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# Photocatalytic Degradation of Oxytetracycline Dihydrate from Aqueous Solution Using Nano ZnO and ZnO.xBaTiO$_3$ (x = 3%, 18%, 33% and 48%)

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## Abstract

Traditional wastewater mostly contains pharmaceutical ingredients. Therefore, the wastewater must be completely free from antibiotics before its release into the environment. In the present study, photocatalytic degradation was done to investigate the removal efficiency of Oxytetracycline Dihydrate (OTC) using ZnO, ZnO/3%BaTiO$_3$ (3 BZ), ZnO/18%BaTiO$_3$ (18 BZ), ZnO/33%BaTiO$_3$ (33 BZ) and ZnO/48%BaTiO$_3$ (48 BZ) under UV light. After the exposure time of 420 min, about 99.57% and 97.87% of OTC was degraded using ZnO and 3 BZ respectively. Further, increasing the amount of BaTiO$_3$ in ZnO prolongs the degradation time. Therefore, faster efficiency was found using ZnO nanoparticles. The observed reaction rate constant using ZnO was 0.00933 min$^{-1}$ which decreased to 0.00532 min$^{-1}$ using 48 BZ, indicating the decrease of reaction rate for increasing the amount of BaTiO$_3$. Hence, the use of ZnO photocatalyst is anticipated to be a promising technique for the photocatalytic degradation of contaminated wastewater with oxytetracycline antibiotics using UV light.

## Keywords

Advanced Oxidation Process (AOP), Antibiotics, Zinc Oxide (ZnO) and Barium Titanate (BaTiO$_3$)

## 1. Introduction

The presence of pharmaceuticals in the aquatic environment has become an emerging environmental issue of concern that scientific researchers are trying to

resolve [1]. Pharmaceutical pollutants can cause incurable harm to animals, the aquatic environment and humans. OTC is an emerging pharmaceutical ingredient that is used for the treatment of infections of the respiratory system, urinary system, soft tissues and skin [2]. Releasing these antibiotics to the surrounding and aquatic environment can cause pollution and various human diseases. As a result of its harmful impact on human health, livestock and aquatic life, feasible techniques with high removal efficiency are highly desirable. However, photocatalytic degradation is one of the most preferred solutions that could play a pivotal role in removing this kind of pollutant from wastewater. Various types of photocatalysts and techniques are developed for efficient degradation and complete removal of pollutants. Various treatment processes such as, biological [3], chemical [4], physical (e.g. membrane separation and adsorption) [5] [6] and advanced oxidation processes (AOPs) have been implemented to remove antibiotics from wastewater [3]. It is not always efficient to use membrane separation and adsorption to completely remove wastewater pollutants [7]. Moreover, chemical processes can produce secondary byproducts that are not desirable for the removal of antibiotics [8]. Due to having toxicity and biopersistent nature, the biological treatment process cannot completely remove the antibiotics [3]. Due to generating different types of radicals such as peroxide, superoxide, hydroxyl and persulfate radicals within the process, AOPs are promising [9] [10] [11] [12]. Many researchers have used photocatalytic degradation of various organic dyes and antibiotics which are the serious wastewater pollutants.

ZnO is a prominent photocatalyst due to its unique properties such as wide bandgap, low cost, high chemical stability, inertness, non-toxicity and low cost [13] [14] [15] [16] [17]. However, there are some limitations such as rapid recombination of electron-hole pairs, not being excited by visible light and deterioration of photocatalytic performances over time due to the loss of photocatalyst particles [18]. ZnO also suffers from photocorrosion [19]. Combining ZnO with other materials such as metals, semiconductors and nanocarbons can solve these limitations. Previously, Maha K. Mohsin *et al.* showed that by applying catalytic ozonation, 94% of oxytetracycline antibiotics were removed using ZnO nanoparticles [20]. Lili Lian *et al.* used Mg@ZnO$Co_3O_4$ to successfully adsorb OTC from polluted water [21].

$BaTiO_3$ is a promising photocatalyst that has been recently used to adsorb pollutants from wastewater. Manjusha Passi *et al.* used Ag-$BaTiO_3$/GO to degrade crystal violet dye and ofloxacin under visible light [22]. Moreover, Joa̋o H.O.S. Pereira *et al.* showed that 100% OTC was removed using $TiO_2$ photocatalyst under natural and simulated solar radiation [23].

Previously, Zeynep Ciğeroğlu *et al.* showed that ciprofloxacin antibiotics can be removed using ZnO/$BaTiO_3$ adsorbent [24]. Moreover, Zeynep Ciğeroğlu *et al.* investigated that adsorption of tetracycline antibiotics can be done successfully from aqueous solutions using g-$C_3N_4$-ZnO-$BaTiO_3$ nanocomposite [25]. To our best knowledge, conducting photocatalytic degradation of OTC antibiotics under UV light using ZnO.x$BaTiO_3$ was not before. In the present study, ZnO,

$ZnO/3\%BaTiO_3$, $ZnO/18\%BaTiO_3$, $ZnO/33\%BaTiO_3$ and $ZnO/48\%BaTiO_3$ photocatalysts were used to degrade oxytetracycline antibiotics under UV light from aqueous solution. The kinetic study, the influence of irradiation time and the influence of different catalysts were investigated.

## 2. Materials and Methods

### 2.1. Materials

The pharmaceutical oxytetracycline dihydrate (OTC) was obtained from Sigma-Aldrich, Chemie GmbH, Kappelwegl, Germany. ZnO (99.99%, product# 30N-0801, Lot# IAM10229ZNON), $BaTiO_3$ (99.95%, 100 nm, Catalog# 5622ON-01, Lot# IAM6070NBTO1) were collected from Inframat® Advanced Materials™, USA. 3 mg $BaTiO_3$ with 97 mg ZnO, 18 mg $BaTiO_3$ with 82 mg ZnO, 33 mg $BaTiO_3$ with 67 mg ZnO and 48 mg $BaTiO_3$ with 52 mg ZnO, respectively, to produce 3 BZ, 18 BZ, 33 BZ and 48 BZ. An ISO Laboratory (Institute of National Analytical Research and Service, BCSIR, Dhaka, Bangladesh) provided the deionized water that was used in this experiment. A Nylon membrane pore size—0.22 μm, dia—25 mm; PTFE; chrodisc by CHM (CHMLAB Group, Barcelona, Spain) was used to filter the samples after a certain experimental time. Ethanol was used to clean all of the glassware used in this experiment. No additional process was demonstrated for further purification of the chemicals.

### 2.2. Characterization of Nanoparticles

At 2˚ $min^{-1}$ of scan rate from 25˚ to 65˚, the powder XRD patterns of the catalysts were characterized using a Bruker D8 Advance XRD (Bruker AXS, Germany) with CuK$\alpha$ radiation, $\lambda$ = 1.5418 Å.

### 2.3. Photocatalytic Experiment

Under UV irradiation, OTC antibiotics were degraded using a simple photocatalytic reactor. Three ultraviolet lights, each of 8W and 254 nm wavelength were used as UV irradiation source. A spacing of 4.5 cm between the radiation source and solution was maintained to utilize the maximum amount of UV light which was explained by the experimental setup represented in our previous work [26].

A stock solution of OTC which was of 30 mg/L concentration, was prepared using deionized water. In this research, the dosage of each catalyst was 0.5 g/L. The photocatalysts were added to a 100 mL OTC solution at the room temperature. Before exposure to the UV radiation, an adsorption control equilibrium of each catalyst was performed. In the dark, the OTC solution mixture was stirred for 20 minitues using an orbital shaker at 250 rpm. After the adsorption and UV experiment, 5 mL of the sample was collected using a syringe after a specific experimental time. The solutions were then kept in the dark for 24 h to settle down the nanoparticles. After removing the nanoparticles via filtration using a Nylon Membrane Filter, the solutions were analyzed. The UV-vis absorption spectra of OTC were measured using a UV1800 spectrophotometer (Shanghai UPG International Trading Co., LTD, China). By investigating the removal of OTC anti-

biotics, the impact of different photocatalysts was investigated.

### 2.4. Data Analysis

For measuring the removal efficiency of OTC antibiotics, the following equation was used—

$$\%\text{Removal efficiency} = \frac{C_0 - C_t}{C_0} \times 100\% \quad (1)$$

where $C_0$ is the initial concentration of OTC and $C_t$ is the concentration of OTC after a distinct time interval.

The photocatalytic degradation kinetics was determined using first-order kinetic model.

First-order kinetic equation:

$$\ln\left(\frac{C_0}{C_t}\right) = K_{obs}t \quad (2)$$

where $K_{obs}$ is the first-order kinetic rate constant.

## 3. Result and Discussion

### 3.1. Characterization of Photocatalysts

Figure 1 shows the XRD patterns of ZnO, $BaTiO_3$ and 48 BZ. The diffraction peaks occurred at 31.66˚, 34.30˚, 36.21˚, 47.49˚, 56.59˚ and 62.89˚ for ZnO. These results matched with JCPS No (36-1451). For $BaTiO_3$, the diffraction peaks were at 31.51˚, 38.99˚, 45.29˚ and 56.29˚. These results matched with JCPDS No (05-0626). For 48 BZ, a group of diffraction peaks were at 31.66˚, 34.44˚ and 36.35˚ indicating the presence of ZnO and another group of diffraction peaks were at 38.84˚, 45.29˚ and 56.14˚ indicating the presence of $BaTiO_3$.

### 3.2. Adsorption-Desorption Activities in the Dark

The adsorption-desorption activities of all catalysts were evaluated after stirring in the dark for 20 min. The concentrations of OTC were 12.55, 14.21, 7.95, 13.82 and 10.46 ppm after 20 min using ZnO, 3 BZ, 18 BZ, 33 BZ and 48 BZ respectively. 58.15%, 52.62%, 73.47%, 53.9% and 65.10% removal of OTC were done via adsorption process using ZnO, 3 BZ, 18 BZ, 33 BZ and 48 BZ respectively. The percentages are higher due to the destruction of the outer layer. However, the highest efficiency was found using 18 BZ in the dark.

### 3.3. Photocatalytic Activity of OTC

Figures 2(a)-(e) show the evolutions of UV-vis absorption spectra of photodegradation of OTC within irradiation time. The topmost curve indicates the initial concentration of OTC (30 mg/L) which has a maximum absorbance at 347.5 nm. As time elapsed, the absorbance peaks were decreased that is, the concentrations of antibiotics were decreased. The peaks were shifted to the right side with the flow of time due to the red shift.

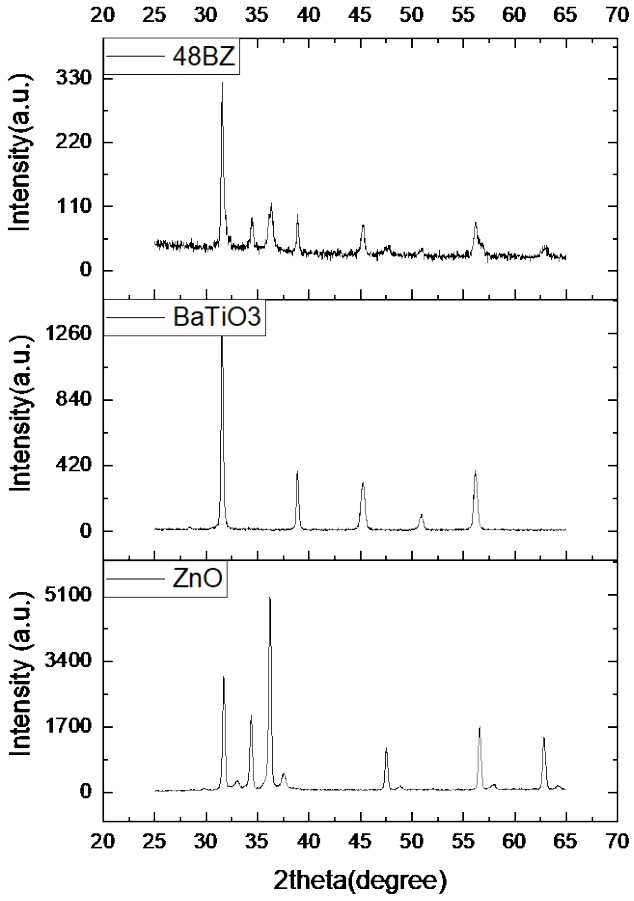


**Figure 1.** XRD patterns of ZnO, $BaTiO_3$ and 48 BZ.

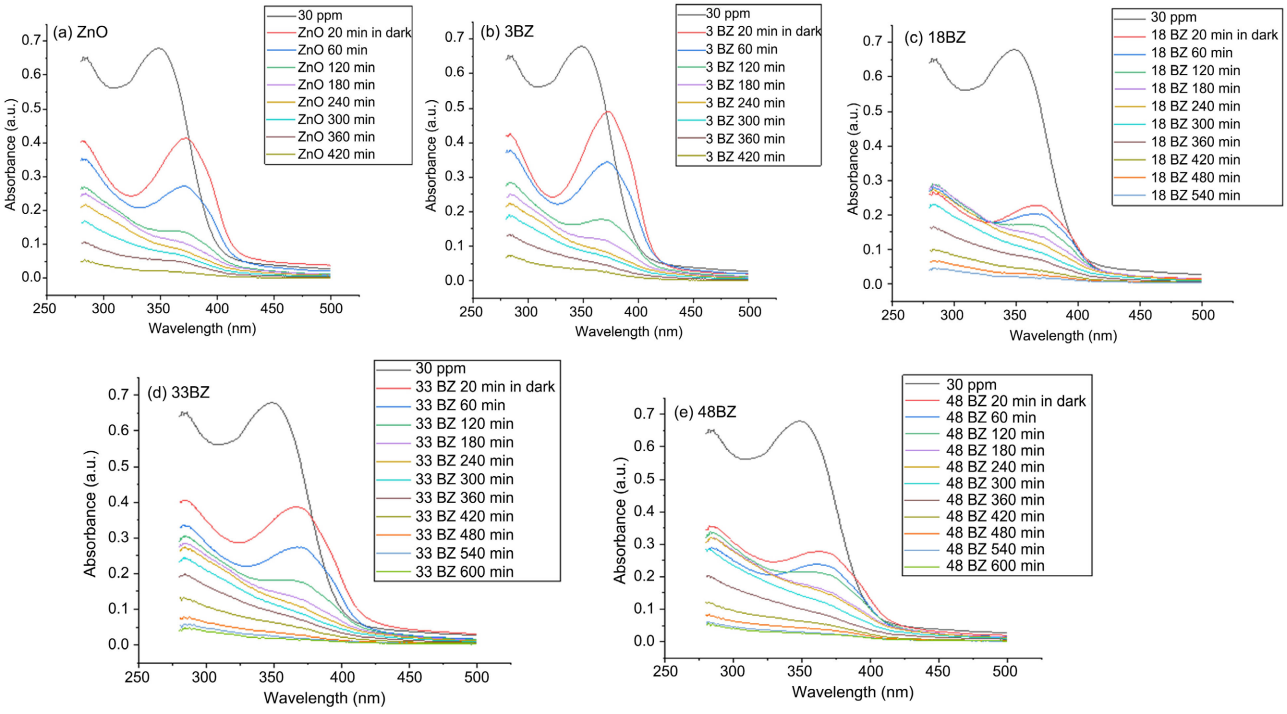


**Figure 2.** Degradation of OTC using (a) ZnO, (b) 3 BZ, (c) 18 BZ, (d) 33 BZ and (e) 48 BZ under dark condition and UV light.

### 3.4. Calibration Curve and Determination of Unknown Concentrations of OTC

5, 10, 15, 20, 25 and 30 ppm standard solutions were prepared for having calibration curve. The solutions were investigated by using a UV-vis spectrophotometer. The wavelengths were recorded at which maximum absorbances were gained. All of the absorbances of OTC solutions were taken at the wavelength of 347.5 nm. Therefore, in Figure 3, a calibration curve of OTC was drawn by graphing the values of absorbance at 347.5 nm against the standard concentrations of OTC solution. The curve was further used for the determination of unknown concentrations of OTC solutions.

### 3.5. Effect of Different Catalysts

70.21% of OTC was degraded after 60 min using ZnO catalyst which can be seen in Figure 4. This reached 99.57% after 420 min whereas 97.87% of OTC was degraded by using 3 BZ. Again, more than 420 min of experiment time was required to get more than 95% efficiency using 18 BZ, 33 BZ and 48 BZ. The use of 18 BZ showed an efficient photocatalytic degradation after 540 min which had 99.72% removal efficiency. Figure 4 shows that when the irradiation time rose, the removal efficiency improved as well. After 600 min of the exposure time, the

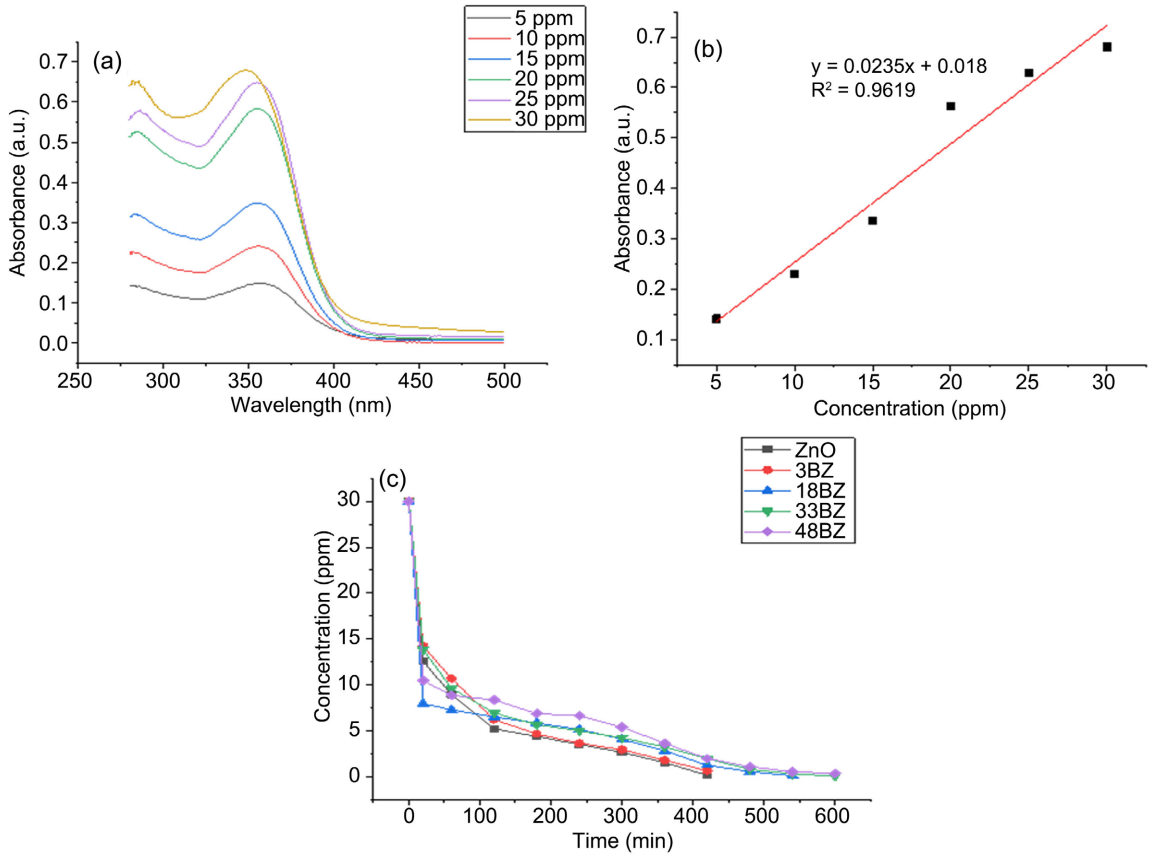


Figure 3. (a) UV-visible spectra of standard OTC solutions at different concentrations, (b) calibration curve of OTC at 347.5 nm and (c) concentration vs. time curve of the experimented solution.

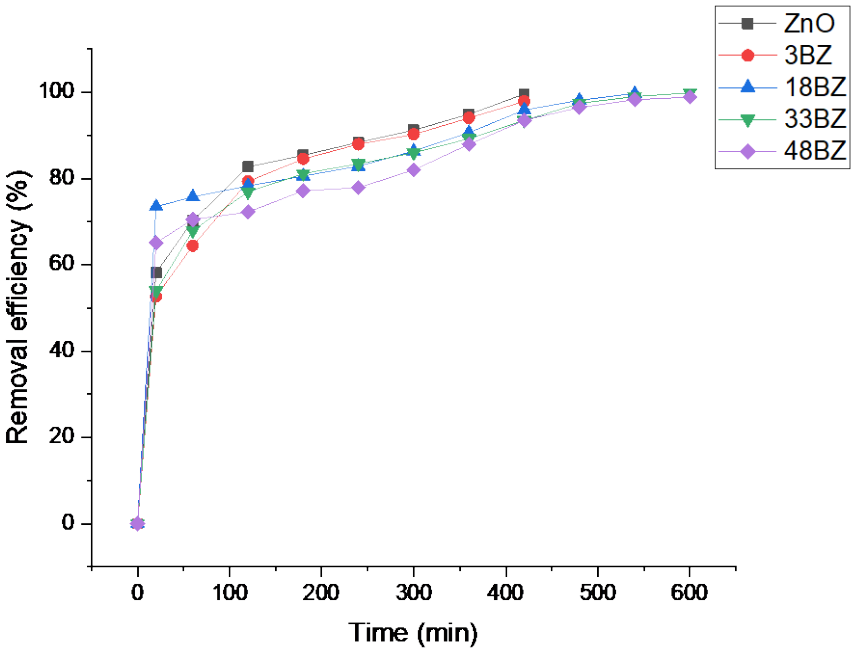


**Figure 4.** Effect of different catalysts with time on the degradation efficiency.

removal efficiencies were 99.86% and 99.87% for using 33 BZ and 48 BZ respectively which were almost the same. Enhancing the amount of $BaTiO_3$ causes a decrease in degradation efficiency. Hence, more than 420 min is required to get almost 99% efficiency. Qinghua Chen *et al.* investigated that almost 96% of OTC can be from aqueous solution after 1 hr of exposure to the simulated solar solution using Au-CuS-$TiO_2$ nanobelts [27].

### 3.6. Reaction Kinetics Study

Kinetic study plays a pivotal role in understanding the degradation rate. **Figure 5** depicts the experimental data of the first-order kinetic curves of OTC. The applicability and validation of the first-order model were determined following the values of the determination of coefficient ($R^2$). The values of $R^2$ observed using the zero and second-order kinetic models were much lower and it can be determined that the degradation of antibiotics does not follow those models. The kinetic reaction rate constant was measured from the slope of the plot of $-\ln(C_t/C_0)$ versus experiment time ($t$). It is evident that the highest $K_{obs}$ value was found for the ZnO photocatalyst which had the value of 0.00933 $min^{-1}$. The high value of the reaction rate constant indicates high degradation of OTC. Furthermore, the lowest value of reaction rate constant was 0.00532 $min^{-1}$ which was observed for 48 BZ photocatalyst which means that $BaTiO_3$ can be used for tailoring the activity of ZnO photocatalyst. Previously, Shuai Zhang *et al.* showed that for the photodegradation of OTC antibiotics using visible light, the first-order reaction rate constant was 0.00765 $min^{-1}$ using nanohybrids of CoFe alloy nanoparticles along with mesoporous carbon codoped with nitrogen-/sulfur where the flow of $N_2$ was at 1000˚C for 3 hr [28].

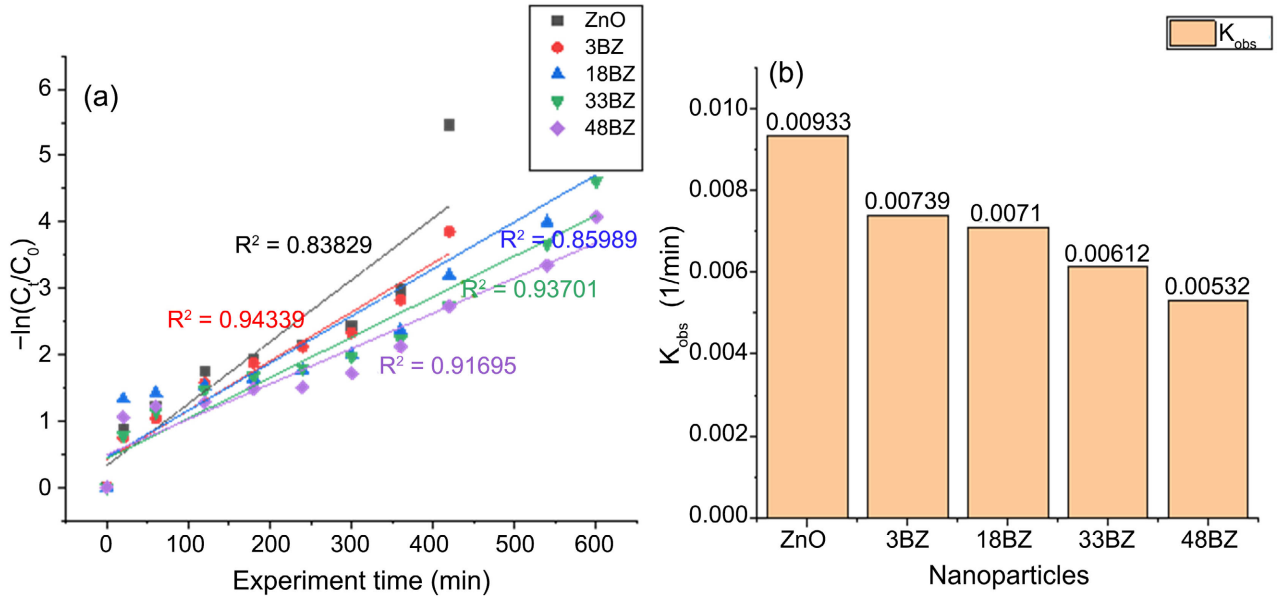


**Figure 5.** (a) First-order kinetic curve of OTC and (b) Reaction rate constant value of different nanoparticles.

## 4. Conclusion

OTC was successfully removed from the aqueous solution with the help of ZnO, 3 BZ, 18 BZ, 33 BZ and 48 BZ using photodegradation technique under UV light. Using ZnO, 99% degradation of OTC was found within 420 min, whereas the percentages of degradation were lower in the case of 3 BZ, 18 BZ, 33 BZ and 48 BZ at the same time. The addition of $BaTiO_3$ to ZnO slows the photocatalytic degradation and thus, extends the degradation time. As the percentage of $BaTiO_3$ increases, the rate of reaction decreases. Moreover, no adjustment of pH was done during this process. The present study demonstrates that ZnO photocatalyst has fast and high performance.

## Credit Authorship Contribution Statement

**Preyanty Sen:** Conceptualization, Data collection, Methodology, Validation, Writing—original draft.

**Surya Subrin Soshi:** Experimentation, Data collection, Methodology, Validation, etc.

**Md. Abdul Gafur:** Project administration, Resources, Supervision, Conceptualization, Writing—review an editing.

## Conflicts of Interest

The authors declare that they have no known competing interests.

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