Investigating the Effect of the Light Source on The Performance of Oxide and Non-Oxide Transition Metal Photocatalysts– A Comparative Study of UV, UV-Vis, and Sunlight

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***Abstract*—**Wastewaters often contain toxic organic micropollutants (OMPs), possibly adversely affecting human health and aquatic life upon exposure. Conventional treatment technologies applied in wastewater treatment have many drawbacks, such as secondary pollution, incomplete removal of OMPs, inefficiency/non-destructiveness of some or most persistent OMPs, and cost implications. Water treatment utilizing photocatalysis (Advanced Oxidation Processes) is a cutting-edge, alternative, and sustainable technology that has recently received a lot of interest, due to its potential for green energy and wastewater remediation. Conventionally, visible-light photocatalysts are either unstable upon illumination with light or possess low activity. Alternative and more efficient visible-light photocatalysts are needed to meet the requirements of future environmental and energy technologies driven by solar energy. Therefore, this study aimed to evaluate the effect of light sources (UV, UV-vis, and sunlight) on various semiconductor photocatalysts for treating municipal wastewater. The photocatalysts considered were Titanium dioxide (TiO_2), Iron III oxide (Fe_2O_3), Zinc Sulphide (ZnS), and Copper Sulphide (CuS). Operating conditions such as catalyst load (1.5 g/L), mixing speed (90 rpm), and exposure time (60 minutes) were investigated using the experimental analysis one-factor-at-a-time (OFAT) approach. The treated effluent's water quality parameter chemical oxygen demand (COD) was analyzed to evaluate the photocatalytic efficacy of the various light sources. It was found that the UV-visible light source favoured the UV-visible absorption wavelength for Fe2O3 and ZnS, with the optimum COD removal efficiency at 72.25% and 70.87% at 10 and 50 minutes, respectively. CuS's best COD removal efficiency was 70.20% at 20 minutes. The comparative study revealed UV-visible irradiation to be the most effective, and sunlight was shown to be more effective than UV light irradiation. This demonstrated that Fe_2O_3, ZnS, and CuS can be used as alternative photocatalysts for wastewater treatment under UV-vis irradiation.

***Keywords*—**Advanced Oxidation Process, Photocatalysis; Semiconductors; Chemical Oxygen Demand; Organic Micropollutants; Wastewater, UV, UV-Vis, Sunlight

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I. Introduction

The duo of water and energy crises are challenges that have sparked the global drive towards sustainable water and energy-renewable remediation techniques to meet the growing demand for clean water and minimize the negative environmental effects [1, 2]. Global Sustainable Development Goals (SDGs) set forth by the United Nations, states that there must be universal and equitable access to safe and affordable drinking water for all by 2030. The latest United Nations statistics revealed that the efforts toward achieving this goal are still slow in most countries [3]. With that being said South Africa is the epitome of these challenges [4]. Uncontrolled disposal of untreated wastewater into water resources is associated with significant hazardous impacts on humans and the environment [5]. The challenges facing this wastewater are the complexity and heterogeneity of the biorecalcitrant pollutants, such as organic micropollutants (OMP) and their derivatives, disinfection byproducts (DBPs), endocrine-disrupting chemicals (EDCs), pharmaceuticals and personal care products (PPCPs), pesticides, E. coli, heavy metals, etc. [6]. Consequentially, the need for highly efficient remediation technologies is essential to reduce the long-lasting harmful impact of these pollutants on the environment and humans and to meet the required standards for water quality [7, 8].

The traditional wastewater treatment techniques have been challenged because of the increased demand for clean water and are progressively failing to satisfy the required criteria. Traditional techniques include biological treatment, physio-chemical treatment, membrane filtering, absorption, and oxidation. However, these techniques possess several drawbacks: (i) they are unable to efficiently break down and remove a wide range of organic pollutants; (ii) there is secondary pollutant generation, slurry, and sludge formation; (iii) they require pre-treatment or post-treatment steps to mineralise the pollutants found in wastewater entirely; (iv) some pollutants or contaminants require external chemicals; (v) these processes usually consume large amounts of energy; (vi) they necessitate routine maintenance and are; (vii) expensive [7].

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Photocatalysis (advanced oxidation process) is acknowledged as a viable eco-conscious solution in wastewater treatment settings with great potential for the remediation of wastewater by the degradation of resistant organic pollutants, non-selectively and swiftly from wastewater using semiconductors as photocatalyst materials [9]. Photocatalysis via semiconductors is revealed to be one of the most efficient, cost-effective, environmentally friendly, and sustainable technologies that utilize solar energy to address energy and environmental crises. It has a wide range of uses in the production of hydrogen clean energy as well as organic degradation, which has been favored among many researchers [10-12]. The photocatalytic semiconductor mechanism is divided into a photoinduced charge separation mechanism and a redox mechanism [13]. This method allows for the removal of a wide range of pollutants while also aiding in the complete mineralization or breakdown of complex pollutants into simpler substances like water, carbon dioxide, and inorganic ions without requiring the use of external chemicals under normal operating conditions [14]. Additionally, photocatalysts can be regenerated and reused for further processing.

According to recently published research, photocatalysis offers a great deal of potential for breaking down harmful metal ions, dyes, hydrocarbons, pesticides, bacteria, and microorganisms in wastewater. Metal semiconductors such as TiO_2, ZnO, CdS, and so on are among the most common. TiO_2 is characterized by its great oxidation ability, low cost, good stability, high activity, low production cost, and non-toxicity. Its excellent hydrolysis activity under ultraviolet (UV) light and its vast applicability in the photolysis of aquatic hydrogen and the degradation of pollutants have garnered a lot of interest. TiO_2 is an ideal photocatalyst. However, due to its large energy band energy (3.2 eV for the anatase phase and 3.0 eV for the rutile phase) and high electron-hole recombination rate, it can only be utilized under ultraviolet light irradiation [12, 15]. Since the majority of photocatalysts are activated by UV light, a constant source of UV light is necessary to maintain the reaction, which is not a financially feasible option and entails high maintenance expenses [7]. Thus, the large-scale industrial photocatalytic implementation of these techniques is presently limited. [12].

Thus, assessing the efficacy of various visible-light photocatalysts that utilize the freely available solar energy for water treatment, and promoting the industrial application of photocatalytic technology is significant for the reclamation of wastewater technology. Traditional visible-light photocatalysts are either unstable upon illumination with light or possess low activity. Therefore, exploring efficient visible-light photocatalysts has become a top priority to meet the requirements of future environmental and energy technologies driven by solar energy. Therefore, this study aimed to investigate the effect of the light source on the performance of oxide and non-oxide transition metal photocatalysts using UV, UV-vis, and sunlight irradiation light sources for municipal wastewater treatment. The photocatalysts considered were Titanium dioxide (TiO_2), Iron (III) oxide (Fe_2O_3), Zinc Sulphate (ZnS), and Copper Sulphate (CuS). The water quality parameter chemical oxygen demand (COD) of the treated effluent was analyzed to evaluate the effectiveness of the various light sources at optimum conditions. A comparative study was also conducted, and it was estimated based on the average COD removal percentage for each respective photocatalyst. The subsequent sections provide a detailed description of the methodology (section II), experimental setup (section III), results and discussion (section IV), and conclusions (section V).

II. Materials and Methods

A. Chemicals

The photocatalysts used in this study were Titanium (IV) Oxide (TiO_2), Iron (III) oxide / Ferric Oxide (Fe_2O_3), Zinc Sulphide (ZnS), Copper Sulphide (CuS) and were supplied by Sigma Aldrich, Durban. The physicochemical characteristics of the semiconductor photocatalysts considered are presented in Table I [16-20].

TABLE I
Properties of Raw Municipal and Synthetic Wastewater before Treatment

Semiconductor Photocatalyst	Band Gap (eV)	Absorbance Wavelength (nm)
TiO_2	3.2	275-405
Fe_2O_3	2.2	320-420
ZnS	3.6	375-575
CuS	1.6-2.2	380-800

B. Effluent sample

Synthetic wastewater was simulated using analytical-grade chemicals, which represent the typical composition of the treatment plant and was used as the basis for analyzing the experimental results obtained. The composition of the chemicals used was adapted from Munien et al. [21]. The raw wastewater was obtained from a local South African municipality wastewater treatment plant based in the Kwa-Zulu Natal province.

C. Effluent sample characterization

The raw and synthetic wastewater was characterized by the following characteristics (Table II).

TABLE II
Properties of Raw Municipal and Synthetic Wastewater before Treatment

Water Quality Parameter	Raw Wastewater	Synthetic Wastewater
pH	8.12	8.63
Colour	1949	3950
Turbidity	124	519
COD	8950	9150

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D. Analytical methods

The COD was analyzed by Spectrophotometer DR 3900 (HACH), using the stored programs 435- COD HR. The COD removal percentages were determined by using Equation (1):

$$\text{COD removal \%} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

where C_i and C_f are the initial and the final COD concentrations (mg/L) before and after treatment, respectively [22].

III. Experimental Setup

A. UV, UV-visible, and sunlight

The effect of the various light sources (UV, UV-visible, and sunlight) on the performance of photocatalytic degradation was investigated. The investigation was conducted in three experiments i.e. UV (experiment 1); UV-visible (experiment 2); and sunlight (experiment 3) using TiO_2, Fe_2O_3, ZnS, and CuS photocatalysts. The investigative conditions were (catalyst load = 1.5 g/L) and (mixing speed = 90 rpm) for 60 minutes [23], using the experimental analysis one-factor-at-a-time (OFAT) approach. The water quality parameter chemical oxygen demand (COD) of the treated effluent was analyzed to evaluate the effectiveness of the various light sources at optimum conditions. The experiments utilizing (UV and UV-visible) were carried out using a laboratory-scale photochemical reactor (Lelesil Innovative Systems) [21]. The sunlight experimental setup consisted of four identical 2 L beakers, where all wastewater was fed, and all equipped with a magnetic stirrer, whilst exposed to sunlight to excite the catalysts to trigger a reaction. The sunlight was incident on the beakers to ensure uniformity of light distribution. The light intensity (LUX) under UV, UV-vis, and sunlight irradiation was recorded as 600x100 (60 000), 1910x100 (191 000), and 1337x100 (133 700) LUX, respectively using a MT940 handheld Lux Meter

IV. Results and Discussion

A. Effect of UV, UV-visible, and Sunlight irradiation on the photocatalytic treatment

The exposure time was varied from 10 to 60 minutes, whilst the mixing speed and catalyst load were kept constant at 90 rpm and 1.5 g/L, respectively for all three experiments. The light intensity (Lux) was noted as 600x100 (60 000) Lux under UV irradiation. As shown in Fig. 1a, ZnS and CuS optimum values (40.78% and 36.02%) were obtained at 50 minutes due to their visible light (long) absorption wavelength (375-575 nm / 380-800 nm) as compared to TiO_2 with a shorter absorbance wavelength (275-405 nm) and large band gap energy (3.2 Ev) which was achieved sooner at a shorter time of 20 minutes and 36.20% under the UV light irradiation (Table I). Fe_2O_3's shorter absorbance wavelength (320-420 nm) was also favored sooner under UV light irradiation. It is also apparent that ZnS's large energy band gap of 3.6 eV enhanced optimum contaminant removal under UV irradiation. Additionally, ZnS and CuS possess higher stability (crystal lattice) and have a large absorbance wavelength of (375-575 nm) and (380-800 nm) respectively. Therefore, an increase in contact time (interaction time) between the pollutant and the surface of the photocatalyst aided a higher COD removal percentage as the availability of hydroxyl radicals for the oxidation of pollutants present in wastewater increases with an increase in contact time [24, 25]. It is also observed that TiO_2 (275-405 nm) and Fe_2O_3 (320-420 nm) with shorter absorbance wavelengths favored faster peaks under UV light, and rightfully so, as these wavelengths fall within the UV-visible regions on the electromagnetic spectrum [26]. It is also known that the larger band gaps and shorter wavelengths absorb UV light. Hence optimum values and UV light response for TiO_2 and Fe_2O_3 were likely to be obtained sooner [27]. A decrease in COD removal percentage is thereafter observed which could be due to the tendency of TiO_2 and Fe_2O_3 (powder) to agglomerate in the water system and therefore the interactive surface of the photocatalyst became saturated and dissociated over time [28, 29].

The light intensity (Lux) under UV-vis irradiation was noted as 1910x100 (191 000) Lux. As shown in Fig. 1b, the UV-visible light source favored the UV-visible absorption wavelength for Fe_2O_3 (320-420 nm) and ZnS (375-575 nm), with the optimum COD removal values at 72.25% and 70.87% at 10 and 50 minutes, respectively. CuS's best COD removal efficiency was 70.20% at 20 minutes, which precisely illustrated its compatibility with the UV-visible light source favoring its visible absorption wavelength (380-800 nm) and at a short time interval of 20 minutes as compared to the optimum COD removal efficiency of 36.02% at 50 minutes in experiment 1 (UV light source). TiO_2 showed the best COD removal efficiency at 46.66% at 30 minutes. This could be attributed to the UV-visible light source promoting a longer absorption wavelength incident on the TiO_2 photocatalyst surface, which elevated the removal efficiency as compared to 36.20% at 20 minutes in experiment 1. Compared to experiment 1 (UV light source), experiment 2 (UV-visible) has higher COD removal percentages.

The light intensity (Lux) under sunlight irradiation was noted as 133 700 Lux. Fig. 1c shows the effect of sunlight on COD removal percentage; sunlight consists of visible light rays, which favoured the Fe_2O_3 -visible light absorption wavelength, with the best COD removal efficiency of 57.14% at 20 minutes. The sunlight also favored CuS's visible light absorption wavelength at a gradual rate, with the optimum COD removal efficiency of 47.16% at 60 minutes. The best COD removal efficiency for TiO_2 and ZnS was 56.56% and 42.50% at 40 and 20 minutes, respectively, and thereafter gradually decreased. This could be due to TiO_2 and ZnS‘s large energy band gap of 3.2 eV and 3.6 eV, respectively, which makes it difficult to oxidize or hydrolyze the organics at a high electron- hole recombination rate as ideally large band gap energy can absorb UV light (shorter wavelength) and small energy band gap can absorb visible light (longer wavelength) [16, 27].

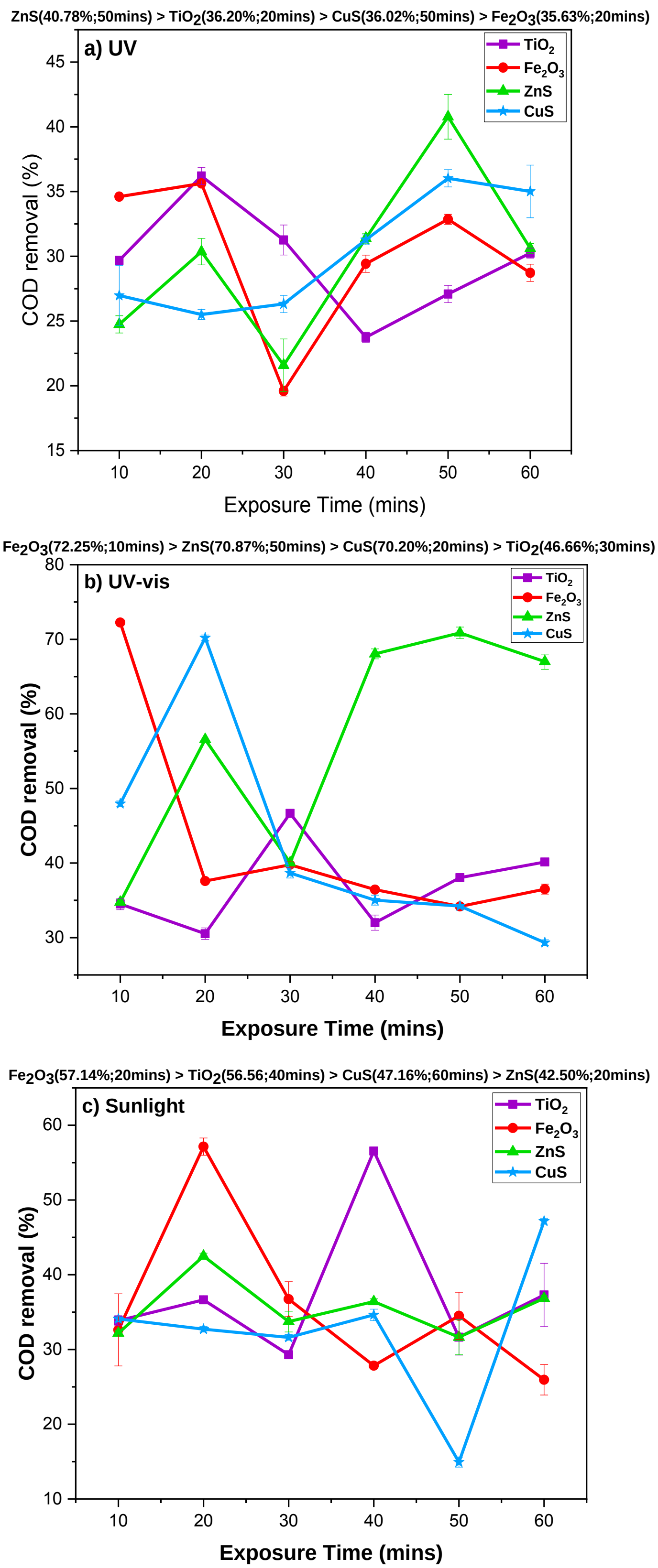

Fig. 1 Effect of (a) UV, (b) UV-visible (c) Sunlight irradiation on COD removal using TiO_2, Fe_2O_3, ZnS, and CuS

B. Comparative study for UV, UV-visible, and Sunlight

The comparative study was estimated based on the average COD removal percentage for each respective photocatalyst. Fig. 2 shows the comparative study for the effect of UV, UV-visible, and Sunlight irradiation on the photocatalytic treatment for all four photocatalysts (TiO_2, Fe_2O_3, ZnS, CuS) with the averaged COD removal efficiency represented. The desirable COD removal performance is at 35%. UV-visible irradiation was estimated to be the most efficient method for COD removal among all three examined. ZnS had an estimated COD removal efficiency of 56.21% and was found to be the best under the UV-visible irradiation, followed by Fe_2O_3 with 42.79%, CuS with 42.57%, and TiO_2 with 36.98% COD removal efficiency.

Sunlight was shown to be more effective than the UV light irradiation. TiO_2 had an estimated COD removal efficiency of 37.55% and was the best under sunlight, followed by Fe_2O_3 with 35.80%, ZnS with 35.55%, and CuS with 32.53% COD removal efficiency. Concerning the solar spectrum, approximately 52-55% of the sunlight reaching the Earth's surface is Infrared (IR), 42-43% is visible, and 3-5% is UV light. Almost half of the sunlight on the Earth's surface falls within the visible region (400-700 nm) [30]. Consequently, this could justify the Sunlight's superiority over UV light in correspondence with the favorable visible absorbance wavelength of photocatalysts and their respective band gaps. The UV light irradiation illustrated the lowest COD removal efficiency compared to UV-visible and Sunlight.

CuS had an estimated COD removal efficiency of 30.18% and was found to be the best under the UV light source, followed by Fe_2O_3 with 30.14%, ZnS with 29.91%, and TiO_2 with 29.70% COD removal efficiency. This could be due to the restriction within the UV region absorbance wavelength, limiting the photocatalysts' potential COD removal efficiency. Also, UV light has a shorter wavelength and relatively higher energy than visible light, leading to phototoxicity or photodamage and lower penetrability in the samples [31]. UV light irradiation might cause problems such as photobleaching, material damage, and reduced fatigue resistance [32, 33]. Furthermore, the light intensity (Lux) under UV-vis irradiation was noted as 1910x100 (191 000) Lux, which was found to be the highest amongst the light sources examined. Therefore, a higher COD removal efficiency was achieved by the respective photocatalysts in comparison to sunlight (133 700 Lux) or UV (60 000 Lux) [UV-vis > Sunlight > UV].

Therefore, UV-visible irradiation was the most effective, with the desirable COD removal performance at 35%, among the light sources examined in this study. These results obtained are in agreement with the literature that suggests a smaller band gap (longer absorbance wavelength) promotes visible light absorption [34]. One of the major challenges or limitations of TiO_2 photocatalysis on OMPs is the need to harvest visible light photons efficiently. Additionally, almost half of the sunlight on the earth's surface falls within the visible region (400-700 nm). Therefore, efficiently capturing visible light photons is of utmost importance in addressing this challenge [30, 35, 36]. This study successfully reveals possibilities for utilising visible light irradiation using alternative photocatalysts, as absorption toward visible light, is the central part of the solar spectrum.

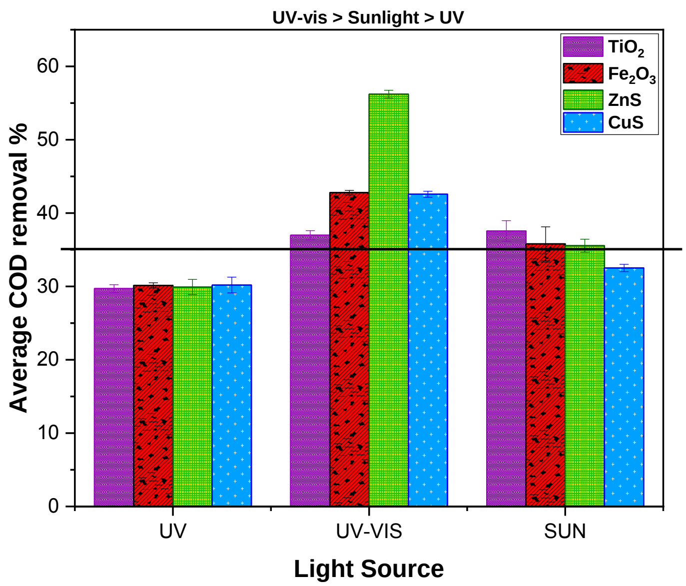

Fig. 2 Comparative study between UV, UV-visible, and Sunlight irradiation for COD removal efficiency (%) using TiO_2, Fe_2O_3, ZnS, and CuS photocatalysts.

V. Conclusion

The UV-visible light source favored the UV-visible absorption wavelength for Fe_2O_3 and ZnS, with the optimum COD removal efficiency of 72.25% and 70.87% at 10 and 50 minutes, respectively. CuS's best COD removal efficiency was 70.20% at 20 minutes which precisely illustrated its compatibility with the UV-visible light source favoring its visible absorption wavelength. The comparative study revealed UV-visible irradiation to be the most effective. The light intensity (Lux) under UV-vis irradiation was noted as 1910x100 (191 000) Lux, which was found to be the highest amongst the light sources examined. Sunlight was shown to be more effective than UV light irradiation as most of the sunlight on the earth's surface falls within the visible region (400-700 nm). Consequently, this could justify the Sunlight's superiority over UV light in correspondence with the favourable visible absorbance wavelength of photocatalysts and their respective band gaps. The shorter wavelength of UV light and relatively higher energy than visible light may have caused phototoxicity or photodamage as well as lower penetrability in the samples

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Authors' Contributions

Conceptualization, C.M., and E.K.T.; methodology, C.M.; software, C.M., and E.K.T.; validation, C.M., and E.K.T.; formal analysis, C.M., and E.K.T.; investigation, C.M..; resources, S.R. and E.K.T.; data curation, C.M.; writing—original draft preparation, C.M., and E.K.T.; writing—review and editing, C.M., S.R. and E.K.T.; visualization, C.M.; supervision, S.R., and E.K.T.; project administration, S.R. and E.K.T. All authors have read and agreed to the published version of the manuscript.

Declaration of Competing Interests

We have no financial or personal affiliations that could have influenced this paper's findings.

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