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Kaolin-Supported Metal Oxides for Efficient Dye Adsorption: Synthesis, Characterisation and Performance Evaluation

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Abstract

As the world is undergoing rapid development industries are also going through many advancements. But with that pollution is also increasing. It is the need of an hour to address this environmental issue. In this research work how the hazardous dyes removed from industries should be treated is explained. Kaolin metal oxides nanoparticles can work very well to address this problem. Kaolin has layered structure, and surface functionality make it an attractive support for metal oxide dispersion which can remove dyes from wastewater very efficiently. This review examines recent advancements in synthesis methods, materials characterization, dye adsorption performance, and underlying mechanisms of kaolin-supported metal oxide composites. The performance of composites against various cationic and anionic dyes under different conditions, including pH, concentration, and temperature, is critically analyzed. Challenges and future directions for scale-up and environmental application are also discussed.

Keywords: *Kaolin clay, metal oxides, dye adsorption, surface modification, wastewater treatment, adsorption mechanisms.*

Introduction

A large amount of wastewater from textile industries, containing dyes enter into water bodies as effulents. These dyes are hazardous to biotic as well as abiotic life. Some of these dyes are Congo red dye, Azo dyes, Methylene blue dyes. [1] Generally, such dyes are classified into cationic, anionic and nonionic dyes. [2][3] The anionic type of dyes i.e. Azo dyes are toxic to human health and causes irritation to eyes, skin and respiratory tract. Azo dyes also causes water borne diseases. Conventional treatment methods such as biological oxidation, coagulation, and membrane filtration often fail to achieve satisfactory dye removal. Adsorption is an eco-friendly process. Kaolin-supported metal oxide composites have high surface area, active sites for dye binding, and improved stability, positioning them as efficient adsorbents. Kaolin posseses very low reactive surface. It has very less adsorption sites. When Kaolin is treated with metal oxide it overcomes it characterstic limitations.

Materials

Materials required for synthesis of Kaolin-metal oxide composite are as follows Natural Kaolin clay, Metal precursors e.g., $FeCl_3$, $TiCl_4$, $Zn(NO_3)_2$), reducing agents (e.g., $NaBH_4$ for nanoparticles) [5], solvents (deionised water, ethanol).

Synthesis Routes

Several synthesis methods reported in the literature include:

Co-precipitation

In this method, metal salts are mixed with kaolin clay suspension (Zn-Kaolin via co-precipitation method) [6]. Kaolin-Cobalt ferrite nanoparticles is effective adsorbent for removal of methylene blue dyes from wastewater.[7]

The co-precipitation method for synthesizing kaolin-metal composites offers a low-cost, simple, and efficient one-pot technique for producing highly uniform Nano-powders with enhanced specific surface areas and improved stability. It is particularly effective for creating active adsorbent materials by facilitating uniform distribution of nanoparticles, which increases reactivity and enhances removal efficiency for water treatment and environmental remediation [8][9]. This method is simple and cost-effective.

Sol-gel Method

In this method Kaolin-metal oxide nanocomposites were studied at different firing temperatures, with soaking time of 30 min to 60 min and at different zeolite concentrations, such as 0%, 10%, 20%, etc. Kaolin-metal oxide nanocomposites thus prepared were functionalized with carboxylic acid group to increase adsorption area.

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Further it was used for removal of acid fuschin dye and methylene blue dye.[10] Sol-gel synthesis of kaolin-metal composites (such as TiO_2 or Zn) leads to high photocatalytic activity and superior antimicrobial efficacy. In this method, metal alkoxides or salts hydrolyse onto kaolin, yielding uniform dispersion on the surface [12]. Kaolin was also encorporated with Titanium propoxide which was used for removal of methylene blue and methyl orange II dyes. In this way these nanocomposites prepared by sol-gel method were used for removal of tannery wastewater. [13][14]

Solvothermal Method

In this method kaolinite was kept in an organic medium such as tetrapropylammonium ion.[15][16][17][18]. Here it was observed that crystal growth was influenced when different parameters such as temperature, soaking time were changed.[19] To remove impurities acid treatment was done.[20] Heat and pressure promote crystalline growth of metal oxides on kaolin, improving structural stability.

Wet Impregnation Method

Recently, researchers have prepared Kaolin-nickel oxide nanocomposite using Wet-impregnation method. The catalytic activity of prepared nanocomposites were studied at variable temperature, amount of oxidant. Wet-impregnation method was used for good dispersion of active phase in dye removal process. Using this malachite green dye was removed. In this method Kaolin was washed thoroughly with distilled water. Then it was dried at 100 degree Celsius. Then precursor dissolved in water was added to it. NaOH was added to it for complete precipitation. The mixture was stirred and sand bath treatment was done. Then the sample was dried and calcined. Like this different samples were prepared with varying metal oxide concentration.[22] Also CuO – Kaolin nanocomposites were prepared using wet impregnation method. Its performance was studied at differentr concentrations of Kaolin and CuO catalyst.[23] In this method, kaolin is soaked in metal precursor solution, followed by calcination to form metal oxides.

Characterization Techniques :

- X-ray Diffraction (XRD): Phase identification of kaolin and metal oxides

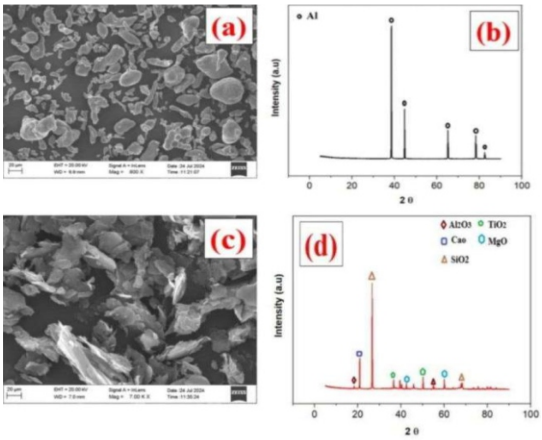

Fig. (a) – SEM of received aluminium
Fig. (b) – XRD of received aluminum
Fig. (c) – SEM of extracted kaolin powders
Fig. (d) XRD of extracted kaolin powders[25]

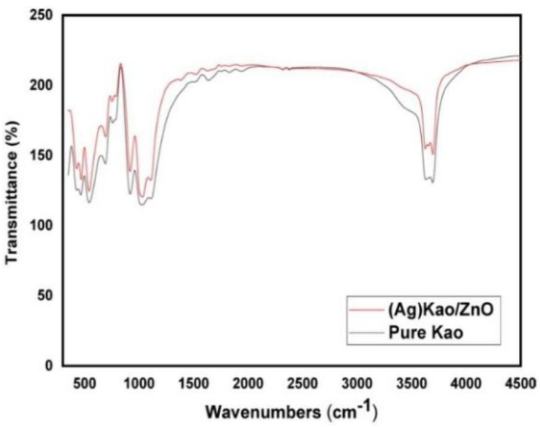

Fig. (f) – FTIR investigations of the (Ag)Kaolin/ZnO nanostructure and pure kaolinite Collectively, these techniques result in successful deposition of metal oxides, enhancing surface area and increasing active adsorption sites.[27][28]

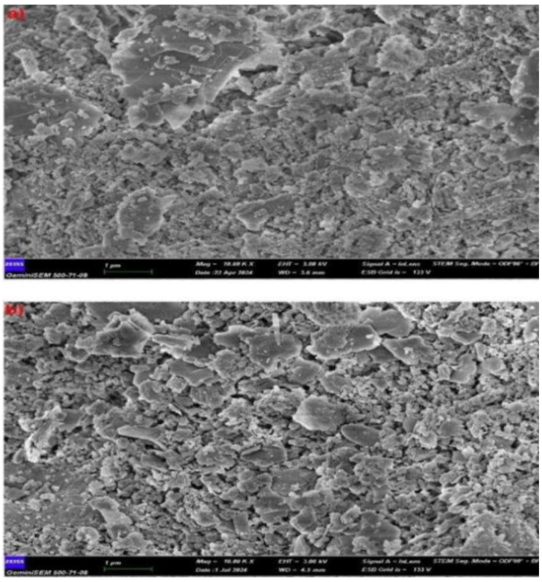

Fig. (e) – Scanning/Transmission Electron Microscopy (SEM/TEM): Surface morphology and particle dispersion [26]

Results

Kaolin - Metal nanomaterials are paramount adsorbents in wastewater treatment and elimination of diverse pollutants such as heavy metal ions and dyes, offering superior adsorption capacity, selectivity, and stability compared to nanoparticles. Clay-based nanocomposites have received substantial scientific and technical attention owing to their biocompatibility, abundance, and extensive chemical proficiency regarding clay interlayer infiltration. Layered silicates can be employed in the construction of polymeric nanocomposites to improve their physical and mechanical properties. Meanwhile, clay soils such as montmorillonite, kaolinite, and mica have shown a perfect framework for polymeric nanocomposites. These minerals can form strong bonds with polymer chains, resulting in superior properties and homogeneous structure [29][30].

Structural Properties

Kaolin altered with metal oxides shows significantly increased surface area and pore volume compared with raw clay alone. Metal oxide nanostructures are uniformly dispersed with an increase in accessible adsorption sites. X-ray diffraction analysis reported in several studies confirms that the characteristic reflections of kaolinite at 2θ ≈ 12.3° and 24.9°, corresponding to the (001) and (002) planes, are preserved after metal modification, indicating that the fundamental layered structure of kaolin remains intact. Additional diffraction peaks corresponding to metal oxides such as
Fe_2O_3, ZnO, CuO, and NiO confirm the successful incorporation of metal species onto the
kaolin. Light peak broadening and intensity variations are observed, which show nanoscale metal oxide dispersion and partial interlayer interaction without structural modification.

Fourier transform infrared spectroscopy further supports these findings. Shifts and intensity reductions in hydroxyl bands indicate coordination or hydrogen bonding between metal ions and surface –OH groups. Scanning electron microscopy images consistently show that raw kaolin possesses plate-like, stacked morphology, whereas metal-modified kaolin composites display rougher and more heterogeneous surfaces due to the deposition of metal oxide nanoparticles. Uniform nanoparticle dispersion enhances surface heterogeneity and active adsorption sites, while excessive metal loading can lead to particle agglomeration, potentially reducing adsorption efficiency.

Reported values show an increase from approximately 10–20 m^2/g for raw kaolin to 40–80 m^2/g for metal-modified composites, with pore size distribution shifting toward the mesoporous range (2–50 nm), favourable for dye diffusion and adsorption. Mixed metal oxide systems, such as Fe–Zn loaded kaolin, further enhance pore structure and interlayer spacing, leading to improved adsorption behavior.

Overall, structural characterisation confirms that kaolin serves as a stable support for metal oxides, providing enhanced surface area, modified surface chemistry, and improved porosity. These synergistic structural features are directly responsible for the superior dye removal performance of kaolin–metal composites compared to pristine kaolin.

Adsorption Performance

Compared to pristine kaolin, metal-modified composites exhibit superior adsorption performance due to enhanced surface area, modified surface charge, and the presence of reactive metal oxide sites [39][40].

Electrostatic Interactions:

Electrostatic interactions controls dye adsorption of these nanocomposites.
Incorporation of metal oxide alters adsorptive behavior. Hence under acidic conditions anionic dyes were adsorbed and under alkaline conditions cationic dyes were adsorbed. [41][42][43].

Several studies have demonstrated that adsorption efficiency increases significantly when electrostatic attraction dominates the interaction between dye molecules and composite surfaces [44].

Surface Complexation and Coordination:

Surface complexation plays a crucial role in metal-modified kaolin systems. Metal oxides such as Fe_2O_3, ZnO, CuO, and NiO act as Lewis acid sites and form coordination bonds with functional groups of dyes, including $–SO_3^-$, $–NH_2$, –OH, and –COOH [45][46]. These inner-sphere complexes contribute to strong and stable adsorption, explaining the higher adsorption capacities observed for kaolin–metal composites compared to raw kaolin [47].

Hydrogen Bonding:

Hydrogen bonding between hydroxyl group of kaolin, metal oxide and electronegative atoms in dye molecules enhance adsorptive performance. [48] FT-IR spectral shifts after adsorption provide evidence of hydrogen bond formation, particularly in dyes containing amine and sulfonate groups [49].

π–π Interactions:

It contributes to improved adsorption in aromatic compounds. Van-der waals interactions support adsorption at higher dye concentrations. [50][51]

Pore Diffusion and Kinetic Behaviour:

Metal encorporation onto Kaolin enhances mesoporosity.[52]

Synergistic Effect of Metal Oxides:

Mixed metal oxide-modified kaolin composites provide synergistic adsorption effects by offering multiple binding sites and improved surface heterogeneity. Such systems often demonstrate enhanced adsorption capacity, faster kinetics, and better regeneration performance, highlighting their potential for practical wastewater treatment applications [53].

Effect of temperature and pH:

The low pH favours anionic dye adsorption as the surface of adsorbent becomes protonated. Hence there is increase in positive sites. So, here anioinic dye adsorption occurs here due to electrostatic attraction.[54][55][56]. At higher pH, surface deprotonation results in more negative sites, which enhances cationic dye adsorption (e.g., methylene blue, crystal violet) [57][58].

For example in case of removal of methylene blue dye coulombic interactions increases when pH is increases beyond 7.. Because of these effects, optimising pH is crucial for maximising dye removal efficiency. Adsorption capacity generally increases with pH for cationic dyes due to negative surface charge.

Effect of Temperature: Temperature influences dye adsorption by affecting kinetics, thermodynamics, and molecular diffusion. The effect of temperature on adsorption is typically explained through thermodynamic parameters:

In exothermic adsorption processes, adsorption capacity decreases with increasing temperature because heat release favours lower thermal conditions, and the strength of physical adsorption interactions weakens at higher temperatures [61][62].

In endothermic processes, adsorption capacity increases with temperature, often associated with chemisorption or systems where energy barriers must be overcome for dye molecules to interact with active sites [63][64].

Several studies confirm these trends. For example, methylene blue adsorption on modified clay adsorbents reduced as temperature increased from 30 °C to 60 °C, indicating an exothermic process. Conversely, some composites showed improved dye uptake at higher temperatures due to enhanced mobility and accessibility of dye molecules to interior pores [65][66]. These temperature effects influence the Gibbs free energy, enthalpy, and entropy values of adsorption systems, which further clarify whether the process is spontaneous or endothermic/exothermic [67][68][69]. In general, understanding temperature dependence is vital for effective application of kaolin–metal composites in real wastewater treatment scenarios where temperature conditions may vary. Temperature impacts kinetics, but most adsorption is favourable at ambient conditions.[70]

Discussion

Dye removal occurs through hydrogen bonding and π–π interactions, Electrostatic attraction (charged dye interacts with oppositely charged surface), hydrogen bonding, Pore filling and physical adsorption. Metal oxides enhance adsorption by Providing active sites, Increasing total surface area, Improving interaction with dye molecules. Unmodified kaolin is a good adsorbent for removal of dyes pollutants from aquatic system. Unmodified (raw) kaolin has been extensively studied affordable and naturally abundant adsorbent for the removal of dyes pollutants aqueous systems [71][72]. Its adsorption ability arises mainly from its layered aluminosilicate structure, surface hydroxyl groups, and moderate ion-exchange capacity. Raw kaolin shows limited adsorption capability [73][74]. Raw kaolin typically exhibits a surface area in the range of 10–20 $m^2\ g^{-1}$, which restricts dye diffusion and surface interaction [75][76][77]. Various studies have shown that chemical or metal modification of kaolin significantly enhances surface area and pore volume due to structural modification [78][79]. Acid-treated or metal-oxide-loaded kaolin develops mesoporous characteristics, leading to improved dye accessibility and higher adsorption efficiency compared to unmodified kaolin. The modified kaolin exhibits additional Lewis acid sites, surface defects, and metal–oxygen functional groups that promote stronger adsorption mechanisms such as surface complexation and coordination bonding [80]. These enhanced interactions result in significantly higher adsorption capacities than those of raw kaolin [81][82].

- pH Responsiveness and Surface Charge Behaviour: Unmodified kaolin shows limited pH-dependent adsorption behaviour due to its relatively fixed surface charge characteristics. [83] Metal- or chemically modified kaolin displays a shifted point of zero charge, allowing better control over electrostatic interactions with both cationic and anionic dyes across a wider pH range [84]. This improved pH sensitivity is a key advantage of modified kaolin in real wastewater treatment applications [85].

- Adsorption Capacity and Kinetics: Comparative studies consistently report that modified kaolin adsorbents exhibit 2–5 times higher dye adsorption capacity than raw kaolin. While adsorption on unmodified kaolin often follows slower pseudo-first-order kinetics dominated by physical adsorption, modified kaolin systems typically obey pseudo-second-order kinetic models, indicating the dominance of chemisorption processes. [86] Kaolin alone shows modest dye uptake (<20 mg/g), while composites often exceed 80 mg/g, highlighting the importance of metal oxide addition.

- Stability and Reusability: Although raw kaolin is thermally stable, it often suffers from poor regeneration efficiency and reduced adsorption capacity after repeated cycles. Modified kaolin composites demonstrate enhanced structural stability and reusability due to stronger dye–adsorbent interactions and reinforced surface structure, maintaining adsorption efficiency over multiple adsorption–desorption cycles.

- Overall Performance Comparison: Overall, while unmodified kaolin remains an economical and environmentally benign adsorbent, its adsorption performance is relatively modest. Modification through metal loading or chemical treatment transforms kaolin into a high-performance adsorbent with improved surface area, porosity, surface chemistry, adsorption capacity, and operational flexibility. Consequently, modified kaolin composites are more suitable than raw kaolin for efficient and sustainable dye removal from wastewater systems.

Future Scope and Challenges

Kaolin –metal nanocomposite are sustainable catalysts also are very efficient dye adsorption, wastewater remediation. However some challenges are needed to be addressed such as nanoparticle agglomeration. Developing sustainable catalysts is important. Because after multiple operational cycles its adsorptive efficiency decreases. The leaching of nanoparticles into aquatic environment during operation is another concern. Further more eco-friendly techniques could be invented for preparation of these nanocomposites.

Conclusion

Kaolin-supported metal oxide composites are promising, low-cost adsorbents for dye removal. Their performance is superior to raw kaolin due to enhanced surface properties and active sites. Future work should focus on real wastewater application, continuous-flow systems, and life-cycle assessment.

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