Effect of Different UV and Solar Radiation Wavelengths on the Photocatalysis Treatment of 2,4,6-Trichlorophenol Contaminated Wastewater: Parametric and Kinetic Studies

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Remediation of 2,4,6-trichlorophenol (2,4,6-TCP) contaminated wastewater was carried out using photocatalysis treatment method in the presence of TiO_2 photocatalyst. Parametric and kinetic studies were investigated, in which, the effect of different UV and solar radiation wavelengths, initial concentrations of 2,4,6-TCP (10, 20, 30, 40 and 50 ppm), TiO_2 dosages (0.02, 0.04, 0.06 and 0.08 mg l^{-1}) and initial pH (4,7, and 10) on the degradation of 2,4,6-TCP were investigated. According to the result obtained, the degradation efficiency of 2,4,6- TCP was directly proportional to the increase in the TiO_2 dosage and initial pH of the solution. However, the degradation efficiency decreased with the increase in the initial concentration of 2,4,6-TCP and irradiation wavelength of UV light or in the presence of solar radiation. UV-C, the shortest wavelength, exhibited the highest degradation percentage (88%) compared to UV-B (83%), UV-A (79%) and solar (30%). The highest TiO_2 dosage (0.08 mg/ml), gave the best degradation effect (97%). Alkaline solution condition, pH 10.0, gave the best degradation removal percentage (98%), followed by pH 7.0 (97%) and acidic condition, pH 4.0 (65%). Degradation of 2,4,6-TCP using TiO_2 achieved remarkable reduction in chemical oxygen demand (COD) about 82% removal efficiency but no change was observed with the biological oxygen demand (BOD) analysis. The photodegradation of 2,4,6-TCP followed the first order reaction kinetics.

Key words: 2,4,6-TCP; TiO_2; photocatalysis; COD; BOD; kinetic study; first order; UV; solar; radiation; contaminated wastewater

In the 21st century, one of the significant threats to the quality of human life is water pollution [1]. Numerous organic contaminants of emerging concerns are found in surface water and groundwater such as aliphatic compounds (chlorobenzene and carbon tetrachloride), chlorinated aromatic, dyes and phenolic compounds (phenol, chlorophenols, and nitrophenol) [2]. Some of the examples of chlorinated compounds which come from industry effluents such as pulp mills [3] that have been detected in treated wastewater [4] include chlorinated guaiacols and catechols [5].

Chlorophenol or chlorinated phenols, is a group of chemicals which contains chlorine atoms between first and fifth carbon atoms in the aromatic ring bonded to the phenol functional group [6]. 2,4,6-trichlorophenol (TCP) (shown in the Figure 1) is one of the 19 chlorinated phenol derivatives and was chosen as the target pollutant in this study. 2,4,6-TCP is normally used in the manufacturing of herbicides, pharmaceutics, pesticides, wood preservatives and defoliants. Apart from that, dye manufacturing, pesticide, wood, paper and pulp industries, pharmaceuticals, and parts of water treatment also generate chlorophenols [7]. 2,4,6-TCP is a highly carcinogenic, toxic, and mutagenic pollutant [8] and have been reported to cause severe health disorders such as gastrointestinal effects and cancer [9]. Besides that, this chemical also causes altered pulmonary functions, chronic bronchitis and cough [10]. Due to the high persistence, toxicity and even carcinogenic properties of 2,4,6-TCP, it's the elimination from any aqueous mediums is highly vital.

In this study, the photocatalysis process, a subset of the Advanced Oxidation Processes, was used to photodegrade the target pollutant. Our previous studies using photolysis process yielded satisfactory results in this effort [11-12]. Photocatalysis works based on the following principal; the photocatalyst (in this study we used TiO_2) is activated by UV or solar light energy and generates electron-hole pairs. These electron-hole pairs react with water and oxygen molecules to produce highly reactive hydroxyl radicals (OH•) and

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the superoxide radicals $O_2\bullet^-$. These reactive oxygen species (ROS) can break down various pollutants in wastewater. The study aims to evaluate the degradation efficiency of 2,4,6-TCP by TiO_2 photocatalysis under different parameters (different wavelength irradiation, an initial concentration of 2,4,6-TCP, TiO_2 dosage and initial pH of solution) and followed by kinetic analysis.
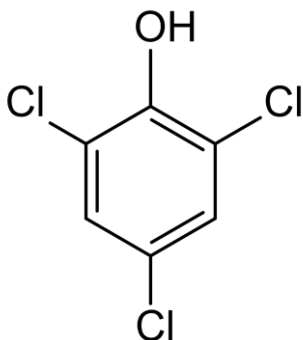

Figure 1. Chemical structure of 2,4,6-trichlorophenol [11].

EXPERIMENTAL SECTION

Preparation of Stock Solution

Powdered 2,4,6-TCP (99% pure), and Degussa P25-TiO_2 nano-particles were obtained from Sigma-Aldrich and used without further treatment. The preparation of the stock solution of 1000 mg l^{-1} 2,4,6-TCP was carried out by adding 2,4,6-TCP to distilled water. 2,4,6-TCP solutions with different initial concentrations were prepared from the stock solution with the addition of distilled water [11].

Photocatalytic Experiment

The experiments were conducted in a batch photoreactor as shown in Figure 2. The degradation of 2,4,6-TCP was monitored by measuring the maximum absorbance value (λ_{max}) of 2,4,6-TCP by using UV-Vis spectrophotometer. All the experiments were carried out based on the aforementioned parameters (wavelength of irradiation, initial concentration of 2,4,6-TCP, TiO_2 dosage and pH of the solution). All the photocatalysis experiments were conducted for one hour with samples collected at every 10 min for further analysis.

Different irradiation sources, UV-A, UV-B, UV-C, and Solar were used to determine their effects on the remediation of 2,4,6-TCP. All the lamps employed were low-pressure mercury lamps with the same specifications. They differ only by their wavelengths; UV-C – 254 nm, UV-B – 311 nm, UV-A – 365 nm and solar lamp-maximum at 610 nm (Joseph *et al.* 2017). The TiO_2 photocatalyst used is the commercially available P25 Degussa [13]. The TiO_2 dosage studied were from 0.02 mg/ml to 0.08 mg/ml with an interval of 0.02 mg/ml. The pH of 2,4,6-TCP solution was adjusted to pH 4.0, pH 7.0 and pH 10.0 by using sodium hydroxide (NaOH) and hydrochloric acid (HCl), accordingly. These pH values were chosen to represent the acidic (pH 4), neutral (pH 7) and alkaline (pH 10) conditions. The initial concentrations of 2,4,6-TCP that were chosen are 10,20,30, 40 and 50 ppm. All the samples were analyzed using UV-Vis Spectrophotometer to study the change in the concentration of 2,4,6-TCP. The degradation efficiency of 2,4,6-TCP was calculated as follows [13]:

$$\text{Degradation efficiency } (\%) = \frac{(A_0 - A_t)}{A_0} \times 100 \qquad (1)$$

Where, A_0 = Initial absorbance of 2,4,6-TCP at $t = 0$ min
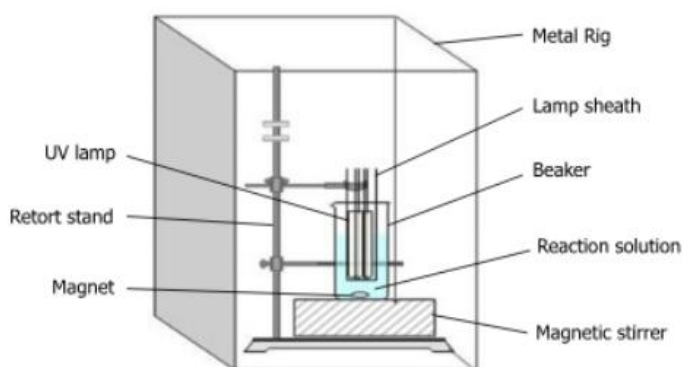A_t = Absorbance of 2,4,6-TCP at t min

Figure 2. Schematic diagram of the batch photoreactor.

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The optimum condition for the highest degradation of 2,4,6-TCP was the determined from each parameter studied and was further analysed with the COD and BOD analysis.

RESULTS AND DISCUSSION

2,4,6-TCP Calibration Spectrum

Maximum absorbance, λ_{max}, of 2,4,6-TCP was determined using UV-Vis spectrophotometer. The λ_{max} of 2, 4, 6-trichlorophenol was determined to be at 285 nm as is shown in Figure 3.

The calibration curve of 2,4,6-TCP at 285 nm showed a good regression coefficient ($R^2 = 0.9999$) and agreed well with the Beer-Lambert Law (not presented).

A dark experiment was carried out as a control experiment. The 2,4,6-TCP solution was kept under similar conditions as the photocatalysis experiment but without any irradiation from the UV or solar lamps [13]. This was conducted to study the effect of adsorption of 2,4,6-TCP by TiO_2. Based on the tabulated graph in Figure 4 above, there was no significant change in the concentration of 2,4,6-TCP solution throughout the 60 min contact time under dark condition. The adsorption of 2,4,6-TCP by TiO_2 under non-irradiated condition was very low (0.12%) and is negligible. This is because, based on previous studies [14], TiO_2 is chemically stable and inert under dark conditions. Therefore, it can be concluded that the adsorption effect by TiO_2 is insignificant.
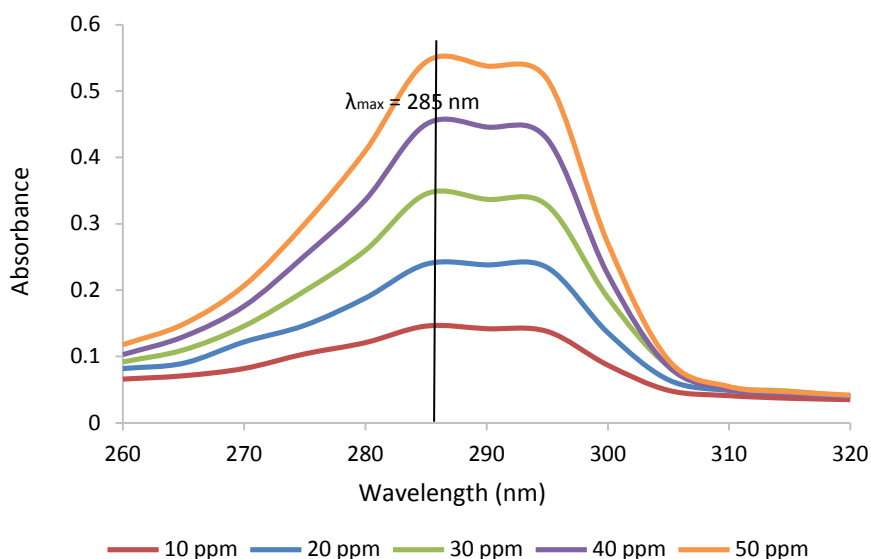

Figure 3. Absorbance spectrum of 2,4,6-TCP at different initial concentrations of 2,4,6-TCP.
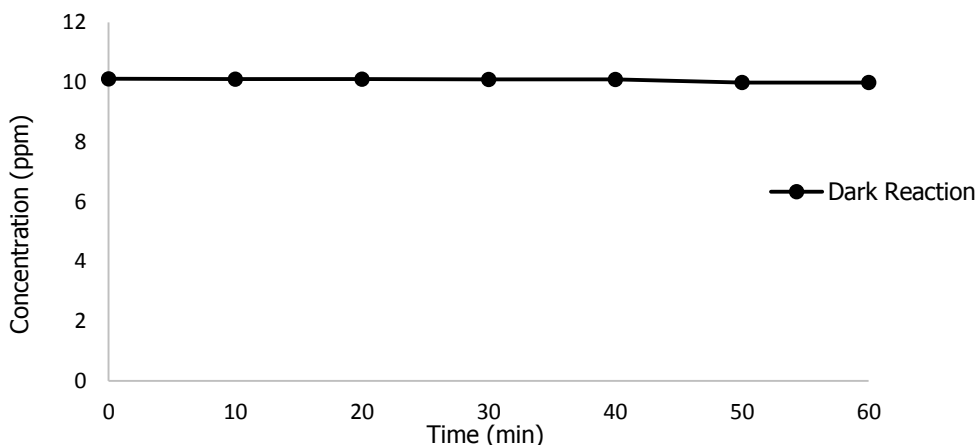
Dark Experiment

Figure 4. Adsorption of 2,4,6-TCP by TiO_2 photocatalyst under non-irradiated experimental condition.

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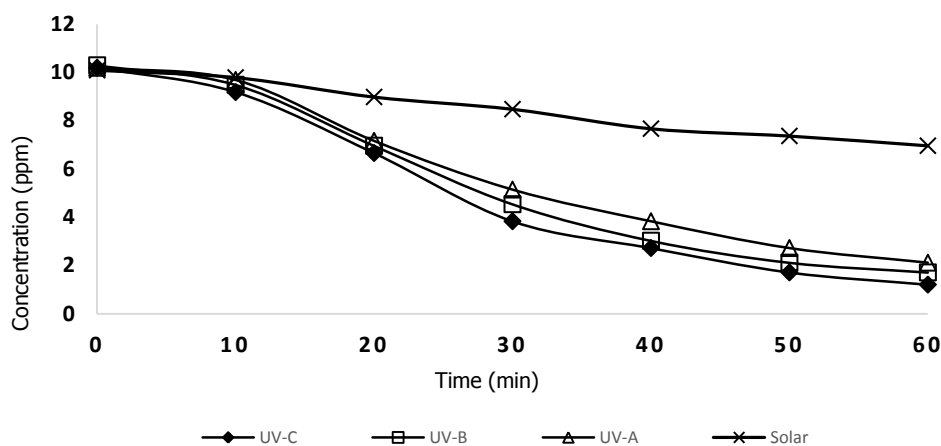nated Wastewater: Parametric and Kinetic Studies

Figure 5. Photocatalysis degradation of 2,4,6-TCP using UV-A,
UV-B, UV-C and solar lamp for 1 h.

The Degradation Effect of 2,4,6-TCP under Different Irradiation Wavelength

The effect of different lamp sources (different wavelength) on the degradation of 2,4,6-TCP was investigated. The graph of concentration against time for all artificial UV lamps and solar lamp is plotted in Figure 5. For this experimental parameter, the initial concentration of 2,4,6-TCP and the TiO_2 dosage were fixed at 10 ppm and 0.02 mg/ml, respectively. It was found that the concentration of 2,4,6-TCP decreased with the increasing irradiation time. The result showed that the UV and solar irradiation wavelength affects the efficiency of the photodegradation of 2,4,6-TCP's [15].

As shown in Figure 5, UV-C irradiation had the highest degradation efficiency whereas solar lamp showed the lowest degradation efficiency of 2,4,6-TCP. UV-C irradiation achieved about 88.12% of degradation efficiency within an hour. While the degradation efficiencies of UV-B and UV-A were 83.33% and 79.21%, respectively. However, only 29.59% of 2,4,6-TCP was degraded in an hour under the solar lamp irradiation.

All of the photodegradation under various wavelengths were unable to achieve 100% of degradation within one hour. This is because photocatalysis is a time-dependent process. However, complete degradation has been reported to be achievable when the experimental time was extended [16]. UV-C with the irradiation wavelength in the range of 100-280 nm had the most effect on the target pollutant due to its higher penetration ability by its high energy photons. Therefore, it can to produce more electron-holes pairs on the surface of TiO_2 which translated into higher production of hydroxyl radicals [17]. As such, UV-C

was chosen as the irradiation wavelength for all the following photocatalytic experiments due to its ability to induce photocatalysis by TiO_2 [11-13,18].

The Degradation Effect of Different Initial Concentrations of 2,4,6-TCP

The relationship of the different initial concentrations of 2,4,6-TCP on its degradation efficiency is shown in the Figure 6. The initial concentration tested were 10, 20, 30, 40 and 50 ppm, while the TiO_2 dosage was fixed at 0.02 mg/ml. These initial concentrations were chosen as they showed good linearity in the calibration curve with the R^2 = 0.9999. Based on Figure 6, the degradation efficiency of 2,4,6-TCP was inversely proportional to the initial concentration of pollutants [19].

When the initial concentration of the 2,4,6-TCP increased, the probability of collision between the hydroxyl radicals and the targeted pollutant molecules increased as well. So, at a low initial concentration such as 10 ppm, the degradation efficiency of 2,4,6-TCP is higher. However, the *in vitr*o half-life of the hydroxyl radicals is in the nanosecond range and is therefore unable to react with the excess amount of 2,4,6-TCP molecules at high concentration (50 ppm). As such, by increasing the initial 2,4,6-TCP concentration will result in a decreased degradation efficiency. In addition to this, the amount of available active sites on the TiO_2 is limited as the photocatalyst dosage was kept constant for different initial concentration. Therefore, when the initial concentration of 2,4,6-TCP increased, there are some leftover 2,4,6-TCP in the solution at the end of the experiment [20] that did not undergo degradation during the experimental duration of 1 h.
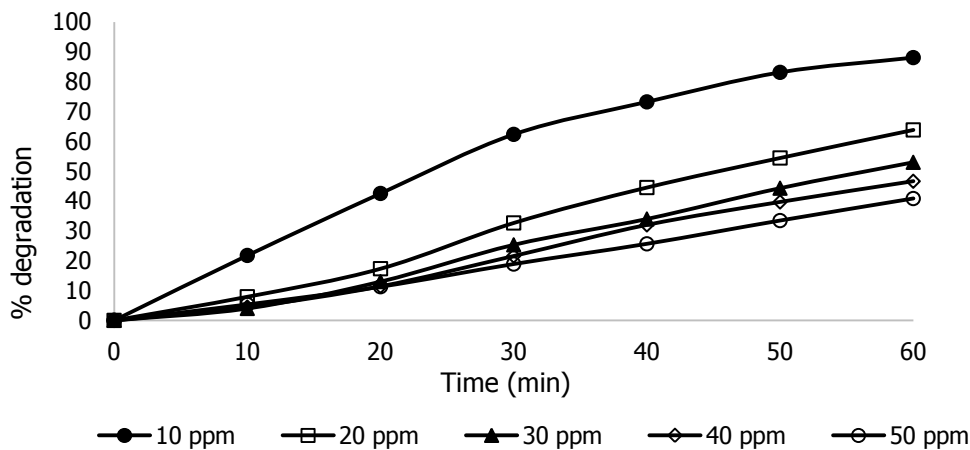
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Figure 6. The degradation efficiency of 2,4,6-TCP with various initial concentrations of 2,4,6-TCP
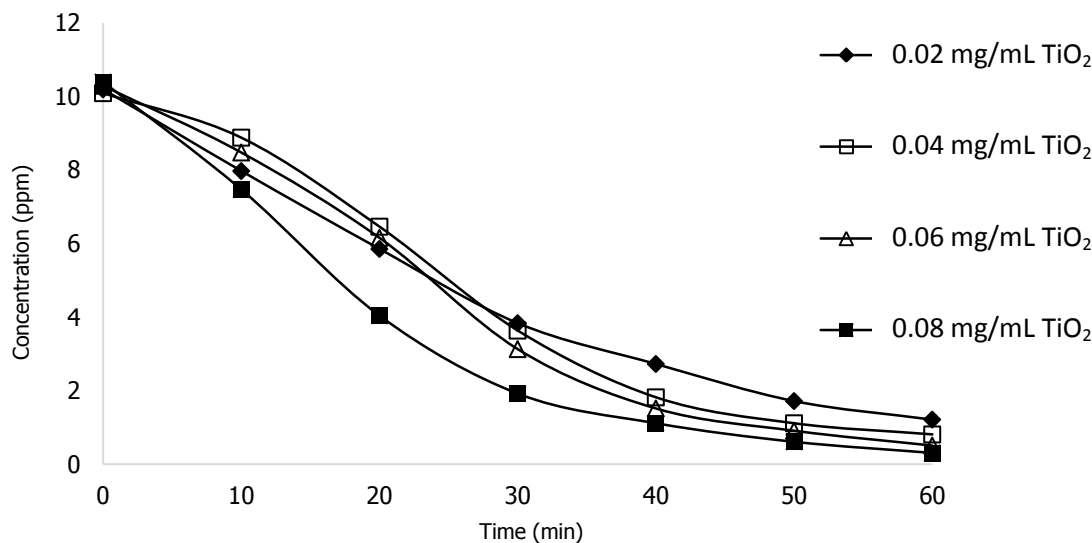under UV-C irradiation for 1 h.

Figure 7. Photocatalysis degradation of 2,4,6-TCP using different TiO_2 dosages under UV-C
irradiation for one hour.

The Degradation Effect of Different TiO_2 Dosage

The effect of different TiO_2 dosages (0.02 mg/ml, 0.04 mg/ml, 0.06 mg/ml and 0.08 mg/ml) on the degradation efficiency of 2,4,6-TCP was investigated. The graph with different initial dosage against time was plotted and is presented in Figure 7. The degradation efficiency of 2,4,6-TCP increases slightly when higher catalyst dosage was used due to availability of more active sites on the surface of TiO_2.

Figure 7 showed that no significant changes in the degradation efficiencies of 2,4,6-TCP between the different dosages were observed. This is because of the dosages used in this parameter only differed a little from 0.02 mg/ml to 0.08 mg/ml. However, the degradation efficiency of 0.02 mg/ml and 0.08 mg/ml TiO_2 dosages were significant at 88.12% and 97.09%, respectively. This was because when the TiO_2 dosages increased, the number of active sites on the TiO_2 photocatalyst increased accordingly. Thus, there are more sites for the adsorption of H_2O or OH^-[17]. This directly increased the amount of $HO^{\bullet}$ conversion, which was the most important oxidant in this photocatalysis process. Therefore, increasing the TiO_2 dosage will increase the degradation efficiency until an optimum level is reached.
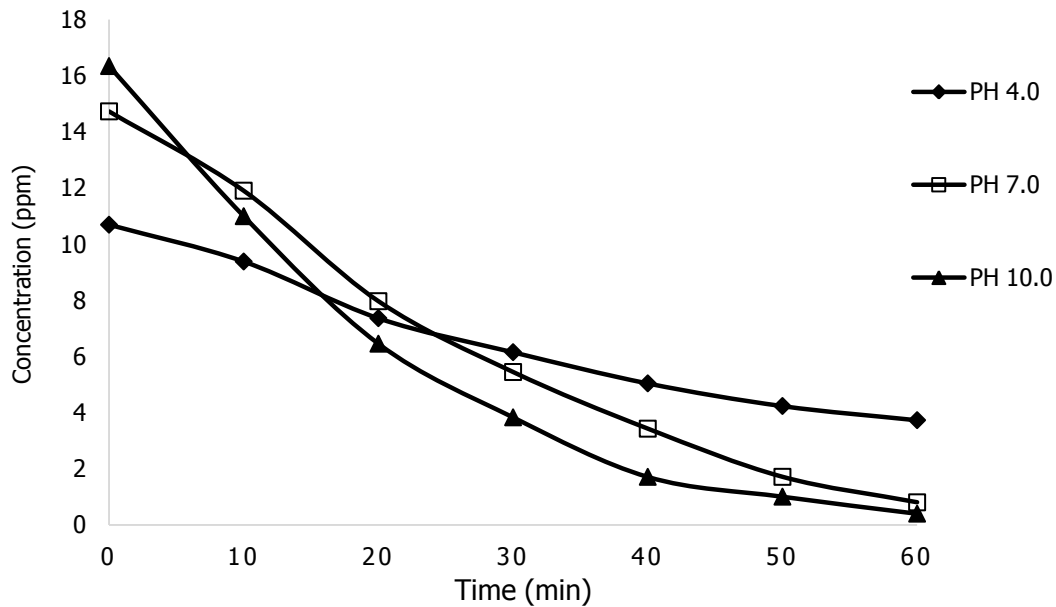
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Figure 8. Photocatalytic degradation of 2,4,6-TCP under different pH
using UV-C irradiation and 0.02 mg/ml TiO_2.

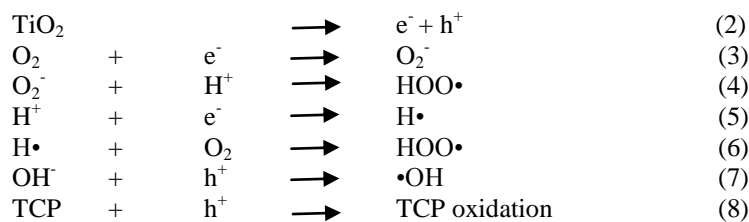## The Degradation Effect of Different pH of Solution

The effect of pH on the degradation of 2,4,6-TCP was investigated. The pH for 2,4,6-TCP aqueous solution were fixed at pH 4.0, 7.0 and 10.0. Figure 8 shows the effect of initial solution pH on the TiO_2 photocatalytic degradation of 2,4,6-TCP.

Photocatalytic degradation efficiency is highly influenced by the pH of the 2,4,6-TCP solution [20]. Based on Figure 8, 2,4,6-TCP had the highest degradation under alkaline condition (pH 10). The photocatalysis at pH 10.0 achieved 97.53% degradation efficiency which was almost 100% whereas pH 4.0 showed the least degradation which was only 65.09%. Therefore, it has been proven that the pH of the 2,4,6-TCP solution was highly influenced by the degradation degree.

When the pH increased, the degradation efficiency increased corres-pondingly as there is an increase in the number of hydroxyl ions in the solution [22]. By trapping the photo-induced holes with OH^- ions, more hydroxyl radicals (•OH) are formed in the solution. In order words, when there are more hydroxyl radicals, photodegradation of TCP will become more efficiently via radical reaction [21]. When the experiment was conducted under acidic condition, the photodegradation is lower due to the lack of OH^- ions [23]. Besides that, chlorophenolate anion species may be degraded by hydroxyl radicals more easily. Therefore, there is a significant increase in degradation efficiency when pH of solution was increased from acidic to alkaline medium.

The higher degradation effect was found at higher pH (pH 10.0), indicating that the generation of hydroxyl radicals played an important role in TCP photocatalysi [21]. The following equation from Equation 2-8 shows the basic mechanism of generation of hydroxyl radicals by using TiO_2 as a photocatalyst in this study [21,24].

$$TiO_2 \longrightarrow e^- + h^+ \qquad (2)$$
$$O_2 + e^- \longrightarrow O_2^- \qquad (3)$$
$$O_2^- + H^+ \longrightarrow HOO• \qquad (4)$$
$$H^+ + e^- \longrightarrow H• \qquad (5)$$
$$H• + O_2 \longrightarrow HOO• \qquad (6)$$
$$OH^- + h^+ \longrightarrow •OH \qquad (7)$$
$$TCP + h^+ \longrightarrow TCP\ oxidation \qquad (8)$$

The degradation reaction proposed is shown in Equation 9 [27].

$$+ \quad •OH \longrightarrow CO_2 + H_2O + Minerals\ [9].$$

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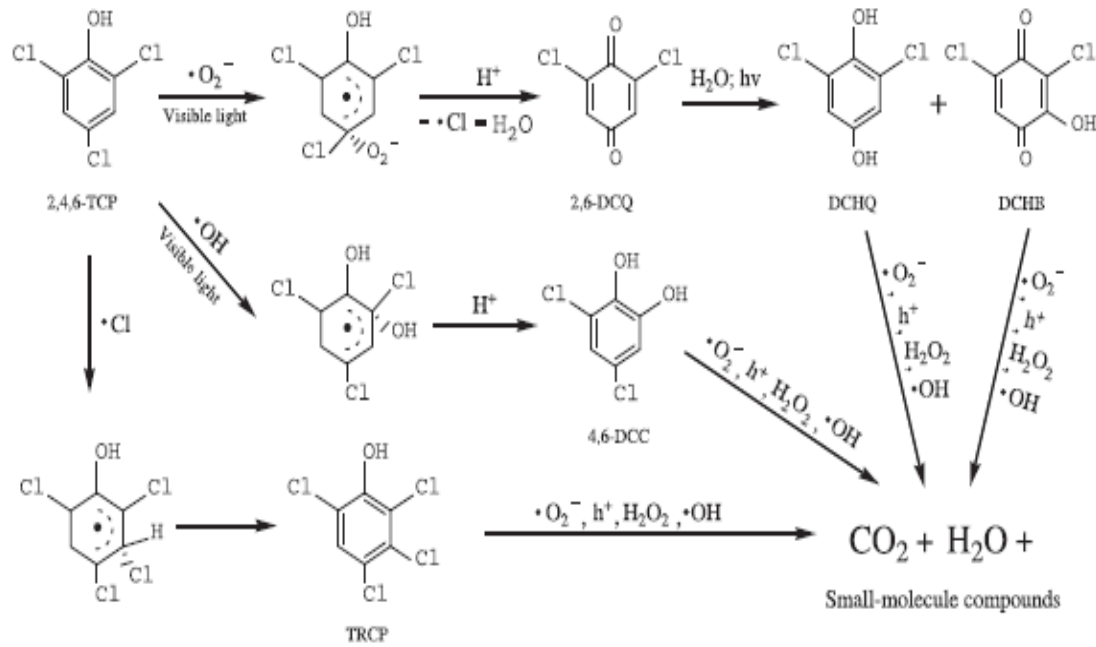

Figure 9. Possible degradation pathways of TCP as reported
by previous research [28].

The 2,4,6-TCP will break into intermediates and further degradation process of organic substance occurs with the aid of •OH which will mineralize the chlorophenol into the final products as shown in Equation 10 [24, 28].
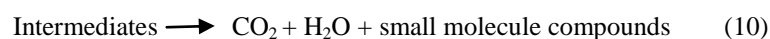
The BOD$_5$ and COD Analysis

For this analysis, the optimum condition with 10 ppm of 2, 4, 6-TCP, pH 10 and UV-C irradiation was used for the further BOD$_5$ and COD analysis.

From Table 1, the initial COD value of 2, 4, 6-TCP was 11 and after the treatment was 2. The percentage removal of COD was calculated according to Equation 11.

The COD removal percentage is 81.82%. The higher the percentage COD removal, it means that the toxic effect of TCP in the solution decreases (the solution become cleaner after treatment) [29]. For the BOD$_5$, the value remained unchanged from beginning until the end of treatment throughout an hour. Since distilled water was used to prepare the 2, 4, 6-TCP solution with 2,4,6-TCP being a biocide and irradiated with UV-C (germicidal lamp), there is a high probability that any microbe activity would have been eliminated before, during and after the experiment was completed. As a result, the BOD$_5$ value did not change [30].

$$\text{Intermediates} \longrightarrow CO_2 + H_2O + \text{small molecule compounds} \qquad (10)$$

$$\text{COD removal \%} = \frac{\text{before treatment} - \text{after treatment}}{\text{before treatment}} \times 100\% \qquad (11)$$

Table 1. Results of analysis of BOD$_5$ and COD before and after treatment separately.

Sample Marking Parameter	1 Before treatment	2 After treatment	Test method	Removal %
BOD$_5$ (mg/l)	2	2	APHA 5210B	0
COD (mg/l)	11	2	HACH 8000	82

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The Kinetic Study

The kinetic study is performed with the aim to determine the rate constants for 2, 4, 6-TCP-TiO$_2$ decomposition reaction. The photocatalytic degradation of 2, 4, 6-TCP followed the first order kinetics [31, 32, 33]. In this study, graph of ln (C_0/C_t) against time was plotted to investigate the reaction kinetics of each parameter studied.

The Effect of Different Irradiation Wavelength

Table 2 shows the excellent regression correlation and the rate constants for the effect of irradiation wavelength on the photocatalytic degradation of 2, 4, 6-TCP as reported by others [34].

Based on the regression correlation values from Table 2 and Figure 10, all of the different light sources fitted well with the first order kinetics. Among all of the radiations, UV-C radiation showed

the best regression correlation value and highest rate constant with 0.9948 and 0.0365 min^{-1}, respectively. The rate constant reflects that UV-C will take the shortest time to complete degradation of 2, 4, 6-TCP. Since the rate constant increases when the irradiation wavelength decreases from solar to UV-C irradiations, it can be concluded that the shorter the irradiation wavelength, the higher the rate constant and degradation efficiency.

The Effect of Initial Concentration of 2,4,6-TCP

Table 3 shows the effect of initial TCP concentration on the photocatalysis of 2, 4, 6-TCP. The experiment conducted with 10 ppm initial concentration of TCP had the highest rate constant and regression correlation with a value of 0.0365 min^{-1} and 0.9948, respectively. Generally, the rate constant reduced as the initial concentration increased. This indicates that the degradation rate of TCP will decrease if the initial TCP concentration was increased [20].

Table 2. The rate constant and regression correlation for the effect of different irradiation wavelength on the photodegradation of 2,4,6-TCP in aqueous solution.

Experiment	Rate constant, k (min^{-1})	Regression correlation (R^2)
UV-A	0.0281	0.9684
UV-B	0.0329	0.9813
UV-C	0.0365	0.9948
Solar	0.0067	0.9733

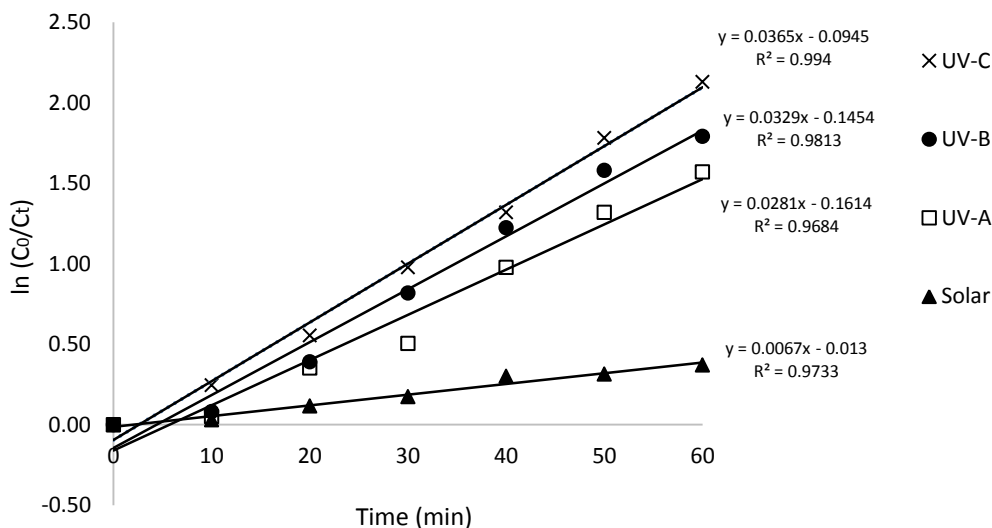**Figure 10.** Kinetics reaction of different irradiation sources of 2,4,6-TCP.

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Table 3. The rate constant and regression correlation for the effects of initial 2, 4, 6-TCP concentration on the photocatalysis of 2, 4, 6-TCP.

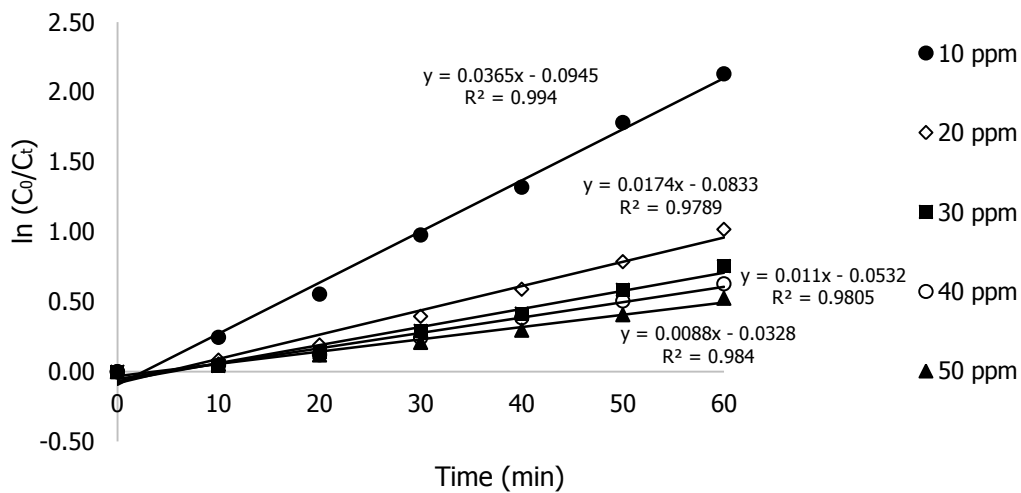| Experiment	Rate constant, k (min^{-1})	Regression correlation (R^2)
10 ppm	0.0365	0.9948
20 ppm	0.0174	0.9789
30 ppm	0.0130	0.9755
40 ppm	0.0110	0.9805
50 ppm	0.0088	0.9840

Figure 11. Kinetics reaction of different initial concentrations of 2,4,6-TCP.

The Effect of TiO$_2$ Photocatalyst Dosage

Table 4 shows the effects of TiO$_2$ dosage on the photodegradation of TCP via photocatalysis within an hour.

The results displayed in Table 4 and Figure 12 shows that all the experiments fitted well in the first order reaction kinetics. The TiO$_2$ catalyst dosage of 0.08 mg/ml gave the highest regression correlation with the value of $R^2 = 0.9945$. A trend can be observed in relation with the rate constants. By increasing the TiO$_2$ dosage, the rate constant increased in tandem. This is because when TiO$_2$ dosage increases, the number of available of active sites on the catalyst increases as well [20].

Table 4. The rate constant and regression correlation for the effects of TiO$_2$ dosage on the photocatalysis of 2, 4, 6-TCP.

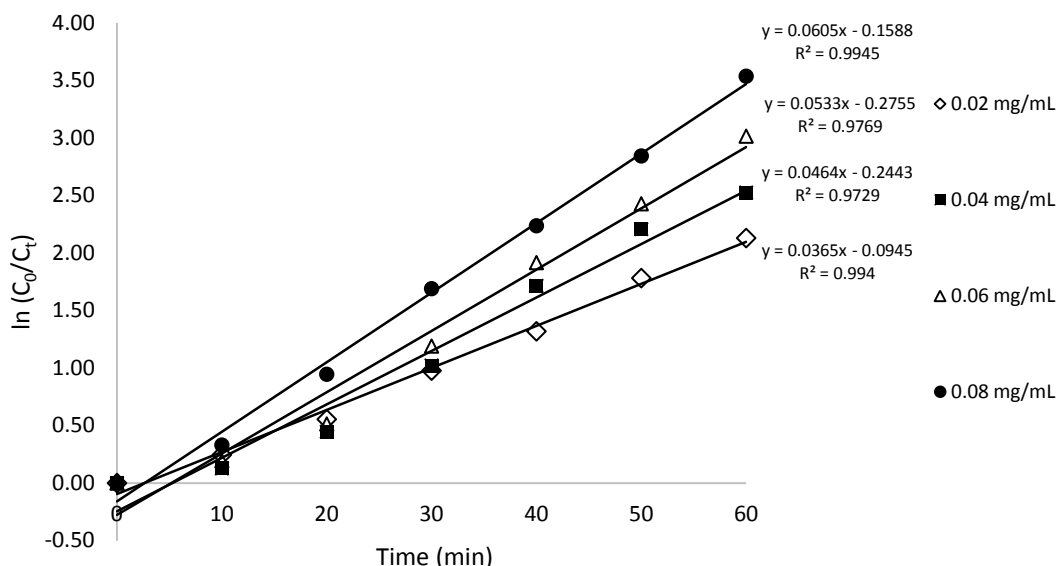| Experiment	Rate constant, k (min^{-1})	Regression correlation (R^2)
0.02 mg/ml	0.0365	0.9940
0.04 mg/ml	0.0464	0.9729
0.06 mg/ml	0.0533	0.9769
0.08 mg/ml	0.0605	0.9945

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Figure 12. Reaction kinetics on the effect of different TiO$_2$ dosage on the degradation rate of 2,4,6-TCP.

CONCLUSION

This study was conducted to investigate the degradation efficiency of TiO$_2$ photocatalysis on 2,4,6-TCP solution, which is, colourless in aqueous solution. Various parameters were investigated which demonstrated that the degradation of 2,4,6-TCP was affected by the initial concentration of TCP, initial pH solution, TiO$_2$ dosage and different types of irradiation sources within the one hour experiment duration. The degradation rate increased with longer contact time but higher initial concentration lowered the degradation rate. When the TiO$_2$ dosage increased, the degradation rate increased as well and UV-C irradiation, gave the best degradation percentage due to direct photolysis coupled with photocatalysis effect on the TCP solution. As a conclusion, the degradation of TCP exhibited the best performance under UV-C irradiation, alkaline condition, higher TiO$_2$ dosage and lower initial concentration of TCP. In our efforts to turn back the tide of environmental degradation, it is important to explore new technology which offers such possibilities.

ACKNOWLEDGEMENT

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