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Evaluation of the Effect of Conductive Nanoparticles MXene, ITO, and CNTs on Enhancing the Performance of a Three-Dimensional Electrochemical Reactor for Congo Red Dye Removal from Industrial Wastewater

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ABSTRACT

The presence of resistant dyes such as Congo Red in industrial wastewater, particularly from the textile industry, poses serious challenges for both environmental safety and treatment processes. Among various treatment methods, three-dimensional electrochemical systems have gained increasing attention due to their unique structure and high oxidation capacity. One promising approach to enhancing the efficiency of these systems is the incorporation of conductive nanoparticles, which can improve electron transfer, increase active surface area, and facilitate the generation of reactive species. This review compiles and analyzes findings from previous studies regarding the use of three widely studied conductive nanomaterials MXene, ITO, and CNTs in these systems. The focus is placed on their structural properties, influential operational parameters, and reported outcomes in the removal of Congo Red dye. The synthesis of existing research suggests that appropriate selection of nanomaterials and operating conditions plays a critical role in improving the performance of three-dimensional electrochemical systems for the treatment of dye-containing industrial wastewater.

Introduction

The widespread use of synthetic dyes in textile, leather, and paper industries has led to the generation of large volumes of dye-contaminated wastewater. Among these dyes, azo dyes represent the largest and most problematic class due to their complex aromatic structures and high chemical stability. These dyes are often resistant to conventional treatment methods such as biological degradation, sedimentation, or simple oxidation. One of the most widely studied examples is Congo Red, an anionic azo dye known for its environmental persistence, toxicity, and potential carcinogenicity(1, 2). The limitations of traditional wastewater treatment technologies have driven increasing interest in advanced oxidation processes (AOPs), particularly electrochemical oxidation. In recent years, three-dimensional electrochemical reactors (3D-ECRs) have emerged as a promising innovation in this field. Unlike conventional two-electrode systems, 3D-ECRs incorporate particle electrodes between the anode and cathode, increasing the available reactive surface area and enabling more efficient pollutant degradation. These systems also offer benefits such as compact design, scalability, and reduced chemical usage(3).To enhance the performance of 3D-ECRs, researchers have explored the integration of conductive nanomaterials into the system architecture. Conductive nanoparticles can significantly improve electron transport, catalyze redox reactions, and facilitate the generation of reactive oxygen species (ROS) such as hydroxyl radicals ($^{\bullet}OH$), which play a key role in breaking down complex organic molecules. This review aims to synthesize existing research on the use of three advanced conductive nanomaterials MXene (e.g., Ti_3C_2Tx), indium tin oxide (ITO), and carbon nanotubes (CNTs) within 3D electrochemical systems for the treatment of dye-laden wastewater. Particular attention is given to the physicochemical characteristics of each nanomaterial, the mechanisms by which they influence electrochemical degradation, and their comparative performance in the removal of Congo Red(4, 5). SM El-Mas et al in 2025 , showed that 2D $Ti_3C_2T_x$ MXene achieved approximately 99% degradation of Congo Red within 6 minutes under optimal conditions. The reaction followed a pseudo-first-order kinetic model, and the catalyst remained stable over five reuse cycles without loss of efficiency(6) .By bringing together recent findings, this article seeks to provide an updated perspective on the role of nano-assisted electrochemical systems in addressing the challenges of industrial dye pollution.

2 . Structure and Function of Three-Dimensional Electrochemical Reactors (3D-ECRs)

Electrochemical treatment systems have gained significant attention as effective technologies for the degradation of persistent organic pollutants in wastewater, especially when conventional biological and physicochemical methods fail. These systems rely on redox reactions occurring at electrode surfaces to oxidize or reduce contaminants into less harmful compounds(7, 8). However, traditional two-dimensional (2D) configurations based on flat anode and cathode plates often suffer from key limitations such as limited active surface area, inefficient mass transfer, and uneven current distribution, which collectively reduce overall treatment efficiency and increase energy consumption. To address these shortcomings, three-dimensional electrochemical reactors (3D-ECRs) have been developed, incorporating a third active phase in the form of electrically conductive particle electrodes dispersed between the main electrodes. These particles, when in contact with the electric field, act as microelectrodes, dramatically increasing the available surface area for electrochemical reactions and enhancing the number of active sites throughout the reactor volume. This 3D configuration allows for more uniform current distribution, accelerated degradation kinetics, and improved interaction between pollutants and reactive zones(9). In addition to increasing surface area, 3D-ECRs enable the in situ generation of reactive oxygen species (ROS) such as hydroxyl radicals ($^{\bullet}OH$), superoxide ($O_2^{\bullet-}$), and hydrogen peroxide (H_2O_2). These species play a central role in breaking down stable molecular structures, including azo dyes like Congo Red, which are notoriously resistant to biodegradation. The ability of 3D-ECRs to simultaneously promote direct electron transfer and indirect oxidation through ROS makes them particularly suitable for treating dye-laden industrial effluents(10, 11). The performance of 3D-ECRs is strongly influenced by the nature of the particle electrodes. Recent research has explored the use of advanced materials including carbon-based supports, conductive ceramics, and metal oxides as carriers or components of the particle bed. Among these, conductive nanomaterials such as MXene, indium tin oxide (ITO), and carbon nanotubes (CNTs) have emerged as promising additives due to their high electrical conductivity, chemical stability, and large specific surface area(12). By incorporating these nanomaterials into the reactor structure either as surface coatings, embedded additives, or functional modifiers researchers have demonstrated marked improvements in pollutant removal rates, energy efficiency, and long-term system stability. Such improvements are particularly significant in applications targeting complex, recalcitrant compounds like azo dyes, where both adsorption and oxidative degradation mechanisms must work synergistically(13). Overall, 3D electrochemical reactors represent a highly adaptable and scalable platform for advanced wastewater treatment. However, their optimal design requires careful selection and engineering of particle electrodes, with particular emphasis on the properties of the conductive nanomaterials employed. Further studies are essential to fully understand the interactions between electrode material, reactor dynamics, and pollutant characteristics in order to unlock the full potential of nano-assisted electrochemical processes.

3. Role of Conductive Nanomaterials in Enhancing the Performance of 3D Electrochemical Systems

The integration of conductive nanomaterials into three-dimensional electrochemical systems has emerged as a promising approach to enhance pollutant degradation, particularly in the treatment of persistent dyes. These nanomaterials offer a unique combination of high electrical conductivity, large specific surface area, and catalytic activity, making them suitable for improving both electron transfer and the generation of reactive oxidative species(14, 15). When introduced into the structure of 3D electrochemical reactors, conductive nanomaterials can serve multiple functions. They enhance the conductivity of particle electrodes, facilitate the rapid movement of electrons throughout the system, and increase the density of electroactive sites. This leads to improved degradation kinetics, especially for recalcitrant compounds such as azo dyes, which require strong oxidizing conditions to break down their stable molecular structures(16-18). Furthermore, some nanomaterials possess intrinsic photocatalytic or electrocatalytic properties, contributing directly to the formation of hydroxyl radicals and other reactive intermediates. For instance, MXenes, with their layered structure and surface functional groups, not only provide excellent electrical pathways but also actively participate in surface redox reactions. Similarly, carbon nanotubes (CNTs) are known for their high surface area and remarkable conductivity, which can boost both adsorption and electrochemical reactions. Indium tin oxide (ITO), while more traditionally used in optoelectronic applications, has also demonstrated promise as a stable and efficient electron conductor in electrochemical settings(19). The choice of nanomaterial, its loading method, and its distribution within the reactor system significantly influence overall performance. Methods such as surface coating, blending with carbon-based materials, or direct deposition onto support structures have been explored. Additionally, operational factors including pH, applied voltage, current density, and reaction time interact closely with the material’s properties to determine efficiency outcomes. the strategic use of conductive nanomaterials in 3D electrochemical reactors enables a synergistic enhancement of degradation pathways. Their ability to improve electrical conductivity, increase surface reactivity, and facilitate advanced oxidation processes underlines their value in next-generation wastewater treatment technologies(20).

3-1 MXene: Structure, Properties, and Applications in 3D Electrochemical Systems

MXenes are a class of two-dimensional transition metal carbides, nitrides, or carbonitrides, typically derived from MAX phases (where M = early transition metal, A = group IIIA or IVA element, and X = carbon and/or nitrogen). Among them, Ti_3C_2Tx is the most extensively studied, where Tx denotes surface terminations such as ^{-}OH, ^{-}O, or ^{-}F. The combination of layered structure, high electrical conductivity, hydrophilicity, and abundant surface functional groups makes MXenes highly attractive for electrochemical applications(21, 22) .In the context of three-dimensional electrochemical reactors, MXenes can be used as active components of particle electrodes, as coatings on conductive supports, or as blended additives within electrode composites. Their role is multifaceted: they enhance electrical conductivity across the reactor medium, improve electron mobility, and increase the formation rate of reactive oxidative species such as hydroxyl radicals, which are crucial for the breakdown of persistent organic pollutants like Congo Red. The surface chemistry of MXenes allows for strong interactions with both pollutants and intermediate species. Their negatively charged surfaces promote the adsorption of cationic dye molecules, while their redox-active sites catalyze oxidation reactions under applied potential. Several studies have shown that integrating MXenes into electrochemical systems can significantly improve color removal efficiency, reduce energy consumption, and shorten treatment time. Moreover, MXenes are structurally stable under a wide range of operating conditions and can withstand variations in pH, temperature, and current density. However, certain challenges remain, such as their tendency to restack, which reduces available surface area, and their sensitivity to oxidation in aqueous environments. To address these limitations, researchers have explored surface modification, hybridization with carbon-based nanomaterials, and the use of MXene-polymer composites to maintain dispersion and electrochemical activity(20, 23-25).

3-2 Indium Tin Oxide (ITO): Structure, Electrochemical Behavior, and Role in Dye Degradation

Indium tin oxide (ITO) is a transparent conductive oxide (TCO) composed primarily of indium oxide (In_2O_3) doped with tin oxide (SnO_2), typically in a 90:10 weight ratio. While ITO is best known for its widespread use in electronics, solar cells, and touchscreens due to its excellent electrical conductivity and optical transparency, its properties also make it a valuable candidate in electrochemical applications particularly as a support or modifier in advanced wastewater treatment systems(25, 26). In three-dimensional electrochemical reactors (3D-ECRs), ITO can serve multiple functions. It offers high electrical conductivity, electrochemical stability, and low overpotential for oxidation reactions. When incorporated into particle electrodes or used as a coating material on substrates such as glass beads or carbon supports, ITO facilitates efficient electron transfer throughout the system and contributes to the generation of reactive oxygen species under applied voltage. One of the advantages of ITO over some other conductive materials is its chemical inertness in a range of pH environments, making it suitable for long-term operation in harsh wastewater conditions. Its smooth surface and stability under oxidative conditions also enable reproducible performance and minimal fouling, which are critical for maintaining reactor efficiency over time(27, 28). Several studies have demonstrated that the inclusion of ITO in electrochemical systems can enhance dye degradation rates, especially when combined with other conductive or catalytic materials. However, ITO alone does not exhibit strong adsorption properties for organic molecules such as dyes, and its catalytic activity is limited compared to more advanced nanomaterials. For this reason, it is often used in conjunction with carbon-based materials or integrated into hybrid electrode designs to achieve synergistic effects(29). Moreover, economic and environmental considerations must be taken into account, as indium is a relatively scarce and expensive element. While this does not preclude its use in laboratory-scale or high-performance systems, scalability and material cost remain open questions for real-world applications. ITO provides excellent electrochemical stability and conductivity in 3D-ECRs, and its integration into hybrid electrodes has shown promising results in the degradation of dye pollutants such as Congo Red. Future research may focus on reducing ITO loading, enhancing its surface reactivity, and combining it with other functional materials to maximize efficiency while minimizing cost(30).

3-3 Carbon Nanotubes (CNTs): Properties, Electrochemical Functions, and Applications in Dye Removal

Carbon nanotubes (CNTs) are cylindrical nanostructures composed of rolled graphene sheets, which can exist in single-walled (SWCNTs) or multi-walled (MWCNTs) forms. Their exceptional electrical conductivity, high surface-to-volume ratio, and remarkable mechanical and chemical stability make them one of the most extensively studied nanomaterials in various fields, including sensors, catalysis, energy storage, and environmental remediation(31). In the context of three-dimensional electrochemical reactors (3D-ECRs), CNTs contribute significantly to the enhancement of pollutant degradation, particularly for persistent organic dyes such as Congo Red. When integrated into the particle electrode matrix or used as surface coatings on support materials, CNTs serve multiple roles: they facilitate efficient electron transfer across the system, provide a large number of electroactive sites, and exhibit high adsorption capacity for organic molecules(32). One of the key features of CNTs is their ability to act as both adsorbents and conductive pathways. Their π–π interactions with aromatic dye molecules enhance dye accumulation near reactive sites, increasing the likelihood of degradation via electrochemical or radical-based mechanisms. Moreover, their porous structure promotes mass transport and allows better access to reactive species, including hydroxyl radicals and superoxide ions generated within the reactor. Functionalization of CNTs by introducing oxygen-containing groups, doping with metals, or combining with other nanomaterials further enhances their dispersion in aqueous media and improves their electrochemical activity(33, 34). A comparison of conductive nanomaterials in three-dimensional electrochemical reactors for Congo red removal is shown in Table 1. Several studies have reported that CNT-modified electrodes demonstrate improved dye removal efficiencies, reduced energy consumption, and greater long-term operational stability compared to traditional carbon-based materials. CNTs also pose some challenges. Their aggregation tendency in solution can reduce effective surface area unless they are properly functionalized. Additionally, concerns about their potential toxicity and environmental persistence, especially in large-scale applications, have been raised and require further study. Despite these limitations, the unique combination of adsorption capacity, electrical conductivity, and structural stability positions CNTs as a leading choice for enhancing the performance of 3D electrochemical systems. Their inclusion in hybrid electrodes alongside materials such as MXene or ITO may further improve dye degradation efficiency while balancing cost, performance, and environmental impact(34).

Table1. Technical Comparison of Conductive Nanomaterials in 3D Electrochemical Reactors for Congo Red Removal

Technical Parameter Nanomaterial	**MXene**	**ITO**	**CNTs**
Base chemical structure	Ti_3C_2Tx (layered with surface functional groups)	$In_2O_3 + SnO_2$ (semiconducting oxide)	Single-/multi-walled tubular graphene
Conductivity type	Metallic to semi-metallic	Semiconducting with metallic behavior	π–π conductive with high electron mobility
Role in dye removal	Adsorption + direct/indirect oxidation	Facilitates electron transfer and ROS generation	Efficient adsorption + fast charge transfer + auxiliary catalysis
Impact on dye removal efficiency	Significant enhancement (>90% under optimal conditions)	Moderate, more effective when combined	Marked improvement, especially in hybrid systems
Reactive oxygen species generated	$^{\bullet}OH$, $O_2^{\bullet-}$, surface-mediated intermediates	H_2O_2, occasionally $^{\bullet}OH$	Boosted $^{\bullet}OH$ generation with metal/oxide presence
Behavior across pH range	Stable in neutral/acidic	Stable across broad pH	Depends on surface functionalization
Operational stability	Relatively high; prone to restacking	High; long-term stable	Moderate; prone to agglomeration unless functionalized
Challenges/limitations	Oxidation in aqueous phase; costly	High cost; limited catalytic activity	Environmental concerns; requires functionalization
Compatibility with other materials	CNTs, graphene, conductive polymers	Activated carbon, CNTs	Metals (Fe, Cu), TiO_2, MXene
Industrial scalability potential	Moderate; emerging	Low; cost-limited	High, if safety and control ensured

4. Application of Conductive Nanomaterials in the Degradation of Congo Red Dye

In recent years, advanced oxidation processes, particularly three-dimensional electrochemical systems (3D-ECRs), have emerged as promising alternatives for treating industrial effluents containing highly resistant dyes. What distinguishes these systems from conventional treatment methods is their ability to generate highly reactive oxidative conditions without relying on biological or chemical catalysts. However, the structural complexity of compounds like Congo Red characterized by extended conjugated aromatic systems and azo linkages often poses challenges for complete degradation through electrochemical oxidation alone(35). Conductive nanomaterials have gained significant attention as next-generation performance enhancers in electrochemical reactors. Unlike bulk materials or traditional electrode surfaces, nanomaterials such as MXene, ITO, and CNTs offer a combination of high surface area, excellent electrical conductivity, and specific molecular interactions that enable more efficient degradation pathways for organic dyes. Generally,Their integration into 3D-ECRs not only facilitates indirect oxidation mechanisms such as the in situ generation of hydroxyl radicals ($^{\bullet}OH$) and other reactive oxygen species but also improves dye adsorption, enhances electron transfer, and reduces internal resistance within the system. Moreover, uniform dispersion of these nanomaterials on conductive substrates or within hybrid particle electrodes contributes to increased operational stability and minimizes performance loss under variable treatment conditions. Empirical data and literature reviews consistently highlight that both individual and combined applications of these nanomaterials can significantly enhance the degradation efficiency of Congo Red. In particular, hybrid or multiphase electrode configurations incorporating combinations of MXene, ITO, and CNTs have demonstrated synergistic effects, leading to faster, more stable, and energy-efficient dye removal. The degradation pathway of Congo Red in a 3D electrochemical system involves both direct and indirect oxidation mechanisms. Initially, hydroxyl radicals ($\bullet OH$) and superoxide anions ($O_2\bullet^-$), generated at the electrode surface, attack the azo bonds (–N=N–) of the dye, leading to their cleavage. This results in the formation of aromatic amine intermediates, which undergo further oxidative degradation through ring-opening reactions. These reactions ultimately lead to the formation of simpler and non-toxic compounds such as carboxylic acids, ammonium (NH_4^+), nitrate (NO_3^-), and carbon dioxide (CO_2). This multi-step mineralization confirms the efficiency of the 3D electrochemical process combined with conductive nanomaterials in breaking down complex dye molecules. The complete degradation sequence and transformation of Congo Red into harmless end products are illustrated in Figure 1.

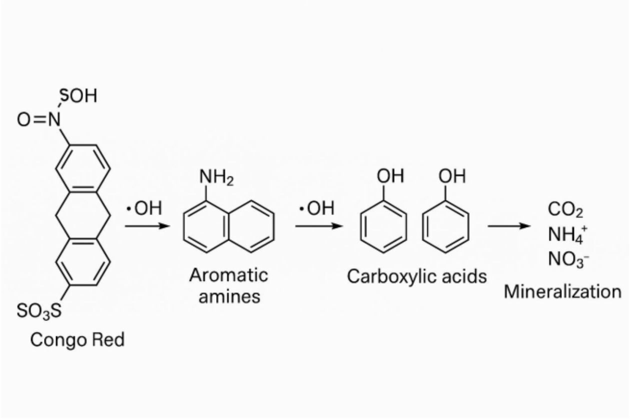

Fig 1. Proposed degradation pathway of Congo Red dye in the presence of reactive oxygen species (ROS) generated in the 3D electrochemical system using conductive nanomaterials.

4.1 Comparative Performance in Previous Studies

Numerous studies have investigated the integration of conductive nanomaterials into electrochemical systems for the degradation of Congo Red and other azo dyes. The comparative performance of MXene, ITO, and CNTs has been evaluated under varying configurations, reactor types, and operational conditions. While the outcomes vary depending on experimental design, several trends are consistently observed .MXenes, particularly Ti_3C_2Tx, have demonstrated superior degradation efficiency in multiple studies, often achieving over 90% removal within short reaction times(36, 37). Their layered structure and surface terminations provide abundant active sites for redox reactions, while their intrinsic conductivity ensures rapid electron transfer. MXenes also contribute to the generation of hydroxyl radicals and other reactive species, enhancing oxidative degradation pathways. CNTs have been extensively applied due to their high surface area, strong π–π interactions with aromatic dye molecules, and exceptional electron mobility. Their dual functionality as both adsorbents and electron conductors makes them particularly effective in hybrid systems. When combined with metal oxides or MXene, CNT-based electrodes have shown enhanced performance, particularly in dye-laden wastewater with complex matrices(38). ITO, though less frequently used as a primary catalytic material, plays a critical supporting role in many electrochemical configurations. Its high electrochemical stability, optical transparency (when relevant), and compatibility with carbon-based supports make it a valuable component in composite electrodes. However, its standalone catalytic performance in Congo Red degradation is typically lower compared to MXene or CNTs(39). Comparative evaluations across different studies suggest that hybrid systems incorporating two or more of these nanomaterials often yield the most efficient and stable results. For example, CNT/MXene composites have shown not only high dye removal rates but also improved operational durability and energy efficiency. Such findings support the notion that material synergy, rather than single-component superiority, is key to optimizing electrochemical degradation systems for industrial applications.

4.2 Influence of Operational Conditions

The efficiency of conductive nanomaterials in three-dimensional electrochemical reactors (3D-ECRs) for Congo Red degradation is strongly influenced by operational parameters. These parameters not only determine the rate of degradation but also affect the stability, reactivity, and interaction of nanomaterials within the system. Proper optimization of these conditions is essential to fully exploit the advantages offered by materials such as MXene, ITO, and CNTs(40).

- **pH of the Solution**

The pH of the solution is a crucial factor that influences both the electrochemical activity of the electrodes and the ionization state of the dye molecules. In mildly acidic conditions (typically pH 4–6), the production of hydroxyl radicals ($^{\bullet}OH$) is enhanced, improving the degradation rate. MXene-based systems generally perform well in this range but may degrade under highly alkaline conditions. CNTs and ITO exhibit greater stability across a wider pH range, though extremely low or high pH may still alter their surface properties or catalytic performance.

- **Current Density**

Current density directly affects the generation of reactive oxidative species at the electrode surfaces. While higher current densities can increase the production of species like $\bullet OH$ and $O_2\bullet^-$, they may also lead to side reactions such as oxygen evolution, which decreases system efficiency. Optimal performance is usually achieved in the range of 10–30 mA/cm^2. Excessive current may also lead to thermal degradation of nanomaterials or unnecessary energy consumption.

- **Reaction Time (Retention Time)**

The contact time between the dye and the electroactive system is another key parameter. With nanomaterial-enhanced electrodes, high degradation efficiency can be achieved in relatively short periods (15–60 minutes), depending on dye concentration, electrode configuration, and flow dynamics. Overextended treatment times may not significantly improve removal but will increase operational costs.

- **Nanomaterial Loading and Distribution**

The amount and method of nanomaterial integration into the electrode system significantly impact performance. Insufficient loading leads to limited electroactive sites, while excessive loading can cause aggregation and reduced surface availability. Uniform dispersion either through coating, impregnation, or composite fabrication is essential for maintaining high electrochemical activity and system stability.

- **Temperature and Conductivity of the Medium**

Although not always reported, temperature and ionic strength of the solution also affect degradation efficiency. Moderate temperatures (25–40°C) typically improve kinetics without compromising nanomaterial integrity. Sufficient ionic conductivity in the electrolyte enhances charge transport, particularly when using low-conductivity nanomaterials .Overall, operational parameters such as solution pH, current density, initial dye concentration, and reaction time play a crucial role in determining the performance of conductive nanomaterials in three-dimensional electrochemical reactors. Variations in these conditions can significantly influence the degradation rate of Congo Red, as well as the structural stability and reactivity of nanomaterials like MXene, ITO, and CNTs. Proper optimization of these parameters is essential to fully harness the unique advantages of these materials, leading to a more efficient, rapid, and environmentally friendly degradation process(41-43). A comparative evaluation of conductive nanomaterials in 3D electrochemical reactors for azo dye removal is presented in Table 2.

Table2. Comparative Evaluation of Conductive Nanomaterials in 3D Electrochemical Reactors for Azo Dye Removal

Parameter / Property	**MXene ($Ti_3C_2T_x$)**	**ITO (Indium Tin Oxide)**	**CNTs (Carbon Nanotubes)**
Nanostructure	2D – Layered	0D – Nanoparticles	1D – Tubular
Role in 3D Reactor	Active electrode, filler	Support coating, secondary electrode	Porous filler, conductivity enhancer
Function in 3D Architecture	Charge transfer, surface area boost	Chemical stability support	Adsorption and electron pathway
Electrical Conductivity (S/cm)	$\sim10^4$	$\sim10^2$	$\sim10^3$
Charge Transfer Resistance (Rct, Ω)	12.4	25.6	9.8
Onset Potential (E_onset, V)	0.45	0.62	0.38
Rate Constant (k_CR, min^{-1})	0.083	0.048	0.077
Azo Dye Removal Efficiency (Congo Red)	94–97%	70–85%	90–95%
Effective Removal Time (min)	25–35	40–50	30–40
Optimal pH Range	5–7	6–9	5–8
Dominant Kinetic Model	Pseudo-1st Order	Pseudo-1st Order	Pseudo-2nd Order
Removal Mechanism	ROS + Adsorption + Charge Transfer	Direct oxidation	π–π adsorption + ROS
Advantages	High conductivity, ROS-active, hybrid-capable	Stable, corrosion-resistant	High surface area, strong adsorption
Limitations	Restacking, oxidation in water	Low surface area, high cost	Aggregation, environmental concerns
Compatibility with Composites	High (polymers, CNTs, TiO_2)	Low	High (MXene, ZnO, metal oxides)
Scalability Potential	Moderate	Low	Moderate to high

4.3 Removal Efficiency, Advantages, and Limitations

The practical applicability of conductive nanomaterials in dye degradation systems is largely determined by their removal efficiency, operational advantages, and intrinsic limitations. While MXene, ITO, and CNTs each show promising performance in 3D electrochemical systems, their efficiency and scalability depend on multiple physicochemical and structural factors.

- **MXene ($Ti_3C_2T_x$)**

MXenes have demonstrated exceptional dye removal capabilities, with removal efficiencies often exceeding 90% under optimized conditions. Their high conductivity, layered structure, and abundance of surface terminations ($^-$OH, $^-$O, $^-$F) facilitate rapid electron transfer and generation of reactive oxygen species. However, MXenes are prone to restacking, which reduces available surface area and limits their long-term activity. Moreover, they are susceptible to oxidation in aqueous environments, especially under highly oxidative conditions, which can degrade their structure over time. Despite these limitations, MXene-based systems show strong potential when used in hybrid configurations with other materials such as CNTs or polymers to enhance dispersion and stability(44).

- **Indium Tin Oxide (ITO)**

ITO exhibits good electrochemical stability and moderate conductivity, with reported dye removal efficiencies ranging from 70% to 85% in most studies. Its major advantage lies in its long-term stability, corrosion resistance, and low fouling tendency. ITO also performs reliably across a wide pH range and under varying current densities. However, its relatively low surface area and catalytic activity make it less effective in standalone systems. Additionally, indium is a rare and expensive element, limiting the cost-effectiveness and scalability of ITO-based systems for large-scale wastewater treatment(45).

- **Carbon Nanotubes (CNTs)**

CNTs are widely used due to their exceptional conductivity, large surface area, and strong adsorption capability for aromatic dye molecules via π–π interactions. Their removal efficiency often reaches over 90% when used alone or in combination with metal oxides or MXene. CNTs not only enhance electron transport but also increase the local concentration of dye molecules near electroactive sites. However, they tend to agglomerate in aqueous environments unless properly functionalized, which reduces their effectiveness. Furthermore, there are ongoing concerns regarding the environmental safety and potential toxicity of CNTs, particularly if released into the ecosystem(46).

4.4 Structural Breakdown and Possible Degradation Mechanisms

The successful degradation of azo dyes such as Congo Red in nanomaterial-assisted three-dimensional electrochemical systems relies on a combination of physical interactions and chemical reactions that work synergistically to break down the dye's molecular structure. Congo Red, characterized by two azo (-N=N-) linkages connected to aromatic rings and sulfonate groups, exhibits high chemical stability and resistance to conventional degradation methods, making its removal from wastewater particularly challenging. Within advanced electrochemical systems, degradation typically proceeds through both direct and indirect oxidation pathways. In direct oxidation, electron transfer occurs between the dye molecule and the active electrode surface. In contrast, indirect oxidation involves the in situ generation of reactive oxygen species (ROS), such as hydroxyl radicals ($^{\bullet}OH$) and superoxide anions ($O_2^{\bullet-}$), which initiate non-selective attacks on the dye structure. Conductive nanomaterials such as MXene, ITO, and CNTs enhance these mechanisms by increasing the reactive surface area, improving charge transfer, and facilitating ROS production. Additionally, their high adsorption capacity allows for the localized concentration of dye molecules near active sites, further accelerating degradation. Literature reports based on spectroscopic and chromatographic analyses suggest a sequential breakdown process. The initial cleavage of azo bonds leads to the formation of aromatic amine intermediates. These compounds undergo further oxidation and ring-opening reactions, forming carboxylic acids and, ultimately, mineralizing into CO_2, NH_4^+, and other harmless end products. The efficiency and completeness of this transformation are strongly influenced by the type of nanomaterial employed and the operational conditions, including pH, current density, and contact time. Overall, the incorporation of conductive nanomaterials not only improves the rate of dye removal but also plays a critical role in directing the degradation pathway toward more effective and complete mineralization something that conventional systems often fail to achieve(46-49).

Conclusion

This review examined the potential of conductive nanomaterials MXene, ITO, and CNTs in enhancing the degradation of Congo Red dye in three-dimensional electrochemical reactors (3D-ECRs). These nanomaterials have demonstrated significant improvements in dye removal efficiency by promoting electron transfer, generating reactive oxygen species (ROS), and increasing surface area for adsorption. MXenes showed excellent conductivity and redox properties, resulting in high degradation efficiency. CNTs, with their large surface area and strong adsorption capacity, also exhibited high performance, although they are prone to agglomeration. ITO, while not as catalytic, contributed to system stability, particularly in hybrid systems. Overall, hybrid systems combining MXene, CNTs, and ITO showed the best performance in terms of efficiency, stability, and sustainability. Despite these advancements, challenges such as cost, long-term stability, and potential environmental concerns remain. conductive nanomaterials, especially in hybrid configurations, provide an effective approach for electrochemical wastewater treatment, offering significant potential for large-scale industrial applications.

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