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Research Article

Pharmaceutical analysis for Novel drug formulation

Degradation Of Amoxicillin In Aqueous Solution By Nano-MgO/UV Photocatalytic Process

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Abstract: Antibiotics, an important type of pharmaceutical product, have attracted many researchers to the study of their removal from aqueous solutions for certain reason. Although the amount of pharmaceuticals in the aquatic environment is low, its continuous input may constitute in the long term potential risk for aquatic and terrestrial organisms. Therefore, over the past few years these compounds are considered to be an emerging environmental problem. In this study, the effects of some operational factors on the degradation efficiency of Amoxicillin (AMO) in aqueous solution were discussed through photocatalytic experiments using Magnesium oxide (MgO) as the photocatalyst. The experiments were carried out by irradiating the aqueous solutions of AMO containing photocatalysts with UV light. Similar experiments were carried out by varying pH (3–11), amount of catalyst (0.1–1 g/L) and initial concentration of AMO (10–100 mg/L) at irradiation time between 10 and 120 min. The experimental results indicated that the maximum degradation of AMO occurred at basic pH and the maximum degradation of AMO was noticed at pH 11. The percentage reduction of AMO was estimated under UV system and it was found that AMO reduction takes place at a faster rate under UV light as compared to darkness. The results indicated that the optimal conditions for the process were the irradiation time of 60, amount of catalyst of 0.6 g/L and AMP concentration of 10 mg/L, which resulted in the maximum efficacy of 100%. In addition, the findings indicated that the process had outstanding efficiency in the removal of the AMO antibiotics.

Keywords: Batch process, Nano-MgO/UV, Amoxicillin, Photocatalytic, Degradation.

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1. INTRODUCTION

Poor sanitation, water scarcity, deterioration of water quality, emergence of waterborne diseases, and lack of clean water supply are all posing global challenges due to the rising demand by an increasing world population [1, 2]. The sustained use of antibiotics in human medicine and veterinary has provoked an alarming increase of antibiotic resistant bacteria in the environment [3, 4]. This resistance can be transmitted to human beings through complex biochemical transference processes [5, 6]. Over the past few years, antibiotics and their metabolites have been detected in surface water, ground water, wastewater, and drinking water at levels ranging from ng/L to mg/L [7, 8]. Wastewater treatment plants are not capable of completely removal these kinds of pharmaceuticals, which in turn can reach superficial waters or sediments constituting a public health concern [9, 10]. Amoxicillin is a semi-synthetic, broad-spectrum medication widely used to treat common human infections [11]. It is an β-lactam antibiotic that has the molecular form of $C_{16}H_{19}N_3O_5S$ and the molecular weight of 365.4 g/mol[12]. It's antimicrobial activity is related to the beta lactam ring system in its structure [13]. It inhibits interactions between linear peptidoglycan chains, resulting in the inhibition of bacterial cellular wall synthesis [14]. Significant amounts of Amoxicillin (AMO) have been detected in superficial, potable water and sludge due to their ineffective removal by the conventional water treatment methods [15]. It seems that traditional biological methods could not effectively eliminate antibiotics [11]. Therefore, it is important to develop effective techniques for rapid elimination of antibiotics[12]. Various methods have been suggested to handle the antibiotic removal from water such as biodegradation, coagulation, adsorption, advanced oxidation processes (AOPs) and the membrane process [16, 17]. All these processes have some advantages or disadvantages over the other methods [18] A balanced approach is therefore needed to look into the worthiness on choosing an appropriate method which can be used to degrade the antibiotic in solution [19, 20]. Recently there has been a considerable interest in the utilization of advanced oxidation processes (AOPs) for the complete destruction of antibiotics [21]. AOPs are based on generation of reactive species such as hydroxyl radicals that oxidizes a broad range of organic pollutants quickly and non-selectively [22]. AOPs include photocatalysis systems such as combination of semiconductors and light, and semiconductor and oxidants [23]. Photocatalysis is a promising method in water treatment for removal and mineralization of organic pollutants. Photocatalysis using a semiconductor is an alternative to conventional methods [24]. Of all the semiconductors used for the process, titanium dioxide has essentially proved itself to be the best material for environmental purification because of its many desirable properties [25]. Magnesium oxide (MgO) is a cheap, readily available material, highly stable chemically and the photo-generated holes are highly oxidizing. In addition, MgO is capable of oxidation of a wide range of organic compounds into harmless compounds such as CO_2 and H_2O [24]. Given the importance of the removal of antibiotics from the pharmaceutical wastewater discharged by industries, the present study was to assess the potential of Magnesium oxide nanoparticles in AMO removal from aqueous solutions.

2. MATERIALS AND METHODS

2.1 Chemicals and reagents

Amoxicillin (AMO) was supplied by Sigma–Aldrich. The chemical structure of AMO is as shown below (Fig 1). All reagents such as sodium hydroxide (NaOH, 98%), and sulfuric acid (H_2SO_4, 99%) and acetonitrile were of analytical grade and purchased from Merck (Germany). All solutions were prepared by using deionized water. The experiments were performed at ambient temperature between 25 °C and 28 °C at solution pH. The AMO solutions were prepared using double distilled water. Similar experiments were carried out by varying pH (3 –11), amount of catalyst (0.1–1 g/L) and initial concentration of AMO (10–100 mg/L) at irradiation time between 10 and 120 min. As for the UV/MgO process, irradiation was performed in a batch photoreactor of 1000 ml in volume with a mercury lamp Philips 30 W (UV-C). A 500 mL aliquot of the antibiotics aqueous solution was placed in a 1000 mL reactor with the required amount of MgO and was mixed by a magnetic stirrer. The pH was adjusted to the required value by 0.1 N H_2SO_4 or 0.1 N NaOH and the mixture was kept for 30 min for dark adsorption. Thereafter, the mixture was subjected to UV irradiation and the source of UV light was a UV lamp (Spectroline model; EA-160/FE, 230V) with a nominal power of 30 W. Samples were taken at pre-selected time intervals using a syringe and filtered through 0.45 μm membrane filter for determination of antibiotic concentration. AMO concentration of all samples was determined by HPLC (C_{18} ODS column) with a UV detector 2006 at a wavelength of 190 nm. The mobile phase was a mixture of buffer phosphate with pH= 4.8 and acetonitrile with a volumetric ratio of 60/40 with an injection flow rate of 1 mL/min.

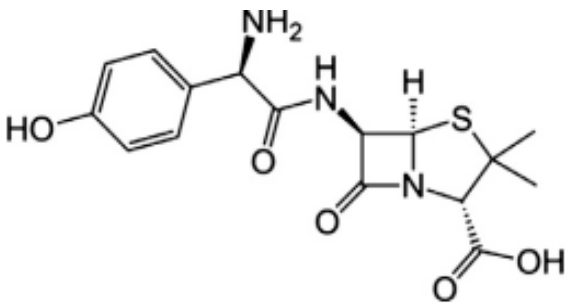

Fig 1. The chemical structure of AMO

3. RESULTS AND DISCUSSION

3.1 Effect of UV irradiation and MgO particles

Fig. 2 shows the effect of UV irradiation and MgO particles on photodegradation of AMO. It can be seen from the Fig. 2. that in the presence of both MgO and light, 99% of AMO was degraded at the irradiation time of 60 min. This was contrasted with 29.5% degradation for the same experiment performed in the absence of MgO, and the negligible 49% when the UV lamp had been switched off and the reaction was allowed to occur in the darkness. These experiments demonstrated that both UV light and a photocatalyst, such as MgO were needed for the effective destruction of AMO. This is due to the fact that when MgO is illuminated with the light of λ<390 nm, electrons are promoted from the valence band to the conduction band of the semiconducting oxide to give electron–hole pairs[26]. The valence band (h^+_{VB}) potential is positive enough to generate hydroxyl radicals at the surface and the conduction band potential is negative enough to reduce molecular oxygen[27]. The hydroxyl radical is a powerful oxidizing agent and attacks pollutants present at or near the surface of MgO. It causes photo-oxidation of pollutants according to the following reactions

$$MgO + h\vartheta \rightarrow e^- + h^+$$
$$e^- + O_2 \rightarrow O^{\bullet 2-}$$
$$e^- + h^+ \rightarrow \text{heat}$$
$$h^+ + H_2O \rightarrow OH^\bullet + H^+$$
$$h^+ + OH^- \rightarrow OH^\bullet$$
$$h^+ + AMO \rightarrow AMO^\bullet + \text{oxidized products}$$
$$OH^\bullet + AMO \rightarrow \text{degradation of the AMO}$$

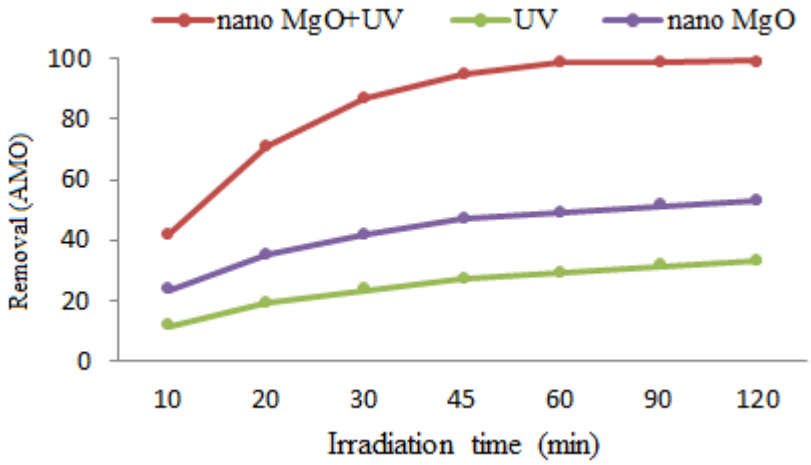

Fig 2. Effect of UV light and MgO on photocatalytic degradation of AMO (pH: 11, initial AMO concentration: 10 mg/L, photocatalyst concentration: 0.6 g/L)

3.2 Effect of changes in Photocatalyst Concentration

MgO loading photocatalytic processes is an important factor that can strongly influence antibiotics wastewater degradation. Experiments were performed using catalyst loadings up to 1 g/L and the extent of AMO wastewater reduction after 60 min of irradiation at effluent ambient pH are shown in Fig. 3. It is evident that removal was sufficiently high (69%) even for the lower MgO loading tested (0.2 g/L) and increased over 96.2% with increasing MgO concentration to 0.6 g/L; above this, catalyst concentration removal remained practically constant. At 1 g/L MgO, which is the higher loading tested, removal was found to be almost 100% resulting in a completely AMO solution. It has been reported in several studies that AMO degradation increases with increasing catalyst concentration up to a value, above which AMO conversion remains practically unchanged, reaching a plateau. This crucial MgO loading depends on several factors like reactor geometry, operating conditions of the photoreactor, wavelength and intensity of the light source, and corresponds to the point where the entire catalytic surface is fully illuminated [28-30].

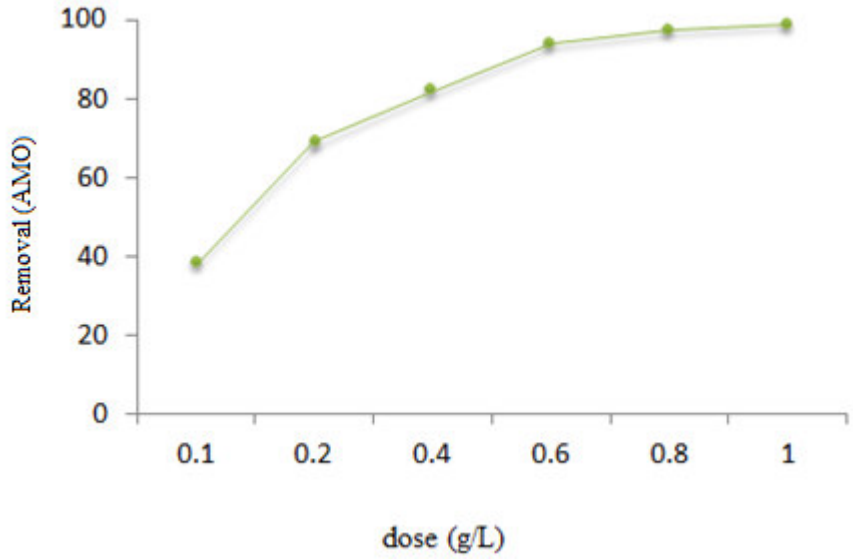

Fig 3. Effects of photocatalytic concentration changes on removal of AMO (pH: 11, initial AMO concentration: 10 mg/L, photocatalyst concentration: 0.6 g/L, reaction time: 60 min)

3.3 Effect of pH

The efficiency rates for removal of AMO at pH 3, 5, 7, 9 and 11 were 51.44, 59.73, 71.4, 91.3 and 98.78%, respectively. In the absence of the nanocatalyst, these values were 34.5, 41.42, 49.31, 58.53 and 68.35%, respectively (Fig 4). The greatest removal rate reported at pH 11 in the presence of the nanocatalyst was caused by a high concentration of hydroxyl radicals in the solution [31]. The impact of pH on the contaminant molecules, the surface charge of a nanocatalyst, and the mechanism of the degree of production of hydroxyl radicals are important [32]. The hydroxyl radical rapidly reacts with the aromatic ring in the side chain of amoxicillin. The effect of pH on antibiotic degradation can be explained by considering the properties of both catalyst and antibiotics at different pH values [30]. At acidic pH, MgO and amoxicillin are positively charged, and hence, adsorption on the surface of MgO is limited. The high degradation of antibiotics at acidic pH compared to that of at neutral pH may be due to the hydrolysis of antibiotics. At alkaline pH, both amoxicillin and the MgO are negatively charged, and so repulsive forces develop between the catalyst and the antibiotics [33]. The high degradation of antibiotics under alkaline conditions may be due to two facts: the enhancement of hydroxyl radical formation at high pH due to the availability of hydroxyl ions on the MgO surface that can easily be oxidized to form hydroxyl radicals and the hydrolysis of the antibiotics due to the instability of the β-lactam ring at high pH values [34]. In the study of Evgenidou et al at photocatalytic removal of the antibiotics amoxicillin from aquatic environments. Results showed that the greatest removal rate was obtained at pH 11. It was also reported that at alkaline pH, more hydroxyl ions had become available to the catalyst, producing more hydroxyl radicals and thus oxidizing the antibiotic [35].

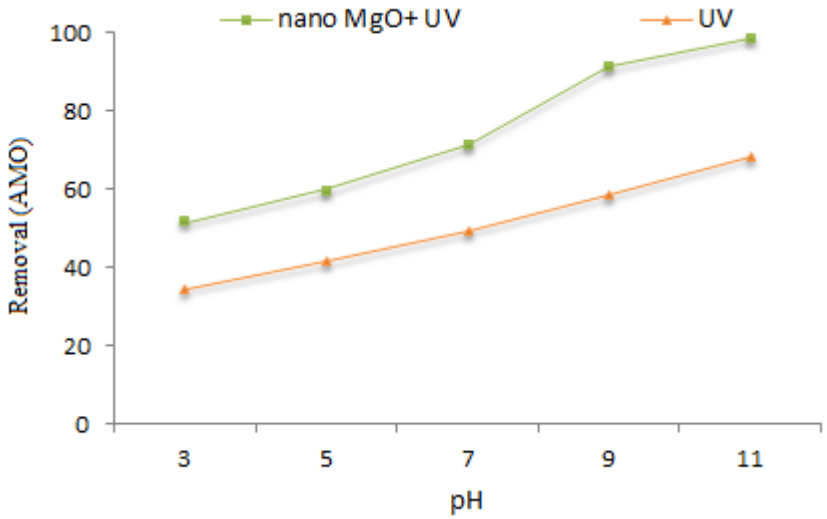

Fig 4. Effect of initial pH changes on removal of AMO (initial AMO concentration: 10 mg/L, photocatalyst concentration: 0.6 g/L, reaction time: 60 min)

3.4 Effect of concentration of AMO

After optimizing the pH conditions, reaction time and catalyst dose (pH 11, time 60 min and catalyst dose 0.6 g/L), the photocatalytic degradation of AMO was carried out by varying the initial concentrations of the AMO from 10 to 100 mg/L in order to assess the appropriate amount of catalyst dose. As the concentration of the AMO was increased, the rate of photo-degradation decreased indicating either to increase the catalyst dose or time span for the complete removal. Fig 5 depicts the time dependent graphs of degradation at different concentrations of AMO solutions (10–100 mg/L). In the case of AMO, for AMO solutions of 10 and 20 mg/L, almost 100 and 96.5 % degradation occurred within 45 min. For 60 mg/L of the AMO solution degradation was 91% in 90 min and it gets further decreased on increasing the concentration of AMO. The possible explanation for this behavior is that as the initial concentration of the AMO increases, the path length of the photons entering the solution decreases and in low concentration the reverse effect is observed, thereby increasing the number of photon absorption by the catalyst in lower concentration [36-38]. The same effect was observed by Napoleon et al.[39] during the photocatalytic degradation of three commercial textile dyes (Reactive Yellow, Reactive Red and Reactive Blue) using TiO_2 as photocatalyst under solar light.

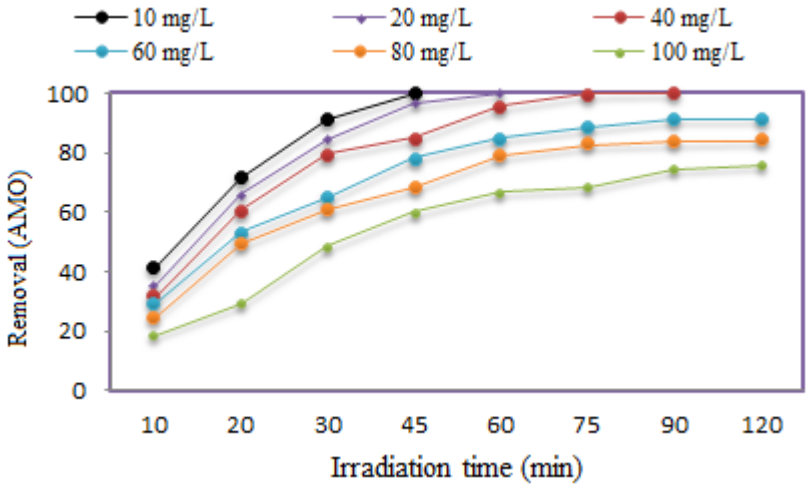

Fig 5. Effect of concentration of AMO on degradation efficiency under UV (catalyst dose 0.6 g/L; pH 11)

The effect of catalyst loading and pH on the reaction rate was ascertained and optimum conditions for maximum degradation was determined. This experimental study was conducted in a photo-reactor with a useful volume of one liter. According to the results, there was a direct correlation between photocatalyst concentration and contact time with the process efficiency in the removal of the AMO. The process efficiency was higher in alkaline conditions compared to neutral and acidic conditions. The results obtained showed that alkaline pH is proper for the photocatalytic removal of AMO.

4. CONCLUSION

In this study, the photocatalytic degradation of AMO was investigated in the UV/MgO system. The amount of AMO antibiotic removal was found to be dependent on catalyst concentration, initial AMO concentration, and irradiation time. Finally, the photocatalytic process with MgO nanoparticle could be used effectively as an advanced oxidation reaction to remove AMO antibiotic.

5. ACKNOWLEDGEMENTS

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6. AUTHORS CONTRIBUTION STATEMENT

Prof. FK. Mostafapour conceived the idea and also reviewed the manuscript. Dr. D. Balarak and Dr K. Chandrika carried out the research study, evaluated the results and drafted the manuscript.

7. CONFLICT OF INTEREST

Conflict of interest declared none.

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