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Research article

Comparison between silver- and copper-modified zeolite-rich tuffs as microbicide agents for *Escherichia coli* and *Candida albicans*L.G. Rossainz-Castro ^{a, b}, I. De-La-Rosa-Gómez ^a, M.T. Olguín ^{b, *}, D. Alcántara-Díaz ^c^a Laboratorio de Investigación en Ingeniería Ambiental, Instituto Tecnológico de Toluca, Av. Tecnológico s/n, Ex. Rancho la Virgen, Metepec, México, C.P. 52140, Mexico^b Departamento de Química y, Instituto Nacional de Investigaciones Nucleares, Carretera México-Toluca s/n, La Marquesa Ocoyoacac, México C.P. 52750, Mexico^c Departamento de Biología, Instituto Nacional de Investigaciones Nucleares, Carretera México-Toluca s/n, La Marquesa Ocoyoacac, México C.P. 52750, Mexico

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ABSTRACT

Zeolite-rich tuff from the State of Chihuahua was modified with silver or copper ions (ZChAg and ZChCu) to evaluate its microbicidal effect against *Escherichia coli* (*E. coli*) and *Candida albicans* (*C. albicans*) suspended in an aqueous solution in order to compare the microbial disinfection kinetics between bacteria and yeast. The zeolite-rich tuff was treated with AgNO₃ or CuCl₂ solutions. The materials obtained were characterized using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD) and the textural properties were also determined by BET-analyses. The concentration of Ag and Cu was verified in the zeolitic materials using neutron activation analysis. The experimental data were adjusted to both Chick and Chik-Watson models to describe the kinetic behavior of the process. It was found that when the mass of ZChAg increased, the survival microorganisms notably decreased. The *E. coli* and *C. albicans* showed higher resistance in contact with ZChCu even when the mass of such material was 10–20 times higher than the mass of ZChAg. Chick and Chik-Watson constants showed that the kinetics of the disinfection process depended on the desorption of the exchange ion that modified the structure of the zeolitic material, its concentration in aqueous medium, its oligodynamic properties, and each microorganism's characteristics (Gram-negative bacteria and yeast). The kinetic desorption of Ag and Cu from the corresponding modified-zeolite-rich tuffs was also considered in this work. In this case, the Higuchi and Korsmeyer-Peppas models were applied.

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1. Introduction

Currently, the microbiological contamination of water is an important factor to be considered due to the ensuing health hazard (Schijven et al., 2016). Because of the variety and numbers of microorganisms present in water, it is not possible to fully analyze them. Therefore, microorganisms such as fecal and total coliforms have been commonly used as contamination indicators due to their rapid replication and small size (Drechsel and Keraita, 2014; Odonkor and Ampofo, 2013). Other larger microorganisms (such as yeasts) that are more complex in their structure show stronger resistance to different disinfection agents (Burrola-Aguilar, 2004), and thus, it is important to consider them in the sanitary control of

water. Among yeast species, the most frequent and pathogenic from an odontological-medical point of view is *C. albicans*, which exhibits a lower decay rate in seawater compared with *E. coli*. *C. albicans* can impact the population's health, causing effects that range from skin allergies to severe systemic infections in immunodeficient patients. In the vegetative state, this yeast is slightly more resistant than bacteria to the action of certain antiseptics, and cultures of this yeast in distilled water can survive for up to 2 years (Mayer et al., 2013; Negroni, 2009).

Total coliform and fecal coliforms are resistant to commonly used disinfectants, such as chlorine (Friedler et al., 2011). This makes it necessary to research new disinfectants that not only have a broad microbicidal effect but also reduce the generation of disinfection by-products to a minimum (Krishnani et al., 2012). Some inorganic compounds can satisfy these requirements; among them are metallic compounds that exhibit microbicidal properties

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(Johari et al., 2016; Lalley et al., 2014; Hrenovic et al., 2012a,b).

The metallic ions of silver, copper, mercury, tin, lead, bismuth, cadmium, chromium, and thallium have microbicidal effects (Top and Ülkü, 2004). In recent years, several studies have been performed using silver as a microbicide against indicator organisms of water quality that are pathogenic agents with respect to human health.

Zeolites modified with different metallic ions (Ag, Cu, Fe, Hg, Sn, Pb, Bi, Cd, Cr, Ti) have been used as microbicide agents (Burrola-Aguilar, 2004; Hrenovic et al., 2012a,b; Lalley et al., 2014). An example are natural zeolites modified with silver or copper, which can eliminate pathogenic microorganisms such as *E. coli*, *Streptococcus faecalis*, and *Staphylococcus aureus* from water (Akhigbe et al., 2014; Hrenovic et al., 2012a). Although it is important to consider whether the silver or copper released from the modified natural zeolite after the disinfection process exceeds the permissible levels in drinking water established by the US EPA (Ag, 0.1 mg/L; Cu, 1.3 mg/L) and the WHO guidelines (Ag, 0.1 mg/L; Cu 2.0 mg/L) to prevent health effects, such as Argyria in the case of silver and liver or kidney damage from copper.

The disinfection processes has been focused on the kinetic (Rodríguez-Chueca et al., 2015; Akhigbe et al., 2014); however, the correlation between both kinetic parameters (disinfection and metallic release) is not well known. The kinetic models give information on the behavior of the disinfection processes, as well as the release of metals from a zeolitic material, which play a role in the efficiency of the antimicrobial systems. It is important to consider a kinetic model to compare the experimental data through the parameters obtained for each case.

Somani and Ingole (2012) mention that Chick gave the principal precepts for a disinfection kinetics model based on the similarity between microbial inactivation by disinfectants and reactions. The Chick-Watson model considers the effect of the microbicide agent concentration on the reaction rate (Rodríguez-Chueca et al., 2015). In the case of the kinetics of the copper and silver release from the zeolitic network, in the present work, the Higuchi and Korsmeyer-Peppas models were considered for describing the release of metal ions from a solid matrix. The former model is based on Fick's law, and the latter model is considered when the release mechanisms are not well known or when more than one type of liberation mechanism could be involved, as was mentioned by Costa and Lobo-Lobo (2001).

Factors such as the mass of the modified zeolitic material and type of metal it contains can affect the time required to achieve total mortality of the microorganisms present in water (Burrola-Aguilar, 2004). Moreover, it is necessary to further study the disinfection process kinetics, considering the desorption of metallic ions from the modified zeolitic materials and the type of microorganisms contaminating the water. Therefore, the aim of this work was to describe the disinfection process for water by applying both Chick and Chick-Watson models. Different masses of the modified zeolitic material (10–200 mg), various metallic ions (Ag^+ and Cu^{2+}), and different pathogenic microorganisms (*E. coli* and *C. albicans*) for comparison purposes were considered. Silver and copper desorption from Ag- or Cu-zeolite-rich tuffs plays a significant role in water disinfection; therefore, the present investigation also describes the desorption kinetics, applying the Higuchi and Korsmeyer-Peppas models.

2. Materials and methods

2.1. Materials

A Mexican zeolite-rich tuff from “Cerro Prieto ejido San Miguel de los Anchondo”, located 7 km from Parral, Chihuahua, with a

cation exchange capacity of 1.09 ± 0.01 meq/g (Díaz-Nava et al., 2005) was used in this work. The particle diameter selected was 0.60 mm. The material was identified as ZCh. The reagents used (NaCl , AgNO_3 and CuCl_2) were of commercial analytical grade from J. T. Baker.

2.2. Zeolite modification

2.2.1. NaCl solution

A sample of ZCh was treated in a reflux system with a 1 M NaCl solution for 24 h. The sample was then washed with deionized water until the presence of chloride ions in the solution was not detected. The zeolite-rich tuff sample modified with sodium (ZChNa) was dried at 85 °C for 5 h.

2.2.2. AgNO₃ solution

A sample of ZChNa was treated in a reflux system with a 0.1 M AgNO_3 solution for 12 h in darkness. Then, the sample was exhaustively washed with deionized water until the presence of NO_3^- ions was not detected in solution. The zeolite-rich tuff sample modified with silver (ZChAg) was dried at 82 °C for 5 h (De la Rosa-Gómez et al., 2008a).

2.2.3. CuCl₂ solution

A sample of ZChNa was treated in a reflux system with a 0.1 M CuCl_2 solution for 12 h. The sample was then washed with deionized water until the presence of chloride ions was not detected in solution. The zeolite-rich tuff sample modified with copper (ZChCu) was dried at 85 °C for 5 h (Burrola-Aguilar, 2004).

2.3. Characterization

2.3.1. Scanning electron microscopy and energy dispersive X-ray spectroscopy

To determine the morphology and a semi-quantitative elemental composition of the different modified zeolites (ZCh, ZChNa, ZChAg, and ZChCu), the samples were mounted directly onto the holders and then observed at 10 and 20 kV in a JEOL JSM 5900 scanning electron microscope (SEM) under vacuum with an Oxford energy dispersive X-ray spectroscopy system (EDS). Tests of three different regions of the zeolitic materials were performed for each zeolitic sample at 500 X to obtain the average weight (%).

2.3.2. X-ray diffraction

To identify the minerals contained in zeolite-rich tuff and to determine the presence of other components or to quantify the reduction in crystallinity of the materials after the different modifications with metallic salts, the samples were analyzed by X-ray diffraction, using SIEMENS D5000 equipment, with a Cu anode. The X-ray diffraction patterns of the zeolitic materials were compared with those of clinoptilolite (JCPDS 01-083-1261), quartz (JCPDS 03-065-0466), AgNO_3 (JCPDS 01-074-0947) and CuCl_2 (JCPDS 00-034-0198).

2.3.3. Neutron activation analysis

The amounts of Na, Mg, Cl, K, Ca, Fe, Cu, and Ag present in the different samples of natural zeolite were determined by neutron activation analysis using the SINCA position of the nuclear reaction TRIGA MARK III of the National Institute of Nuclear Research (Instituto Nacional de Investigaciones Nucleares), with an approximate flux of 10^{12} n/(cm²s), during different irradiation times. The radioactivity of the samples was measured in a Ge/hyper-pure device and compared with the certified standard material. The nuclear characteristics of the isotopes generated, as well as the detection limits for each element, are listed in [Supplementary](#)

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