



Research Article

Enhanced photocatalytic degradation of perfluorooctanoic acid (PFOA) by using MoS₂/GO/CMC composites: Impact of irradiation conditions, solution pH, and stability for sustainable water treatment

Syafarina Farisa Sateria¹, Kavirajaa Pandian Sambasevam^{2,3}, Ahmad Husaini Mohamed¹, Zulhatiqah Zolekafeli¹, and Siti Nor Atika Baharin^{1,2*}

¹School of Chemistry and Environment, Faculty of Applied Sciences, Universiti Teknologi MARA, Cawangan Negeri Sembilan, Kampus Kuala Pilah, 72000 Kuala Pilah, Malaysia

²Advanced Material for Environmental Remediation (AMER) Research Group, Faculty of Applied Sciences, Universiti Teknologi MARA Cawangan Negeri Sembilan Kampus Kuala Pilah, 72000 Kuala Pilah, Negeri Sembilan, Malaysia

³Electrochemical Material and Sensor (EMas) Group, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia

*Corresponding author: atikabaharin@uitm.edu.my

Received: 29 September 2024; Revised: 7 April 2025; Accepted: 9 April 2025; Published: 1 June 2025

Abstract

This study investigated the removal of perfluorooctanoic acid (PFOA) by using molybdenum disulphide-graphene oxide-carboxymethyl cellulose (MoS₂/GO/CMC) composites as a photocatalyst under various irradiation conditions and solution pH value levels. The performance of MoS₂/GO/CMC was compared with MoS₂/CMC and GO/CMC composites. Under LED light irradiation (12 W, 400–700 nm spectrum range), the MoS₂/GO/CMC composite achieved a maximum PFOA degradation efficiency of 92.26% within 2 h, outperforming other photocatalysts. The incorporation of cellulose improved nanoparticle stability and increased surface-active sites, enhancing degradation efficiency. pH 5 was found to be optimal for PFOA degradation due to favorable hydrophobic interactions, while higher pH levels hindered degradation due to Coulombic repulsion. Increasing LED wattage to 12 W maximised degradation efficiency by enhancing photodecomposition. These findings provide valuable insights into optimising PFOA degradation under different environmental conditions, highlighting the potential of MoS₂/GO/CMC composites for sustainable water treatment solutions.

Keywords: carboxymethyl cellulose, perfluorinated compounds, photocatalyst, graphene oxide, hydrogels

Introduction

Perfluorooctanoic acid (PFOA) belongs to a class of per- and polyfluoroalkyl substances (PFAS), which have been widely used in various industrial and consumer applications due to their exceptional thermal, chemical, and biological stability [1,2,3]. However, these same properties contribute to their persistence in the environment and potential bioaccumulation, raising significant health and ecological concerns [4,5,6]. The strong carbon-fluorine (C-F) bonds make PFOA highly resistant to natural degradation, leading to its widespread detection in water bodies, soil and even human biological [7,8,9]. A study conducted by Mohamad Haron found PFOA concentrations that ranged from 0.31 ng/g to 3693.96 ng/g in household dust from

urban areas, such as Petaling Jaya, Putrajaya and Kuala Lumpur, demonstrated its prevalence in residential environments [10]. Additionally, PFAS, particularly PFOS, have been detected in almost all food categories, with the highest concentrations observed in canned foods (0.18 ng/g – 34.5 ng/g). Alarming, hazard quotient (HQ) values exceeding 1 suggest a significant risk to human health [11,12].

The persistent nature of PFOA necessitates the development of effective remediation techniques to mitigate its environmental and health risks. Several methods have been explored for PFOA removal, including adsorption [13], membrane separation [14], microbial degradation [15] and electrochemical degradation [16]. Among these, photocatalysis has

emerged as a promising and environmentally friendly approach due to its ability to degrade PFOA without secondary pollution [17,18]. In a photocatalytic system, light energy excites the surface electron of photocatalyst, leading to the generation of reactive species that will break down pollutants into less harmful compounds [19].

Amongst various photocatalysts, two-dimensional (2D) semiconductor-based materials have gained attention due to their visible-light activity and high surface-to-volume ratio, enabling efficient charge transfer and light absorption. Transition-metal oxides such as ZnO, TiO₂, WO₃ as well as sulfides like MoS₂ and CdS, have been explored for their photocatalytic applications [20,21,23]. Molybdenum disulfide (MoS₂) has attracted particular interest due to its tunable bandgap, strong visible-light absorption, and high adsorption capacity, making it an excellent candidate for organic pollutant degradation [24]. However, to enhance its photocatalytic efficiency and reduce electron-hole recombination, MoS₂ is often combined with other materials such as carbon particles [25], titanium oxide nanoparticles [26], and zinc oxide [27]. Amongst these, graphene oxide (GO) has emerged as a highly effective co-catalyst due to its high electrical conductivity, large surface area, and ability to act as an electron acceptor, thereby facilitating charge separation and enhancing photocatalytic activity [28]. In this study, MoS₂ was integrated with GO to improve its overall photocatalytic performance.

To address challenges associated with nanoparticle aggregation and recovery, immobilisation within a hydrogel matrix was explored as a strategy for enhancing photocatalyst stability and reusability. Amongst natural polysaccharides, carboxymethyl cellulose (CMC) stands out due to its water solubility, biocompatibility, and ability to form hydrogels with excellent mechanical stability [29-36]. Unlike other polysaccharides such as chitosan and lignin, CMC offers superior dispersibility, which facilitates uniform photocatalyst distribution and enhances availability of surface-active site. Previous studies had demonstrated the successful integration of cellulose-based hydrogels with various nanomaterials, such as nano chitosan/TiO₂ [37], carbon dot/ZnO [38], Ag/AgCl [39], and polyaniline/GO [40], yielding efficient photocatalysts for pollutant degradation. The presence of CMC in MoS₂/GO composites is expected to further enhance photocatalytic efficiency by stabilising nanoparticles and preventing their agglomeration [41].

Additionally, the choice of light source plays a crucial role in determining photocatalytic efficiency. Light-emitting diodes (LEDs) have gained popularity

as an energy-efficient alternative to conventional UV lamps and mercury-based fluorescent lights due to their longer lifespan, low energy consumption, and tunable spectral output [29]. Unlike traditional sources, LEDs emit a narrow spectrum of visible light, ensuring optimal energy utilisation for photocatalysis while minimising heat loss [30]. Their compact design also allows for flexible reactor configurations, making them suitable for scalable water treatment applications. Despite these advantages, few studies had explored the application of LED-activated photocatalysts for PFOA degradation, highlighting the need for further research in this area.

This study aims to develop and characterise MoS₂/GO photocatalysts immobilised in a CMC hydrogel matrix for the efficient photodegradation of PFOA under visible-light LED irradiation. The photocatalytic activity of MoS₂/GO/CMC is evaluated under different reaction conditions, including pH and LED wattage, to determine the optimal degradation parameters. Additionally, the recyclability of the photocatalyst is assessed to demonstrate its potential for sustainable water treatment applications. By integrating an energy-efficient light source with a heterogeneous MoS₂/GO/CMC hydrogel system, this study seeks to provide an effective and environmentally friendly approach for PFOA remediation.

Materials and Methods

Materials

In this research, sodium molybdate (Na₂MoO₄, 98%), sulfuric acid (H₂SO₄, 98%), graphite powder (particle size: 20 µm), potassium permanganate (KMnO₄, 99%), sodium carboxymethyl cellulose (MW: ~250,000 g/mol, 99.5%), and perfluorooctanoic acid (PFOA) were obtained from Sigma-Aldrich Co. (St. Louis, Missouri, USA). Thiourea (CH₄N₂S, ≥99.0%), sodium hydroxide (NaOH), hydrogen peroxide (H₂O₂), and sodium nitrate (NaNO₃) were procured from R&M Chemicals (United Kingdom).

Instrument

A Spectrum 100 FTIR Spectrometer from Perkin Elmer, Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR)-FTIR was utilised to examine the functional group in hydrogels with wavenumber ranges of 4000-650 cm⁻¹.

Preparation of MoS₂/GO/CMC hydrogels

MoS₂ and GO were prepared according to [18]. The synthesis started with the addition of 0.1 g of MoS₂ and 0.1 g of GO nanoparticles to 2.5 mL of isopropyl alcohol. This mixture was rapidly agitated at 60°C for 1 h. Another solution was created simultaneously by mixing 0.4 g of CMC with 2.5 mL of deionised

water and vigorously swirled. After both solutions were stirred for 1 h, the MoS₂ and GO dispersed solution was carefully introduced into the CMC solution while stirring it for 20 min. Then, the resulting gel was processed to form flat beads. The product was finally dried in a desiccator for 48 h [31]. For control samples MoS₂/CMC and GO/CMC hydrogels were prepared by adding 0.1 g of MoS₂ nanoparticles to 2.5 mL of isopropyl alcohol and agitated vigorously at 60°C for 1 h. Consequently, 0.4 g of CMC was added to 2.5 mL of deionised water and subjected to intense stirring at the same temperature for 1 h. The MoS₂ solution was carefully poured into the CMC solution and stirred for another 20 min. The resulting gel was moulded into flat beads. Finally, the product was dried in a desiccator for 48 h. The preparation of GO/CMC hydrogels followed a similar procedure, with the key difference.

Photocatalytic degradation studies

The photodegradation experiments were conducted in a custom-designed photoreactor equipped with an LED light source ($\lambda = 420\text{--}700\text{ nm}$, visible light spectrum) with a maximum power of 12 W. The irradiation time for each experiment was set at 180 min to achieve optimal degradation. To evaluate the photocatalytic efficiency of MoS₂/CMC, GO/CMC, and MoS₂/GO/CMC in degrading PFOA, a photoreactor (**Figure 1**) which contained multiple quartz tube reactors was used. In a typical experiment, 0.01 g of the photocatalyst was added to a 10 mL PFOA stock solution (50 mg/L). Before exposure to light, the solutions were stirred in the dark for 30 min to establish an adsorption/desorption equilibrium. Then, the photocatalytic reactions were initiated under LED irradiation (420 nm –700 nm) at varying power levels (7 W, 9 W, 12 W). After treatment, the solutions were filtered and the

concentration of PFOA was analysed by using HPLC-UV at 210 nm.

Effect of light source

The photocatalytic performance of MoS₂/CMC, GO/CMC, and MoS₂/GO/CMC was evaluated under different light sources (dark, UV, fluorescent, and LED). Each composite (0.01 g) was added to 20 mL of 50 mg/L PFOA solution in separate photoreactor tubes. The mixtures were exposed to the respective light sources for degradation, followed by filtration. The PFOA concentration was analysed by using HPLC-UV at 210 nm.

Effect of pH value

The effect of pH value on PFOA degradation was investigated by using a 50 mg/L PFOA solution across a pH value range of from 2.0 to 12.0. The pH value was adjusted by using HCl or NaOH at 25°C. Then, hydrogels were introduced to the pH-adjusted solution, sonicated for 30 min, and transferred to a glass cuvette for photocatalytic degradation in a UV photoreactor. Afterward, the solutions were filtered, and the PFOA concentration was analysed by using HPLC-UV at 210 nm.

Zero potential analysis

The zero point of charge (pHZPC) of MoS₂/GO/CMC was determined by using the salt addition method. A solution of 0.1 M NaCl (40 mL) was prepared and adjusted to pH 2–10 by using HCl or NaOH. Then, 0.1 g of the material was added, and the suspensions were stirred at 250 rpm overnight. The final pH values were recorded, and the ΔpH ($\text{pH}_f - \text{pH}_i$) was plotted against pH_i to determine pHZPC [32–33].

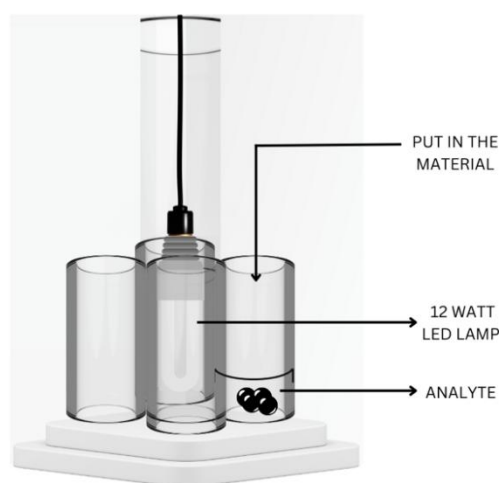


Figure 1. A customised schematic measurement

Effect of power watt

The photocatalytic performance of MoS₂/GO/CMC was evaluated under different power watt (7, 9, 12). Each composite (0.01 g) was added to 20 mL of 50 mg/L PFOA solution in separate photoreactor tubes. The mixtures were exposed to the respective light sources for degradation, followed by filtration. The PFOA concentration was analysed by using HPLC-UV at 210 nm.

Analytical technique

A High-Performance Liquid Chromatography (HPLC) with a UV detector system was used to measure the concentration of PFOA in the aqueous phase. The Agilent 1100 series HPLC system used had a manual sample injector with a degasser, a 20-L injection volume, a column oven that was kept at 40°C, and a pump. A C18 separation column was utilised for the analysis. The samples were eluted at 1.0 mL/min with acetonitrile and aqueous sodium dihydrogen phosphate (5 mMol, pH 7.0) as the mobile phase in a 50:50 (v/v) ratio. of 210 nm was used for detection. Equation (1) was used to calculate the degradation percentage:

$$\text{Degradation (\%)} = \frac{(C_0 - C)}{C_0} \times 100 \quad (1)$$

Where, C₀ is initial PFOA concentration and C is the PFOA concentration at the end of experiment [17].

Results and Discussion

FTIR spectra for CMC, MoS₂/CMC, GO/CMC, and MoS₂/GO/CMC are shown in **Figure 2**. The FTIR spectrum of CMC powder revealed absorption peaks which corresponded to the vibrational frequencies of chemical bonds within the CMC structure. The absorption peak at 1600 cm⁻¹ corresponded to the carboxylate group (COO⁻), while the peak at 1438 cm⁻¹ was associated with the asymmetric stretching vibration of carboxylate compound (COO⁻Na). Additionally, strong absorption between 950 cm⁻¹ and 1250 cm⁻¹ indicated the presence of ether (-C-O-C-) linkages in the CMC compound [34]. The broad band at 3400 cm⁻¹ corresponded to O-H stretching, while the peak at 1321 cm⁻¹ was attributed to the symmetric bending of CH₂ groups.

For MoS₂, the FTIR spectrum displayed peaks between 692 cm⁻¹ and 1137 cm⁻¹, which were characteristic of Mo-S and S-S bond vibrations, confirming the presence of MoS₂. The broad peak around 3405 cm⁻¹ corresponded to O-H stretching and bending modes, likely due to residual water or hydroxyl groups adsorbed on the surface [35]. Meanwhile, for GO, the FTIR spectrum exhibited a peak at 3453 cm⁻¹, which attributed to O-H

stretching vibrations and indicated the presence of hydroxyl functional groups. Peaks at 1740 cm⁻¹ and 1589 cm⁻¹ corresponded to carbonyl (C=O) and C=C stretching vibrations, respectively, confirming the oxidation of graphite. Additionally, the peak at 1137 cm⁻¹ represented the epoxy (C-O-C) functional group [36-38].

In the FTIR spectrum of the MoS₂/GO/CMC composite, all the characteristic peaks of CMC, MoS₂, and GO were present, confirming the successful formation of the composite material [39]. The skeletal vibration peak for the C-O-C bond appeared at 1057 cm⁻¹, while a peak at 865 cm⁻¹ corresponded to Mo-S and S-S linkages, indicating the incorporation of MoS₂ within the composite matrix. The presence of these characteristic functional groups suggested strong interactions between the components, which might enhance the stability and photocatalytic activity of MoS₂/GO/CMC composite [40].

In this study, the photocatalytic process and performance of MoS₂/CMC, GO/CMC, and MoS₂/GO/CMC composites in degrading aqueous PFOA solutions were evaluated. **Figure 3** illustrates the C/Co ratio for these composites under different photocatalyst and irradiation conditions, whereby a higher C/Co ratio indicated lower degradation efficiency. The impact of various radiation sources, including dark, UV, fluorescent, and LED irradiation, was examined for over 120 min. Under dark conditions, all photocatalysts displayed insignificant degradation efficiency, primarily due to physical adsorption mechanisms [41].

Meanwhile, under LED light, MoS₂/GO/CMC exhibited the highest degradation efficiency (0.08) as compared to MoS₂/CMC (0.28) and GO/CMC (0.29). Notably, fluorescent light showed better degradation performance than UV light for GO/CMC (0.28) and MoS₂/GO/CMC (0.25). This enhanced performance was attributed to the synergistic effect of MoS₂ and GO within the composite matrix. The inclusion of cellulose improved nanoparticle stability, reducing the likelihood of their separation from the polymer matrix and enhancing the ability of composite to effectively separate photogenerated electron pairs. Additionally, GO increased the number of surface-active sites, further improving photocatalytic activity. These findings highlighted the promising potential of MoS₂/GO/CMC composites for effective photocatalytic degradation, particularly under LED light.

The study also examined the effect of solution pH value on PFOA degradation at pH 5, 7, and 9 (**Figure 4**) [42]. Results indicated that pH 5 was the most

effective condition, yielding a C/Co ratio of 0.08. This optimal pH value facilitated interaction between PFOA and MoS₂/GO/CMC, primarily due to hydrophobic interactions between graphene oxide and the hydrophobic tail of PFOA [13]. At higher pH levels, PFOA deprotonation resulted in a negatively charged species, leading to electrostatic repulsion with the negatively charged MoS₂/GO/CMC surface, and thus reducing degradation efficiency.

For MoS₂/GO/CMC, the study measured the pH value at the point of zero charge (pHpzc), which was determined to be 5.24 (Figure 5). This value indicated a nearly neutral surface charge at this pH value. At higher pH value levels, the presence of excess OH⁻ ions led to deprotonation of MoS₂/GO/CMC, resulting in a negatively charged surface which further hindered interaction with the deprotonated PFOA anions [43,44].

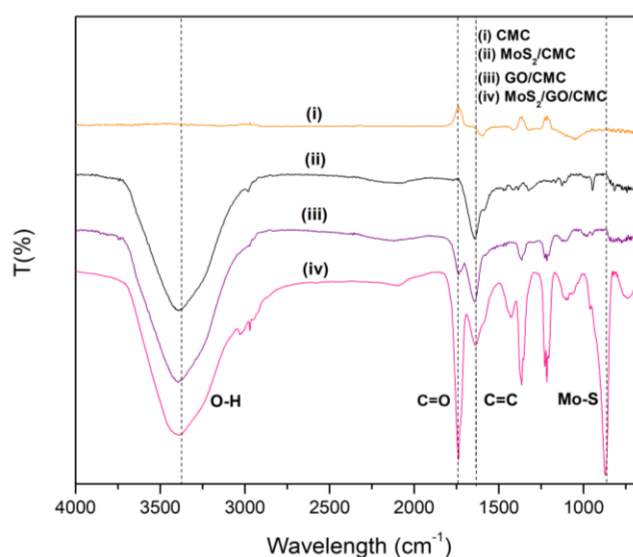


Figure 2. FTIR spectra of (i) CMC, (ii) MoS₂/CMC, (iii) GO/CMC, and (iv) MoS₂/GO/CMC composites

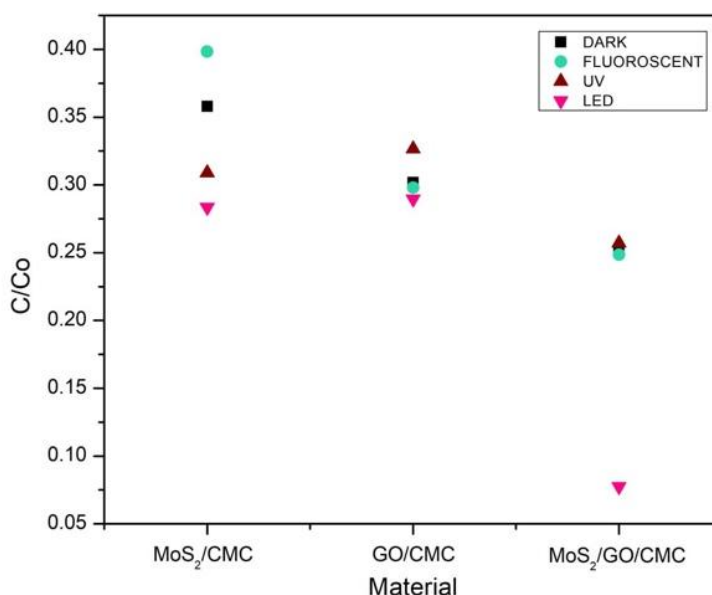


Figure 3. Light sources' influence on PFOA degradation (50 ppm PFOA, pH 2 and 1 g/L of photocatalysts)

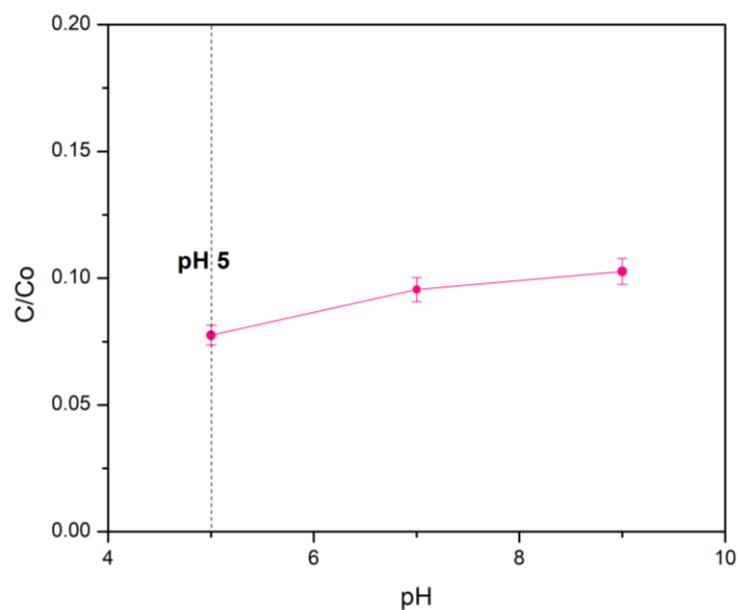


Figure 4. Influence of pH on PFOA photocatalytic degradation (50 ppm of PFOA, 12 watts of LED light, and 1 g/L of loading catalyst)

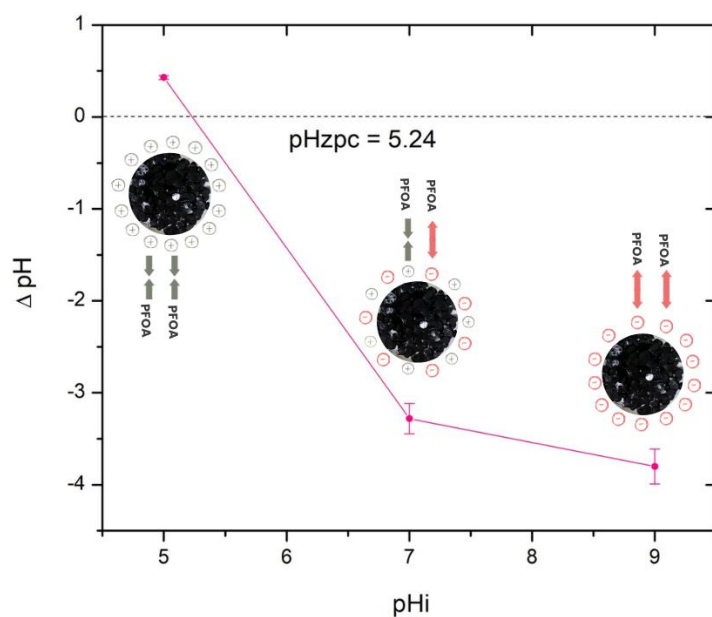


Figure 5. Zero potential (pHpzc) of MoS₂/GO/CMC (5, 7, 9)

The effect of different wattage levels of emitting diodes (7, 9, and 12 W) on the photocatalytic degradation of PFOA was also investigated. Results showed that 12 watts of power yielded the highest percentage degradation of PFOA (**Figure 6**). This increase in power enhanced the number of quanta

yields available for photodecomposition, thereby improving decomposition efficiency. Overall, these findings offered valuable insights into optimising the photocatalytic degradation of PFOA in various environmental conditions, considering factors such as irradiation, pH value, and power levels [2].

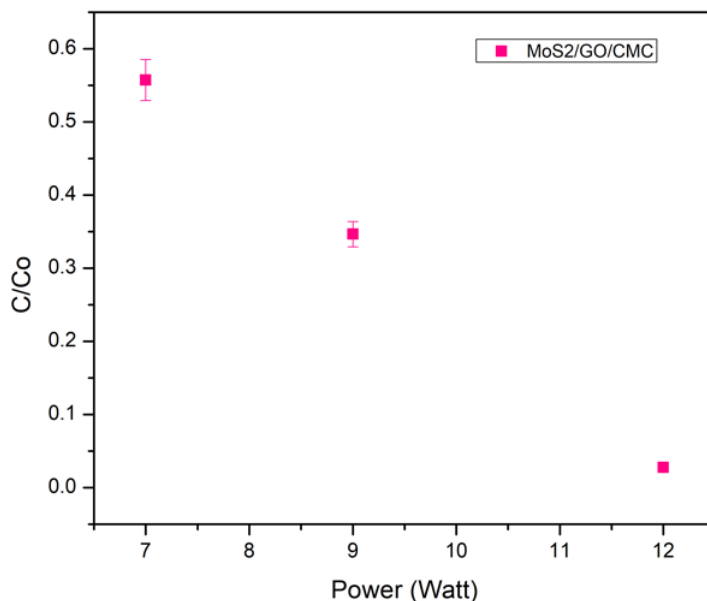


Figure 6. Effect of light source power (watt) of PFOA (1 g/L of loading catalyst, and pH 5) on photocatalytic degradation of PFOA.

The most noteworthy result of photodegradation experiments was a viable photocatalyst that may be renewed. After a few applications, a good photocatalyst ought to be able to maintain good deteriorating performance. The photocatalyst was washed with deionised water and methanol before the experiment, and it was put in oven at 70 °C for 2 h. **Figure 7(a)** shows that after seven cycles, the deterioration efficiency was decreased by about 12%. Therefore, it was determined that it was allowed to reuse the MoS₂/GO/CMC for up to seven cycles [45-46]. MoS₂/GO/CMC structures did not show a significant reduction in efficiency after seven cycles, as seen in **Figure 7(a)**. After seven cycles, the PFOA degradation efficiency decreased from 92.26% to 89.02%, but the deterioration was still significant [47]. Therefore, MoS₂/GO/CMC revealed high recyclability.

The FTIR analysis of the MoS₂/GO/CMC composite after the seventh cycle provided insight into the factors which contributed to its reduced photocatalytic degradation efficiency. As shown in **Figure 7(b)**, a noticeable decrease in peak intensity indicated a diminished presence of functional groups

essential for photocatalytic activity. This suggested that repeated use led to the saturation or deactivation of active sites, limiting the functional groups responsible for radical generation. As a result, the ability of composite to generate reactive oxygen species (ROS) declined, reducing its affinity for pollutant molecules and ultimately decreased the degradation rate [48].

Additionally, the absence of the characteristic peak between 1000 cm⁻¹ and 800 cm⁻¹ after multiple cycles suggested structural modifications, likely due to the repeated washing of the composite with deionised water [49]. This process may have resulted in the partial removal of functionalised GO components, further reducing available surface-active sites. Despite these changes, the composite still maintained a high degradation efficiency after seven cycles, demonstrating its relative stability and reusability. However, the observed decline highlighted the need for potential regeneration strategies or modifications to enhance long-term catalytic performance.

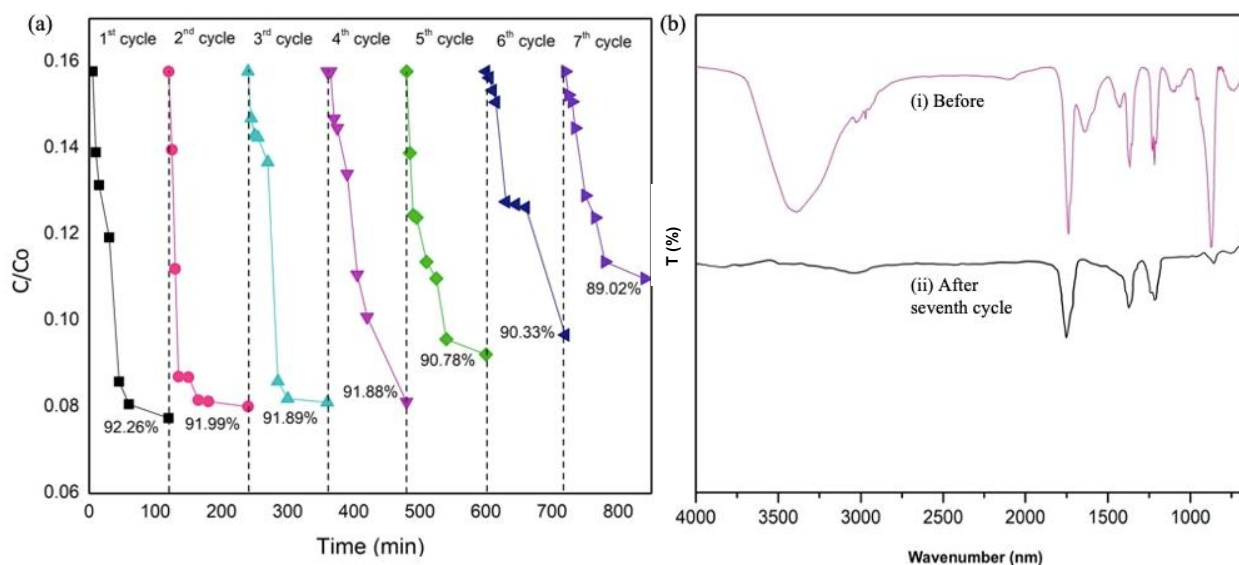


Figure 7. (a) The recyclability study of MoS₂/GO/CMC and (b) FTIR Spectrum of MoS₂/GO/CMC (i) before and (ii) after the seventh cycle

Conclusion

In conclusion, this study demonstrates the effectiveness of MoS₂/GO/CMC composites in degrading PFOA under LED light irradiation, outperforming MoS₂/CMC and GO/CMC composites. The incorporation of cellulose enhanced nanoparticle stability and increased surface-active sites, leading to improved degradation efficiency. The optimal degradation conditions were observed at pH 5, whereby favorable hydrophobic interactions facilitated degradation, whereas higher pH value levels impeded the process. Additionally, increasing the LED wattage to 12W significantly boosted photodecomposition, maximising efficiency. These findings provided valuable insights into optimising PFOA degradation for real-world applications. Future research should explore the long-term stability and reusability of these composites, assess their performance in complex water matrices, and investigate large-scale implementation strategies to advance sustainable water treatment solutions.

Acknowledgement

We acknowledge the School of Chemistry and Environment, Faculty of Applied Science UiTM Negeri Sembilan, Kuala Pilah Campus for the facilities that have been provided for the research. This project was supported by financial grants such as the Fundamental Research Grant Scheme [FRGS/1/2022/STG05/UITM/02/12] by the Ministry of Higher Education Malaysia.

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