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PRELIMINARY STUDIES ON SUNLIGHT ASSISTED DEGRADATION OF 2-CHLOROPHENOL USING MoS_2/GO AS PHOTOCATALYST

(Kajian Awal Mengenai Cahaya Tampak Dituruti Penyusutan Sinaran 2-klorofenol Menggunakan MoS_2/GO Sebagai Pemangkin Cahaya)

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Abstract

This study highlighted preliminary studies on photodegradation of 2-chlorophenol using MoS_2/rGO photocatalyst with different weight ratios under sunlight irradiation. The 2-chlorophenol is known as one of the most dangerous pollutant due to its hazardous properties of mutagenicity, carcinogenicity and eco-pollutant. The MoS_2/rGO photocatalysts were synthesized using the hydrothermal method and used for the degradation of 2-chlorophenol under solar light irradiation. Three different compositions of MoS_2/rGO (1%, 5% and 15%) were synthesized with different weight ratio of MoS_2. The composites material of MoS_2, GO and MoS_2/rGO was characterized by FTIR and XRD indicating successful formation of MoS_2/rGO composites. The photodegradation of the 10 ppm of 2-chlorophenol were observed under the exposure of sunlight for 180 minutes and analyzed using UV-VIS to measure percent degradation of 2-chlorophenol. 1% MoS_2/rGO has been chosen as the composite to degrade 2-chlorophenol due to having the highest percent degradation of 51.41% among other photocatalysts.

Keywords: graphene oxide, molybdenum disulfide, photocatalytic degradation, semiconductor photocatalyst, solar irradiation

Abstrak

Kajian in menekankan kajian awal mengenai fotodegradasi 2-klorofenol dengan menggunakan berat ratio pemangkin cahaya MoS_2/rGO yang berlainan dibawah radiasi matahari. 2-klorofenol dikenali sebagai salah satu bahan pencemar yang mempunyai ciri-ciri merbahaya seperti mutagenisiti, karsinogenik dan eko-pencemar. Komposit MoS2/rGO telah disintesis menggunakan kaedah hidroterma. Kaedah fotokatalisis yang menggunakan MoS_2/rGO sebagai pemangkin cahaya untuk merosotkan 2-klorofenol dibawah sinaran matahari. Tiga komposisi MoS_2/rGO yang berlainan (1%, 5% and 15%) telah disintesis menggunakan nisbah berat MoS_2 yang berbeza. Bahan komposit MoS_2, GO dan MoS_2/rGO yang dicirikan menggunakan FTIR dan XRD telah menunjukkan hasil jaya pembentukan komposit MoS_2/rGO dengan jayanya. Penyusutan sinaran oleh 10 ppm 2-klorofenol telah diperhatikan dibawah sinaran matahari selama 180 minit telah dianalisiskan dengan UV-VIS untuk mengukur peratus penyusutan sinaran 2-klorofenol. 1% MoS2/rGO telah dipilih untuk merosotkan 2-klorofenol kerana mempunyai peratusan penyosotan yang tinggi iaitu 51.41%.

Kata kunci: grafena oksida, molibdenum disulfida, degradasi fotokatalitik, pemangkin cahaya semikonduktor, penyinaran suria

Introduction

2-chlorophenol is one of the chemical groups derives from chlorophenol that were made by electrophilic halogenation of phenol with chlorine. It is widely known as one of the most priority pollutants by the US Environmental Protection Agency and World Health Organization (WHO) based on its properties of strong odor, mutagenicity, carcinogenicity and eco-persistent [1]. It is highly toxic and difficult to degrade due to its high thermal and chemical stability [2]. Even with its hazardous characteristics, it is largely used in industrial and commercial scales such as plastic, pesticide, textile, steel, pharmaceutical, iron, paper and wood preserving that were reported to be a major source of chlorophenol compound found in the wastewater [3]. It can affect organisms when it released to the environment. Thus, many studies have been made to eliminate it such as biodegradation, adsorption and Fenton-like advanced oxidation processes. However, due to utilization of extra oxidants, vigorous conditions and intensive energy input, studies are made to search for sustainable technology to degrade 2-chlorophenol. Hence, to overcome the environment issues, solar-driven photocatalysis method is being used to eliminate the contaminant in the wastewater, ground water and rivers.

Photocatalysis is known as one of the advanced oxidation processes which is a method of removing contaminants concerning wastewater effluents. Photocatalysis is a technique to degrade pollutant by using photocatalyst to utilize photogenerated carriers such as electrons and holes in order to start the redox reaction for the conversion of solar to chemical energy [4]. Photocatalysis is one of the advanced treatment technologies to degrade contaminant by using radicals produced from transition-metal oxide photocatalysts involving UV irradiation. To promote indirect hole oxidation, the electron-hole pairs are generated on the photocatalyst surface and the valence band edge of photocatalysts must be better of the oxidation potential of hydroxyl radical, •OH [5].

Table 1 examines the different types of photocatalyst materials for degradation of 2-chlorophenol.

Table 1. Degradation of 2-chlorophenol using photocatalytic degradation

Photocatalyst	Source of Light	Degradation Time	Percentage Degradation	References
Ru/TiO_2	UV light	180 minutes	50%	[6]
Cu doped $BiFeO_3$ and Ba-Cu co-doped $BiFeO_3$	Visible light	70 minutes	98% and 100%	[7]
C_3N_4/TiO_2-NTs in pH of 9 and 12	UV/ Visible light	180 minutes	25.02% and 70.25%	[8]
ZnO-ZnS@PANI	Visible light	240 minutes	89%	[9]
5% Ag-TiO_2	UV light	150 minutes	74%	[10]

Meanwhile, photocatalyst is material that absorbs light energy to facilitate chemical reaction. Semiconductor photocatalyst uses light energy to get desirable chemical transformation through reduction-oxidation (redox) reaction. The photocatalytic semiconductors are commonly used for wastewater treatment, surfaces with self-cleaning and antifogging properties, purification of outdoor air, indoor air deodorization and cancer therapy [11]. It uses suitable band gap to produce strong visible light response with a high photocatalytic presentation, non-toxicity, good thermal, inexpensive and have chemical stability [12].

Graphene is a 2-D structure of hexagonal carbon atom in the sp^2 hybrid orbital that is widely used in electrochemical energy storage, optical and optoelectronics bioengineering due to its outstanding electrical, optical, thermal and mechanical properties. Graphene and its derivatives consist of large surface area, thermal conductivity and carrier mobility. Thus, it has the high potential in energy sensors and microelectronic devices. Integration of GO with other semiconductor can effectively improve the structural stability of the composites and enhance the photocatalytic activity by hindering the recombination of photo-generated carrier. Moreover, GO-based composite can alleviate the serious agglomeration of nanoparticles to a certain extent [13, 14] . Graphene is also known as electron cocatalyst that can improve hydrogen performance of photocatalysts due to its excellent of conductivity [15]. It also has capacity for adsorption of organic pollutants and potential mechanical strength and electrical conductivity [16]. GO can give productive route for highly dispersive, band tunable and few-layer structured of graphene-based materials thus it acts as supportive material for photocatalyst semiconductor [17].

MoS_2 is a 2-D layered n-type semiconductor and has narrow bandgap of 1.20 eV for its few layers where each layers have gap value of 1.90 eV. Thus, it has large specific surface area complex edge structure and abundant unsaturated active sites. It has high thermal stability and strong absorption in visible spectrum region. MoS_2 have limitations as it has limit in energy storage and photocatalytic properties due to high recombination rate of photo-generated electron hole pairs, low energy density and poor electronic conductivity. Combining with other metal oxide nanostructures, it can improve the disadvantages of MoS_2 such as GO can enhance its photocatalytic performances [18, 19]. This can be explained by transfer of photo-generated electrons from MoS_2 conduction band to the graphene [20]. When the band gap of MoS_2 increases due to quantum confinement effects of MoS_2, it will change the redox potential of MoS_2 thus photo-generated electron can be easily migrate [21]. Therefore, this research focusses on preliminary studies on the ability of MoS_2-GO photocatalyst to degrade 2-chlorophenol from aqueous solution under sunlight.

Materials and Methods

Materials

2-chlorophenol, sulphuric acid (H_2SO_4), sodium nitrate ($NaNO_3$), graphite powder, hydrogen peroxide (H_2O_2), hydrochloric acid (HCl), sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$), thiourea (H_2NCSNH_2) were purchased from Merck Sdn. Bhd. Finally, deionized water was supplied from Purite machine model Fusion system (United Kingdom) that has ultrapure water 18.2 M Ω at a flow rate of 48 L/hr.

Synthesis of GO

Graphene oxide was prepared by using modified Hummers method from pure graphite powder. Solution of 23 mL sulphuric acid, H_2SO_4, 0.5 g of sodium nitrate, $NaNO_3$ and 1.0 g of graphite powder were mixed. The solution was stirred for an hour in ice bath until it becomes dark green solution. The solution was stirred for another 4 hours after being removed from the ice bath where the solution turned into dark grey solution. The solution was transferred into a water bath and the temperature was raised to 35˚C and stirred for 30 minutes. The solution was added 250 mL of deionized water and was stirred for another 15 minutes while heated up to 70˚C then added dropwise of 10 mL of hydrogen peroxide, H_2O_2. Cool the solution as there will be exothermic reaction occurs. The solution was centrifuged three times at 3000rpm for 10 minutes and the supernatant drained, and the residual was washed HCl to obtain GO solution. The GO solution was dried

the using the oven at ˚C for 24 hours to gain powdered form of GO [22].

Synthesis of MoS_2

Molybdenum disulfide, MoS_2 was synthesized by using hydrothermal method. Firstly, 3.0 g sodium molybdate dihydrate, $Na_2MoO_4.2H_2O$, 3.8 g of thiourea, H_2NCSNH_2, 80 mL of deionized water were mixed and stirred for 30 minutes. 10 mL of 1M HCl was added to the solution and transferred into 100 mL Teflon stainless steel autoclave and heated in the oven at 120˚C for 24 hours. The solution was kept in the room temperature to let it cooled before centrifuged three times and washed with deionized water. The solution was dried in the oven at 70˚C for 24 hours [23].

Synthesis of MoS_2/GO

The solution was prepared by using ex-situ and hydrothermal method by adding 0.001 g of molybdenum disulfide, MoS_2 and 20 mL of deionized water were mixed and sonicated for 30 minutes. Another solution was prepared of 0.1 g of graphene oxide, GO and added with 20 mL of deionized water before sonicated for 30 minutes. Both solutions were added together and sonicate for another 30 minutes before transferred into the Teflon stainless steel and dry in the oven overnight at 120˚C. Centrifuged and washed the solution with deionized water three times after cooling at room temperature and dried the product at 80˚C for 24 hours [24].

Characterisations

Perkin Elmer Fourier Transform model 100 was used to identify the functional group of the sample by producing infrared absorption spectrum in the range 600-4000 cm^{-1}. Ultraviolet-visible spectroscopy solid Perkin Elmer model was used to measure light absorbance of the sample at the wavelength range of 200 to 500 nm. The model Oxford Instruments of X-ray Diffractometer (XRD) was used to determine the crystallographic structure of composites.

Degradation of 2-Chlorophenol by using MoS_2/ GO

The composites undergo screening process to determine the optimum sorbent for the extraction of 2-chlorophenol by exposing under the sunlight for 3 hours. 20 mL of 10 ppm of 2-chlorophenol was added to 10 mg photocatalyst. The degradation of 2-chlorophenol was measured using UV-visible in wavelength between 230 nm to 500 nm. Degradation percentage of 2-chlorophenol was determined by using the formula:

Degradation of 2-chlorophenol(%)

$$= \frac{Ao - A}{Ao} \times 100 \qquad (1)$$

where A_o is an initial absorption of 2-chlorophenol and A is an absorption of 2-chlorophenol at sampling time after the degradation process [25]. The natural sunlight photocatalysis was measured from April to May 2021 (Wednesday to Friday) at (2° 47' 20.3892" N, 102° 13' 4.6992" E) in Kuala Pilah, Negeri Sembilan Malaysia, (10AM- 1PM). The daily solar irradiance could reach 4.21–5.56 $kWhm^{-2}$ [26].

Results and Discussion

Figure 1 below show IR spectra of the representative composites within the range of 650 to 4000 cm^{-1}. The broad peak at 3353.94 cm^{-1} in Figure 1 represented the hydroxyl group stretching vibration of graphene oxide composite. Besides, there is also C=O stretch of carboxylic acid at 1737.46 cm^{-1}. The peak at 1163.52 cm^{-1} and 1042.35 cm^{-1} assigned to C-O-C, epoxy and C-O stretch. Next, the peak at 1588.21 cm^{-1} were corresponding to the C=C aromatic stretch group. The peaks at 862.28 cm^{-1} that were observed at the MoS2 composite corresponds to the Mo-O bond. [24]. FTIR spectroscopy in Figure 1 confirms the presence of MoS_2 and GO which indicates successful preparation of MoS_2/GO in the experiment. Moreover, there are band shifts and intensified of the spectra for different concentration of the 1%, 5% and 15% MoS_2/GO composites.

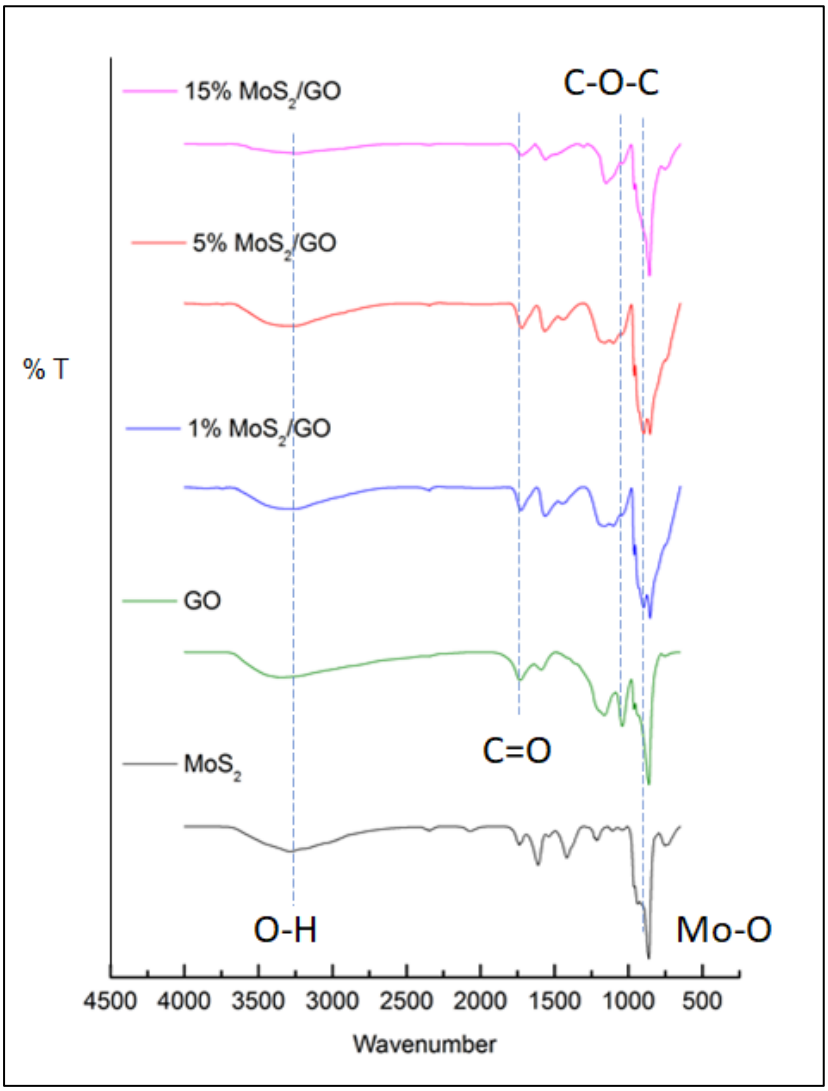

Figure 1. FTIR spectrum of MoS_2, GO and MoS_2/GO composites (1%, 5% and 15%)

Figure 2 shows diffractogram of MoS_2, GO and MoS_2/GO composites (1%, 5% and 15%). Based on the figure, the diffraction peak of GO can be seen at 2^{θ} = 24.92˚ indicating interplanar space of the GO sheet (001) [25]. MoS_2 also showed peak corresponding to (100) plane at 2^{θ} = 32.65˚ which successfully appeared at all of the mass ratio of MoS_2/GO (1%, 5% and 15%)[27][28]. Thus, it confirmed that GO and MoS_2 composites were successfully integrated.

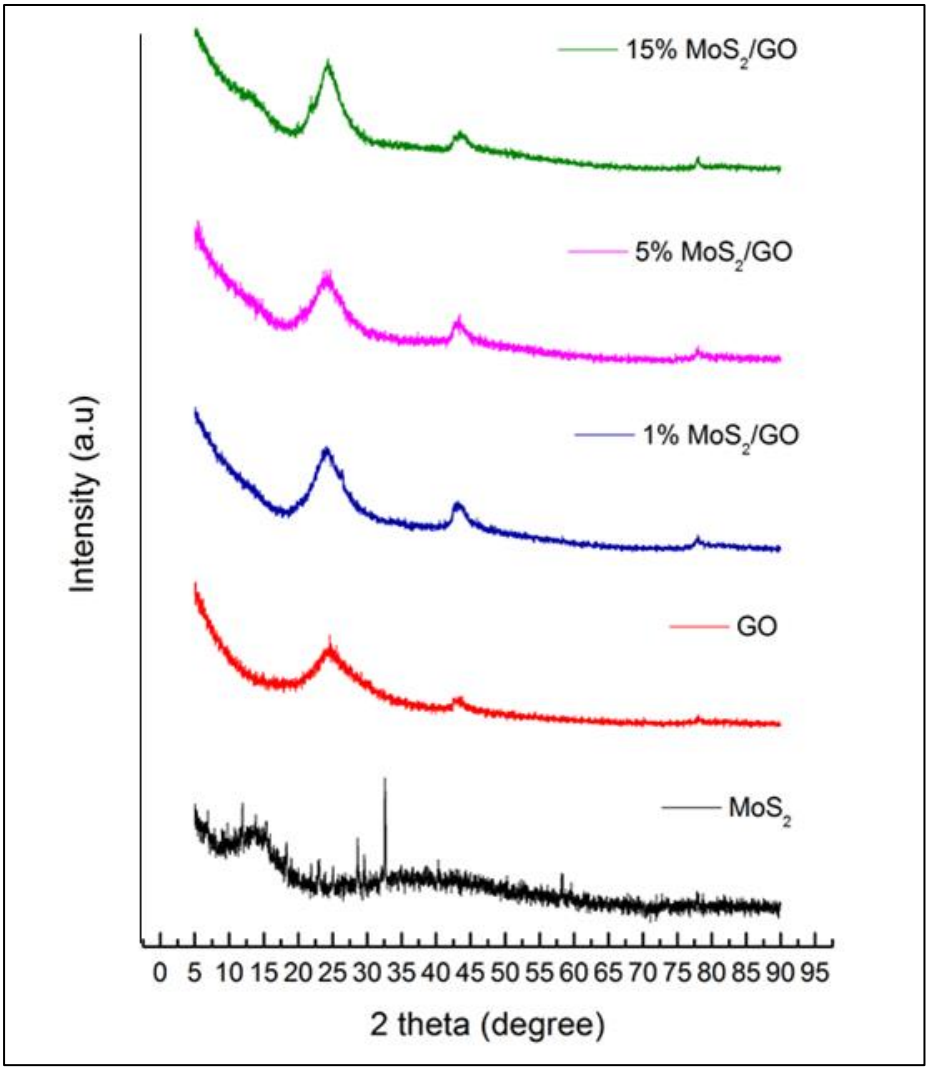

Figure 2. XRD patterns of MoS_2, GO, MoS_2/GO composite (1%, 5% and 15%)

Figure 3 reveals the photodegradation of 2-chlorophenol under sunlight using MoS_2, GO, MoS_2/GO composite (1%, 5% and 15%) photocatalyst. Successful photodegradation was monitored by decreasing in the intensity of absorbance with time irradiation. Hence, 1% MoS_2/GO proved for having lowest absorbance of 0.086 Abs. Based on the Figure 3, the MoS_2 showed an increase in absorption at 0.198 Abs of the visible light in the range of ~275 nm due to the absorption of MoS_2 in the 2-chlorophenol pollutant as it hinders complete usage of sunlight for charge carrier generation due to having wider band gap and have low mobility preventing charge transfer [29]. Moreover, rapid recombination of photogenerated electron hole pairs can decline its photocatalytic activities and poor dispersion happened when the nanomaterials are accumulated [30]. Meanwhile, Figure 4 presents the degradation percentage of all composites where the highest percent degradation is 51.41% for 1% MoS_2/GO composite due to the compatibility between graphene and MoS_2 as it enhanced the catalytic activities of amorphous producing highly efficient hydrogen production under visible light irradiation [31].

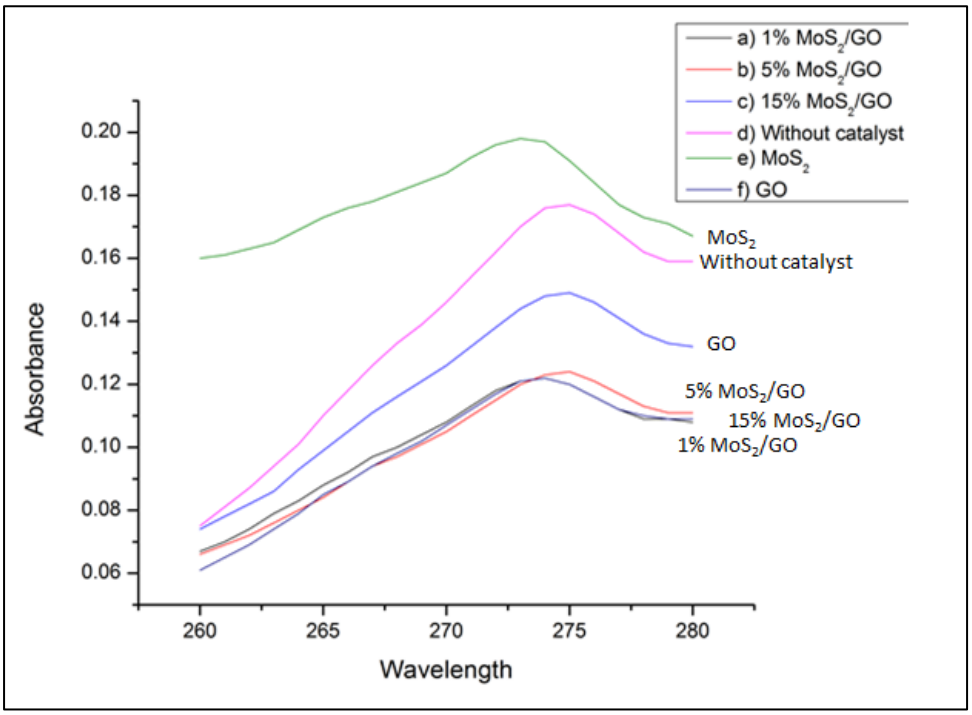

Figure 3. Different absorbance for different catalyst for 3 hours

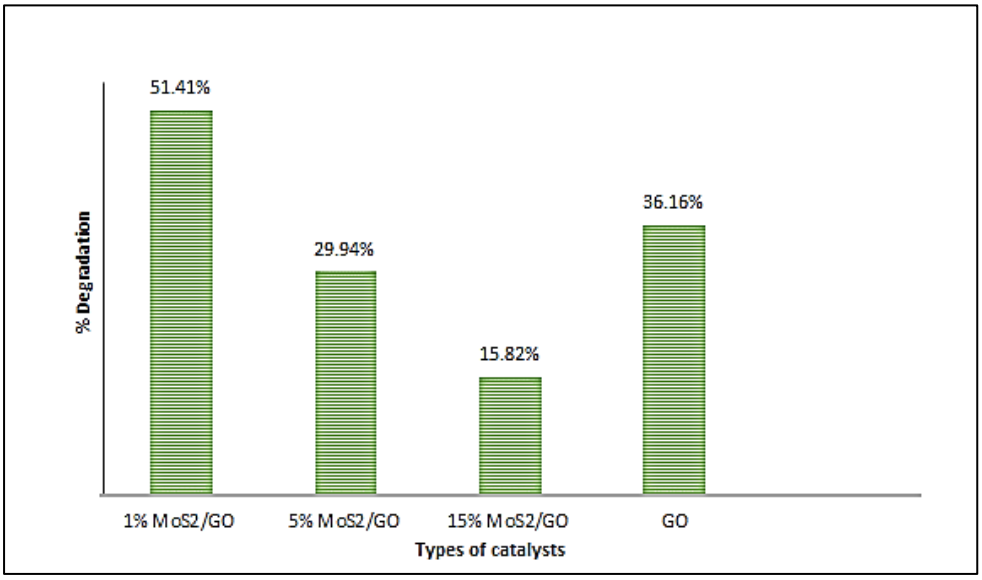

Figure 4. Degradation percentage of 2-chlorophenol for different catalysts under sunlight

Conclusion

MoS_2/GO composites were successfully prepared using ex-situ and hydrothermal method. This can be seen from the analyses of the FTIR, UV-Vis and XRD which confirmed the functional group and incorporated of MoS_2 and GO composites. From the findings, all of the

composites prepared have the potential in degrading 2-chlorophenol under sunlight assisted degradation. 1% MoS_2/GO was revealed as the best composite to degrade 2-chlorophenol with the percentage degradation of 51.41%. Further optimization and controlled experiments can be done to tackle the most prominent condition for 2-chlorophenol to degrade with sunlight as the main source of light to achieve highest degradation percentage within a short time.

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