin composites to improve material characteristics [14]. Bati et al. studied the kaolin ratio effect on wear rate, mechanical properties, water absorption, and acidic resistance of unsaturated polyester resins, using the pin-on-disk wear mechanism. Results show kaolin improves wear property [4]. Baghloul et al. evaluated the mechanical behaviour of a composite material consisting of unsaturated polyester reinforced by recycled marble waste. Results indicated that the reinforcement could contribute to improving wear resistance [15]. Xiao et al. investigated kaolin-reinforced natural rubber composites using a wet mixing method, in order to generate high wear resistance and low heat. Kaolin improvement in wear resistance 64%. The wet mixing process contributed to enhanced wear resistance compared to dry mixing [16]. Wang et al. utilized MOFs, COFs, metal sulfides, oxides, biomass, and polymers for the adsorption of Ag¹⁺ from wastewater, with results indicating an improvement in metal removal efficiency [17]. Mubarak et al. prepared porous activated metakaolinite-reinforced polyethylene nanocomposite membranes as adsorbents for the removal of metal ions from industrial wastewater. The composite showed high removal efficiencies for Cu²⁺, Cd²⁺, pb²⁺ ions were (82.5%, 69.3%,

and 76.2%), respectively. The results confirmed improved hydraulic resistance, porosity, and hydrophilicity [18]. Medri et al. used K-based metakaolin geopolymers to remove ammonium from wastewater, achieving higher recovery efficiency than natural zeolite in producing ($\text{NH}_4\text{NO}_3/\text{KNO}_3$) fertilizers [19]. Similarly, Wei et al. applied chemisorption and ion exchange with metakaolin-based geopolymer microspheres for F^- removal from wastewater, reaching an 89.18% removal ratio and demonstrating treatment potential for acidic wastewater [20]. Building on these findings, this research aims to study the wear behaviour of unsaturated polyester composites reinforced with varying metakaolin content, and their correlation with the recovery of Ni^{+2} from nickel nitrate $\text{Ni}(\text{NO}_3)_2$ solution, thereby expanding the understanding of metakaolin-based composites in environmental purification.

2. Materials and Experimental Methods

2.1. Materials

Methyl ethyl ketone peroxide (MEKP) was added at 2% to unsaturated polyester resin (UP), which was supplied by the Saudi Industrial Resins Company Ltd. (SIR). Kaolin clay, with a particle size smaller than $0.45 \mu\text{m}$, was supplied by the Iraqi Geological Survey. Nickel powder was obtained from Hopkin and Williams. Potassium chloride (KCl) and buffers were sourced from Reidel de Haene to reach pH 7, as measured by a pH meter. Nitric acid (HNO_3) was acquired from Central Drug House (CDH) company, and deionized water was provided by the College of Applied Sciences laboratories. To form amorphous metakaolin with high reactivity, 5 g of kaolin was placed in a laboratory oven at an optimized calcination temperature of 700°C for two hours [21].

2.2. Preparation of nickel nitrate $\text{Ni}(\text{NO}_3)_2$ Aqueous Solution

Prepared Solution standard Ni solution (1000ppm) by dissolution in a dilute HNO_3 (1:1). Added calculated volumes of Ni solution, preprepared, KCl (1M) with buffer by pipetting to pH was adjusted to 7. The final volume was then completed to 40ml. All these amounts were placed in a 50-ml container [22].

2.3 Manufacturing Process of Metakaolin-Enhanced Unsaturated Polyester Composites

Unsaturated polyester resin and 2% hardener were mixed to obtain a homogeneous mixture. Metakaolin (0, 1, 1.5, 3, 5 wt.%) was measured using a sensitive balance, then added to the mixture with continuous stirring by the hot plate stirrer for 30 min to obtain the composite. The unsaturated-metakaolin composite was dried for 24 hours in the lab. temperature.

2.4 Preparation of Experimental Specimens for Metal Recovery Test

Recovery test samples (length 10 mm, width 10mm, and thickness 3 mm) on ASTM D2240 were placed in a 15 ml $\text{Ni}(\text{NO}_3)_2$ solution. Stirred samples by an electric vibrator (30, 60, 90, and 120 min), after which the solutions were filtered with filter paper. A flame atomic absorption spectroscopy measured the Ni^{+2} ions concentration. Recovery % calculated from equation (1) [23]:

$$MR\% = \frac{(C_i - C_f) \times 100}{C_i} \quad \dots (1)$$

MR: metal recovery. C_i , C_f : initial and final Ni^{+2} concentration.

2.5 Preparation of Experimental Specimens for Wear Test

Composite material was cut according to ASTM (D695M-89, 08.01) into samples in a cylindrical shape (10mm diameter, 20mm length). The Pin-on-Disk method was used to measure the wear rate, with a disc rotational speed of 510 rpm. Hardness of disc 32 HV measured by Vickers macro hardness model Z323, while specimens' hardness measured in shore-D. Sample weight is one of the important factors to calculate the wear rate from equation (2) [24]:

$$\text{Wear Rate} = \frac{(W_{\text{initial}} - W_{\text{final}}) \times t}{W_{\text{initial}}} \quad \dots (2)$$

W_{initial} , W_{final} : weight (g) before and after wear process, respectively, t : time (min), test time is constant = 5 min.

3. Results and discussion

3.1. Recover of Ni^{+2} from neutral $\text{Ni}(\text{NO}_3)_2$ solution

Fig. 1 exhibits the recovery of nickel as a function of contact time for various concentrations of the metakaolin-reinforced unsaturated polyester adsorbent. The adsorbent concentration influences the nickel recovery %. At 5 wt.% adsorbent consistently the highest recovery rates exceeding 60%, and at 3 wt.% adsorbent which achieved recovery % in a range 53-58%. Lower concentrations, specifically 1.5 wt.% and 1 wt.%, exhibited lower recovery efficiencies, with the 1 wt.% concentration showing the least effective performance. A higher concentration of adsorbent provides a large number of available active sites for metal ion binding, leading to an enhancement of the total removal capacity [25]. A contact time effect varied depending on the adsorbent concentration. At higher concentrations (5 wt.% and 3 wt.%), Ni^{+2} recovery % increased and stabilized. However, a slight decrease in recovery was observed at 90 min for 3 wt.%, likely due to factors such as desorption processes and the saturation of active sites occurring at longer contact times. At (1.5 wt.% and 1 wt.%), the nickel recovery decreased with increasing contact time. At 1.5 wt.%, a peak appeared at 60 min, while 1 wt.% showed a continuous decrease, indicating that extended contact times are not beneficial for lower adsorbent concentrations due to the rapid saturation of the limited active sites [26]. In comparison with recent studies, metakaolin has gained much importance as a cost-effective and efficient adsorbent material for heavy metals removal [27]. Studied metakaolin-based geopolymers (MKG) for the adsorption of Ni^{+2} ions from aqueous solutions. Results indicated to increase in Ni^{+2} ions adsorbed with an increase in the contact time, needed to reach equilibrium, approximately 50 min [28]. Consistent with the present study, higher adsorbent concentrations (3 and 5 wt.%) yielded the best recovery percentage at 60-90 minutes. The slight difference in equilibrium time might be due to differences in the metakaolin modification, the preparation method of the geopolymer, or the presence of the polyester component in the composite.

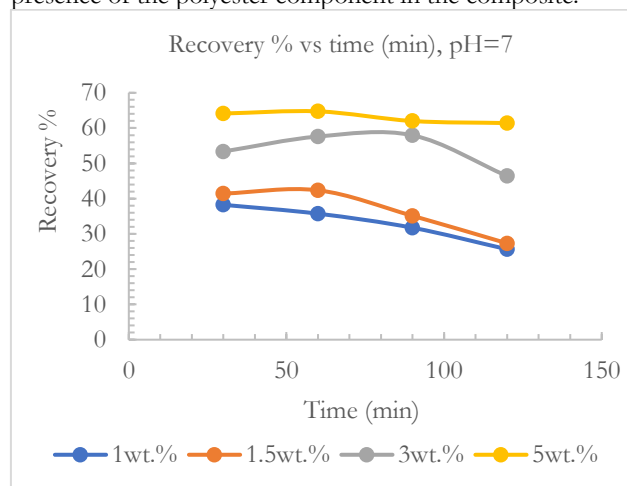


Figure 1. Recovery% of nickel as a function of contact time for various concentrations of the metakaolin reinforced unsaturated polyester adsorbent.

3.2 Analysis of Variance (ANOVA) and Comparison with Recent Studies

To validate the experimental results and study the influence of process parameters on the recovery of Ni^{+2} ions, a statistical analysis was performed on the results. The experimental design involved two independent variables: time (at four levels: 30, 60, 90,

and 120 minutes) and metakaolin concentrations (at four levels: 1.0, 1.5, 3.0, and 5.0 wt.%). A two-way Analysis of Variance (ANOVA) was the selected statistical method to evaluate the main effects of these parameters.

To examine the mean recovery percentages for each factor:

3.2.1 Effect of Concentration:

- 1wt. %: Average recovery is $(38.241 + 35.726 + 31.733 + 25.633) / 4 = 32.83$
- 1.5wt. %: Average recovery is $(41.426 + 42.361 + 35.12 + 27.317) / 4 = 36.56$
- 3wt. %: Average recovery is $(53.361 + 57.599 + 57.947 + 46.398) / 4 = 53.83$
- 5wt. %: Average recovery is $(64.126 + 64.731 + 61.997 + 61.415) / 4 = 63.07$

There is a clear, strong trend showing that as the concentration (wt.%) increases, the Ni+2ion recovery percentage also increases. This increase shows particularly significant between 1.5wt.% and 3wt.%.

3.2.2 Effect of Time:

- 30 min: Average recovery is $(38.241 + 41.426 + 53.361 + 64.126) / 4 = 49.29$
- 60 min: Average recovery is $(35.726 + 42.361 + 57.599 + 64.731) / 4 = 50.10$
- 90 min: Average recovery is $(31.733 + 35.12 + 57.947 + 61.997) / 4 = 46.70$
- 120 min: Average recovery is $(25.633 + 27.317 + 46.398 + 61.415) / 4 = 40.19$

The effect of time is not straightforward. Recovery seems to peak around 60 minutes and then decreases significantly at 90 and 120 minutes, especially at lower concentrations. This suggests that a shorter duration (around 30-60 minutes) might be more effective, or that after a certain point, the process becomes less efficient or reverses.

3.2.3 Interaction between Time and Concentration: The effect of time appears to depend on the concentration.

- At low concentrations (1wt.% and 1.5wt.%), recovery steadily decreases over time.
- At high concentrations (3wt.% and 5wt.%), recovery is more stable, peaking at 60-90 minutes before seeing a smaller drop. It indicates that the optimal time for the process changes depending on the concentration used.

3.2.4 Comparison with Recent Studies:

Recent studies are focus on removing Ni+2from wastewater using an adsorption process. These studies show that the removal efficiency increases with the initial concentration of Ni+2ions until the active sites on the adsorbent material become saturated [26,29].

3.3.Effect of load and metakaolin concentration on wear rate composite material

As observed from Fig. 2, a trend across all concentrations of composite is that the wear rate increases with the applied load, due to the higher contact pressure that results from sliding, leading to increased frictional forces [31]. In addition, dissipated energy through sliding wear contributes to localized heating and polymer matrix degradation [32]. Unsaturated polyester (UP) shows a high relative wear rate, particularly under increased applied loads, reflecting its wear behavior under operational conditions. At 1 wt.% and 1.5 wt.% metakaolin, wear rate curves are nearly that of unreinforced polymer, particularly at higher loads, where they show a slightly higher wear rate because at low loadings, metakaolin particles were not well-dispersed to form a network in the polymer matrix [33], leading to limited improvement in the wear rate. The composite reinforced with 3 wt.% metakaolin had the lowest wear

rate at all applied loads. The notable improvement in the tribological properties of unsaturated polyester is achieved by metakaolin. At this concentration, metakaolin particles are perfectly dispersed in the UP matrix, providing effective load transfer and acting as physical barriers to resist wear mechanisms. In addition, the fine particle size and hardness of metakaolin contributed to increasing the surface hardness of the composite [34]. Increasing the metakaolin concentration to 5 wt. This results in the lowest wear rate compared to the 3 wt.% composite, but it is still better than pure UP and the lower concentration composites. This rise in performance at higher filler helps to reduce the effective interfacial area between the filler and the matrix [35]. In comparison with recent studies, it was observed that the optimal concentration of metakaolin influences the metakaolin in enhancing the wear resistance of unsaturated polyester composites [4]. Investigated the effect of kaolin ratio on the wear of thermoset composites; results show that the specific wear rate decreased with an increasing reinforcement rate.

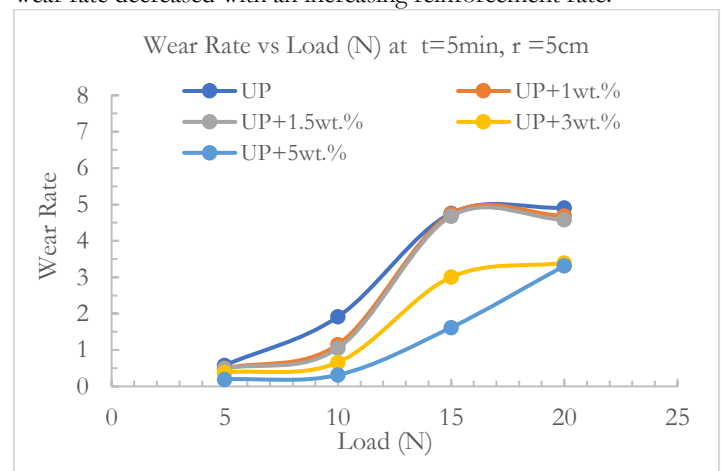


Figure 2. Wear rate of metakaolin- unsaturated polyester at time 5min, and radius of disk $r = 5\text{cm}$

3.4. Effect of various wt.% metakaolin on the hardness of the composite material

As observed from Fig. 3, the Shore-D hardness of the composites generally increases with increasing wt.% metakaolin. This is because metakaolin particles have a stiffer property than the polyester matrix. Their presence decreases the deformation of the polymer chains under stress [36]. Results show an increase in hardness between 3 wt.% and 5 wt.% metakaolin. In contrast, there are only smaller increases at lower concentrations. This greater increase occurs due to good interfacial adhesion between the filler particles and the polymer matrix [37]. The correlation coefficient between wt.% and Shore-D hardness is approximately 0.929, indicating a strong positive correlation. The highest hardness value, 71.4869, occurs at 5 wt.%. At this point, higher filler loadings achieve a more compact packing density, reducing the interface within the composite and further contributing to increased hardness [38].

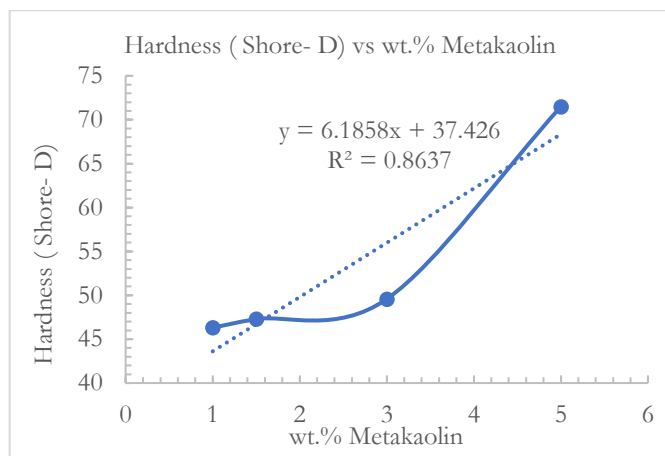


Figure 3. The influence of various wt.% metakaolin on the hardness of the composite material

3.5. Surface morphology (SEM) examine

3.5.1 Surface morphology after Ni+2 ion recovery

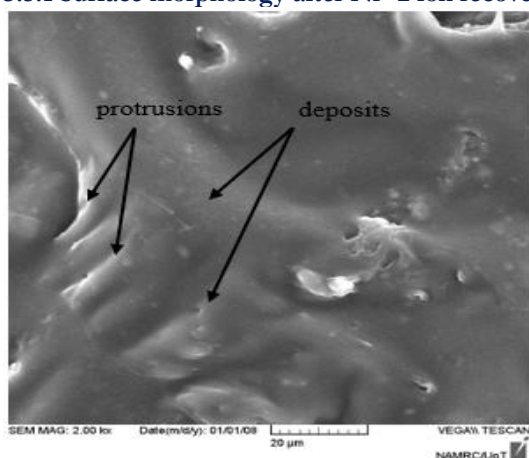


Figure 4. Surface morphology (SEM) of the adsorbent material at 5wt.% metakaolin after Ni+2 ion recovery

As shown in Fig. 4, the SEM micrograph reveals a relatively smooth surface morphology with localized deposits and irregular protrusions, which are indicative of Ni+2 ion recovery from the Ni (NO₃)₂ solution. The appearance of these agglomerates refers to the adsorption of metal ions. The presence of micro-clusters and patches indicates the presence of deposited metal on the adsorbent surface. The smooth regions between the deposits indicate that the adsorption process occurred at preferential active sites.

3.5.2. Surface morphology and wear direction

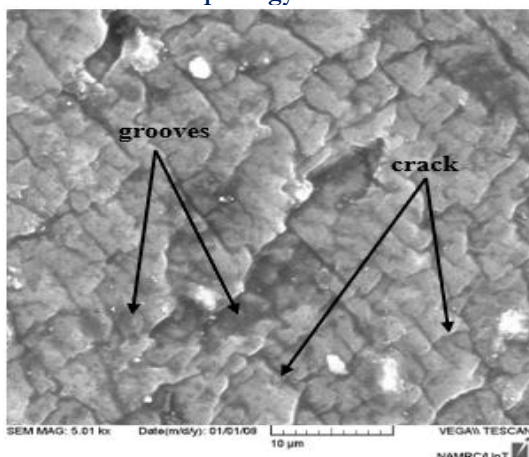


Figure 5. Surface morphology (SEM) and wear direction of the composite material at 5wt.% metakaolin

Observed clearly from Fig. 5, the SEM micrograph reveals a surface morphology characterized by irregular plates. The surface exhibits formation of cracks and grooves, which indicates that the nanocomposite material was subjected to a sliding wear process and material loss under the wear conditions. The elongated lineaments indicate the sliding path of abrasive particles during the wear process. This directional alignment confirms that the wear mechanism is not random but rather governed by the motion of particles across the surface, with the wear direction clearly determined by the alignment of grooves.

4. Conclusion

This study focuses on the metakaolin-reinforced unsaturated polyester composite and its efficacy in Ni²⁺ ion recovery from a Ni(NO₃)₂ solution and its wear resistance characteristics. The results show that the incorporation of metakaolin was found to substantially enhance the composite's performance. The highest Ni²⁺ recovery of 61.415% was consistently achieved at the maximum reinforcement ratio of 5 wt.% metakaolin. This reinforces the role of the clay content in providing and supporting active sites necessary for efficient metal ion adsorption. Similarly, the best results in both wear and hardness were attained at the same 5 wt.% metakaolin, where the highest values 3.3153 and 71.4869 were observed for wear rate and the hardness, respectively. Suggest significant potential for using this composite material in two distinct application areas :

- 1-Wastewater treatment by removing heavy metal ions from industrial effluents.
2. Applications requiring enhanced wear-resistant materials, such as in coatings and structural components subjected to friction and mechanical stress.

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6. References

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