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Synthesis and Characterization of Optical Properties of Talc/Montmorillonite Nanocomposites via Sol-Gel and Ball Milling Methods

Sefrilita Risqi Adikaning Rani[1]*, & Eka Cahya Muliawati[2]
[1*]Universitas Islam Negeri Alauddin Makassar, Indonesia, [2]Institut Teknologi Adhi Tama Surabaya, Indonesia
*Co e-mail: sefrilita.rani@uin-alauddin.ac.id

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ABSTRACT

Nanocomposites derived from talc ($Mg_3Si_4O_{10}(OH)_2$) and montmorillonite (MMT) have gained considerable attention due to their tunable optical, mechanical, and thermal properties. This study systematically compares two synthesis techniques—sol-gel processing and ball milling—for fabricating talc/MMT nanocomposites, with a focus on their optical characteristics. The sol-gel method promoted homogeneous nanoparticle dispersion, while ball milling enhanced exfoliation and reduced particle size. Comprehensive characterization via X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM), ultraviolet-visible (UV-Vis) spectroscopy, and photoluminescence (PL) spectroscopy revealed that the nanocomposites exhibit strong UV absorption in the 200–400 nm range, a reduced optical bandgap from 4.5 eV to 3.8 eV, and enhanced PL intensity compared to pristine materials. These findings suggest promising applications in UV shielding, optoelectronics, and photocatalytic systems. The study concludes that the choice of synthesis method plays a pivotal role in tailoring the nanocomposites' structural integrity and optical functionality, with sol-gel favoring intercalation and uniformity, while ball milling enhances exfoliation and defect-mediated performance. This comparative study highlights the critical influence of synthesis method on the structural, morphological, and optical properties of talc/MMT nanocomposites, providing valuable insights for optimizing layered silicate-based materials for advanced functional applications.

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INTRODUCTION

Layered silicates such as talc and montmorillonite (MMT) have attracted increasing interest in materials science due to their distinctive layered architectures and multifunctional properties. Talc, a trioctahedral magnesium silicate, is composed of stacked sheets of silicate layers held together by weak van der Waals forces. This structure imparts several valuable properties, including excellent thermal stability, hydrophobicity, chemical inertness, and lubricity. Owing to these attributes, talc has been widely employed as a reinforcing and functional filler in a range of industries such as polymers, ceramics, coatings, and pharmaceuticals (Bergaya & Lagaly, 2013). In contrast, montmorillonite a member of the smectite group features a 2:1 layer structure consisting of two tetrahedral silica sheets surrounding a central octahedral alumina sheet. This configuration confers a high cation exchange capacity (CEC), significant swelling behavior in aqueous environments, and a large specific surface area, making MMT suitable for applications in catalysis, adsorption, ion exchange, and nanocomposite formulation (Chen et al., 2020; Nguyen et al., 2019).

When talc and MMT are combined at the nanoscale, the resulting hybrid system can exhibit synergistic properties that surpass those of the individual components. Talc contributes thermal and chemical stability, while MMT introduces reactivity and ion exchange capabilities. The integration of these two materials can lead to nanocomposites with enhanced structural, optical, and electronic functionalities, particularly useful in advanced materials applications. At the nanoscale, quantum confinement effects may arise due to the restricted motion of charge carriers, leading to altered electronic band structures and modified optical properties such as enhanced absorption and emission behavior (Kumar et al., 2019; Sharma et al., 2020). Furthermore, the high surface area and interfacial interactions within such nanocomposites can significantly enhance charge separation and light scattering, both of which are vital for optoelectronic and photocatalytic performance (Gupta et al., 2022).

The successful development of talc/MMT nanocomposites largely depends on the synthesis method employed. Among the most widely adopted techniques for clay-based nanomaterial synthesis are the sol-gel method and mechanical ball milling. The sol-gel process offers molecular-level control over composition and enables homogeneous mixing of precursors, often resulting in highly dispersed nanocomposites with uniform phase distribution and controlled crystallinity. This method involves the hydrolysis and condensation of metal alkoxides or other inorganic precursors to form a colloidal suspension (sol), which then transitions into a gel and is eventually converted into a nanocomposite through drying and calcination (Li et al., 2020; Yang et al., 2020). On the other hand, ball milling is a mechanical alloying approach that utilizes kinetic energy from milling media to induce solid-state reactions, reduce particle size, and enhance dispersion. It is a cost-effective, scalable, and environmentally friendly method, capable of producing nanocomposites with modified surface structures and improved interfacial bonding through mechanical activation (Wang et al., 2021; Park et al., 2019).

Despite the widespread use of these synthesis techniques, there is a noticeable gap in the current literature concerning a direct, systematic comparison of the sol-gel and ball milling methods for preparing talc/MMT nanocomposites. A comprehensive comparative study is essential to

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understand how these synthesis routes influence the final properties of the nanocomposites, including their crystal structure, particle morphology, optical bandgap, and functional performance in real-world applications. Addressing this knowledge gap could provide valuable insights into the structure–property relationships in clay-based hybrid systems and guide the selection of appropriate synthesis strategies for targeted technological applications.

In response to this gap, the present study aims to investigate the synthesis and characterization of talc/MMT nanocomposites using both sol-gel and ball milling techniques. Following synthesis, the resulting materials will be thoroughly characterized using a range of analytical tools, including X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and UV-Vis spectroscopy. These techniques will allow the evaluation of crystallinity, chemical bonding, particle size and morphology, elemental distribution, and optical absorption properties (Zhang et al., 2018; Smith et al., 2017). Furthermore, the functional potential of these nanocomposites will be assessed in optoelectronic and photocatalytic contexts by analyzing their band gap energies, photoluminescence behavior, and photocatalytic degradation efficiency under UV and visible light exposure (Thomas et al., 2018; Rodriguez et al., 2022; Lee et al., 2021).

By combining materials with complementary properties and evaluating their synthesis through two contrasting techniques, this work seeks to advance the development of multifunctional layered nanocomposites with potential utility in next-generation energy, environmental, and optical technologies.

METHODS

1. Materials

The materials utilized in this study include talc powder with a purity greater than 98 percent, supplied by Sigma-Aldrich, and sodium montmorillonite (Na-MMT) obtained from Nanocor Inc. Ethanol with a purity of 99.9 percent from Merck was used as the dispersion medium. Tetraethyl orthosilicate (TEOS), also from Sigma-Aldrich, served as the silica precursor in the sol-gel synthesis process. Hydrochloric acid (HCl) from Merck was employed to adjust the pH of the solution during hydrolysis.

2. Synthesis Methods

Two different approaches were used to synthesize talc/MMT nanocomposites, namely the sol-gel method and the ball milling method, in order to compare their effects on material structure and properties.

In the sol-gel synthesis, one gram of talc and one gram of Na-MMT were ultrasonically dispersed in fifty milliliters of ethanol for thirty minutes to ensure a homogeneous suspension. Following this, five milliliters of TEOS were added dropwise under constant stirring. To initiate hydrolysis, five milliliters of distilled water adjusted to a pH of three using HCl were introduced into the system. The resulting mixture was stirred at a temperature of sixty degrees Celsius for six hours to promote condensation and gel formation. The obtained gel was dried at one hundred

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degrees Celsius for twelve hours and subsequently calcined at five hundred degrees Celsius for two hours to achieve thermal stability and eliminate organic residues.

For the ball milling synthesis, talc and Na-MMT were combined in equal weight ratios and loaded into a planetary ball mill, model Retsch PM 100. The milling process was carried out at a rotational speed of three hundred revolutions per minute for ten hours. Zirconia balls were used as the grinding media, maintaining a ball-to-powder weight ratio of ten to one. After milling, the resulting powder was sieved using a mesh with openings smaller than fifty micrometers to ensure uniform particle size distribution.

3. Characterization Techniques

The synthesized nanocomposites were characterized using a variety of analytical techniques to assess their structural, morphological, and optical properties. X-ray diffraction measurements were conducted using a Bruker D8 Advance diffractometer equipped with Cu-Kα radiation, with a wavelength of 1.5406 angstroms, over a scanning range of 2θ from 5 to 70 degrees, in order to determine the crystallographic phases and estimate crystallite sizes. Fourier-transform infrared spectroscopy was performed using a PerkinElmer Spectrum Two instrument in attenuated total reflectance mode across the wavenumber range of 400 to 4000 inverse centimeters, to identify functional groups and chemical bonding.

Ultraviolet–visible diffuse reflectance spectra were recorded with a Shimadzu UV-2600 spectrophotometer in the wavelength range of 200 to 800 nanometers. These data were used to calculate the optical band gap using the Kubelka–Munk function. Photoluminescence spectroscopy was carried out with a Horiba Fluorolog-3 system using an excitation wavelength of 325 nanometers and collecting emission spectra between 350 and 600 nanometers, to evaluate charge carrier recombination behavior. Transmission electron microscopy imaging was performed using a JEOL JEM-2100 microscope operating at 200 kilovolts to observe particle morphology, phase dispersion, and nanoscale structural features.

RESULTS

This section presents a comprehensive analysis of the structural, morphological, and optical properties of talc, montmorillonite (MMT), and their nanocomposites synthesized via sol-gel and ball milling methods. The comparative evaluation aims to elucidate the effects of different synthesis techniques on the material characteristics and to assess their potential suitability for optoelectronic and photocatalytic applications. Detailed characterization results obtained from X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM), and UV–visible spectroscopy are discussed to provide insights into the compositional and functional differences among the samples.

Summarizes the XRD results, highlighting the key diffraction peaks for talc, MMT, and their nanocomposites. The shift of the (001) peak in the sol-gel composite confirms intercalation and increased interlayer spacing, whereas the broadening of peaks in the ball-milled sample indicates reduced crystallite size and partial structural disorder caused by mechanical milling.

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Table 1. XRD Analysis of Talc, MMT, and Nanocomposites

Sample	Main Peaks (2θ, °)	Notable Features	Crystallite Size (nm)
Talc	9.6 (001), 19.3 (002), 28.6 (004)	Matches PDF #00-029-1493	~45
MMT	7.2 (001)	Typical basal reflection for Na-MMT	~40
Sol-gel Composite	6.8 (001)	Peak shift indicates intercalation (d = 13.0 Å)	~22
Ball-milled Composite	Broad peak (~7.0–9.5)	Peak broadening due to reduced crystallinity	~15

Presents FTIR spectral data where changes in the Si–O–Si stretching and hydroxyl group vibrations reveal structural modifications. The sol-gel method introduces silica incorporation and stronger hydrogen bonding, as seen by broader bands, while ball milling weakens the hydroxyl signals due to disruption of the clay layers.

Table 2. FTIR Spectra of Talc, MMT, and Nanocomposites

Sample	Si–O–Si Stretching (cm^{-1})	OH Stretching (cm^{-1})	Notes
Talc	1018	3675	Sharp bands characteristic of crystalline talc
MMT	1035	3620	Strong OH vibration from interlayer water
Sol-gel Composite	1042	3640 (broad)	TEOS incorporation, hydrogen bonding present
Ball-milled Composite	1025	3635 (weakened)	Weaker OH due to structural disruption

Lists UV–Vis absorption peaks and the estimated optical bandgap energies. Both nanocomposites show a decrease in bandgap compared to pure talc and MMT, with the ball-milled sample exhibiting the lowest bandgap. This bandgap narrowing suggests enhanced optical activity potentially beneficial for photocatalytic and optoelectronic applications.

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Table 3. UV–Vis Absorption and Optical Bandgap Values

Sample	Absorption Peaks (nm)	Estimated Bandgap (eV)	Remarks
Talc	220, 280	4.5	Wide bandgap insulator
MMT	220, 280	4.2	Slightly narrower due to layered structure
Sol-gel Composite	220, 280	3.9	Bandgap reduced by interfacial effects
Ball-milled Composite	220, 280	3.8	Smallest bandgap due to exfoliation, defects

DISCUSSION

The XRD results demonstrate clear structural differences between talc, montmorillonite (MMT), and their nanocomposites synthesized by sol-gel and ball milling methods. The shift of the basal reflection peak in the sol-gel sample suggests effective intercalation of silica species within the MMT layers, leading to an expansion of the interlayer spacing. This observation indicates a strong interaction between the inorganic matrix and the clay layers, which likely enhances the composite's mechanical stability and functional properties. In contrast, the ball milling process causes significant peak broadening, reflecting a reduction in crystallite size and partial disruption of the layered structure. This mechanical treatment produces exfoliated nanosheets with smaller dimensions, as confirmed by TEM imaging, which may contribute to improved surface area and reactivity. The associated bandgap reduction observed in both composites suggests improved charge carrier mobility, which is beneficial for optoelectronic applications (Li et al., 2020).

Photoluminescence (PL) spectroscopy further supports these findings, with emission peaks observed at 420 nm for the sol-gel composite and 435 nm for the ball-milled sample. The PL intensity increased by approximately 30% compared to pure talc, indicating enhanced radiative recombination. This enhancement is attributed to defect states introduced during processing, which act as recombination centers, as previously reported by Gupta et al. (2022). These defect states can improve charge separation efficiency, an important factor for photocatalytic performance.

FTIR analysis corroborates the structural changes revealed by XRD and PL. The sol-gel nanocomposite exhibits broadened Si–O–Si bands and stronger hydroxyl stretching vibrations, indicative of TEOS-derived silica integration and enhanced hydrogen bonding networks. These modifications likely facilitate charge transfer processes and improve photocatalytic activity. In contrast, the ball-milled composite shows weakened hydroxyl peaks, reflecting a loss of ordered hydrogen bonding due to mechanical forces, which may affect thermal stability but simultaneously introduce defect sites that enhance optical properties.

Optical measurements reveal a notable decrease in the bandgap energies of both nanocomposites relative to the pristine materials. The bandgap narrowing in the sol-gel composite can be explained by the intimate contact between talc, MMT, and silica, which creates new electronic

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states at the interfaces. The ball-milled sample exhibits an even lower bandgap, likely due to increased defect densities and quantum confinement effects stemming from reduced particle sizes and exfoliated structures. These optical improvements confirm that both synthesis routes effectively tune the material properties, making these nanocomposites promising candidates for applications requiring efficient light absorption and charge separation, such as optoelectronics and photocatalysis.

Overall, the choice of synthesis method significantly influences the structural integrity, morphology, and optical behavior of the nanocomposites. Sol-gel processing favors intercalation and chemical bonding while preserving the layered structure, whereas ball milling enhances particle size reduction and exfoliation, boosting surface-related properties. Future studies could optimize synthesis parameters to balance structural order and defect density for targeted applications and evaluate device-level performance to fully exploit the potential of these materials.

CONCLUSIONS

Talc/MMT nanocomposites were successfully synthesized using both sol-gel and ball milling techniques. The sol-gel method produced nanocomposites with a more homogeneous and well-intercalated structure, while ball milling resulted in smaller particle sizes with exfoliated morphology. Optical characterization demonstrated enhanced ultraviolet absorption, a significant reduction in bandgap energy, and improved photoluminescence intensity compared to the pristine materials.

This study addresses a critical gap in the literature by systematically comparing sol-gel and ball milling methods for the synthesis of talc/MMT nanocomposites. The findings demonstrate that the choice of synthesis method significantly influences the structural arrangement and optical performance of the composites. Specifically, sol-gel synthesis enhances interfacial integration and uniformity, while ball milling promotes exfoliation and defect-induced optical activity. These insights provide a foundation for the rational design and optimization of layered silicate-based nanomaterials for advanced applications in optoelectronics, UV shielding, and photocatalysis.

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