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Disinfection of biologically treated industrial wastewater using montmorillonite/alginate/nanosilver hybrids

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ABSTRACT

Different techniques have been developed to eliminate microorganisms from industrial wastewater and improve its final quality for reuse. Among them, the use of silver nanoparticles (AgNPs) anchored onto montmorillonite (MMT) and dispersed in a sodium alginate (ALG) polymeric matrix appears to be a good alternative. In this sense, the aim of this work was to prepare MMT–ALG–AgNPs hybrids and then characterize them using UV–vis spectrophotometry, transmission electron microscopy, scanning electron microscopy, Fourier transform infrared spectroscopy and X-ray diffraction, to develop a new bactericidal material for the disinfection of industrial wastewater intended for reuse. The results confirmed the presence of spherical AgNPs anchored onto MMT, which was dispersed in the ALG matrix. The evaluation of the bactericidal potential of the MMT–ALG–AgNPs hybrids was performed by the agar diffusion test using two microorganisms (*E. coli* and *S. aureus*). These tests showed the formation of a prominent zone of inhibition for both bacteria. The MMT–ALG–AgNPs hybrids were then used to disinfect industrial wastewater. The results indicated a reduction of up to 98.5% of the total coliforms. For comparison, the industrial wastewater was subjected to UV radiation, which is commonly used in the disinfection of water and wastewater. In this case, the removal of the total coliforms using the MMT–ALG–AgNPs hybrids was slightly higher when compared to the use of UV radiation. This result illustrates the potential of these materials for the disinfection of industrial wastewaters.

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1. Introduction

Growing concerns regarding environmental conservation and the implementation of increasingly stringent laws on waste disposal have led many researchers to develop products and processes with a lower pollution potential. Thus, the development of studies that aim to minimize the environmental impact of industrial wastewater is of great interest [1].

The reuse of industrial wastewater has been encouraged in Brazil because this practice brings numerous economic and environmental benefits. However, there is no legislation in Brazil that specifies acceptable physicochemical and microbiological parameters for this practice. Thus, these parameters depend on the future application of effluent to be reused [2,3].

Metal nanoparticles anchored onto solid substrates have found applications in a broad range of fields, including solar batteries, catalysts, photocatalysts, and sensors, among others [4]. In the last decade, silver nanoparticles (AgNPs) have been widely used as antibacterial agents for the disinfection of water and the microbiological treatment of industrial wastewater due to a number of advantages over traditional disinfectants, such as chemical stability, safety, and efficiency [5,6]. Recent literature indicates the role of silver species as the principal actors to explain AgNPs antimicrobial effects [6]. However, they also have a harmful influence on eukaryotic species [7]. In order to reduce damage on non-target organisms and avoid environmental pollution, the controlled application and kinetics of release are of prime importance [8].

Clay minerals possess large surface areas, which gives them good adsorbent properties. Therefore, the silicates represent an excellent substrate for the attachment of metallic nanoparticles [4,9] such as AgNPs on the surface of montmorillonite (MMT). Moreover, hybrid materials based on MMT and AgNPs may be an inexpensive and interesting alternative to the conventional

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disinfection processes used currently for the treatment of wastewater intended for reuse [5,10,11]. However, due to the small particle size, the removal of such materials from the medium after the disinfection process may be difficult if they are not supported on another substrate.

Polymeric matrices such as poly(methacrylic acid), poly(vinyl pyrrolidone), poly(methyl methacrylate), chitosan, and polyamide 6.6 have been investigated as support materials for AgNPs [12–14]. Alginate (ALG), a biopolymer extracted predominantly from brown algae, has been tested in the removal of contaminants in aqueous media [9].

In this context, the aim of the present work was to prepare hybrid materials from MMT, ALG and AgNPs for application in the disinfection of industrial wastewater intended for water reuse. Initially, the morphology and physicochemical characteristics of the hybrids were analyzed. Then, the bactericidal potential of the hybrids against two types of microorganisms (*E. coli* and *S. aureus*) was evaluated using the agar diffusion test. Finally, the MMT–ALG–AgNPs hybrids were used in the disinfection of industrial wastewater, and the results were compared to those obtained from the treatment of wastewater using UV radiation. The amount of silver within the hybrid material and in the effluent was quantified before and after disinfection using inductively coupled plasma optical emission spectroscopy (ICP-OES).

2. Experimental

2.1. Materials

All chemical reagents used in this work are of analytical grade: silver nitrate ($AgNO_3$, Merck), calcium chloride dihydrate ($CaCl_2{\cdot}2H_2O$, Merck), sodium borohydride ($NaBH_4$, Merck), medium viscosity sodium alginate (Vetec), and sodium citrate tribasic dihydrate ($Na_3C_6H_5O_7$, Sigma–Aldrich). Sodium MMT was obtained from Argel T. The following solutions: $AgNO_3$, $Na_3C_6H_5O_7$, $NaBH_4$, and $CaCl_2{\cdot}2H_2O$ were prepared using deionized water (resistivity 18.2 MΩ cm) obtained from a Direct-Q 3 UV (Milli-Q Millipore) system.

2.2. Synthesis of the AgNPs

The AgNPs were synthesized by the chemical reduction of silver ions by $NaBH_4$ in the presence of $Na_3C_6H_5O_7$. The method used was based on the work of Jana et al. [15]. Briefly, a 100 mL solution was prepared by mixing $AgNO_3$ (50 mL, 0.25 mmol L^{-1}) and $Na_3C_6H_5O_7$ (50 mL, 0.25 mmol L^{-1}), and stirred for 30 s. Then, 10 mL of a 1.00 mmol L^{-1} solution of $NaBH_4$ (freshly prepared) was added to the mixture. The synthesis was performed at $23.0 \pm 2.0\,^{\circ}C$ and protected from light. Finally, the system was stirred for 60 s and subsequently stored at 10 °C.

2.3. Characterization of the AgNPs

The AgNPs were characterized using UV–vis spectrophotometry and transmission electron microscopy (TEM). The AgNPs solution was analyzed in a Thermo Scientific Evolution 60S spectrophotometer between 300 and 550 nm using a quartz cuvette with an optical path of 10 mm. TEM was performed with a Jeol JEM-1200 Ex II microscope at 80 kV. The AgNPs were previously dispersed in deionized water (1 drop of the colloidal silver solution in 20 mL of water) using an Unique USC 1400 ultrasound for 30 min. This solution was then dropped onto a 300 mesh copper grid coated with a Formvar film and allowed to dry for 48 h before analysis. The particle size distribution of the AgNPs was determined using *Image Tool for Windows* software from the analysis of 200 particles on average.

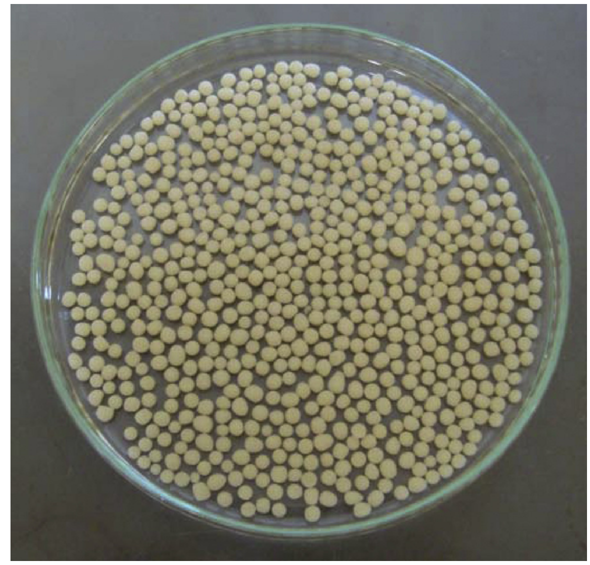

Fig. 1. Morphology of the MMT–ALG–AgNPs hybrids.

2.4. Preparation of the MMT–ALG–AgNPs hybrids

The method used to prepare the MMT–ALG–AgNPs hybrids was based on the previous work of Liu et al. [9]. Initially, MMT (5.0 g) was mixed with 100 mL of the AgNPs dispersion for 12 h. ALG (1.0 g) was then added to the suspension and the system was mixed for a further 12 h. After this, the mixture was slowly dripped into a $CaCl_2{\cdot}2H_2O$ solution (4.0% m/v) using a syringe.

The hybrid material formed (Fig. 1) remained in this solution for a further 48 h at 10 °C away from light. Then, the hybrid material was washed repeatedly with deionized water to remove the calcium chloride, and stored in deionized water at 10 °C until required for the microbiological tests.

It is noteworthy that the optimal amount of MMT and ALG used in the preparation of the MMT–ALG–AgNPs hybrids was previously investigated via parametric studies considering two experimental designs. In the first case, the amount of ALG was maintained constant (1.0 g) while the amount of MMT was varied (1.0, 2.0, 3.0, 4.0 and 5.0 g); in the second case, the amount of MMT was maintained constant (1.0 g) while the amount of ALG was varied (2.0, 3.0, 4.0 and 5.0 g). In both cases, it was used the same volume of the AgNPs dispersion, i.e., 100 mL. The mixture time and concentration of the $CaCl_2{\cdot}2H_2O$ solution were also maintained constant (12 h and 4.0% m/v, respectively). The best result in terms of stability of the material during the disinfection process of the industrial wastewater was obtained with the hybrids which were prepared using 5.0 g of MMT and 1.0 g of ALG. In the other cases, the hybrid materials did not acquire the pellet form (Fig. 1) or they crumbled during the stirring in the orbital shaker. Concerning the AgNPs dispersion, the volume and concentration were not changed in the experimental design because the removal percentage of total coliforms was up to 95%.

2.5. Characterization of the MMT–ALG–AgNPs hybrids

The MMT–ALG–AgNPs hybrids were characterized by TEM, scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared (FT-IR) spectroscopy. TEM analysis was performed as previously described in Section 2.3. The surface of the

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