



# Development and Performance Evaluation of a Silver Oxide–Titanium Dioxide–Nanoclay Composite Filter for the Treatment of Mining Wastewater

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## Abstract

Mining activities constitute one of the major contributors to environmental contamination worldwide due to the generation of wastewater containing elevated concentrations of heavy metals, suspended solids, toxic ions, and other persistent pollutants. In many developing countries, particularly those with extensive artisanal and small-scale mining operations, untreated mining effluents are frequently discharged into nearby water bodies, resulting in severe ecological degradation and potential public health risks. Conventional treatment technologies, including chemical precipitation, reverse osmosis, ion exchange, and activated carbon adsorption, have demonstrated varying degrees of effectiveness; however, their widespread application is often constrained by high operational costs, membrane fouling, excessive sludge production, and reduced efficiency in treating complex wastewater matrices. The present study investigated the development of a silver oxide–titanium dioxide–nanoclay composite filter as an innovative and sustainable material for mining wastewater remediation. The composite filter was designed by integrating the adsorption capabilities of nanoclay with the photocatalytic and antimicrobial properties of titanium dioxide (TiO<sub>2</sub>) and silver oxide (Ag<sub>2</sub>O) nanoparticles. Nanoclay was prepared through beneficiation, sedimentation, drying, and mechanical milling processes, while metal oxide nanoparticles were synthesized using environmentally benign approaches. The nanoparticles were subsequently incorporated into the clay matrix through wet impregnation and thermal sintering to produce a porous ceramic nanocomposite filter. Physicochemical characterization of the developed filter was performed using standard analytical techniques to determine its morphology, porosity, crystalline structure, and surface functional groups. Mining wastewater samples collected from active mining sites were analyzed before and after treatment to evaluate the removal efficiency of selected heavy metals and other physicochemical parameters. Results demonstrated substantial reductions in contaminant concentrations, accompanied by significant improvements in water clarity and overall quality. The enhanced performance of the composite filter was attributed to synergistic interactions among adsorption, photocatalytic oxidation, ion exchange, and surface complexation mechanisms. The findings indicate that silver oxide–titanium dioxide–nanoclay composite filters possess considerable potential as cost-effective and environmentally sustainable materials for the treatment of mining wastewater. The developed filtration system offers a promising alternative to conventional treatment technologies, particularly in resource-constrained regions where affordable and efficient water purification solutions are urgently required.

**Keywords:** Mining wastewater, nanocomposite filter, nanoclay, silver oxide, titanium dioxide, heavy metals, adsorption, photocatalysis, water treatment

## 1. INTRODUCTION

Water pollution arising from mining operations remains one of the most pressing environmental challenges confronting both developed and developing nations. The extraction and processing of mineral resources generate substantial volumes of wastewater

containing a complex mixture of dissolved metals, suspended solids, sulfates, nitrates, acids, and other potentially hazardous contaminants. These pollutants often find their way into surface water and groundwater systems through direct discharge, seepage, accidental



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spillages, or inadequate wastewater management practices. Consequently, mining wastewater has become a significant source of environmental degradation, threatening aquatic ecosystems, agricultural productivity, and human health (Akcil & Koldas, 2006).

The environmental implications of mining wastewater are particularly severe in developing countries where regulatory enforcement and wastewater treatment infrastructure remain inadequate. In Nigeria, artisanal and small-scale mining activities have expanded considerably during the last two decades owing to increasing demand for mineral resources and economic opportunities. Despite their economic significance, many of these operations discharge untreated or poorly treated wastewater into nearby rivers, streams, and agricultural lands. Such practices contribute to the accumulation of toxic heavy metals in environmental compartments and increase the risk of contamination of drinking water sources.

Heavy metals are among the most problematic contaminants associated with mining effluents because of their persistence, toxicity, and tendency to bioaccumulate within biological systems. Unlike organic pollutants, heavy metals cannot be degraded into harmless products and therefore remain in the environment for extended periods. Metals such as lead (Pb), copper (Cu), manganese (Mn), iron (Fe), cadmium (Cd), arsenic (As), chromium (Cr), and mercury (Hg) have been reported in mining wastewater at concentrations exceeding permissible regulatory limits (Tchounwou et al., 2012). Exposure to these contaminants has been linked to neurological disorders, renal dysfunction, developmental abnormalities, carcinogenic effects, and ecosystem disruption.

Various technologies have been developed to address heavy metal contamination in wastewater. Conventional approaches such as chemical precipitation, coagulation-flocculation, ion exchange, membrane filtration, electrochemical treatment, and activated carbon adsorption have demonstrated effectiveness under specific operating conditions (Fu & Wang, 2011). Nevertheless, many of these technologies suffer from limitations that hinder their widespread adoption. Chemical precipitation generates large quantities of sludge requiring further treatment and disposal. Membrane-based systems, including reverse osmosis, are associated with high energy consumption and membrane fouling. Similarly, commercial adsorbents such as activated carbon often involve significant operational costs, making them economically impractical

for large-scale implementation in resource-limited settings.

Recent advances in nanotechnology have opened new opportunities for the development of highly efficient materials for environmental remediation. Nanomaterials possess unique physicochemical properties resulting from their exceptionally small particle size, high surface-area-to-volume ratio, enhanced surface reactivity, and tunable structural characteristics. These attributes enable superior adsorption and catalytic performance compared with many conventional materials (Qu et al., 2013). Consequently, nanotechnology-based treatment systems have attracted substantial attention for applications involving heavy metal removal, disinfection, organic pollutant degradation, and wastewater purification.

Among the various nanomaterials investigated for water treatment applications, nanoclay has emerged as a particularly attractive candidate due to its abundance, low cost, environmental compatibility, and excellent adsorption capacity. Clay minerals possess layered aluminosilicate structures characterized by high cation exchange capacities and large specific surface areas, making them effective adsorbents for metal ions and organic contaminants. Furthermore, the modification of clay materials with functional nanoparticles has been shown to significantly enhance their treatment performance by introducing additional reactive sites and catalytic functionalities (Abukhadra et al., 2019).

Titanium dioxide (TiO<sub>2</sub>) nanoparticles represent one of the most extensively studied photocatalytic materials in environmental engineering. Their popularity stems from their chemical stability, non-toxicity, corrosion resistance, and strong oxidative capability under ultraviolet irradiation. Upon activation, TiO<sub>2</sub> generates reactive oxygen species capable of degrading organic pollutants and facilitating the oxidation of various contaminants (Hashim et al., 2011). However, TiO<sub>2</sub>-based systems often exhibit limited antimicrobial properties and may experience rapid electron-hole recombination, which can reduce photocatalytic efficiency.

To overcome these limitations, researchers have increasingly explored the incorporation of silver-based nanomaterials into TiO<sub>2</sub> systems. Silver oxide nanoparticles exhibit notable antimicrobial properties and can enhance photocatalytic activity through improved charge separation mechanisms. The synergistic interaction between silver oxide and titanium dioxide has been reported to improve contaminant degradation, microbial inactivation, and overall treatment performance in aqueous systems (Rai et al., 2009).



The integration of silver oxide nanoparticles and titanium dioxide nanoparticles within a nanoclay matrix presents a promising strategy for developing multifunctional filtration materials capable of simultaneously removing heavy metals, reducing microbial contamination, and improving overall water quality. The porous structure of clay facilitates water permeability and adsorption, while the incorporated nanoparticles provide catalytic and antimicrobial functionalities. Despite growing interest in nanocomposite materials for water treatment applications, limited studies have investigated the development and application of silver oxide–titanium dioxide–nanoclay composite filters specifically for mining wastewater treatment.

Therefore, this study aims to develop and evaluate a silver oxide–titanium dioxide–nanoclay composite filter for the treatment of mining wastewater. The study focuses on the synthesis and characterization of the composite material, assessment of its physicochemical properties, and evaluation of its effectiveness in removing heavy metals and other contaminants from mining effluents. The outcomes are expected to contribute to the development of affordable, efficient, and environmentally sustainable technologies for wastewater remediation, particularly in regions affected by artisanal and small-scale mining activities.

## 2. LITERATURE REVIEW

### 2.1 Mining Wastewater Characteristics and Environmental Implications

Mining wastewater is a complex effluent generated during mineral extraction, ore processing, washing, beneficiation, and tailings management operations. Its composition varies according to the type of mineral being extracted, geological conditions, extraction techniques employed, and wastewater management practices adopted at mining sites. Common contaminants found in mining wastewater include dissolved heavy metals, suspended solids, sulfates, chlorides, nitrates, acids, and residual processing chemicals (Akcil & Koldas, 2006).

One of the major environmental concerns associated with mining wastewater is the presence of potentially toxic elements. Heavy metals such as iron, lead, copper, cadmium, chromium, manganese, arsenic, and mercury frequently occur in elevated concentrations in mining effluents. These contaminants are of particular concern because they are persistent, non-biodegradable, and capable of accumulating within food chains through

bioaccumulation and biomagnification processes (Tchounwou et al., 2012).

When released into aquatic ecosystems without adequate treatment, mining wastewater can alter water chemistry, reduce dissolved oxygen levels, increase turbidity, and adversely affect aquatic organisms. Long-term exposure to heavy metals has been associated with reduced biodiversity, physiological stress in aquatic species, and disruption of ecosystem functions. Furthermore, contamination of groundwater and surface water resources poses significant public health challenges, especially in communities dependent on untreated water sources for domestic and agricultural purposes.

Acid mine drainage (AMD) represents another major challenge associated with mining operations. AMD occurs when sulfide minerals, particularly pyrite ( $\text{FeS}_2$ ), react with oxygen and water, producing sulfuric acid and mobilizing toxic metals into surrounding water bodies. The resulting acidic conditions accelerate metal dissolution and increase contaminant mobility, thereby intensifying environmental risks (Akcil & Koldas, 2006).

The growing environmental burden associated with mining wastewater has intensified the search for effective, affordable, and sustainable treatment technologies capable of addressing multiple contaminants simultaneously.

### 2.2 Conventional Technologies for Mining Wastewater Treatment

Numerous technologies have been developed for the treatment of mining wastewater. These technologies can generally be classified into physical, chemical, biological, and hybrid treatment systems.

Chemical precipitation remains one of the most widely applied methods for heavy metal removal due to its simplicity and relatively low initial cost. The process involves converting dissolved metals into insoluble hydroxides, sulfides, or carbonates through the addition of chemical reagents. Although effective under controlled conditions, precipitation often generates substantial quantities of sludge that require additional handling and disposal (Fu & Wang, 2011).

Ion exchange technology has also been employed for the selective removal of dissolved metal ions. The process relies on synthetic resins capable of exchanging undesirable ions with less harmful ions present on the resin surface. Despite its effectiveness, ion exchange systems are often expensive and sensitive to wastewater composition, limiting their application in large-scale mining operations.



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Membrane-based technologies, including nanofiltration, ultrafiltration, and reverse osmosis, have demonstrated high removal efficiencies for dissolved contaminants. Reverse osmosis, in particular, can achieve near-complete removal of dissolved solids and heavy metals. However, membrane fouling, high energy requirements, and significant operational costs remain major barriers to widespread adoption (Hashim et al., 2011).

Adsorption has emerged as one of the most versatile treatment approaches due to its simplicity, effectiveness, and adaptability. Activated carbon is the most commonly used adsorbent; however, its relatively high cost has motivated the exploration of alternative low-cost adsorbents such as agricultural wastes, biochar, zeolites, and clay minerals. Recent studies have demonstrated that modified clay materials can exhibit adsorption capacities comparable to those of conventional commercial adsorbents under appropriate conditions (Crini & Lichtfouse, 2019).

Although these conventional technologies have achieved varying degrees of success, many suffer from economic, operational, and environmental limitations. Consequently, researchers have increasingly focused on advanced materials capable of enhancing treatment performance while reducing operational costs.

#### 2.3 Nanotechnology Applications in Wastewater Treatment

Nanotechnology has emerged as a transformative field in environmental remediation due to the unique properties exhibited by materials at the nanoscale. Nanomaterials possess exceptionally large specific surface areas, enhanced surface reactivity, tunable pore structures, and unique catalytic characteristics that significantly improve contaminant removal processes (Qu et al., 2013).

The application of nanotechnology in wastewater treatment has expanded rapidly over the past two decades. Nanomaterials have been investigated for adsorption, photocatalysis, membrane enhancement, disinfection, and contaminant sensing applications. Their ability to interact effectively with pollutants at the molecular level provides opportunities for developing highly efficient treatment systems capable of addressing complex wastewater streams.

Several categories of nanomaterials have been evaluated for environmental applications, including metal

oxides, carbon-based nanomaterials, magnetic nanoparticles, nanocomposites, and clay-based nanostructures. Among these materials, metal oxide nanoparticles have attracted considerable attention due to their chemical stability, catalytic activity, and environmental compatibility.

The integration of nanomaterials into filtration systems has been shown to improve contaminant removal through multiple mechanisms, including adsorption, photocatalytic degradation, redox reactions, and antimicrobial activity. These multifunctional properties make nanotechnology particularly attractive for mining wastewater treatment, where multiple contaminants often coexist.

#### 2.4 Nanoclay as an Adsorbent Material

Nanoclay refers to clay minerals processed into nanoscale particles or modified to exhibit nanoscale properties. The most common clay minerals used in environmental applications include montmorillonite, kaolinite, bentonite, and halloysite. These materials possess layered aluminosilicate structures characterized by large surface areas and high cation exchange capacities.

The adsorption capacity of nanoclay is primarily attributed to its negatively charged surface, which facilitates the attraction and retention of positively charged metal ions. In addition, hydroxyl groups located on clay surfaces contribute to metal binding through complexation reactions. These properties enable nanoclays to effectively remove various heavy metals from contaminated water (Abukhadra et al., 2019).

Several studies have reported successful removal of lead, cadmium, copper, chromium, and zinc ions using modified clay adsorbents. Researchers have also demonstrated that clay modification through acid activation, polymer incorporation, or nanoparticle impregnation can substantially enhance adsorption performance.

The abundance and low cost of clay deposits in many developing countries further increase their attractiveness as sustainable treatment materials. Nevertheless, clay-based adsorbents may suffer from limitations such as reduced mechanical strength and limited antimicrobial activity. These shortcomings have motivated the development of clay-based nanocomposites incorporating functional nanoparticles.



## 2.5 Titanium Dioxide Nanoparticles in Water Treatment

Titanium dioxide ( $\text{TiO}_2$ ) is one of the most extensively studied photocatalytic materials in environmental engineering. Since the pioneering work of Fujishima and Honda (1972),  $\text{TiO}_2$  has been recognized for its ability to induce photocatalytic reactions under ultraviolet irradiation.

The photocatalytic mechanism of  $\text{TiO}_2$  involves the excitation of electrons from the valence band to the conduction band upon absorption of photons with sufficient energy. This process generates electron-hole pairs capable of producing highly reactive oxygen species such as hydroxyl radicals and superoxide radicals. These species participate in oxidation-reduction reactions that facilitate contaminant degradation and microbial inactivation.

$\text{TiO}_2$  possesses several advantages that make it attractive for wastewater treatment applications. These include chemical stability, low toxicity, corrosion resistance, environmental compatibility, and long-term durability. Numerous studies have demonstrated its effectiveness in degrading organic pollutants, reducing microbial contamination, and facilitating metal removal from aqueous systems (Hashim et al., 2011).

Despite these advantages,  $\text{TiO}_2$  exhibits certain limitations, including rapid recombination of photogenerated charge carriers and limited effectiveness under visible-light conditions. Consequently, researchers have explored strategies for improving  $\text{TiO}_2$  performance through doping and composite formation.

## 2.6 Silver Oxide Nanoparticles and Their Environmental Applications

Silver-based nanomaterials have received significant attention due to their strong antimicrobial properties and catalytic activity. Silver oxide nanoparticles exhibit broad-spectrum activity against bacteria, fungi, and viruses through mechanisms involving membrane disruption, oxidative stress induction, and interference with cellular metabolism (Rai et al., 2009).

Beyond antimicrobial applications, silver oxide nanoparticles have demonstrated potential in environmental remediation. Their incorporation into filtration systems can improve contaminant removal efficiency by enhancing adsorption processes and facilitating catalytic reactions.

Several studies have reported that combining silver-based nanoparticles with  $\text{TiO}_2$  enhances photocatalytic efficiency by reducing electron-hole recombination. The resulting synergistic effects contribute to improved contaminant degradation and increased treatment performance.

The integration of silver oxide into clay-based filtration systems may therefore provide dual functionality through simultaneous contaminant removal and microbial control.

## 2.7 Nanocomposite Filters for Wastewater Treatment

Recent advances in materials science have facilitated the development of multifunctional nanocomposite filters capable of addressing complex wastewater treatment challenges. Nanocomposites combine the desirable properties of multiple materials into a single structure, resulting in enhanced mechanical strength, adsorption capacity, catalytic activity, and durability.

Clay-based nanocomposites incorporating metal oxide nanoparticles have shown considerable promise in environmental applications. The clay matrix provides structural support and adsorption sites, while metal oxide nanoparticles contribute photocatalytic and antimicrobial functionalities. Such synergistic interactions often produce superior treatment performance compared with individual components used independently.

Despite the increasing number of studies on nanocomposite materials, research specifically focusing on silver oxide–titanium dioxide–nanoclay composite filters for mining wastewater treatment remains limited. Most existing investigations have focused on municipal wastewater, dye-contaminated water, or laboratory-prepared synthetic solutions. Consequently, further investigation is required to evaluate the performance of these materials under actual mining wastewater conditions.

## 2.8 Research Gap

A review of existing literature reveals substantial progress in the application of nanomaterials for wastewater treatment. However, significant knowledge gaps remain regarding the combined utilization of silver



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oxide, titanium dioxide, and nanoclay within a single filtration system for mining wastewater remediation.

Previous studies have primarily examined individual components or binary composites, with limited attention given to multifunctional ceramic nanocomposites designed specifically for heavy metal removal from mining effluents. Furthermore, there is insufficient information regarding the synergistic interactions among adsorption, photocatalysis, ion exchange, and antimicrobial mechanisms within such systems.

Therefore, the present study seeks to address these gaps by developing and evaluating a silver oxide–titanium dioxide–nanoclay composite filter and assessing its effectiveness in treating real mining wastewater. The study contributes to the growing body of knowledge on sustainable nanotechnology-based solutions for environmental remediation.

### 3. MATERIALS AND METHODS

#### 3.1 Study Area and Wastewater Sampling

Mining wastewater samples used in this study were collected from an active artisanal mining site characterized by intensive mineral extraction and ore-processing activities. The site was selected based on evidence of wastewater discharge into surrounding water bodies and the presence of visible contamination associated with mining operations. Sampling was conducted in accordance with standard environmental monitoring procedures to ensure the collection of representative wastewater samples.

Wastewater samples were collected in pre-cleaned high-density polyethylene (HDPE) containers previously rinsed with deionized water and sample water. The containers were filled completely to minimize oxidation reactions during transportation. Samples intended for heavy metal analysis were preserved immediately with concentrated nitric acid to maintain a pH below 2, following standard procedures recommended by the American Public Health Association (APHA, 2017). All samples were transported to the laboratory in ice-packed containers and stored at 4°C prior to analysis.

#### 3.2 Materials and Reagents

The materials used for the preparation of the nanocomposite filter included raw clay, silver nitrate

(AgNO<sub>3</sub>), titanium precursor compounds, distilled water, sodium hydroxide, nitric acid, and other analytical-grade reagents. All chemicals used in the study were of analytical grade and were used without further purification.

The clay utilized in this study was sourced locally and subjected to beneficiation processes to remove impurities and improve its suitability for filter fabrication. The selection of clay was based on its abundance, low cost, environmental compatibility, and known adsorption characteristics.

#### 3.3 Preparation of Nanoclay

The raw clay was initially air-dried for seven days to remove excess moisture. The dried clay was manually crushed and sieved through a 2 mm mesh to remove coarse particles and foreign materials. Beneficiation was performed by dispersing the clay in distilled water and allowing sedimentation to occur. The finer clay fraction was separated from larger particles and collected for further processing.

The beneficiated clay was oven-dried at 105°C for 24 h and subsequently pulverized using a laboratory ball mill. Milling was continued until nanoscale particle sizes were achieved. The resulting nanoclay powder was stored in airtight containers to prevent moisture absorption and contamination.

The reduction of particle size was expected to increase surface area, enhance adsorption capacity, and improve interaction between the clay matrix and incorporated nanoparticles.

#### 3.4 Synthesis of Silver Oxide Nanoparticles

Silver oxide nanoparticles were synthesized using a green synthesis approach to minimize the use of hazardous chemicals and reduce environmental impacts associated with conventional nanoparticle production techniques.

An aqueous silver nitrate solution was prepared and mixed with a plant extract serving as a reducing and stabilizing agent. The mixture was stirred continuously under controlled conditions until a visible color change indicated nanoparticle formation. The synthesized nanoparticles were separated through centrifugation and repeatedly washed with distilled water to remove residual impurities.



The resulting material was dried in an oven and subsequently calcined to obtain silver oxide nanoparticles suitable for incorporation into the nanocomposite filter.

The adoption of green synthesis methods aligns with increasing global efforts toward environmentally sustainable nanomaterial production and reduces the generation of toxic by-products commonly associated with chemical synthesis routes.

### 3.5 Synthesis of Titanium Dioxide Nanoparticles

Titanium dioxide nanoparticles were prepared using a controlled hydrolysis process involving titanium precursor compounds. The precursor solution was prepared under continuous stirring, followed by hydrolysis and condensation reactions leading to nanoparticle formation.

The resulting suspension was aged under controlled conditions to facilitate crystal growth and improve particle uniformity. The precipitated nanoparticles were recovered through filtration and repeatedly washed to remove residual reactants.

Subsequently, the nanoparticles were dried and calcined at elevated temperatures to obtain crystalline TiO<sub>2</sub> nanoparticles with enhanced photocatalytic properties.

Titanium dioxide was selected due to its demonstrated photocatalytic efficiency, chemical stability, non-toxicity, and extensive use in environmental remediation applications.

### 3.6 Fabrication of Silver Oxide–Titanium Dioxide–Nanoclay Composite Filter

The nanocomposite filter was prepared through a wet impregnation technique. Appropriate quantities of silver oxide nanoparticles and titanium dioxide nanoparticles were dispersed in distilled water and mixed thoroughly to achieve uniform distribution.

The nanoparticle suspension was gradually introduced into the prepared nanoclay under continuous stirring. The resulting mixture was homogenized until a consistent paste was obtained.

The composite paste was molded into filter shapes and allowed to air-dry for several days. After drying, the filters were subjected to thermal treatment in a gas kiln at 850°C for 2 h to improve mechanical strength, structural stability, and pore formation.

The sintering process facilitated the development of a reticulated porous structure essential for water permeability and contaminant retention. The finished filters were cooled gradually and stored under dry conditions before characterization and performance evaluation.

### 3.7 Characterization of the Nanocomposite Filter

Comprehensive characterization was conducted to determine the structural, morphological, and physicochemical properties of the developed filter.

#### 3.7.1 X-Ray Diffraction (XRD)

X-ray diffraction analysis was performed to identify crystalline phases present in the nanocomposite and confirm successful incorporation of silver oxide and titanium dioxide nanoparticles within the clay matrix.

Diffraction patterns were recorded over an appropriate scanning range and compared with standard reference databases to determine mineralogical composition and crystallinity.

#### 3.7.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy was employed to investigate the surface morphology and pore structure of the fabricated filter. SEM images provided information regarding particle distribution, pore connectivity, surface roughness, and nanoparticle dispersion within the clay matrix.

#### 3.7.3 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was conducted to identify surface functional groups responsible for adsorption and contaminant interactions. Spectra were recorded over the relevant wavenumber range to detect hydroxyl groups, metal-oxygen bonds, silicate structures, and other functional moieties.

#### 3.7.4 Porosity and Water Absorption Analysis

Porosity measurements were performed to evaluate the suitability of the filter for wastewater treatment applications. Water absorption tests were conducted



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according to established ceramic testing procedures. The porosity of the filter was calculated using Equation (1):

$$P = [(W_2 - W_1)/V\rho] \times 100$$

where:

P = Porosity (%)

$W_1$  = Dry weight of filter (g)

$W_2$  = Saturated weight of filter (g)

V = Volume of filter ( $\text{cm}^3$ )

$\rho$  = Density of water ( $\text{g}/\text{cm}^3$ )

High porosity values were expected to facilitate water flow and increase contact between contaminants and active adsorption sites.

### 3.8 Physicochemical Analysis of Mining Wastewater

The collected wastewater samples were analyzed before and after treatment using standard methods described by APHA (2017).

Parameters analyzed included:

- pH
- Electrical conductivity
- Total dissolved solids (TDS)
- Turbidity
- Color
- Dissolved oxygen
- Heavy metal concentrations

The analyses provided baseline information regarding wastewater quality and enabled assessment of treatment effectiveness.

### 3.9 Heavy Metal Determination

Heavy metal concentrations were determined using Atomic Absorption Spectrophotometry (AAS).

The metals investigated included:

- Iron (Fe)
- Copper (Cu)
- Lead (Pb)
- Manganese (Mn)
- Zinc (Zn)
- Cadmium (Cd)

Appropriate calibration standards were prepared, and instrument calibration was performed before analysis.

The concentrations obtained were compared with national and international regulatory standards to

evaluate environmental risks associated with the untreated wastewater.

### 3.10 Filtration Experiment

Performance evaluation experiments were conducted using a laboratory-scale filtration system. A predetermined volume of mining wastewater was passed through the fabricated nanocomposite filter under controlled operating conditions. Samples were collected at designated time intervals to monitor changes in contaminant concentrations.

The treated wastewater was subsequently analyzed using the same procedures employed for the untreated samples.

The effectiveness of the filtration process was assessed based on reductions in heavy metal concentrations and improvements in physicochemical water quality parameters.

#### 3.11 Determination of Removal Efficiency

Removal efficiency was calculated using Equation (2):

$$\text{Removal Efficiency (\%)} = [(C_0 - C_e)/C_0] \times 100$$

where:

$C_0$  = Initial contaminant concentration (mg/L)

$C_e$  = Final contaminant concentration after treatment (mg/L)

The calculated values were used to compare the removal performance of the developed filter for different contaminants.

### 3.12 Statistical Analysis

Experimental analyses were performed in triplicate to ensure reliability and reproducibility of results. Data obtained were expressed as mean  $\pm$  standard deviation.

Statistical analyses were conducted using appropriate software packages. Analysis of variance (ANOVA) was employed to determine significant differences between treatment conditions at a significance level of  $p < 0.05$ .

Correlation analyses were also performed to evaluate relationships among treatment parameters and contaminant removal efficiencies.

#### 3.13 Mechanisms of Contaminant Removal

The removal of contaminants by the silver oxide–



titanium dioxide–nanoclay composite filter was expected to occur through multiple mechanisms operating simultaneously.

These mechanisms include:

1. Adsorption onto nanoclay surfaces through electrostatic attraction and surface complexation.
2. Ion exchange between dissolved metal ions and exchangeable ions within the clay structure.
3. Photocatalytic oxidation facilitated by titanium dioxide nanoparticles.
4. Catalytic enhancement resulting from silver oxide incorporation.
5. Physical entrapment of suspended particles within the porous filter network.

## 4. RESULTS AND DISCUSSION

### 4.1 Physicochemical Characteristics of the Developed Silver Oxide–Titanium Dioxide–Nanoclay Composite Filter

The successful fabrication of the silver oxide–titanium dioxide–nanoclay composite filter was confirmed through comprehensive physicochemical characterization. The characterization results revealed that the incorporation of silver oxide ( $\text{Ag}_2\text{O}$ ) and titanium dioxide ( $\text{TiO}_2$ ) nanoparticles into the clay matrix significantly modified the structural and surface properties of the filter, thereby enhancing its suitability for wastewater treatment applications.

The nanocomposite exhibited a mechanically stable porous structure with adequate water permeability and structural integrity. The sintering process at  $850^\circ\text{C}$  promoted particle bonding and pore development without causing excessive densification of the ceramic matrix. The resulting filter structure was characterized by interconnected pores that facilitated water flow while providing sufficient surface area for contaminant adsorption.

These observations are consistent with previous studies indicating that clay-based ceramic filters possess desirable hydraulic and adsorption properties when appropriately modified with functional nanomaterials (Abukhadra et al., 2019).

### 4.2 X-Ray Diffraction Analysis

The X-ray diffraction (XRD) patterns of the fabricated

nanocomposite filter confirmed the presence of crystalline phases associated with clay minerals, titanium dioxide, and silver oxide nanoparticles.

Characteristic diffraction peaks corresponding to  $\text{TiO}_2$  were observed, indicating successful incorporation of titanium dioxide within the clay matrix. Peaks attributable to silver oxide were also identified, confirming the retention of  $\text{Ag}_2\text{O}$  during thermal processing.

The clay component exhibited diffraction patterns typical of aluminosilicate minerals commonly found in natural clay deposits. The persistence of these peaks after nanoparticle incorporation demonstrated that the structural integrity of the clay matrix was preserved during fabrication.

The observed crystalline phases are important because crystal structure influences adsorption behavior, photocatalytic activity, and overall treatment efficiency. High crystallinity of  $\text{TiO}_2$  is particularly desirable because photocatalytic performance is strongly dependent on crystal phase composition and structural order (Hashim et al., 2011).

The XRD results therefore confirmed the successful synthesis of a multifunctional composite material containing active adsorption and catalytic components.

### 4.3 Surface Morphology and Structural Characteristics

Scanning Electron Microscopy (SEM) analysis revealed significant morphological modifications resulting from nanoparticle incorporation.

The micrographs showed a highly porous surface characterized by interconnected pore networks and irregular surface topography. Numerous cavities and channels were observed throughout the filter matrix, providing pathways for water movement and contaminant interaction.

The dispersion of silver oxide and titanium dioxide nanoparticles on the clay surface appeared relatively uniform, indicating effective distribution during the wet impregnation process. The nanoparticles occupied both external surfaces and internal pore regions, thereby increasing the number of available active sites for contaminant removal.

The rough surface texture observed in the SEM images is advantageous for adsorption processes because it increases surface area and enhances the probability of contaminant–surface interactions.



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Similar observations have been reported in previous investigations involving clay-based nanocomposite adsorbents, where increased surface roughness and porosity were associated with improved adsorption performance (Qu et al., 2013).

#### 4.4 Fourier Transform Infrared Spectroscopy Analysis

FTIR spectra provided important information regarding the functional groups present on the surface of the nanocomposite filter.

Broad absorption bands associated with hydroxyl (-OH) groups were observed, indicating the presence of surface-bound water molecules and hydroxyl functionalities capable of participating in adsorption reactions.

Absorption bands corresponding to Si-O-Si and Al-O-Si vibrations confirmed the presence of aluminosilicate structures characteristic of clay minerals. Additional bands associated with Ti-O and Ag-O bonds verified the successful incorporation of titanium dioxide and silver oxide nanoparticles.

The presence of hydroxyl groups is particularly important because these groups serve as active sites for metal adsorption through surface complexation mechanisms. The abundance of oxygen-containing functional groups also contributes to electrostatic interactions between the filter surface and dissolved metal ions.

The FTIR results suggest that the developed nanocomposite possesses a diverse range of surface functionalities capable of supporting multiple contaminant removal mechanisms.

#### 4.5 Physicochemical Characteristics of Untreated Mining Wastewater

Analysis of the untreated mining wastewater revealed significant contamination levels consistent with wastewater generated from mineral extraction activities.

The wastewater exhibited dark coloration, objectionable odor, elevated turbidity, and high concentrations of dissolved solids. These characteristics are commonly associated with mining effluents containing suspended mineral particles, dissolved metals, and residual processing chemicals.

The pH values indicated deviation from neutral conditions, suggesting the influence of mineral dissolution and possible acid-generating reactions within the mining environment. Elevated conductivity values reflected the presence of substantial concentrations of dissolved ionic species.

Heavy metal analysis revealed detectable concentrations of iron (Fe), copper (Cu), lead (Pb), manganese (Mn), and other metallic contaminants. In several cases, measured concentrations exceeded recommended environmental discharge limits established by regulatory agencies.

These findings support previous reports indicating that mining wastewater constitutes a major source of environmental contamination when discharged without adequate treatment (Akcil & Koldas, 2006).

#### 4.6 Heavy Metal Removal Performance

One of the primary objectives of the present study was to evaluate the effectiveness of the developed nanocomposite filter in removing heavy metals from mining wastewater.

The results demonstrated substantial reductions in metal concentrations following filtration treatment. The removal efficiencies varied among individual metals, reflecting differences in ionic properties, adsorption affinities, and interaction mechanisms.

Iron ( $\text{Fe}^{3+}$ ) exhibited the highest removal efficiency and was completely removed within the treatment period. The rapid removal of iron may be attributed to its strong affinity for clay adsorption sites and its tendency to form surface complexes with hydroxyl groups present on the nanocomposite surface.

Copper ( $\text{Cu}^{2+}$ ) and lead ( $\text{Pb}^{2+}$ ) also showed exceptionally high removal efficiencies. The observed behavior may be explained by electrostatic attraction, ion exchange processes, and complexation reactions occurring at active surface sites.

Manganese ( $\text{Mn}^{2+}$ ) removal was similarly effective, although slightly lower than that observed for iron, copper, and lead. The lower removal efficiency may reflect differences in ionic radius, hydration energy, and adsorption kinetics.

Overall, the developed nanocomposite filter demonstrated excellent capability for removing heavy metals from mining wastewater.

The observed performance compares favorably with



previously reported values for clay-based adsorbents and many conventional treatment systems (Fu & Wang, 2011).

#### **4.7 Mechanisms Responsible for Heavy Metal Removal**

The superior treatment performance of the developed filter can be attributed to the simultaneous operation of several contaminant removal mechanisms.

##### **4.7.1 Adsorption**

Adsorption represents one of the primary mechanisms responsible for heavy metal removal. The large surface area of the nanoclay component provided abundant adsorption sites capable of attracting dissolved metal ions.

Electrostatic interactions between negatively charged clay surfaces and positively charged metal ions facilitated contaminant retention.

##### **4.7.2 Ion Exchange**

Clay minerals possess exchangeable cations located within their interlayer regions. These cations can be replaced by dissolved heavy metal ions through ion exchange reactions. The ion exchange capacity of the clay component therefore contributed significantly to overall contaminant removal efficiency.

##### **4.7.3 Surface Complexation**

Hydroxyl groups present on the surfaces of clay, titanium dioxide, and silver oxide nanoparticles provided binding sites for metal ions. These interactions resulted in the formation of stable surface complexes that enhanced contaminant immobilization.

##### **4.7.4 Photocatalytic Activity**

Titanium dioxide nanoparticles introduced

photocatalytic functionality into the filtration system. Although the primary focus of the study was heavy metal removal,  $\text{TiO}_2$  may also contribute indirectly to contaminant reduction through oxidation of organic compounds and modification of surface chemistry.

Photocatalytic processes can enhance overall treatment performance by reducing competing contaminants and maintaining active adsorption sites.

##### **4.7.5 Synergistic Nanocomposite Effects**

The combined incorporation of silver oxide, titanium dioxide, and nanoclay generated synergistic effects not achievable by individual components alone.

The clay matrix provided structural support and adsorption capacity, while titanium dioxide contributed photocatalytic activity and silver oxide enhanced surface reactivity and antimicrobial functionality.

This synergistic interaction is likely responsible for the exceptional treatment efficiency observed during performance evaluation.

#### **4.8 Changes in Physical Appearance of Wastewater**

Visual examination of the treated wastewater revealed substantial improvements in overall water quality.

Prior to treatment, the wastewater exhibited dark coloration, elevated turbidity, and objectionable odor. Following filtration through the nanocomposite filter, the treated water appeared significantly clearer, with marked reductions in color intensity and turbidity.

The disappearance of objectionable odor further indicated effective removal of dissolved contaminants and suspended materials contributing to aesthetic deterioration.

These observations suggest that the filter was capable of improving not only chemical water quality but also aesthetic characteristics important for environmental acceptability.

#### **4.9 Comparison with Previous Studies**

The findings obtained in the present investigation are consistent with previous reports highlighting the effectiveness of clay-based nanocomposite materials for wastewater treatment.



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Fu and Wang (2011) reported that adsorption-based technologies exhibit high efficiency for heavy metal removal when suitable adsorbents are employed. Similarly, Qu et al. (2013) emphasized the significant advantages offered by nanomaterials due to their enhanced surface area and reactivity.

The incorporation of titanium dioxide and silver oxide nanoparticles appears to have further improved treatment performance beyond what is typically achieved using unmodified clay materials. This observation supports previous findings demonstrating the benefits of combining adsorption and catalytic functionalities within a single treatment system.

Compared with conventional treatment technologies such as chemical precipitation and membrane filtration, the developed nanocomposite filter offers several advantages, including reduced chemical consumption, lower sludge generation, simplified operation, and potential cost-effectiveness.

These characteristics make the technology particularly attractive for deployment in developing regions where access to advanced wastewater treatment infrastructure remains limited.

### 4.10 Implications for Sustainable Mining Wastewater Management

The results of this study have important implications for sustainable environmental management in mining communities.

The use of locally available clay resources combined with environmentally compatible nanomaterials provides a promising pathway toward affordable wastewater treatment solutions. The ability of the developed filter to remove heavy metals efficiently suggests potential applications in decentralized treatment systems serving artisanal and small-scale mining operations.

Furthermore, the technology aligns with global sustainability objectives emphasizing pollution prevention, water resource protection, and the development of environmentally responsible treatment systems.

The successful implementation of such filtration systems could contribute significantly to reducing the environmental impacts associated with mining activities while promoting safer water management practices in affected communities.

## 5. CONCLUSION, LIMITATIONS, AND RECOMMENDATIONS

### 5.1 Conclusion

The increasing generation of mining wastewater and the environmental risks associated with its discharge have necessitated the development of innovative, efficient, and sustainable treatment technologies. This study investigated the development and performance evaluation of a silver oxide–titanium dioxide–nanoclay composite filter for the treatment of mining wastewater. The study was motivated by the limitations of conventional treatment methods, including high operational costs, excessive sludge production, membrane fouling, and reduced efficiency when treating complex wastewater matrices.

The developed nanocomposite filter combined the adsorption capacity of nanoclay with the photocatalytic and antimicrobial properties of titanium dioxide and silver oxide nanoparticles. The successful integration of these materials resulted in a multifunctional filtration system possessing favorable structural and physicochemical characteristics for wastewater treatment applications.

Characterization studies confirmed the successful incorporation of silver oxide and titanium dioxide nanoparticles into the clay matrix. X-ray diffraction analysis verified the presence of the desired crystalline phases, while scanning electron microscopy revealed a highly porous and interconnected microstructure suitable for filtration processes. Fourier transform infrared spectroscopy further demonstrated the existence of functional groups capable of participating in adsorption and surface complexation reactions.

The treatment performance evaluation demonstrated that the developed filter was highly effective in removing heavy metals from mining wastewater. Significant reductions were observed in the concentrations of iron, copper, lead, manganese, and other contaminants present in the wastewater. The filtration process also improved the physical quality of the wastewater, resulting in reduced turbidity, elimination of objectionable odor, and improved visual appearance.

The enhanced treatment efficiency observed in this study can be attributed to the synergistic interaction among several removal mechanisms. These mechanisms include adsorption onto nanoclay surfaces, ion exchange within the clay structure, surface complexation reactions



involving functional groups, and catalytic contributions from silver oxide and titanium dioxide nanoparticles. The combination of these processes created a highly effective treatment system capable of addressing multiple contaminants simultaneously.

The study therefore demonstrates that silver oxide–titanium dioxide–nanoclay composite filters represent a promising alternative for mining wastewater remediation. The use of locally available clay resources combined with advanced nanomaterials offers a practical approach toward developing affordable and environmentally sustainable water treatment technologies. The findings contribute to the growing body of knowledge on nanotechnology-based environmental remediation and provide a foundation for future research and practical implementation.

### 5.2 Scientific Contributions of the Study

This study makes several important contributions to the fields of environmental engineering, wastewater treatment, and nanomaterial applications.

First, it demonstrates the feasibility of integrating silver oxide and titanium dioxide nanoparticles within a clay-based ceramic matrix to create a multifunctional filtration material. The resulting nanocomposite combines adsorption, photocatalytic, and antimicrobial functionalities within a single treatment system.

Second, the study provides evidence supporting the application of nanocomposite filters for the treatment of actual mining wastewater rather than synthetic laboratory solutions. This distinction is important because real wastewater typically contains a complex mixture of contaminants that may influence treatment performance.

Third, the research highlights the potential utilization of locally sourced clay materials in the development of low-cost treatment technologies suitable for deployment in developing countries. The approach contributes to ongoing efforts aimed at promoting resource-efficient and sustainable environmental management practices.

Finally, the findings contribute to the understanding of contaminant removal mechanisms operating within hybrid nanocomposite systems and provide valuable information for future optimization of filter design and performance.

### 5.3 Practical Implications

The outcomes of this study have important practical

implications for wastewater management and environmental protection.

The developed filtration technology may be particularly beneficial in regions where access to advanced treatment facilities is limited. Artisanal and small-scale mining communities often lack the financial resources and technical capacity required for the operation of sophisticated wastewater treatment systems. The use of clay-based nanocomposite filters offers a potentially affordable and accessible alternative.

Furthermore, the incorporation of photocatalytic and antimicrobial functionalities may reduce maintenance requirements and improve long-term operational performance. The technology may therefore contribute to sustainable water management practices in rural and resource-constrained environments.

The findings also suggest potential applications beyond mining wastewater treatment, including industrial wastewater treatment, groundwater remediation, stormwater management, and drinking water purification.

### 5.4 Limitations of the Study

Despite the promising findings obtained, several limitations should be acknowledged.

First, the study was conducted primarily under laboratory conditions. Although laboratory-scale experiments provide valuable insights into treatment performance, actual field conditions may introduce additional variables capable of influencing filter effectiveness. Factors such as fluctuating wastewater composition, temperature variations, hydraulic loading rates, and prolonged operational periods may affect treatment efficiency.

Second, the study focused primarily on heavy metal removal and selected physicochemical parameters. Additional investigations are required to evaluate the effectiveness of the filter for the removal of other contaminants commonly present in mining wastewater, including sulfates, nitrates, cyanides, and organic pollutants.

Third, long-term durability and regeneration characteristics of the filter were not extensively investigated. Understanding filter lifespan and reusability is essential for assessing economic feasibility and practical implementation.

Fourth, the photocatalytic performance of the titanium dioxide component under varying light conditions



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was not comprehensively evaluated. Future studies should examine the influence of solar irradiation and other environmental factors on treatment efficiency.

Finally, a detailed economic assessment was beyond the scope of the present investigation. Cost-benefit analyses will be necessary before large-scale implementation can be recommended.

#### 5.5 Recommendations

Based on the findings of this study, the following recommendations are proposed:

##### 1. Pilot-Scale Evaluation

Future studies should evaluate the performance of the developed nanocomposite filter under pilot-scale and field-scale conditions. Such investigations will provide valuable information regarding operational stability, treatment efficiency, and maintenance requirements under real-world conditions.

##### 2. Long-Term Performance Assessment

Extended operational studies should be conducted to assess filter durability, fouling behavior, regeneration potential, and lifespan. These factors are critical for determining the practical viability of the technology.

##### 3. Optimization of Nanoparticle Loading

Additional research should investigate the influence of varying silver oxide and titanium dioxide concentrations on filter performance. Optimization of nanoparticle loading may further enhance contaminant removal efficiency while minimizing production costs.

##### 4. Evaluation of Additional Contaminants

Future investigations should assess the capability of the filter to remove a broader range of contaminants, including arsenic, chromium, mercury, sulfates, cyanides, and emerging pollutants.

##### 5. Solar-Assisted Photocatalytic Studies

The photocatalytic properties of titanium dioxide suggest potential benefits under natural sunlight conditions.

Future studies should evaluate the performance of the filter under solar irradiation to determine its suitability for energy-efficient treatment applications.

##### 6. Economic Feasibility Analysis

Comprehensive economic assessments should be performed to compare the developed technology with existing treatment systems. Such analyses should include material costs, production expenses, operational requirements, and maintenance considerations.

##### 7. Scale-Up and Commercialization

Successful pilot-scale results should be followed by technology scale-up and commercialization studies. Collaboration among researchers, industry stakeholders, and regulatory agencies will be essential for facilitating practical implementation.

#### 5.6 Future Research Directions

Future research should focus on the development of next-generation clay-based nanocomposite filters incorporating advanced functional materials capable of enhancing adsorption, catalytic activity, and contaminant selectivity.

Potential areas for further investigation include:

- Development of visible-light-responsive photocatalytic composites.
- Integration of magnetic nanoparticles for simplified regeneration and recovery.
- Incorporation of bio-based materials to improve sustainability.
- Application of machine learning techniques for process optimization.
- Development of hybrid treatment systems combining adsorption, photocatalysis, and membrane technologies. Such advancements have the potential to further improve treatment efficiency and support global efforts toward sustainable water resource management.

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