



# Sonocatalytic Degradation of Humic Substances From Aquatic Environments Using MgO Nanoparticles

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## Abstract

Humic substances are considered as one of the major natural organic contaminants in water resources. Presence of such substances in the drinking water supply resources results in poor water quality and jeopardizes the consumer's health. In the present study, sonocatalytic decomposition of the humic substances by using MgO nanoparticles was investigated under the radiation of the ultrasonic waves. Accordingly, the X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques were used to determine characteristics of the nanoparticles. Furthermore, the effects of various parameters, including the amount of nanoparticles, initial pH, initial concentration of humic substances, contact time, as well as the power and frequency of ultrasonic waves on the efficiency of the sonocatalytic decomposition of humic substances were investigated. Results of the present study showed that the efficiency of sonocatalytic decomposition of humic substances was reduced by increasing the initial pH and initial concentration of the humic substances; while, the increased amount of the nanocatalyst resulted in the increased efficiency of decomposition. Furthermore, the obtained results indicated that the MgO sonocatalytic process could decompose the humic substances with the efficiency of 78.5% under optimal conditions (pH:7, initial concentration of humic substances: 20 mg/L, concentration of nanoparticles:1 g/L, power: 100%, frequency: 37 kHz and contact time:120 min). Based on the obtained results, the sonocatalytic method using MgO can remove humic substances and other similar organic matters from the aquatic environments with a high efficiency.

**Keywords:** MgO nanoparticles, Humic substances, Sonocatalyst, Ultrasonic

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## 1. Introduction

Humic acid substances (HS) are very complex organic compounds derived from the decomposition of animal and plant tissues because of microbial activities (1,2). These substances constitute the main part of the natural organic matters (NOMs) in water resources (3). Many of the qualitative problems of drinking water, such as color, odor, and taste are attributed to the presence of humic substances (4). Humic substances can result in the increased biological re-growth within the water distribution networks, membrane fouling, and increased amount of chlorine required for water disinfection (5,6). Additionally, the biggest problem with the humic substances in water is their reaction

with disinfectants and, consequently, the production of carcinogenic byproducts such as trihalomethanes (THMs) and haloacetic acids (HAAs) (6,7). The United States Environmental Protection Agency (USEPA) has determined the maximum concentration level (MCL) for THM and HAA as 80 and 60 µg/L, respectively (7).

So far, various methods, including adsorption (6), ion exchange (8), nanophotocatalysis (9), membrane (10,11) and ultrasonic technologies (11), electrocoagulation (12), coagulation and enhanced coagulation (13) have been employed for the removal of humic substances from water, among which the advanced oxidation processes (AOPs) have been introduced as a suitable option for the removal or destruction of the humic substances compared

to other common methods (coagulation, ion exchange, membrane filtration, and adsorption) (6). Synthesis of hydroxyl radicals with high oxidative power in AOP has caused these treatment processes to be known as one of the most practical physicochemical treatment procedures (14). Due to the non-selective function and high oxidative potential at normal temperature and pressure, hydroxyl radical is capable of oxidizing almost all the reduced materials unlimitedly (15). Among all the AOPs, today, the focus is on utilizing the sonocatalytic oxidation process as a result of its high efficiency in the removal of resistant industrial contaminants, high potential for mineralizing the target organic contaminants, as well as easiness and safety of exploitation (non-exposure to UV, similar to what happens in photocatalytic processes) (16). Decomposition of the contaminants by ultrasonic waves occurs because of the formation of bubbles in the aquatic environment and their destruction due to the ultrasonic waves, which results in a very high pressure and temperature. Such high temperature produces hydroxyl radicals because of water dissociation (17,18). The use of mere ultrasonic waves for a long time, by spending a considerable amount of energy would result in the decomposition of the contaminants; thus, nowadays, the use of semiconductors as catalysts for the sonocatalytic decomposition of contaminants has been highly considered (19,20). Due to their small size and unique surface characteristics, the use of nano-size catalysts for the decomposition of various bioenvironmental contaminants in different sonocatalytic processes has been taken into account (21,22). In various studies on sonocatalytic procedures, various semiconductors such as ZnO (zinc oxide) and TiO<sub>2</sub> (titanium dioxide) have been used as catalysts (16,19,23). In comparison with other existing catalysts, MgO as an alkaline earth metal oxide has been considered as a promising catalyst due to its remarkable features such as low cost, simple synthesis, and non-toxicity to the environment (especially aquatic environment) (24,25). Low cost and simple synthesis justify the cost-effectiveness of the use of MgO catalyst in a full-scale exploitation. For instance, this catalyst can be easily synthesized through magnesium hydroxide dehydration (25). Moreover, the MgO nanocatalyst with the high specific surface area would have a high catalytic reactivity potential besides its unique physicochemical properties and high thermodynamic stability (24,26).

Several studies have investigated the use of sonocatalytic oxidation process for the decomposition of humic substances in aquatic environment using various catalysts; nevertheless, to the best of our knowledge, no research has been conducted on the utilization of the sonocatalytic process for the decomposition of humic substances using MgO nanoparticles. On the basis of this, the present study aimed to use nanoscale MgO semiconductor under the ultrasonic waves for the sonocatalytic treatment of the humic substances in the aquatic environment.

## 2. Materials and Methods

### 2.1. Synthesis of MgO Nanoparticles

In order to obtain MgO nanoparticles as catalysts through a sonochemical process, firstly, 5.128 g of magnesium nitrate hexahydrate (with formula of Mg(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O) was solved as a precursor in 100 mL of deionized water; then, 25 mL of NaOH 1N was added to the solution within 10 minutes under mixing conditions to form the magnesium hydroxide sediments as a result of environment alkalization (pH >10). The resulting mixture was mixed for 5 minutes to form the gel-like suspension of magnesium hydroxide. The obtained suspension was sonicated in an ultrasonic bath for 120 minutes at 50°C to form the MgO nanoparticles with more uniform size distribution and lower agglomeration in the environment. Afterwards, the suspension was allowed to be precipitated for 1 hour, and then the remaining water on the suspension was removed. The sediments obtained by filtration were washed by deionized water and alcohol for several times; next, in order to form MgO, the sample was dried at 80°C in the oven for 48 hours (27).

### 2.2. Sonocatalysis Experiments

The sonocatalysis was performed using a batch reactor with the volume of 100 mL. The MgO sonocatalyst-containing batch reactor was placed in the ultrasonic bath under the radiation of ultrasonic waves (Elma, P30H, Germany). In order to control the effect of adsorption on the removal efficiency of humic substances, the adsorption efficiency of humic substances onto Mg nanoparticles was solely measured prior to performing the tests. In addition, the removal efficiency of humic substances was examined by using only ultrasonic waves. Having determined the role of each process involved in the removal of humic substances, the effect of different effective operational parameters on the efficiency of sonocatalytic process was evaluated.

### 2.3. Analyses

Total organic carbon (TOC) was determined as indicators of humic substances. The value of TOC was determined by the method of 5310 using a TOC analyzer (TOC/TN analyzers, Skalar, Netherlands). Besides, pH was measured using a pH-meter device. Moreover, in order to determine the morphology and surface characteristics of the catalyst, and scanning electron microscopy (SEM) (Model: MIRA3, Czech Republic) was used. Furthermore, X-ray diffraction (XRD) (Model: X'Pert PRO MPD, the Netherlands) was used to determine the structural features of the catalyst.

## 3. Results and Discussion

### 3.1. Characteristics of Nanophotocatalyst

Fig. 1 shows the SEM image of MgO. As shown in Fig. 1, structure of MgO consisted of dense elliptical crystals with the average size of 20-60 nm, which was normally

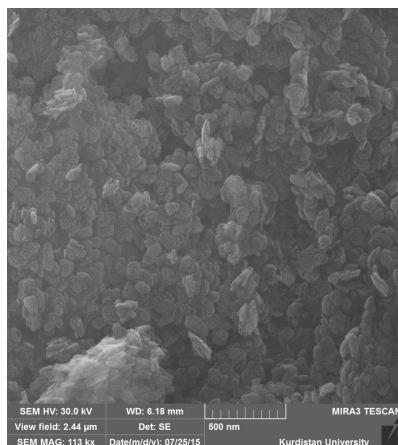


Fig. 1. SEM Image of MgO.

distributed. This size was in agreement with the particle size obtained from Scherrer formula in XRD analysis. This structure represented non-accumulation of the obtained nanoparticles, and also indicated appropriateness of the produced catalyst for implementing a sonocatalytic process with high efficiency and low rate of pore-electron recombination.

Fig. 2 shows the XRD pattern for the MgO nanoparticles. As seen in this figure, the XRD for MgO exhibited an intense peak at the  $2\theta = 24^\circ$  angle, which indicated the formation of the functional groups, such as carboxyl, hydroxyl, and epoxy. Moreover, the peaks with high intensities at 100, 002, 101, 110, 200, 112, 201, and 202 implied the high purity as well as the hexagonal Wurtzite structure of MgO. According to Formula (1) in Scherrer equation, the nanoparticles' size was obtained as approximately 45 nm.

$$D = (K \times \lambda) / (\beta \cos \theta) \quad (1)$$

where,  $D$  is the size of crystallite in nanometers,  $\beta$  is the peak width at half of the maximum intensity in radians,  $\theta$  is the Bragg's angle for the peak in degrees, and  $\lambda$  is the wavelength of X-ray in nanometers.

### 3.2. Effects of Operational Parameters

#### 3.2.1. Effect of pH

Effect of the initial pH on the efficiency of the sonocatalytic decomposition of humic substances by using MgO nanoparticles under the radiation of ultrasonic waves is represented in Fig. 3. This investigation was performed at various values of pH (3, 5, 7, 9 and 11), humic substance concentration of 20 mg/L, and 1 g/L of MgO nanoparticles. Based on the results, increasing the pH from 2 to 7 led to the increased removal efficiency of the humic substances; while, by increasing the pH from 7 to 11, the decomposition efficiency reduced.

The highest percentage of the photocatalytic decomposition of humic substances at pH of 7 was obtained equal to 78.55%. Effect of pH on the sonocatalytic decomposition efficiency depended on the type of

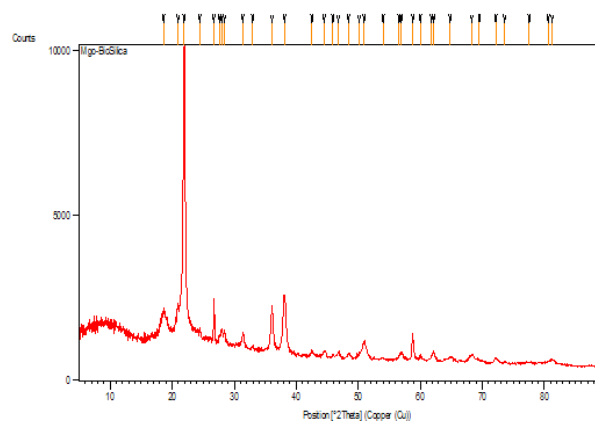


Fig. 2. XRD Pattern for MgO Nanoparticles.

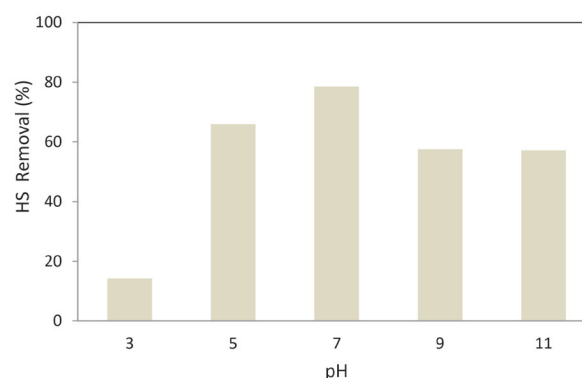


Fig. 3. Effect of Initial pH on Sonocatalytic Decomposition of Humic Substances (Initial concentration of humic substances: 20 mg/L, concentration of nanoparticles: 1 g/L, power: 100%, frequency: 37 kHz and contact time: 120 min).

contaminant and PZC (point of zero charge) of the catalyst (26). As indicated by previous studies, the  $pK_a$  value of the humic substances was equal to 6 (18) and at pHs above this value, the humic substances were negatively charged, while the nanoparticles had positive charge at lower pHs. Therefore, when the pH value ranged between the  $pK_a$  and  $pH_{ZPC}$  values, the positively charged nanoparticles adsorbed negatively charged humic substances, leading to the increased sonocatalytic decomposition efficiency of these substances (9). However, at higher pHs, the repulsion between the nanoparticles and humic substances caused by their negative charge would result in the reduced sonocatalytic removal efficiency of the humic substances (4).

#### 3.2.2 Effect of Nanocatalyst Dosage

Fig. 4 shows the effect of nanocatalyst dosage (0.5, 1, 2, and 3 g/L) on the decomposition of the humic substances under the ultrasonic wave, humic substance concentration of 20 mg/L, pH of 7 and contact time of 120 minutes. Results showed that the sonocatalytic decomposition efficiency of humic substances was increased by increasing the dosage of the MgO nanocatalyst. Increasing the

nanocatalyst content value from 0.3 to 3 g/L increased the decomposition efficiency, to such a degree that, the highest removal efficiency of the contaminant (83.45%) was obtained at nanocatalyst content value of 3 g/L. Furthermore, increasing the nanocatalyst content value led to the increase in the catalyst surface area as well as the number of the accessible active sites for sonocatalytic reactions (9).

### 3.2.3. Effect of Initial Concentration of Humic Substances

The initial concentration of contaminants is considered as one of the major and effective parameters in the sonocatalytic decomposition process. Fig. 5 represents the effect of the initial concentration of humic substances (10, 20, 40, 80, and 100 mg/L) on the sonocatalytic decomposition efficiency using MgO nanoparticles. The obtained results showed that increasing the initial concentration of the humic substances led to the reduced sonocatalytic decomposition efficiency. This was probably due to the fact that, regarding the constant value of the nanoparticles, increasing the concentration of the humic substances caused the increase in their adsorption by MgO and, as a result, the reduction in the surfactant sites for the adsorption of hydroxyl ions, which ultimately resulted in the reduced production of hydroxyl radicals and, consequently, the reduced decomposition efficiency. So far, similar results have been reported by some other researchers (23,25).

### 3.2.4. Effect of Power and Frequency of Waves

Fig. 6 represents the results of sonocatalytic decomposition efficiency of the humic substances at various powers (30%-100%) and frequencies of 37 and 80 kHz. These results showed that the sonocatalytic decomposition efficiency of humic substances was increased by increasing the power of the ultrasonic waves. At higher powers, increasing the cavitation led to the increased production of hydroxyl radicals and, as a

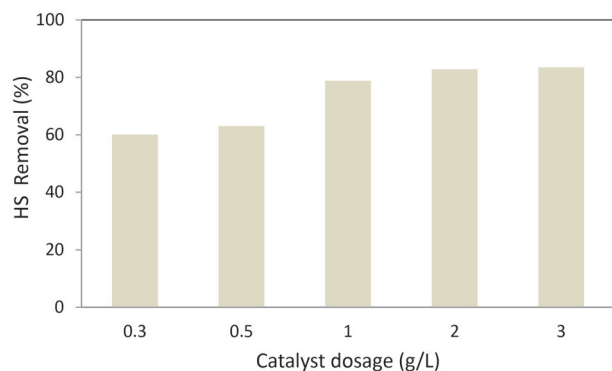


Fig. 4. Effect of Nanocatalyst Dosage on Sonocatalytic Decomposition of Humic Substances (Initial concentration of humic substances: 20 mg/L, pH: 7, power: 100%, frequency: 37 kHz and contact time: 120 min).

result, the increased decomposition efficiency (26). By increasing the power from 30 to 100, the decomposition efficiency was increased from 45% to 80%, and 25% to 45% at the frequencies of 37 and 80 kHz, respectively. As seen in Fig. 6, increasing the frequency led to the decrease in the sonocatalytic decomposition of humic substances. Considering the time of 120 minutes and catalyst value of 1 g/L, the removal efficiency of humic substances at the frequencies of 37 and 80 was obtained equal to 80.7% and 45.21%, respectively.

### 3.2.5. Comparison Processes

Fig. 7 demonstrates the removal efficiency of humic substances using MgO nanoparticles, ultrasonic, and sonocatalytic processes. As seen in this figure, the removal efficiency of humic substances using MgO nanoparticles, ultrasonic, and sonocatalytic processes were obtained as 25.43%, 35.57% and 78.35%, respectively at the contact time of 120 minutes. As shown in Fig. 7,

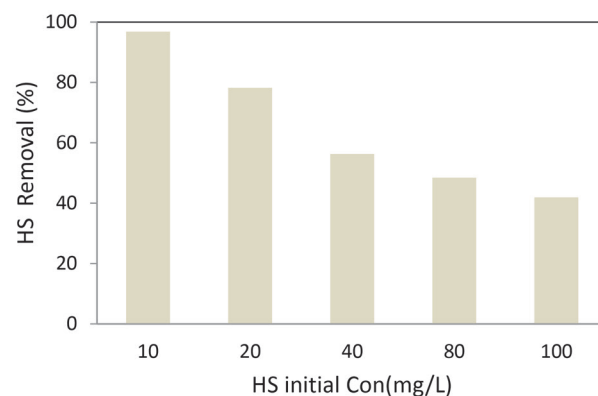


Fig. 5. Effect of Initial Concentration of Humic Substances on Sonocatalytic Decomposition Using MgO Nanoparticles (pH: 7, concentration of nanoparticles: 1 g/L, power: 100%, frequency: 37 kHz and contact time: 120 min).

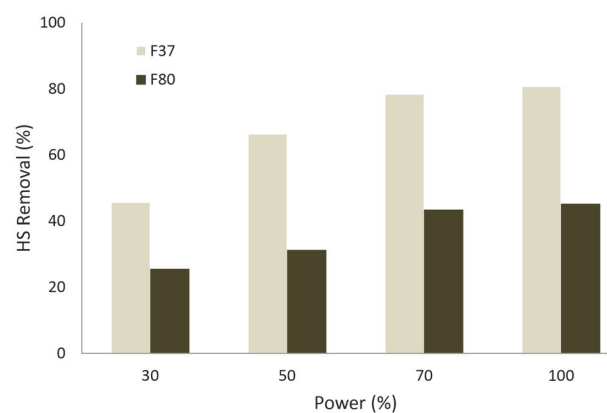
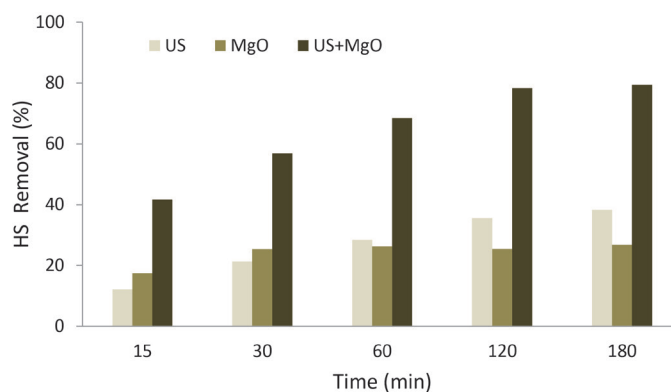


Fig. 6. Effect of Power and Frequency of Waves on Sonocatalytic Decomposition Using MgO Nanoparticles (Initial concentration of humic substances: 20 mg/L, concentration of nanoparticles: 1 g/L, pH: 7 and contact time: 120 min).



**Fig. 7.** Comparison processes (US, MgO, US+MgO) and Effect of Contact Time on Removal of Humic Substances in These Processes (pH: 7, initial concentration of humic substances: 20 mg/L, concentration of nanoparticles: 1 g/L, power: 100% , frequency: 37 kHz and contact time: 120 min).

MgO nanoparticles and ultrasonic process alone had no significant removal efficiency for the humic substances. However, the combination of ultrasonic process and MgO nanoparticles (sonocatalytic processes) led to the increased removal of humic substances. In agreement with the results of our study, Darvishi Cheshmeh Soltani et al observed that the removal efficiency of BR46 dye using ultrasound alone (25.4%) and MgO nanostructures alone (10%) was lower than that of the sonocatalytic processes (84.6%) (28). Fig. 7 also shows that by increasing the contact time, the removal efficiency of humic substances was increased in all processes especially sonocatalytic processes. The high rate of sonocatalytic decomposition of humic substances due to the increase in the contact time could be attributed to the increased production of hydroxyl radicals as well as the increased formation of electron pores (23,24).

#### 4. Conclusion

In the present study, the MgO nanoparticles were synthesized using mild hydrothermal method; then, the efficiency of these nanoparticles for the sonocatalytic decomposition of humic substances in aquatic environments was investigated. Characteristics of the MgO nanoparticles were determined using XRD and SEM analyses. The obtained results indicated that increasing the initial pH from 2 to 7 led to the increase in the removal efficiency of the humic substances, while by increasing the pH from 7 to 11, the decomposition efficiency was reduced. Moreover, the results showed that the sonocatalytic decomposition efficiency of humic substances was low in the absence of the MgO nanocatalyst; whereas, increasing the nanocatalyst content value from 0.5 to 3 resulted in the increased decomposition efficiency. Additionally, the results showed that with an increase in the initial concentration of the humic substances, the sonocatalytic decomposition efficiency was reduced.

#### Conflict of Interest Disclosures

The authors declare that they have no conflict of interests.

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