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PHOTOCATALYTIC PERFORMANCE OF MOLYBDENUM DISULPHIDE-GRAPHENE OXIDE IN PERFLUOROOCTANOIC ACID (PFOA) DEGRADATION UNDER INDOOR LIGHT EMITTING DIODE (LED) IRRADIATION

(Prestasi Fotokatalitik Molibdenum Disulfida-Grafena Oksida dalam Degradasi Asid Perfluorooktanoik (PFOA) di bawah Penyinaran Diod Pemancar Cahaya Dalaman (LED))

Zulhatiqah Zolekafeli[1], Kavirajaa Pandian Sambasevam[1,2,3], Siti Nor Atika Baharin[1,3*]

[1]*Advanced Materials for Environmental Remediation (AMER), Faculty of Applied Sciences, Universiti Teknologi MARA Cawangan Negeri Sembilan Kampus Kuala Pilah, 72000 Kuala Pilah, Negeri Sembilan, Malaysia*
[2]*Electrochemical Material and Sensor (EMas) Group, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia*
[3]*Advanced Biomaterials & Carbon Development (ABCD), Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia*

Corresponding author: atikabaharin@uitm.edu.my

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Abstract

In this research, molybdenum disulphide-graphene oxide (MoS_2-GO) was used as photocatalyst for the degradation of perfluorooctanoic acid (PFOA) under 12-watt light emitting diode (LED) irradiation. The photocatalyst performance was evaluated by a series of experiment; effect of contact time, trapping experiments and recyclability studies. Effect of contact time revealed MoS_2-GO degraded PFOA with 80.77% at 120 equilibrium time. Langmuir-Hinshelwood (L-H) kinetic model was applied to study the kinetic model with (R^2) of 0.8726 at half-life ($t_{1/2}$) 2.31 h under LED light irradiation. The trapping experiment shows that photocatalytic process was dominant by $\bullet O_2^-$, where $\bullet O_2^-$, supported by proton ion (h^+), is the major active species whereas •OH only played a minor part in the entire photocatalytic process. The recyclability study revealed the MoS_2-GO can be reused for up to six cycles. The employed photocatalyst in the sixth cycle was analyzed using FTIR and the FTIR spectrum shows that the peak diminishes its intensity after reusability at peaks of 1300 cm^{-1} to 1800 cm^{-1} for carbonyl (C=O), epoxy, the C=C stretching mode and carbonyl (>C=O), 750 cm^{-1} to 1250 cm^{-1} indicates the S-S bond, and at 3038 cm^{-1} that is attributed to the O-H stretching vibrations. Thus, this research proposed a performance evaluation of MoS_2-GO as photocatalyst for photocatalytic degradation of PFOA under indoor LED light irradiation.

Keywords: molybdenum disulphide, photodegradation, perfluorooctanoic acid, light emitting diode

Abstrak

Dalam penyelidikan ini, molibdenum disulfida-grafena oksida (MoS_2-GO) digunakan sebagai pemangkin foto untuk degradasi asid perfluorooktanoik (PFOA) di bawah penyinaran diod pemancar cahaya (LED) 12 watt. Prestasi pemangkin foto telah dinilai oleh satu siri eksperimen; kesan masa sentuhan, eksperimen perangkap dan kajian kitar semula. Kesan masa sentuhan mendedahkan MoS_2-GO merendahkan PFOA dengan 80.77% pada 120 masa keseimbangan. Model kinetik Langmuir-

Hinshelwood (L-H) telah digunakan untuk mengkaji model kinetik dengan (R^2) sebanyak 0.8726 pada separuh hayat ($t_{1/2}$) 2.31 h di bawah penyinaran cahaya LED. Eksperimen perangkap menunjukkan bahawa proses fotokatalitik didominasi oleh $•O_2^-$, di mana $•O_2^-$, disokong oleh ion proton (h^+), adalah spesies aktif utama manakala •OH hanya memainkan peranan kecil dalam keseluruhan proses fotokatalitik. Kajian kebolehkitaran semula mendedahkan MoS_2-GO boleh digunakan semula sehingga enam kitaran. Pemangkin foto yang digunakan dalam kitaran keenam telah dianalisis menggunakan FTIR dan spektrum FTIR menunjukkan bahawa puncak mengurangkan keamatannya selepas kebolehgunaan semula pada titik 1300 cm^{-1} hingga 1800 cm^{-1} untuk karbonil (C=O), epoksi, mod renggangan C=C dan karbonil (>C=O), 750 cm^{-1} hingga 1250 cm^{-1} mewakili ikatan S-S, dan di titik 3038 cm^{-1} yang mewakili ikatan renggangan O-H. Oleh itu, penyelidikan ini mencadangkan penilaian prestasi MoS_2-GO sebagai pemangkin foto untuk degradasi fotokatalitik PFOA di bawah penyinaran cahaya LED dalaman.

Kata kunci: molibdenum disulfida, fotodegradasi, asid perfluorooktanoik, diod pemancar cahaya

Introduction

Earth's surface is about 71% covered with water and over 96.5% of the Earth's water is held by oceans. In addition to these places, water may be found in air as water vapour, in water bodies such as rivers and lakes, icecaps and glaciers, Earth's soil moisture and aquifers. When harmful chemicals or microbes are released into water resources directly from industrial, commercial, or residential sources, water pollution occurs. Additionally, polluted soils can leak pollutants into streams and lead to environmental problems [2]. Therefore, non-conventional chemical emissions into water supply, surface water and drainage have resulted in significant distress.

Perfluorinated compounds (PFCs) have sparked alarm for the environment and human health because of their persistence and tendency to accumulate heavy metals. PFCs also affect hormones and induce immunotoxicity, neurotoxicity as well as developmental issues [13]. A member of PFC family, called perfluorooctanoic acid (PFOA) has raised controversy amongst the nation due to its necessity in the manufacture of Teflon, high temperature lubricants, protective coatings and surfactants [8]. Likewise, PFOA provides advantages, including improvement of stain, grease and in waterproofing. According to the US Environmental Protection Agency these pollutants are frequently found in industrial, consumer and food products. But, unlike other persistent pollutants, PFOA is easily soluble in water and readily migrated into aquifers [19]. As a result, it can bioaccumulate in the bloodstream and liver instead of fatty tissues [17].

In addition, owing to its carbon-fluorine (C-F) bond, PFOA is a remarkably stable and lasting compound which does not naturally break down. For this reason, the environment or anybody exposed to PFOA might possibly be harmed. Considering the harmful consequences that it has on the environment and to protect as well as nurture the environment for all living organisms, an innovation that can properly break down PFOA is required. The photocatalytic breakdown of PFOAs by photo-oxidative and photo-reductive degradation appears to be more suitable than the other ways outlined above. Additionally, photocatalysis oxidation has become a very promising technology due to its wide range of uses in photocatalytic process, powerful oxidising capacity, and absence of secondary pollutants. Additionally, it has advanced significantly in theory and application [7, 9, 22]. Previously, studies by Hao et al. with $BiPO_4$ photocatalyst and Sahu et al. with Ag-TiO_2 photocatalyst degraded PFOA by 72% and 57.7% when exposed to fluorescent lamp radiation for two hours and seven hours respectively. Meanwhile, Trojanowicz et al. used TiO_2 as a photocatalyst to degrade PFOA with 55.9% efficiency over the course of two hours [3, 13, 15].

Heterogeneous photocatalytic oxidation of PFOA appears as a potent, economical alternative strategy due to the ease of generating active oxygen-containing species under UV-visible irradiation, ease of photocatalyst separation and ability to recycle the catalyst. Heterostructure photocatalysts with semiconductor are employed in various fields, including electronics, catalysts, solar panels, and supercapacitor materials. According to Li et al., integrating a semiconductor with another semiconductor can enhance light absorption and reduce photo-generated electron hole recombination [7]. Two-dimensional (2D) nanostructures have drawn attention as substitutes to photocatalytic materials in the pursuit for highly efficient photocatalytic materials due to their photocatalytic performance being driven by visible light [16]. This is because the outstanding optical and electrical features of the 2D materials demonstrate the capacity to confine electrons in their ultrathin layer. Moreover, due to their high electrochemical nature on the basal planes and edges of 2D materials in energy conversion (e.g., TMDs), well-balanced hydrogen binding Gibbs free energy on the basal sites (e.g., 1T′ MoS_2) high specific surface area, abundance of surface-active sites as well as their superior external performance, the 2D materials of MoS_2 have great structural advantages in photocatalysis [11]. Additionally, the large specific surface area of the 2D design makes surface reactions possible.

A good charge carrier for semiconductors may be found in the molecularly thick, 2D nanomaterials known as graphene, which are composed of sp2 carbon atoms that are arranged in a honeycomb structure [12]. Graphene is employed as a remarkable electron-acceptor or transport material in photocatalysis because of its capacity to decrease photogenerated electron hole recombination and increase light adsorption [6]. In addition, it has been shown that the enormous specific surface area of graphene and effective electron transport properties has made it into a fantastic co-catalyst for photocatalytic activity. [5].

While PFOA is chemically stable, photodegradation is observed to be effective when exposed to UV light. The limitations of light sources like halogen or xenon bulbs are high cost, short lifespan, poor light penetration, and low luminous efficiency. Therefore, the advantages of light emitting diode (LED) sources include the requirement for a separate water-cooling facility removal to maintain a constant temperature, flexibility of LED source placement within the designed reaction system and addition of a co-catalyst; hence, securing the enhancement of photocatalytic activity under the visible-light region. Consequently, in this work, MoS2-GO was used as a photocatalyst material to break down PFOA by using indoor LED light. As per of our knowledge, there is no attempt had been made utilizing indoor LED light as the source of light for photodegradation of PFOA.

Materials and Methods

Materials

The MoS_2-GO composites were synthesised via hydrothermal method under acidic conditions using the sodium molybdate (Na_2MoO_4) and thiourea as starting materials and successfully characterised in prior studies as outlined in literature [9].

Analytical method

The amount of PFOA in the aqueous phase was determined by using the High-Performance Liquid Chromatography (HPLC) instrument. The manual sample injector (injection volume: 20 μL), degasser, pump, and column oven (40ºC) comprised the HPLC system (Agilent 1100 series HPLC). The separating column was served by C18. The samples were isocratically eluted with acetonitrile (ACN) and aqueous NaH_2PO_4 (5 mMol, adjusted to pH 7.0) (50:50, v/v) as the eluents at a flow rate of 1.0 mL min^{-1} and 210 nm, respectively.

Contact time and kinetic study

In this work, the kinetic research of MoS_2-GO composites on the PFOA degradation was studied by using the Langmuir-Hinshelwood (L-H) kinetic model. By breaking down PFOA while being exposed to a 12-watt LED lamp's light, the MoS_2-GO photocatalytic activity was assessed. By oxidising PFOA while being illuminated by a 12-watt LED light, the MoS_2-GO photocatalytic activity was assessed. A photoreactor with several quartz tube reactors served as setting for the photocatalytic testing. In this photocatalytic experiment, 0.002 g of MoS_2-GO composite was placed in a beaker along with 20 mL of a 50 mg/L PFOA stock solution. Then, to guarantee that adsorption/desorption equilibrium was reached, the solution was stirred continuously for 30 min. At this time (t = 0), 20 mL of each solution that contained 50 mg/L (C_o) was introduced to the UV photoreactor. The mixture was subsequently centrifuged and filtered to get rid of the composite. HPLC-UV detection was used to measure the amount of PFOA present during the photocatalytic degradation process. The C/C_o ratio was calculated by using PFOA absorption measurements for each sample, whereby C_o was the PFOA concentration at t = 0 min and C was the PFOA concentration at a specified time.

Trapping experiment

The trapping experiment was to examine the reactive species (such as h^+, $\bullet OH$, e^-, and $\bullet O_2^-$) which could be formed during photocatalysis and interact with PFOA. Trapping tests were carried out under identical experimental circumstances as those in the photodegradation experiments, with the exception that certain scavengers were introduced to the suspensions before being exposed to LED irradiation. The hydroxyl radical ($\bullet OH$) scavenger was isopropanol (IPA, 1 mmol/L) [7], while the superoxide radical ($\bullet O_2^-$) scavenger was p-benzoquinone (BQ, 0.1 mmol/L) [20].

Recyclability of the photocatalyst

Prospect of recycling and renewing the photocatalyst was examined over six cycles. After cleaning with methanol and water and drying for 12 h in a vacuum at 70˚ C, the photocatalyst was recycled. FTIR analysis was applied to check for any chemical modifications in the regenerated photocatalyst.

Results and Discussion

Photodegradation experiment: Contact time and kinetic study

To further study the impact of contact duration on the kinetic behaviour of the photodegradation process, the materials were evaluated with LED irradiation from 0 min to 120 min and in the dark for 30 min to further study the impact of contact duration on the kinetic behaviour of the photodegradation process. Figure 1 shows little deterioration in the dark area with the changing trend shown when the adsorption/desorption equilibrium was established. Between 0 and 30 minutes, there was a noticeable rapid decrease; between 60 min and 120 min, there was a gradual decline and continual stagnation. As the active sites of photocatalyst were initially unoccupied, the increase happened quickly at first. Thereafter, PFOA was instantly degraded by the photocatalysts. Most active sites were interacting, slowing the rate at which PFOA degraded since there were less opportunities for holes and electrons to join. Many research studies on heterogeneous photocatalysts in photodegradation demonstrated a finding that was consistent with the Langmuir-Hinshelwood (L-H) kinetic model for organic pollutants. (Equation 1) [9].

$$ro = -\frac{dC}{dt} = \frac{kKc}{1 + Kc} \tag{1}$$

Where, *ro* is the reactant degradation rate, *k* is reaction rate constant, *K* is adsorption equilibrium constant, and *C* is reactant concentration. $Kc < < 1$ in the L-H equilibrium can be simplified to a pseudo-first-order kinetic model by considering the low concentration of PFOA utilised in the experiment (Equation 2).

$$-\frac{dC}{dt} = kKc = k_{obs}C \tag{2}$$

Further set the limit of *C* = Go at *t*=0, the Equation 2 can be integrated, which resulted in Equation 3.

$$-Ln\frac{C}{C_o} = k_{obs}t \tag{3}$$

According to a plot of Ln C/Co vs irradiation time presented in Figure 1, the kinetic constants for the observed variables pseudo-first-order rate constant k, coefficient of determination R^2, and half-life $t_{1/2}$ were respectively, 0.005 min^{-1}, 0.8726, and 138.6 min. Table 1 displays the kinetic characteristics (pseudo-first order) of various photocatalyst types in the PFOA degradation. Most degrading techniques utilise the use of fluorescent, UV, visible or simulated solar radiation. By using indoor LED lighting as a source of light in the PFOA degradation process, inadequate information was found. The data revealed that PFOA was rapidly degraded under visible light without having Sb_2O_3 as the photocatalyst since Sb_2O_3/TiO_2 had a bigger k constant (0.0126 min^{-1}) and a shorter t half-life (6.612 h) than TiO_2. Further, $NaIO_4$ was shown to have better kinetic performance than Ag-TiO_2 with 0.0096 min^{-1} k constant than 0.00217 min^{-1}. While Ag-PFOA TiO_2 content was reduced by 72% under fluorescent light, $BiPO_4$ was discovered to have good degrading efficiency. Notably, kinetic results from this study showed that MoS_2-GO could break down PFOA when exposed to indoor LED light, with a 2.31-hour reduction in half of PFOA concentration from its original concentration [1, 6, 13, 20].

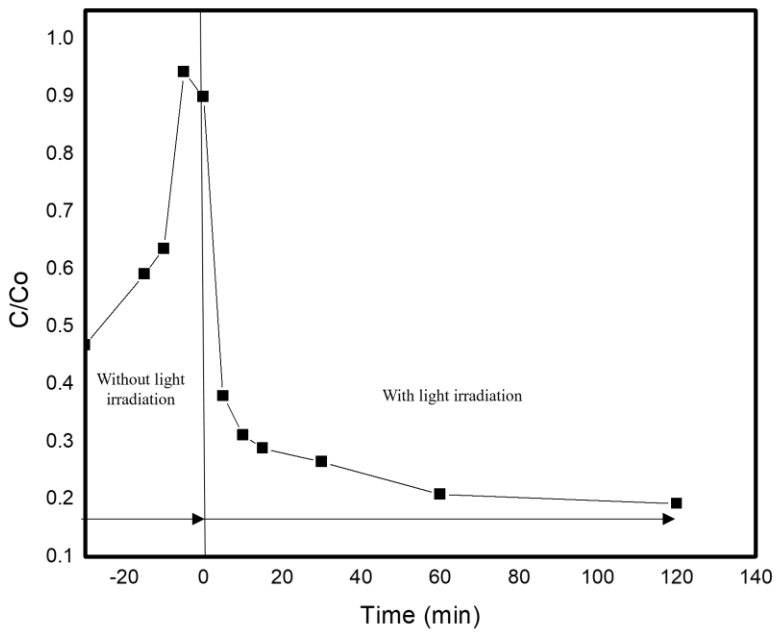

Figure 1. Effect of contact time for photocatalytic degradation of PFOA (50 mg/L PFOA, 12-watt LED light, 2 mg/L of catalyst loading)

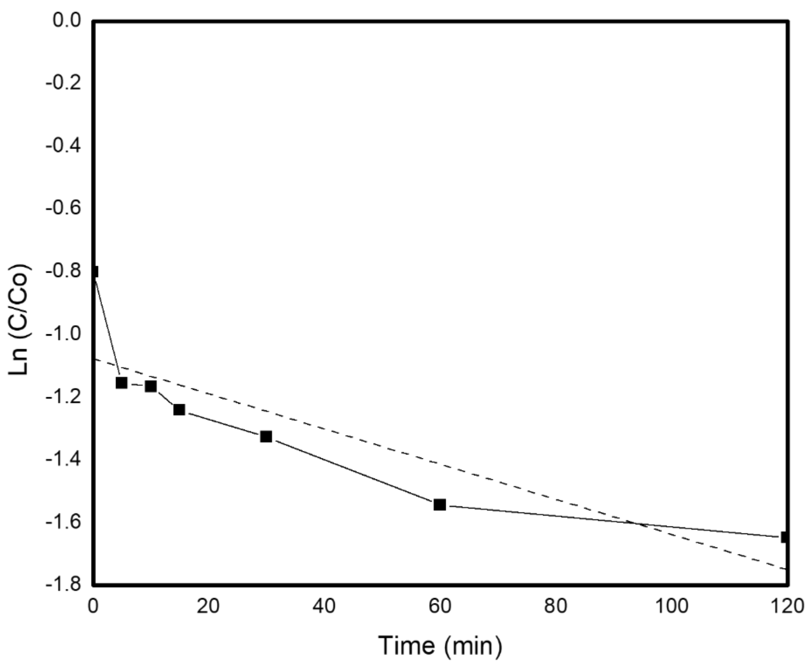

Figure 2. Kinetic plot of *Ln* (C/C_o) versus irradiation time for photocatalytic degradation of PFOA

Table 1. Kinetic parameters (pseudo-first order) of different types of photocatalyst in degrading PFOA

Type of Photocatalyst	Light Source	Contact Time (h)	Initial Concentration (mg/L)	R^2	k (min^{-1})	$t_{1/2}$ (h)	Degradation (%)	Ref.
$Ag-TiO_2$	Fluorescent lamp	7	60	0.7267	0.00217	Not mention	57.7	[13]
$NaIO_4$	UV	Not mention	0.004	Not mention	0.0096	Not mention	70	[16]
TiO_2	UV	2	5	Not mention	0.0063	18.78	55.9	[15]
Sb_2O_3/TiO_2	UV	2	5	Not mention	0.0126	6.612	81.7	[15]
$BiPO_4$	Fluorescent lamp	2	0.05	Not mention	0.00667	Not mention	72	[3]
MoS_2-rGO-15	Indoor fluorescent	3	50	0.972	0.0758	1.524	98	[9]
MoS_2-GO	LED irradiation	2	50	0.8726	0.005	2.31	80.77	This study

Trapping experiment

Many reactive species, including proton ion (h^+), •OH, e^-, and $•O_2^-$ may be generated during photocatalysis and may react with PFOA. Trapping investigations into the active species during the photocatalytic PFOA degradation were conducted to better understand the photocatalysis process. According to Figure 3, there was no discernible difference in the photocatalytic PFOA degradation with isopropanol (IPA, 1 mmol/L, a scavenger of •OH) as compared to without a scavenger. This suggested that •OH was not the main active component. The photodegradation was completely quenched when p-benzoquinone (BQ, 0.1 mmol/L, quencher of $•O_2^-$) was added, indicating that $•O_2^-$ was the main active species and crucial to the photocatalytic activity. It was clear from this that the important oxidative species was $•O_2^-$ produced by the interaction of photogenerated electrons and O_2, while the photogenerated h^+ also supported the absolute term for PFOA degradation in the nanohybrid composite photocatalyst system. It might be presumed that $•O_2^-$ played a significant part in the photocatalytic process based on the trapping analysis (Figure 3). These findings implied that $•O_2^-$, supported by h^+, was the major active species and the fundamental mechanism for the powerful photocatalytic activity, whereas •OH only played a minor part in the entire photocatalytic process [3, 10, 18].

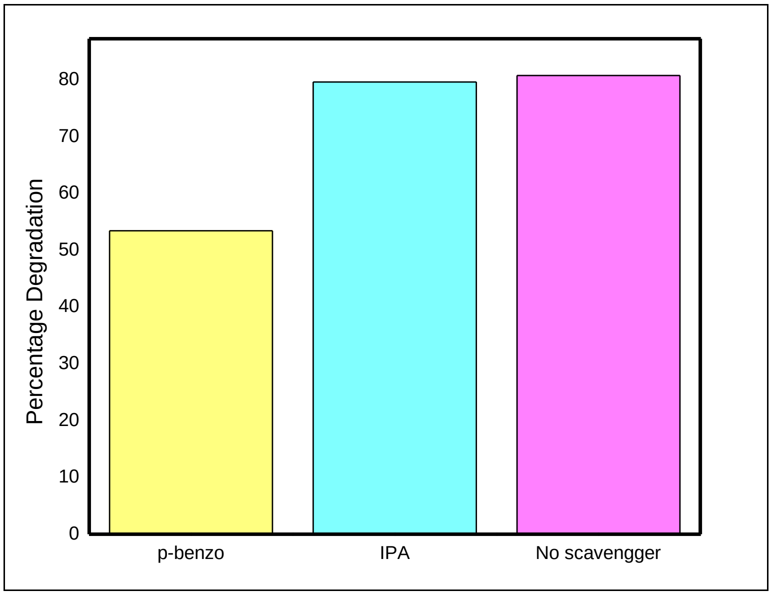

Figure 3. Trapping experiment of the active species during photocatalytic degradation of PFOA over nanocomposite alone and with the addition of BQ (quencher of $•O_2^-$) and IPA (quencher of •OH) under indoor LED light irradiation

Recyclability of study

The most noteworthy aspect of photocatalytic degradation studies is a reusable, sustainable photocatalyst. After a few cycles of use, a promising photocatalyst must be able to maintain good degrading performance. The photocatalyst was cleaned with ethanol and water prior to the experiment and then dried in a vacuum for 2 h at 70 °C. Figure 4 shows that after six further experiments, the deterioration efficiency was reduced by about 30%. As a result, it was determined that the MoS_2-GO might be reused and renewed for up to six cycles. The six cycles of photocatalyst were examined by using FTIR to demonstrate the causes of MoS_2-GO's reduced degradation efficiency. According to Figure 5, the FTIR spectrum demonstrated that the peak intensity decreased after reusability due to lesser affinity as most active sites interact, reducing the production of free radicals and, consequently, the degradation process. They might be seen in the missing peaks at 1300 cm^{-1} to 1800 cm^{-1} for carbonyl (C=O), epoxy, the C=C stretching mode and carbonyl (>C=O), 750 cm^{-1} to 1250 cm^{-1} indicates the S-S bond, and at 3038 cm^{-1} is attributed to the O-H stretching vibrations that resulted from the repeated washing recyclability of the composites [14,15,21]. The recyclability of study may be extended up to seven cycles or more however, the increasing of missing peaks which indicated the availability of MoS_2-GO nanocomposites showed that the degradation process was performed by absorption of light source since the LED irradiation range was applied in a broad wavelength of 275nm to 950nm.

Conclusion

Under the 12-watt indoor LED light irradiation, GO, MoS_2, and MoS_2-GO composites were successfully fabricated, characterised and used as a catalyst for PFOA photodegradation. With increasing loading and contact duration, MoS_2-GO demonstrated the greatest PFOA degradation. The Langmuir-Hinshelwood model employed a pseudo-first order rate law to define the kinetics of PFOA photodegradation. According to the stability and reusability studies, MoS_2-GO may be used up to six times without losing its properties. The greatest degrading effectiveness of photocatalysts in PFOA had proved that MoS_2-GO was successfully used as a photocatalyst. Moreover, by adding the p-benzoquinone scavenger to MoS_2-GO composites it accelerated the breakdown of PFOA, whereby $•O_2^-$ was the main active species and an important player in the photocatalytic reaction. Overall, the research findings showed that MoS_2-GO as a versatile photocatalysts that can degrade the organic wastes by using low cost and safe indoor LED light.

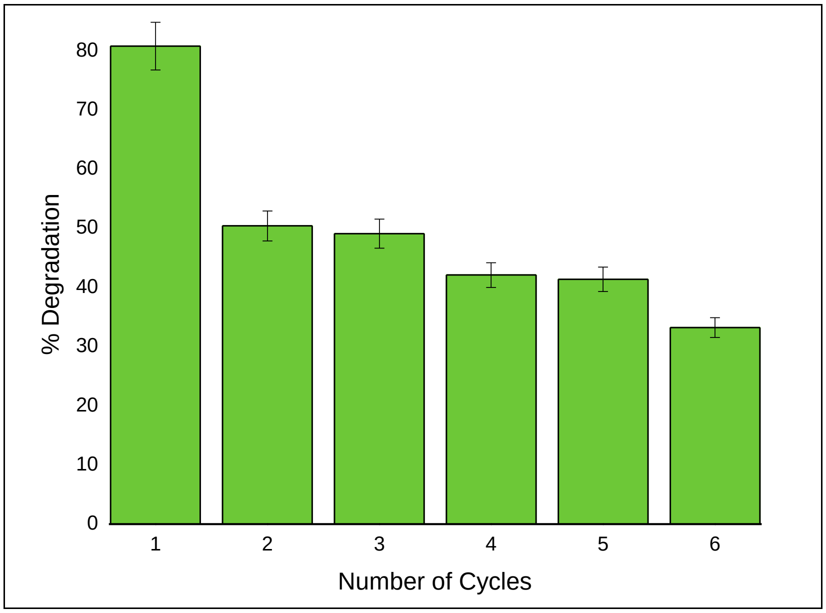

Figure 4: The recyclability study of degradation of PFOA

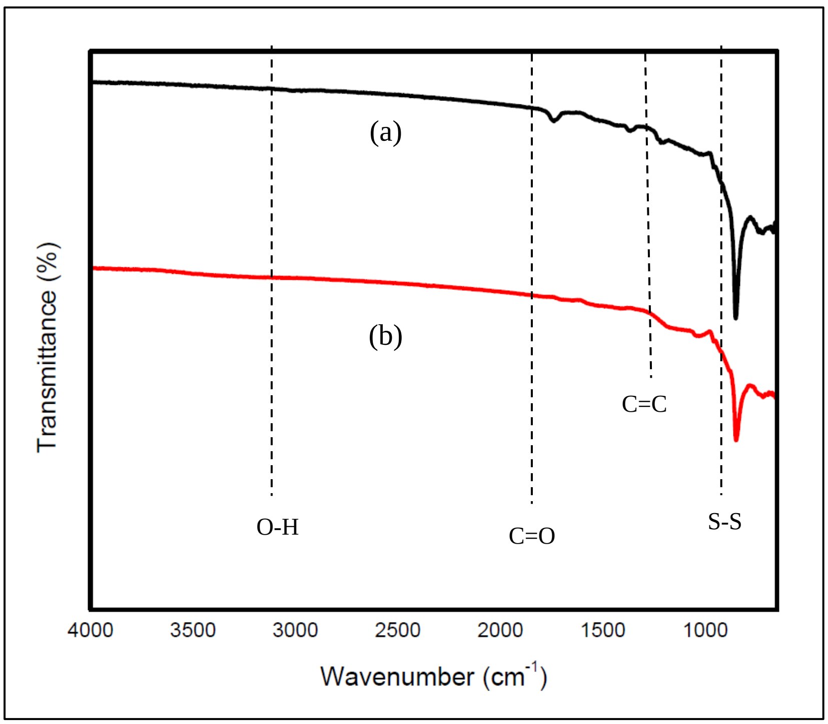

Figure 5. FTIR spectrum of (a) MoS_2-GO before and (b) MoS_2-GO after recyclability study

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