

## RESEARCH ARTICLE

# Optimization of the Photodegradation of Metoclopramide Drug using Super Nanoparticles in Aqueous Solutions

Wesam AL H Alhaidry<sup>1</sup>, Farah Aloraibi<sup>2</sup>, Jasim M Abbas<sup>3</sup>, Hawraa AZ Alhussein<sup>4</sup>, Ruaa Sattar<sup>5\*</sup>

<sup>1</sup>College of Pharmacy, National University of Science and Technology, Dhi Qar, Iraq

<sup>2</sup>Al-Manara College For Medical Sciences, (Maysan), Iraq

<sup>3</sup>Department of Medical Laboratories Technology, AL-Nisour University College, Baghdad, Iraq

<sup>4</sup>Mazaya University College, Iraq

<sup>5</sup>Al-Hadi University College, Baghdad, Iraq.

Received: 18<sup>th</sup> August, 2023; Revised: 09<sup>th</sup> September, 2023; Accepted: 22<sup>th</sup> November, 2023; Available Online: 25<sup>th</sup> December, 2023

## ABSTRACT

In the present research carry out to remove pharmaceutical pollutants as a model metoclopramide (MCP) drug from an aqueous medium utilizing a photocatalytic degradation method with super nanoparticles called TiO<sub>2</sub> nanoparticles (NP). synthesis of TiO<sub>2</sub> nanoparticles by utilizing the hydrothermal processes. The physical characterizations of materials by X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), thermogravimetric analysis (TGA), and transmission electron microscope (TEM). The results showed that the efficiency of the TiO<sub>2</sub> nanoparticle increased by 88.88%. It also showed that the photocatalytic degradation rate increased with decreasing concentration of MCP drug increasing the intensity of the light led to an increase in the rate of photocatalytic degradation. Study effect of several factors, concentration on the drug, mass of nanoparticle, light intensity that the best conditions for the photocatalytic degradation of 0.3 g of the nanocomposite, MCP drug concentration of 50 m/L, pH solution 8, and light intensity 1.7 mW/Cm<sup>2</sup>. Thus, the photocatalytic degradation increased with decreasing the concentration of MCP drug. The obtained TiO<sub>2</sub> nanoparticle exhibits best photocatalytic degradation than the commercial photo-catalysts TiO<sub>2</sub> NPs. Finally, it is essential to evaluate the recycled photocatalytic degradation efficiency, which is one of the utmost significant factors towards photodegradation.

**Keywords:** Photodegradation, Pharmaceutical pollutant, Drug, Metoclopramide, Nanocomposite.

International Journal of Drug Delivery Technology (2023); DOI: 10.25258/ijddt.13.4.38

**How to cite this article:** Alhaidry WALH, Aloraibi F, Abbas JM, Alhussein HAZ, Sattar R. Optimization of the Photodegradation of Metoclopramide Drug using Super Nanoparticles in Aqueous Solutions. International Journal of Drug Delivery Technology. 2023;13(4):1365-1369.

**Source of support:** Nil.

**Conflict of interest:** None

## INTRODUCTION

One of the main records of using nanoparticles in literature dates back to the middle of the 19<sup>th</sup> century when Micheal Faraday was studying gold colloids in the nanometer range. The nanomaterial with less than 100 nm ones at least in one dimension, which means they have less size than that of micro-scale physiochemical properties than the bulk material which inherently depends on their size and shape.<sup>1</sup> The nanomaterials produce unique characters with new characteristics and capabilities by modifying the shape and size at the nano-scale level. Nanomaterials may be of different shapes like nanorods, nanoparticles, and nanosheets which can be characterized based on their dimensional. Nano partials show the potential of many applications such as water treatment, agriculture, medical applications, and building material and there are several methods to produce NPs including condensation, corrosion, culture ionic and hydrothermal synthesis.<sup>2-4</sup>

Hydrothermal synthesis is one of the most commonly used methods for the preparation of nanomaterial by using single or heterogeneous phase reactions in aqueous media at high temperatures (T>25°C) and pressures (P>100 k Par).<sup>5,6</sup> In the middle of the 19<sup>th</sup> century, hydrothermal research was first conducted. It depends on the solubility of aqueous solution under hot water and higher temperature levels in steel pressure vessels called autoclaves.<sup>7,8</sup> One of the main advantages of hydrothermal technique is economies. Another significant advantage of synthesis hydrothermal is that the purity of hydrothermally synthesized powders meaningfully exceeds the purity of the starting materials. Perhaps because hydrothermal crystallization is a self-purifying process throughout the growing crystals/crystallites tend to reject impurities present in the growth environment. The impurities are removed from the system to gather with the crystallizing solution.<sup>9-12</sup> The hydrothermal method is thought to be eco-friendly since

\*Author for Correspondence: ruaa90@huc.edu.iq

the reactions are carried out in a closed system and the contents can be recovered and reused after cooling down at a temperature room. Furthermore, proper and careful control of the hydrothermal method condition allows for control over the physical properties of titania such as crystallite size and form, surface area, contamination morphology and phase uniform distribution and high dispersion and stronger interfacial adsorption properties.<sup>13-16</sup> Titanium dioxide is a robust semiconductor material with a band gap of 3.2 eV which has been extensively explored for heterogeneous photocatalysis due to its non-toxic nature and easy availability. Nevertheless, wide band gap of TiO<sub>2</sub> restricts its operational range to only UV region that is less than 5% of whole solar spectrum, relatively fast recombination of photo-exactions (e<sup>-</sup>, h<sup>+</sup>) is another drawback associated with TiO<sub>2</sub> that lowers its photo catalytic performance. One constraint associated with doping approach is that it can sometimes also reduce the redox potential of charge carrier species that results in decline of photo catalytic efficiency.<sup>17-20</sup>

Metoclopramide (MCP) drug utilized for esophageal, stomach problems. drug is usually used to treatment to prevent vomiting and nausea, used to emptying of the stomach in people with delayed stomach emptying, and to help with gastroesophageal reflux disease and utilized to treatment migraine headaches.<sup>21</sup> Chemical formula C<sub>14</sub>H<sub>22</sub>ClN<sub>3</sub>O<sub>2</sub>, molar mass 299.80 g·mol<sup>-1</sup> and chemical structure as show in Figure 1.

## METHOD AND MATERIALS

All of the chemical reagents used (Sigma-Aldrich). Aqueous ammonia NH<sub>3</sub>, ethanol CH<sub>3</sub>CH<sub>2</sub>OH purity% (99.3%), commercial titanium dioxide TiO<sub>2</sub> purity% (98.5%), methanol CH<sub>2</sub>OH purity% (99.2%), hydrogen peroxide H<sub>2</sub>O<sub>2</sub> purity% (23%), titanium dioxide nanoparticles were previously prepared by the hydrothermal method.<sup>14</sup>

### Photocatalytic Activity

The photocatalytic method was determine utilizing (UVA-meter) with 1.21 mw/Cm<sup>2</sup>. Antibiotic pollutant (Metoclopramide (MCP) drug as a model removal from aqueous solution by photodegradation chosen as higher effective photocatalyst TiO<sub>2</sub> NPs. after that, 200 mL of solution metoclopramide (MCP) drug (50 mg/L) was combined with 0.3 g of TiO<sub>2</sub> NPs photocatalyst. Prior to light exposure, the solution was agitated magnetically in dark for 10 minutes to initiate the adsorption-desorption equilibrium among solution of drug and TiO<sub>2</sub> NPs photocatalyst. Next, aliquots 5 mL was collected for analysis after 10 minutes. During the photoreaction, a UV-vis spectrophotometer was used to drug concentration degradation.

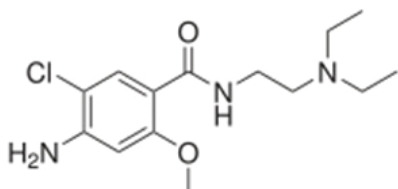


Figure 1: Chemical structure metoclopramide (MCP) drug

To ensure that the photodegradation was triggered only *via* the photocatalyst, the experiment was conducted in two ways: without a catalyst (just drug) and with a photocatalyst (pure TiO<sub>2</sub> NPs)

## RESULT AND DISCUSSION

### Characterization

The morphology of the surface TiO<sub>2</sub> nanoparticles pure have been studied in terms of the size, shape of particles and clusters among them, in addition to the distribution of these particles utilizing the scanning electron microscope (SEM) technique. Figure 2 a shows SEM image of TiO<sub>2</sub> NPs, the nanoparticles have anatase shape with a mean size of 500 nm with highly of agglomerate.<sup>22,23</sup>

The transmission electron microscopy (TEM) gives a more accurate picture of the structure of prepared samples; the crystalline, distribution and the particles size of nanoparticles. The micrographs of TiO<sub>2</sub> nanoparticles were clearly observed in the TEM images as the most intense dark spots on the surface.<sup>20</sup> The nanoparticles deposited on the surface quite small and well deposited as show in Figure 2b.

In Figure 3 an X-ray diffraction (XRD) pattern of TiO<sub>2</sub> there are no other summits indicating the presence of the impurity due to the effect of calcination (500°C), the diffraction peaks were observed at 25.3°, 37.9°, 48.2°, 54.5°, 55.3°, 62.02° and 68.7, which correspond to the (101), (108), (004), (112), (211), (200), (106) and (212) lattice planes of anatase TiO<sub>2</sub> the crystal shape of the diffraction peaks appear clear and sharp.<sup>24,25</sup> Thermogravimetric analysis (TGA) technique in which the mass of a sample is monitored against temperature or time, to study the thermal stability of nanoparticles and indicate

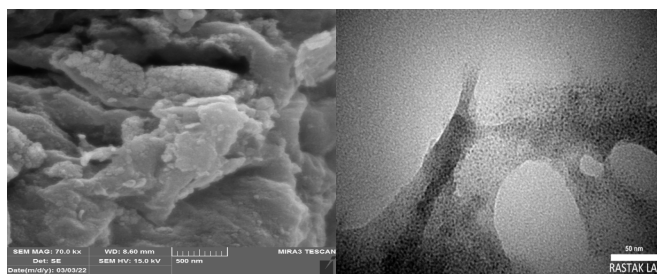


Figure 2: Effect of a) SEM of TiO<sub>2</sub> NPs, and b) TEM of TiO<sub>2</sub> NPs

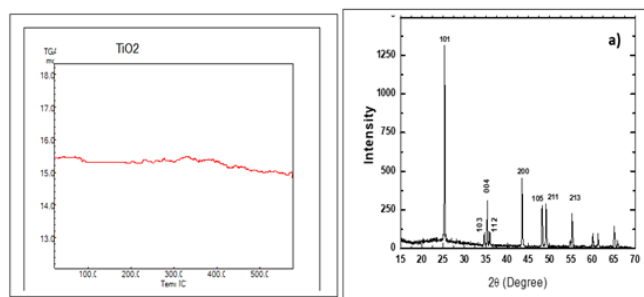


Figure 3: Effect of a) X-ray diffraction of TiO<sub>2</sub> NPs, b) TGA of TiO<sub>2</sub> NPs

the purity of nanoparticles, the TGA curve of  $\text{TiO}_2$  NPs in the temperature range 20 to  $600^\circ\text{C}$ <sup>26</sup> as show in Figure 3b .

### Photocatalytic Activity of MCP drug

#### Effect weight of $\text{TiO}_2$ NPs

To study the effect of mass  $\text{TiO}_2$  NPs on the photocatalytic process of MCP drug, under experimental conditions of MCP drug concentration of 50 mg/L, pH solution 8. A different weight of  $\text{TiO}_2$  NPs (0.1–0.4 g) was used at  $25^\circ\text{C}$  as shown in Figure 4. The photocatalytic efficiency increases with increasing weight of  $\text{TiO}_2$  NPs up to 0.3 g. This is due to the increase in the number of active sites responsible for generating highly reactive radicals. Therefore, photocatalytic efficiency of MCP drug decomposition increases with increasing weight of  $\text{TiO}_2$  NPs, and this may be due to increased penetration. Photon in MCP drug solution, and thus the rate of photo degradation increases.<sup>19,27</sup>

#### Effect of solution pH

The pH solution show very important part in photo catalytic activity method of  $\text{TiO}_2$  NPs. The influence of solution pH on the photo degradation of MCP drug was studied the range pH 3 to 10. The solution pH was adjusted HCl or NaOH (0.1N) before experimentation. The pH meter was calibrated with buffers 3.0, 7.0 and 10.2. The concentration of MCP drug was maintained at 50 mg/L. The influence of solution pH on photocatalytic degradation of MCP drug utilizing  $\text{TiO}_2$  NPs It is observed that the elimination of drug via  $\text{TiO}_2$  NPs was maximum at pH 10, as show in Figure 5

#### Effect concentration of MPC drug

Photocatalytic activity of MPC drug was conducted utilizing several initial concentration of MCP drug from (2–150 mg/L). All experiments were performed at mass of  $\text{TiO}_2$  NPs of about (0.3 g/200 mL). the suspension solution was irradiated with intensity of light  $2.1 \text{ mW/cm}^2$ , 0.3 g/200 mL at  $25^\circ\text{C}$ . Among the results shown in Figure 6, that of photodegradation efficiency depends essentially on the concentration MPC drug. As the concentration of MPC drug increases, the photo catalysis efficiency decreases. This is because increasing the concentration prevents light photons from entering in solution. Therefore, increasing concentration make to block light from penetrating drug into the solution. This prevents the process of photocatalysis. Thus, as the concentration increases, the process of scattering light photons on the surface of drug solution increases.<sup>28</sup>

#### Effect of light intensity

The influence of light intensity ( $1.12$ – $2.33 \text{ mW/cm}^2$ ) has an important factor on the photocatalytic process by determining the distance between the surface and the light source using surface such as a semiconductor ( $\text{TiO}_2$  NPs). The photo degradation of a drug was studied under the influence of light intensity in the presence of a catalyst of 0.3 g, an oxygen flow rate of  $5 \text{ mL/min}^{-1}$  and a pH of 8. Therefore, when the light intensity increased, the photolysis efficiency increased because the high light intensity caused the  $\text{TiO}_2$  molecules

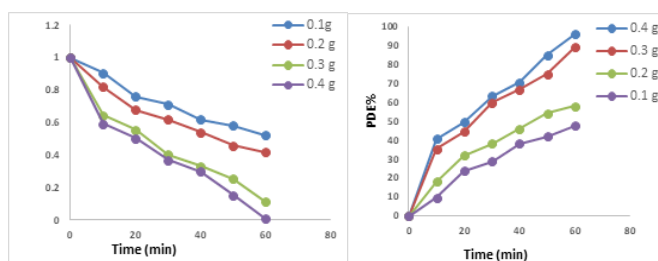


Figure 4: Effect of mass  $\text{TiO}_2$  NPs on photocatalytic process MPC drug.

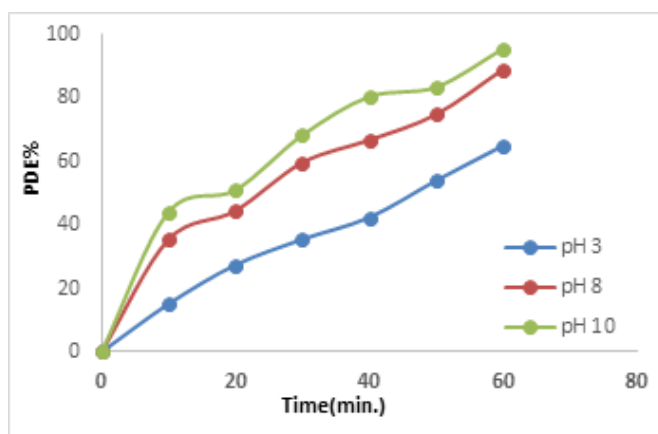


Figure 5: Photocatalytic degradation of MCP drug in the presence of several solution pH on to  $\text{TiO}_2$  NPs.

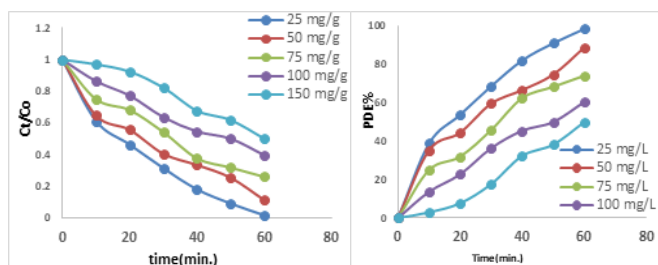


Figure 6: Photocatalytic degradation at several concentrations of MPC drug

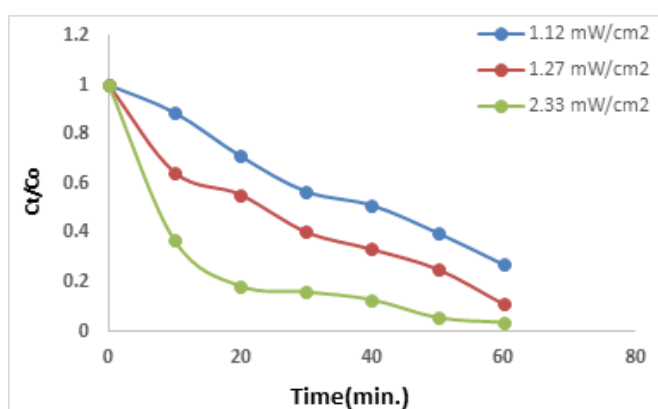


Figure 7: Effect of Effect of light intensity on photocatalytic degradation of MPC drug

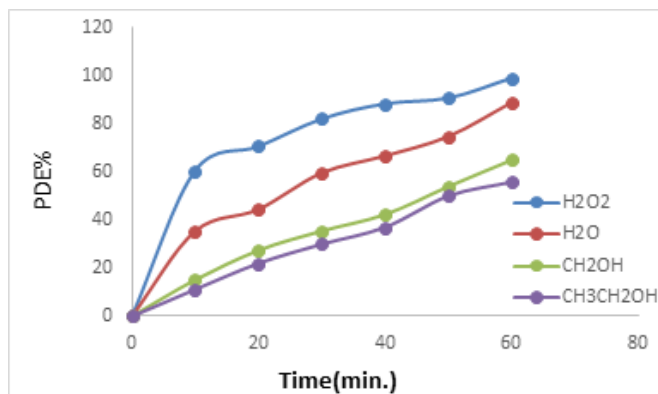


Figure 8: Effect of ROS on to photocatalytic process

Table 1: Comparison between TiO<sub>2</sub> NPs, commercial TiO<sub>2</sub> and without a catalyst (just drug)

Type of catalyst	PDE%
TiO <sub>2</sub> NPs	88.89
Commercial TiO <sub>2</sub>	82.87
without a catalyst (just drug)	12.22

to be stimulated to generate hole-electron pairs.<sup>12</sup> It is also possible that the photocatalytic efficiency decreases as the light intensity decreases because the low light intensity reactions involving electron hole formation are dominant and electron hole recombination is negligible or very minimal, as show in Figure 7.

### Roles of reactive oxygen species

The MPC drug photocatalytic degradation with 0.3 gm TiO<sub>2</sub> NPs; 50 mg/L at 1-hour, the effects of MCP drug (2.3 mW/Cm<sup>2</sup>; at 25°C) at several solvents like ethanol CH<sub>3</sub>CH<sub>2</sub>OH, methanol CH<sub>2</sub>OH, hydrogen peroxide H<sub>2</sub>O<sub>2</sub>. The experimental result could be analyzed assuming first order kinetics because the reaction kinetics were comparable to those seen without the oxidant; the data appear in Figure 8. From the results shown, hydrogen peroxide is the best solvent as it works to photocatalytic process the pollutant faster than other solvents because it works to increase the free radicals inside the solution, and thus increases the photocatalytic process.<sup>29-32</sup>

### Comparison between TiO<sub>2</sub> NPs, Commercial TiO<sub>2</sub> and without a catalyst (just drug)

A comparison between the prepared surface TiO<sub>2</sub> NPs and the commercial TiO<sub>2</sub>, according to the results, there is a clear difference in the results of photo catalysis between the prepared surface 88.88% and the commercial 82.8.8% to remove a MPC drug. To ensure that the photodegradation was triggered only via the photocatalyst, the experiment was conducted in two ways: without a catalyst (just drug) and with a photocatalyst (pure TiO<sub>2</sub> NPs). It was clear from the results that with a photo catalyst (TiO<sub>2</sub> NPs) there was a very clear increase in the photocatalytic process, so that it had a clear effect compared to the absence of a catalyst, as show in Table 1.

## CONCLUSION

Synthesis of approach by use TiO<sub>2</sub> NPs by a simple hydrothermal method and used as a photo-catalyst degradation of drug. The visible-light nanoparticle photo-catalyst was first used to degradation drug by photoreaction after 1-hour. The nanoparticle appears the best photo catalytic as PDE% = 88.17 under best conditions (concentration of drug 50 mg/L nanocomposite dose = 0.3 g and irradiation time = 1 hour). The reuse study appear that nanocomposite photo catalyst exhibited a promising photo-stability, Results appear that the degradation of drug solution via nanocomposite H<sub>2</sub>O<sub>2</sub> system increased PDE% more than 50% after irradiation 1-hour. A comparison between the prepared surface TiO<sub>2</sub> NPs and the commercial TiO<sub>2</sub>, according to the results, there is a clear difference in the results of photocatalysis between the prepared surface 88.88% and the commercial 82.8.8% to remove a MPC drug.

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