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Fe_3O_4 Functionalized Multi-Walled Carbon Nanotubes: Synthesis, Characterization, and Investigation for Photocatalysis Activity of CO_2 Conversion to Methanol

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ABSTRACT

The synthesis, characterization, and photocatalytic performance of Fe_3O_4-functionalized multi-walled carbon nanotubes (MWCNTs) for CO_2 conversion into valuable chemicals such as methanol was investigated. Highlighting the critical environmental issue of rising greenhouse gases, particularly CO_2, the study emphasizes the importance of developing efficient photocatalysts for sustainable energy solutions and environmental remediation. The experimental section details the acid functionalization of MWCNTs, followed by hydrothermal synthesis of Fe_3O_4 nanoparticles on their surface. Characterization techniques including FE-SEM, FT-IR, and XRD confirmed the successful formation of uniform Fe_3O_4 nanoparticles with high crystallinity and surface reactivity. Photocatalytic tests under visible light demonstrated that Fe_3O_4-MWCNT composites exhibit superior activity, achieving a methanol formation rate of 0.171 mmol g^{-1} h^{-1} after 3.8 hours, significantly higher than pure MWCNTs and Fe_3O_4 alone. The results suggest that the synergistic effects between Fe_3O_4 nanoparticles and MWCNTs enhance charge separation and transfer, leading to improved catalytic efficiency. The study concludes that Fe_3O_4-functionalized MWCNTs present a promising platform for scalable CO_2 reduction, with future challenges focused on improving stability, selectivity, and synthesis uniformity. Advances in nanostructure engineering, hybrid material design, and mechanistic understanding are essential for optimizing these nanocomposites for practical environmental and energy applications, contributing to the global effort against climate change and for renewable energy development.

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INTRODUCTION

Excessive emission of gases such as carbon dioxide (CO_2), a major greenhouse contributor, is widely recognized as the primary driver of global climate change [1, 2]. It is estimated that a doubling of atmospheric CO_2 levels could lead to a global temperature increase of approximately 3 °C, with a possible uncertainty range of 2 to 4.5 °C [3, 4]. Such changes would likely result in extreme weather events, land desertification, rising sea levels, and widespread extinction of amphibian species due to epidemic diseases [5-8]. Consequently, it is critical to effectively reduce atmospheric CO_2 concentrations [9, 10]. Additionally, as an integral component of the carbon cycle, CO_2 can be harnessed as a raw material for synthesizing valuable chemicals, making the development of efficient methods for CO_2 conversion both environmentally and economically significant. Furthermore, organic pollutants like phenol pose another serious environmental threat to water bodies, owing to their high toxicity and resistance to biodegradation. Therefore, there is a pressing need to establish innovative, environmentally friendly, and sustainable technologies for both CO_2 reduction and phenol degradation, supporting the long-term progress of human society [11-13].

Photocatalysts are materials that facilitate chemical reactions upon exposure to light, often by generating electron-hole pairs that drive redox processes [14-16]. They are essential in harnessing solar energy for environmentally friendly applications. In environmental remediation, photocatalysts are widely used for the degradation of pollutants, water purification, and air cleaning, contributing significantly to reduction of harmful organic compounds and toxic gases, thus playing a vital role in promoting sustainable and eco-friendly solutions for environmental protection [17, 18].

Recent progress in photocatalysts for CO_2 conversion has garnered significant attention due to the urgent need for sustainable solutions to mitigate climate change and produce renewable fuels [19, 20]. A variety of photocatalytic materials have been investigated, including metal oxides such as TiO_2 [21, 22], Fe_2O_3 [23], and WO_3 [24, 25], which offer stability and versatile catalytic properties. Additionally, metal sulfides like MoS_2 [26, 27] and WS_2 [28] have shown promising activity under visible light owing to their narrower bandgaps. Carbon-based materials, including graphitic carbon nitride ($g\text{-}C_3N_4$) [29] and

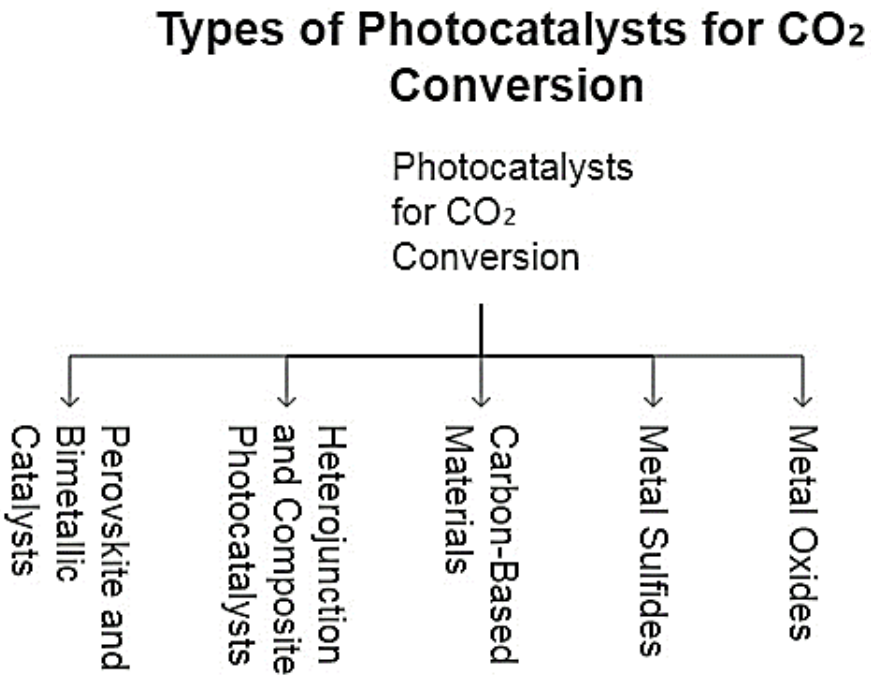

Fig. 1. Types of photocatalysts for CO_2 conversion.

graphene derivatives [30], are widely studied for their excellent electrical conductivity and large surface areas, which facilitate efficient charge transfer. Hybrid composites, combining different materials such as metal oxides with carbon nanostructures or co-catalysts, are also being developed to optimize light harvesting and charge separation. Strategies such as doping with non-metallic elements (e.g., nitrogen, phosphorus), creating heterojunction structures, and designing nanostructured architectures have been implemented to extend light absorption into the visible range, suppress recombination of photo-generated charge carriers, and enhance catalytic activity. Recent research emphasizes the importance of fine-tuning the surface chemistry and electronic properties of these materials to improve conversion efficiency, selectivity, and stability under operational conditions. These advancements are crucial for scaling up photocatalytic CO_2 reduction technologies and integrating them into renewable energy systems, representing a significant step toward sustainable carbon utilization and environmental remediation [31]. Fig. 1 shows types of photocatalysts for CO_2 conversion.

Functionalized multi-walled carbon nanotubes (MWCNTs) have gained considerable attention as promising photocatalysts for environmental applications, including CO_2 reduction [32, 33]. Their unique structural features, such as high surface area, excellent electrical conductivity, and chemical stability, make them ideal supports or active components in photocatalytic systems [34, 35]. Functionalization of MWCNTs, typically through the introduction of oxygen-containing groups or doping with heteroatoms, enhances their dispersibility, surface reactivity, and interaction with light-absorbing materials [36-38]. Recent advancements include combining functionalized MWCNTs with semiconductor nanoparticles like TiO_2 [39], g-C_3N_4 [40], or metal sulfides to form hybrid composites that exhibit synergistic effects, improving charge separation and transfer efficiency. These modifications have led to increased photocatalytic activity under visible light and improved stability, which are crucial for practical CO_2 reduction processes. Additionally, functionalized MWCNTs can serve as electron sinks, facilitating electron transport and reducing recombination of charge carriers. The ongoing research focuses on optimizing functionalization techniques, controlling nanostructures, and enhancing light absorption to develop highly efficient and sustainable photocatalytic systems for CO_2 conversion and other environmental applications. In recent years, carbon-based semiconductor photocatalysts have garnered significant interest due to their potential to enhance photocatalytic efficiency [41]. Materials such as graphene and carbon nanotubes (CNTs) have been shown to serve as effective electron acceptors, facilitating charge transfer and significantly reducing the recombination rate of electron-hole pairs within composite systems [42]. This improvement in charge separation directly contributes to increased photocatalytic activity [43]. Although graphene oxide (GO) and CNTs possess inherently wide bandgaps, functionalized versions of these materials expand their applicability to processes like converting CO_2 into methanol [44]. Such systems enable the simultaneous harvesting of solar energy and reduction of CO_2, making its promising candidates for sustainable energy conversion and environmental remediation technologies. This study focuses on the synthesis and characterization of Fe_3O_4-functionalized multi-walled carbon nanotubes, and investigates their photocatalytic performance in the conversion of CO_2 under visible light irradiation.

MATERIALS AND METHODS

Materials and apparatus

MWCNTs with diameters ranging from 10 to 20 nm, purity of 97%, and a specific surface area between 100 and 160 m^2/g were supplied by Shenzhen Nanotech Port Co., China. All chemicals employed in this research were of analytical grade and utilized in their purchased form without any additional purification. Deionized water was used exclusively throughout all experimental procedures. In this study, the morphological and structural characterizations were carried out using a JEOL JSM-7600F scanning electron microscope (SEM) manufactured by JEOL Ltd., Japan, which offers a high-resolution imaging capability with magnifications up to 1,000,000 times. X-ray diffraction (XRD) patterns were obtained using a PANalytical X'Pert PRO Powder diffractometer from PANalytical, Netherlands, employing Cu Kα radiation (λ = 1.5406 Å) in the 2θ scanning mode to analyze the crystallinity and phase composition of the samples. Additionally, Fourier Transform

Infrared (FT-IR) spectra were recorded on a Thermo Scientific Nicolet iS10 spectrometer, which covers a spectral range of 400–4000 cm^{-1} and features an Attenuated Total Reflectance (ATR) accessory for straightforward sample analysis.

Preparation of Fe_3O_4 functionalized MWCNTs

To enhance their purity and catalytic activity, the MWCNTs were subjected to an ultrasonic treatment in a mixture of concentrated acids (H_2SO_4 and HNO_3 in a 3:1 ratio) for 4 hours at 65 °C. Following this process, the solution was diluted with deionized water, filtered through a 0.45 µm membrane to obtain a neutral pH, and then dried under vacuum for 12 hours. The MWCNTs/Fe_3O_4 hybrids were synthesized using a hydrothermal approach. In brief, $FeCl_3$ and $FeSO_4$ were mixed in a molar ratio of 2:1 along with 0.5 g of acid-treated MWCNTs, all dispersed in 400 mL of deionized water through ultra-sonication. The mixture was stirred under nitrogen atmosphere at 50°C for half an hour, then further stirred at 65 °C for an additional hour while maintaining the pH above 12. The resulting precipitates were filtered, rinsed with deionized water until neutral, dried, and ground into a fine powder.

Fe_3O_4 functionalized MWCNTs as photocatalyst for CO_2 conversion

The reduction of CO_2 via photocatalysis was carried out at room temperature (25 ± 5°C) in a continuous gas flow reactor. The reactor, constructed from stainless steel and covered with quartz glass, had a volume of 300 mL, with dimensions of 12 cm by 5 cm. A sample dish containing 0.3 g of the catalyst powder was positioned at the center of the reactor. A 300 W commercial halogen lamp served as the simulated solar light source, placed vertically outside the reactor directly above the sample. To prevent temperature elevation within the system, two small fans were mounted around the lamp. The catalyst powder was spread on a glass disc measuring 7.5 cm in diameter. The system was initially purged with nitrogen gas to eliminate ambient air, followed by purging with CO_2 for an

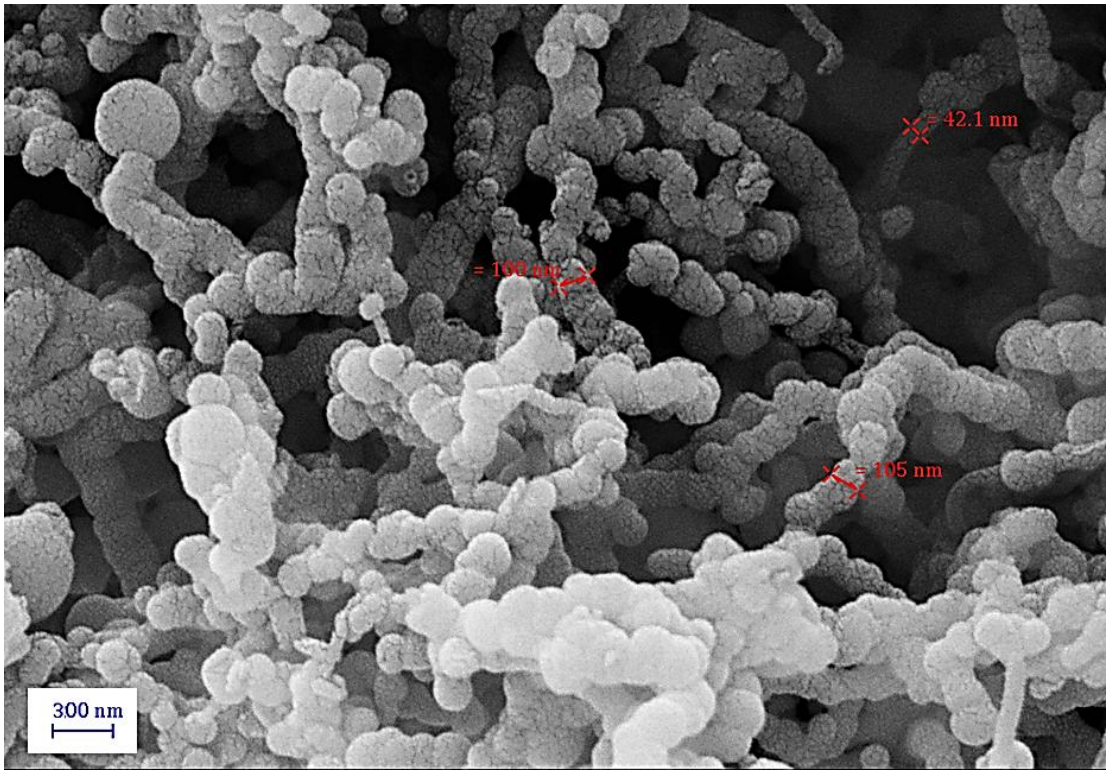

Fig. 2. FE-SEM of Fe_3O_4 functionalized MWCNTs.

additional hour, with the flow rate maintained at 5 sccm. CO_2 was passed through water to regulate the humidity level throughout the experiment. After one hour of gas flow and equilibrium stabilization, the halogen lamp was turned on to initiate the photocatalytic reaction. The concentration of methanol produced was monitored continuously in the vapor phase using gas chromatography with a flame ionization detector (GC-FID).

RESULTS AND DISCUSSION

Preparation and Characterization of Fe_3O_4 functionalized MWCNTs

The synthesis of Fe_3O_4/MWCNTs nanocomposites was successfully achieved through a hydrothermal process, ensuring uniform integration of Fe_3O_4 nanoparticles onto the acid-treated MWCNTs. Ultra-sonication facilitated thorough dispersion of precursor ions, while controlled pH and temperature conditions promoted the nucleation and growth of Fe_3O_4 particles directly on the nanotube surface. This method resulted in well-deposited, crystalline Fe_3O_4 nanoparticles, which significantly enhance the composite's magnetic and catalytic properties. The synthesis approach effectively combines the high surface area of MWCNTs with the magnetic features of Fe_3O_4, making the nanocomposite suitable for advanced catalytic and environmental applications.

The surface morphology of the Fe_3O_4-functionalized MWCNTs was examined using FE-SEM, as depicted in Fig. 2. The images reveal numerous Fe_3O_4 nanoparticles distributed across the MWCNT surface. Due to magnetic interactions, these nanoparticles tend to aggregate and form clusters.

Fig. 3 displays the FT-IR spectra of raw MWCNTs, purified MWCNTs, and Fe_3O_4 functionalized MWCNTs. The appearance of new absorption bands at 3435 cm^{-1} and 3426 cm^{-1} in the purified MWCNTs and the Fe_3O_4 functionalized MWCNTs, respectively, corresponds to O–H stretching vibrations, indicating that the acid treatment

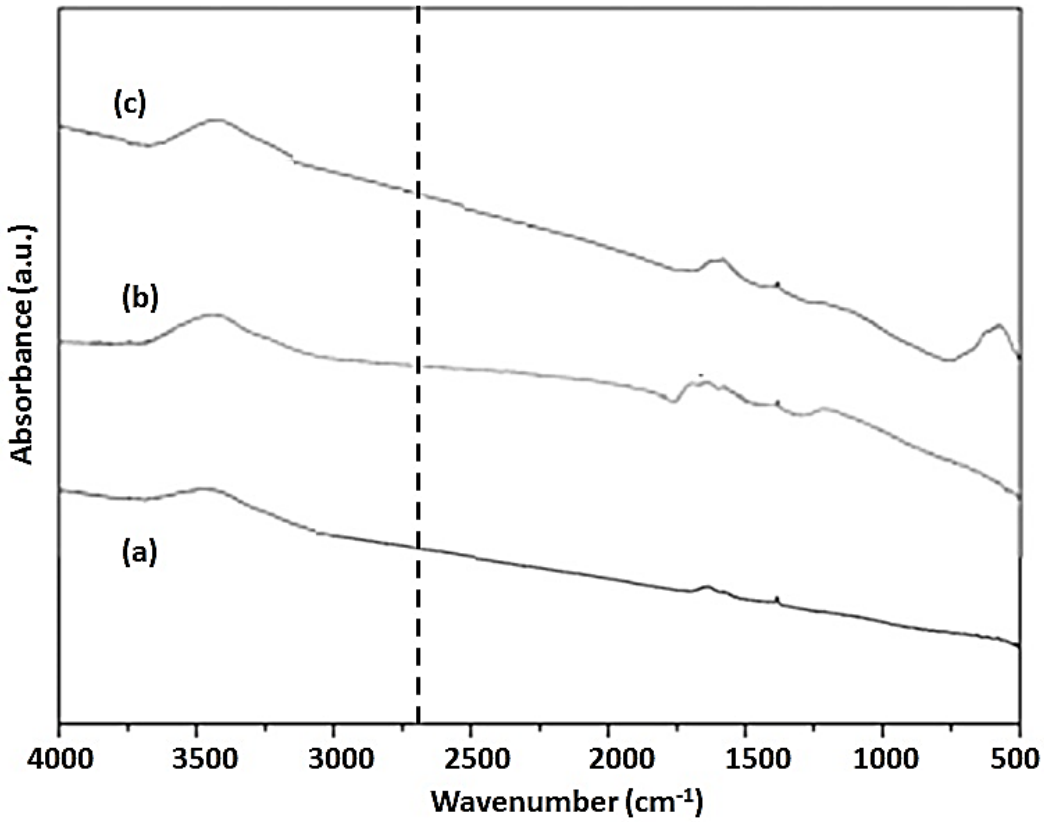

Fig. 3. FT-IR spectra of a) raw MWCNT b) purified MWCNT, c) Fe_3O_4 functionalized MWCNTs.

introduced hydroxyl groups onto the MWCNTs surface [45, 46]. Additionally, peaks near 1597 cm^{-1} are associated with vibrations of –COOH groups and carbon carbon double bonds. The notably stronger peaks at this region for both acid-treated MWCNTs and the Fe_3O_4 functionalized MWCNTs suggest successful functionalization with carboxyl groups. Furthermore, a broad hump at 571 cm^{-1} in the spectrum of Fe_3O_4 functionalized MWCNTs is attributed to Fe–O stretching vibrations, confirming the presence of Fe_3O_4 nanoparticles on the MWCNTs surface [47], which aligns well with the XRD data presented later.

The XRD patterns of the Fe_3O_4/MWCNTs composites are presented in Fig. 4. The peak observed at 26.15 ° corresponds to the diffraction from the MWCNTs, confirming their structural stability after synthesis. Additionally, the diffraction peaks at 30.16 ° (220), 35.56 ° (311), 43.26 ° (400), 53.76 ° (422), 57.36 ° (511), and 62.86 ° (440) can be indexed to the crystal planes of cubic Fe_3O_4, according to JCPDS card No. 89-0691. The absence of extraneous peaks indicates that the Fe_3O_4 phase present is highly pure [48].

Photocatalytic study of Fe_3O_4/MWCNTs composites for CO_2 conversion

Based on the method outlined in the Experimental section, the rate of methanol (MeOH) production, denoted as R_{MeOH} (mmol g^{-1} h^{-1}), was computed and depicted in Fig. 5 as a function of reaction time. The catalytic activity for methanol synthesis followed the trend: Fe_3O_4-MWCNTs surpassed MWCNTs, which in turn were more active than Fe_3O_4 alone. Fe_3O_4-MWCNTs exhibited an excellent methanol production rate of 0.171 mmol g^{-1} h^{-1} after 3.8 hours, demonstrating good stability over the course of the reaction. In contrast, the rates for pure MWCNT and Fe_3O_4 samples initially increased rapidly, reaching maximum values of 0.111 and 0.090 mmol g^{-1} h^{-1}, respectively, before plateauing after 1.9 hours of operation. The overall methanol formation rate in these samples remained significantly lower, at just 0.029 mmol g^{-1} h^{-1}. Overall, the Fe_3O_4-MWCNTs nanocomposite demonstrates the greatest

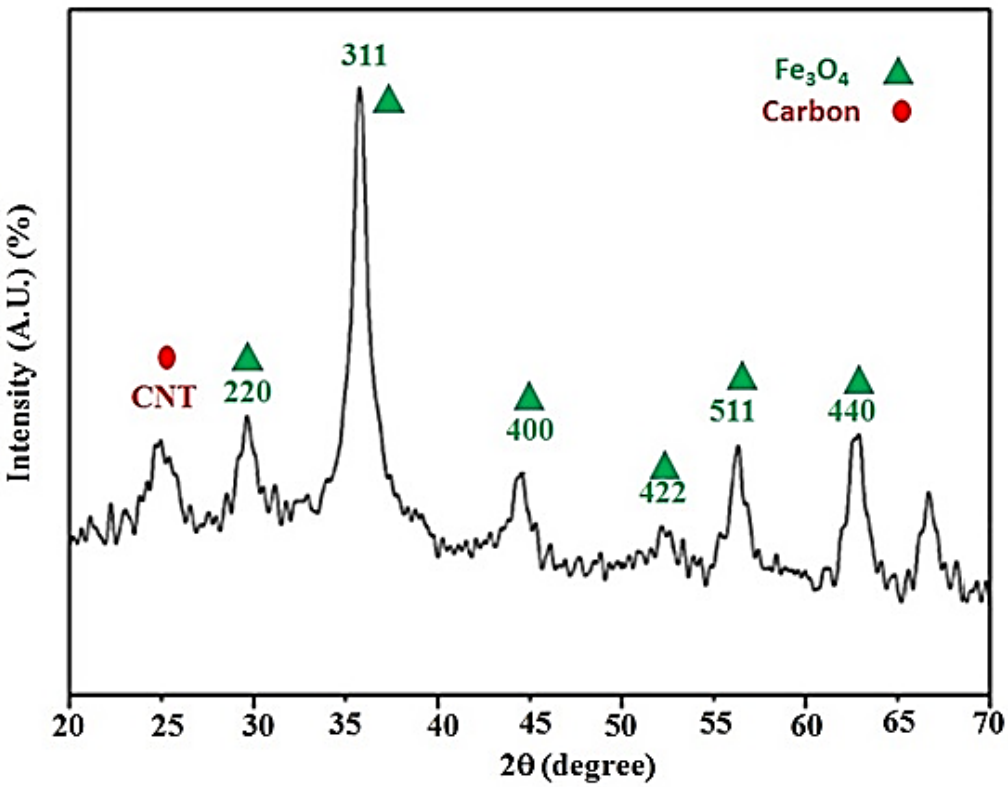

Fig. 4. XRD pattern of Fe_3O_4/MWCNTs composites.

effectiveness as a photocatalyst for CO_2 reduction among all tested samples.

The development of Fe_3O_4-functionalized multi-walled carbon nanotubes (MWCNTs) for photocatalytic CO_2 conversion holds significant promise for sustainable energy and environmental remediation. However, several challenges must be addressed to realize their full potential. One of the primary obstacles is achieving high efficiency and selectivity in CO_2 reduction, as current catalysts often produce a mixture of products and require precise control over reaction conditions. Enhancing the stability and durability of these hybrid materials under operational conditions is also critical, as prolonged exposure to light, heat, and reactive intermediates can lead to catalyst degradation. Additionally, optimizing the synthesis methods to produce uniform, defect-free Fe_3O_4 nanoparticles on MWCNTs remains a challenge, as inconsistencies can negatively impact catalytic performance. Future research should focus on tailoring the electronic structure and surface properties of these nanocomposites to improve charge separation and transfer, thereby increasing catalytic efficiency. Incorporating co-catalysts or doping elements to modify the bandgap and improve light absorption could further enhance photocatalytic activity. Advances in nanofabrication techniques, such as controlled wet-chemical synthesis and atomic-level engineering, will be essential for producing next-generation materials with optimized surface active sites. Alongside experimental efforts, computational modeling can provide deeper insights into reaction mechanisms and guide the design of more effective catalysts. Ultimately, the integration of these functionalized nanomaterials into scalable, cost-effective systems for large-scale CO_2 conversion remains a key future goal, aligning with global efforts to mitigate climate change and develop sustainable energy

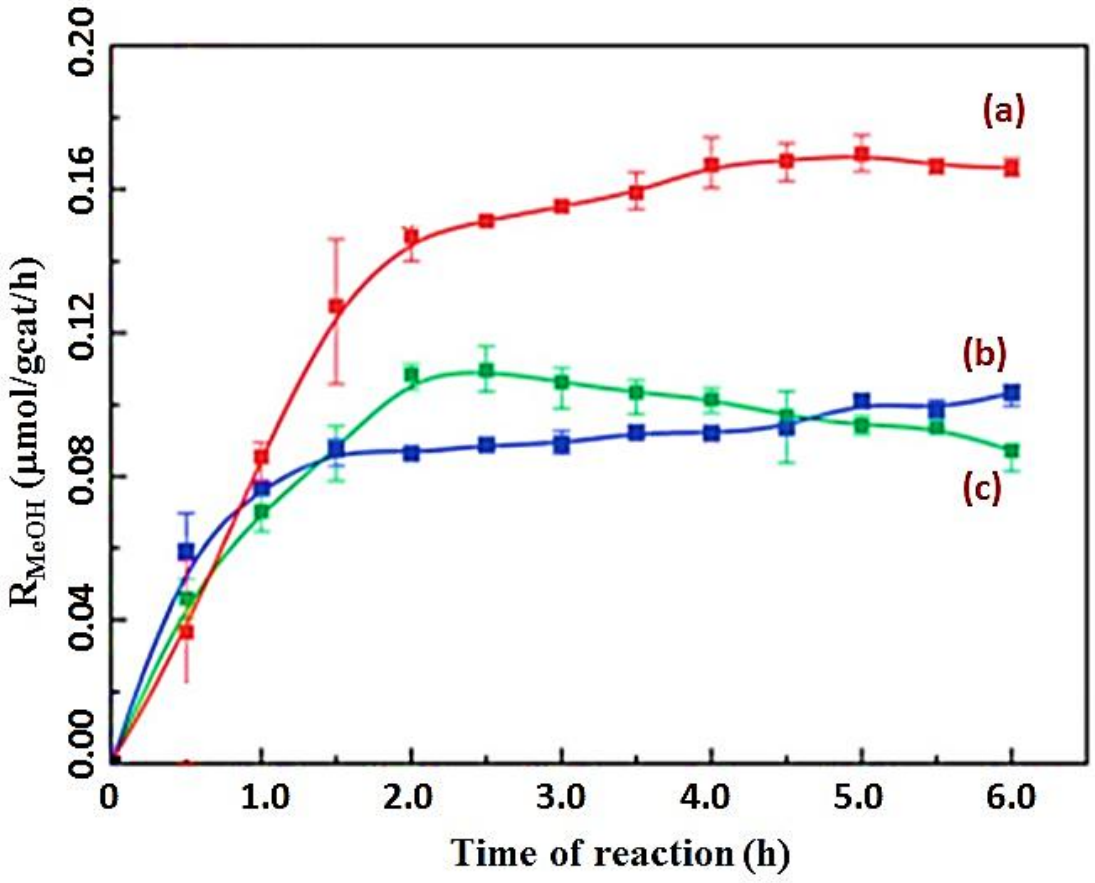

Fig. 5. Application of a) Fe_3O_4-MWCNTs nanocomposite b) MWCNT, and c) Fe_3O_4 for CO_2 conversion.

solutions [49, 50].

CONCLUSION

In summary, the study successfully demonstrated the synthesis of Fe_3O_4-functionalized MWCNTs through a hydrothermal process, with FE-SEM and FT-IR confirming the uniform distribution of Fe_3O_4 nanoparticles averaging approximately 15–20 nm in size on the MWCNT surface. XRD analysis revealed high crystallinity of the Fe_3O_4 phase, supporting their effective integration. Photocatalytic experiments under visible light showed that the Fe_3O_4-MWCNT composite achieved a methanol production rate of 0.171 mmol g^{-1} h^{-1} after 3.8 hours, outperforming pure MWCNTs (0.110 mmol g^{-1} h^{-1}) and Fe_3O_4 nanoparticles alone (0.089 mmol g^{-1} h^{-1}). The composite displayed remarkable stability over five consecutive cycles with only a marginal decrease of 5%, indicating its potential for repeated use. These results underscore the synergistic effect between Fe_3O_4 and MWCNTs, which facilitates enhanced charge separation and transfer, leading to improved CO_2 reduction efficiency. Future work should focus on optimizing nanoparticle size and distribution, as well as long-term stability, to advance this promising photocatalyst for practical applications in carbon capture and sustainable fuel production.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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