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Study of the adsorption-desorption of Cr (III) on green silica obtained from sodium silicate

Estudio de la adsorción-desorción de Cr (III) en sílice verde obtenida a partir del silicato de sodio

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Abstract

Mesoporous silica (MS) is nowadays one of the most widely used materials for the removal of contaminants in water; however, its main disadvantage is its production cost, since it is obtained by the sol-gel process using tetraethyl orthosilicate (TEOS) as silica former, which is not an economical reagent. In this article, we propose the synthesis route of SM from industrial sodium silicate; Na_2SiO_3 by passing it through an ion exchange column (DOWEX-50WX8-100) to obtain silicic acid ($Si(OH)_4$). The acid fraction is collected from the ion exchange column at pH 1–3 and allowed to age for 24 h. The SM silicate is formed from the gelation of the aged silicic acid and was characterized by infrared spectroscopy. The porosity was determined by N_2 desorption adsorption isotherms and scanning electron microscopy. The $Cr(^{3+})$ removal capacity was evaluated by the effect of the initial $Cr(^{3+})$ concentration. Obtaining that the maximum removal capacity was 108.7 mg/g for a Cr^{3+} solution of 2794 ppm.

Low cost, Sodium silicate, Mesoporous silica, Cr^{3+}, Adsorption

Resumen

La sílice mesoporosa (SM) hoy en día es uno de los materiales más empleados para la remoción de contaminantes en agua; sin embargo, su principal desventaja es el costo de producción de la misma; ya que se obtiene a partir del proceso sol-gel empleando Tetraetil-ortosilicato (TEOS) como formador de la sílice; el cual es un reactivo no económico. En este proyecto se propone la ruta de síntesis de la SM a partir de silicato de sodio de uso industrial; Na_2SiO_3 haciendolo pasar por una columna de intercambio iónico (DOWEX-50WX8-100) para obtener ácido silícico ($Si(OH)_4$). De la columna de intercambio iónico se recolecta la fracción ácida ente un pH 1-3 y se dejó envejecer por 24 hr. La SM-silicato se forma de la gelificación del ácido silícico envejecido y se caracterizó por espectroscopia de infrarrojo, la porosidad se determinó con las isotermas de adsorción desorción de N_2 y por microscopía electrónica de barrido. La capacidad de remoción de $Cr(^{3+})$ se evalúo en un batch; determinando el efecto de la concentración inicial de $Cr(^{3+})$; la capacidad máxima de remoción fue de 108.7 mg/g para una solución de Cr^{3+} de 2794 ppm.

Sílice de bajo costo, Sílice mesoporosa, Silicato de sodio, Cr^{3+}, Adsorción

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Introduction

Water quality is essential to life, as it is required for almost every aspect of life. However, the planet's water supply is known to be finite and threatened by pollution [1]. One of the main sources of water pollution is the release of toxic wastewater from various industries, as according to UNESCO (United Nations Educational, Scientific and Cultural Organization), more than 80% of wastewater in developing countries is discharged untreated [2]. In 2019, studies on global water pollution were conducted and the results show that heavy metals are the main chemical pollutants in wastewater, accounting for 31% [3]. Figure 1 shows a study of water quality in Mexico in 2017, where only 18.5% is in excellent condition and 19.15% is in good condition [4].

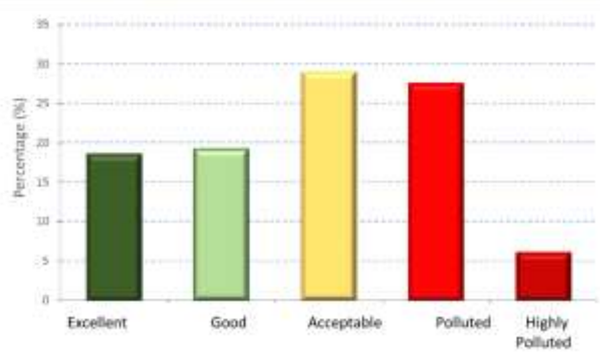


Figure 1 Water quality reported by CONAGUA in 2017 [8]

One of the most common contaminants in wastewater from processes such as electroplating, leather tanning, textile pigments, metallic coatings, and others is chromium [5]. It is a metal that has several carcinogenic and mutagenic properties and its high levels in water bodies can seriously affect humans, animals and aquatic life [6]. In general, some of the major uses and applications of chromium (III and VI) are:

1. In the metallurgical industry, where it is mainly used to provide corrosion resistance and a bright finish.

2. In refractory materials, in the manufacture of magnesite and chrome-based bricks for metallurgical furnaces, and in the use of granular chromite for many other applications requiring heat resistance.

3. In the chemical industry for chrome plating, pigment and dye production, leather tanning and wood treatment.

Therefore, this project proposes the use of a mesoporous silica obtained from sodium silicate as an adsorbent for the removal of Cr^{3+} in wastewater; the silica is obtained by a synthetic method using an inexpensive silica-forming precursor, industrial grade sodium silicate.

Methodology

Synthesis of MS-silicate

SM-silicate was obtained by passing a solution of industrial grade Na_2SiO_3 (10 g of silicate in 50 mL of distilled water) through a DOWEX 50WX8-100 ion exchange column and collecting the acidic fraction at pH 1–3. The collected fraction is silicic acid ($Si(OH)_4$), which is aged at 25°C for 24 h. The silica is then added to a solution of Pluronic P-123 (EO20-PO170-EO20; PMav=5800; Sigma Aldrich), which acts as a structural director. The P-123 dilution was prepared by dissolving 66.2 g of the polymer in 600 mL of deionized H_2O. After mixing P-123 with aged $Si(OH)_4$ (10 mL), the mixture was refluxed for 24 hours until silica gel was formed. P-123 was then extracted by refluxing with acidic ethanol for 12 hours, and the solid obtained was filtered, washed with ethanol, dried at 80ºC for 6 hours, and finally calcined at 200ºC for 3 hours to remove any trace of P-123 present in the silica.

Characterization of MS-silicate

The SM-silicate was characterized by infrared spectroscopy using a Nicolet-iS10 Thermoscientific instrument, obtaining the average of 16 scans, with a resolution of 4 cm^{-1} and a spectral window from 4000 to 600 cm^{-1}. The textural properties of the material (porosity) were determined with the N_2 adsorption-desorption sysotherm at 77K, using a Micromeritics ASAP-2010 instrument. The samples were degassed for 12 hours at 180 °C and 71 mmHg prior to measurement. Finally, the morphology of the material was observed by scanning electron microscopy in a Joel-6510 plus SEM microscope.

Cr^{3+} removal tests with MS-silicate

A solution of chromium sulfate (Karal; 99%) in distilled water was used to determine the Cr^{3+} removal capacity of MS-silicate. Table 1 shows the concentrations used in the study. Adsorption was performed with 0.1 g of silica and 10 mL of Cr^{3+} solution at 25ºC with constant stirring.

The concentration of residual Cr^{3+} was quantified using the Beer-Lambert law by measuring the absorbance at a wavelength of 413 nm in an SQ-2800 single beam UV-visible spectrophotometer.

Cr^{3+} concentration (mgL^{-1})	247	437	837	1636

Table 1 Conditions used for the Cr3+ removal study

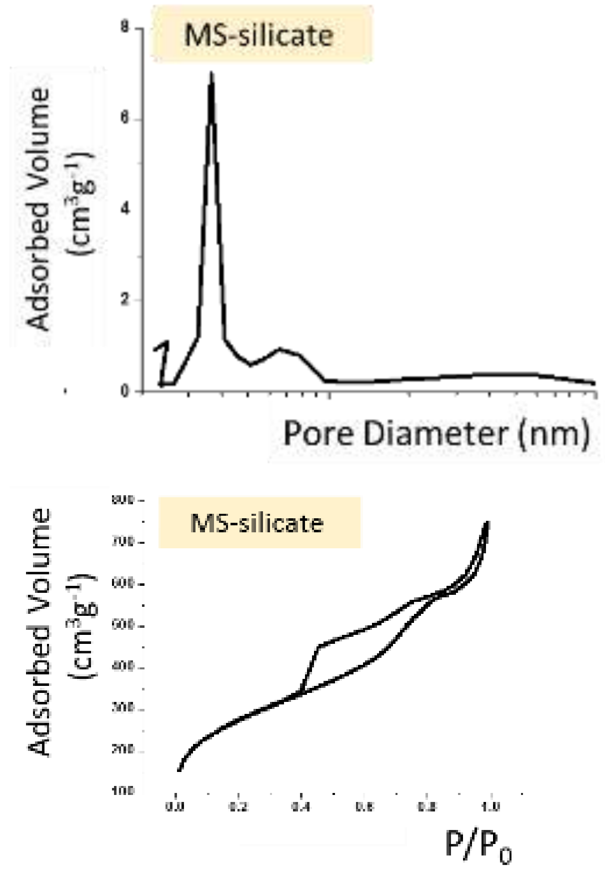
Results

Figure 1a shows the characterization of SM-silicate by infrared spectroscopy, where the silica skeleton (SiO_2) is observed free of organic matter and with a significant amount of physisorbed water broadband at 3500 cm^{-1},□ and 1600 cm^{-1} □. The silica skeleton is identified by the signal at 1100 cm^{-1},□□(Si–O–Si) and free silanols, Si–OH at 950 cm^{-1} [7].

According to the N_2 adsorption-desorption isotherms (Figure 1b) we have a mesoporous material with isotherm type IV according to IUPAC and H_2 hysteresis as well; the BET surface area was calculated to be 924.12 m^2g^{-1} with a BJH desorption pore volume of 0.99 cm^3g^{-1}.

The pore distribution shows the formation of two pores at 4.3 and 8.6 nm, while the SEM shows the formation of a material by agglomerates of spherical particles in the nanometer range.

a



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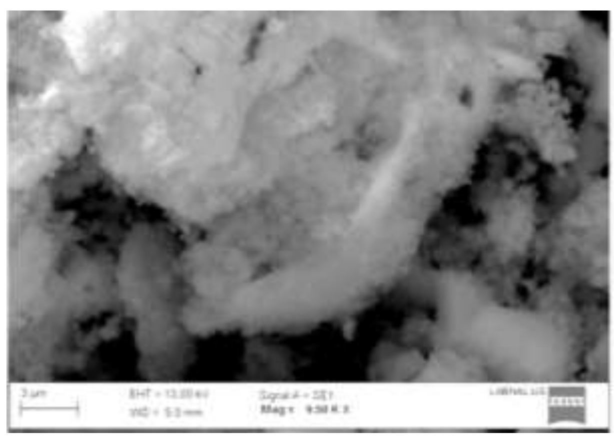
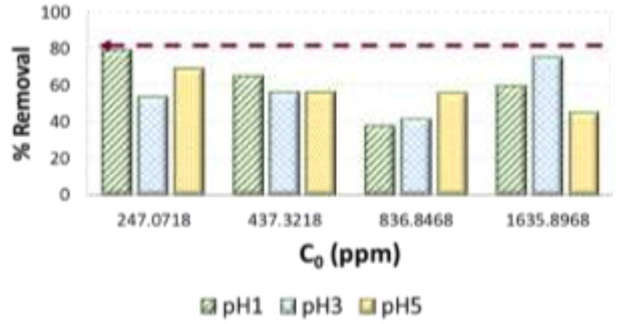


c

Figure 1 Characterization of MS-silicate (a) Infrared spectroscopy (b) N_2 adsorption-desorption isotherms (c) SEM

To quantify the Cr^{3+} concentration in the removal tests, the calibration curve was obtained in a range giving an R^2 of 0.9995. Figure 2a shows the removal percentage calculated according to eq. 1, where C_0 is the initial concentration and C_e is the concentration of the remaining chromium that has not been adsorbed (equilibrium concentration); depending on pH and concentration, up to 80% removal can be achieved at pH 1 and pH 3.

$$\%Removal = \frac{C_0 - C_e}{C_0} X100 \quad (1)$$



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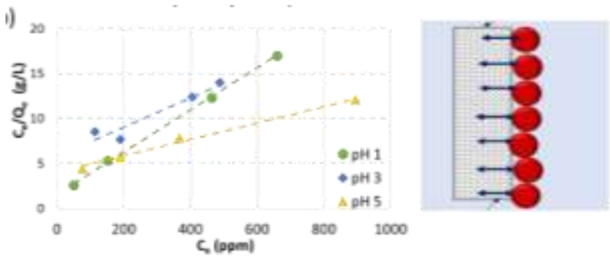

b

Figure 2 Cr^{3+} Removal Study (a) Percent Removal (b) Maximum Adsorbed Load

On the other hand, the data were fitted to the Langmuir adsorption isotherm model (eq. 2), which considers an ideal adsorption condition, where one analyte molecule is adsorbed in each active site until the monolayer is formed. In this model, Q_0 is the maximum adsorption charge in mgg^{-1}, q_e is the charge at equilibrium ($8mgg^{-1}$), C_e is the sorbate concentration in the solution at equilibrium and K_L is the Langmuir constant. The partition coefficient, R_L (eq. 3), determines the type of adsorption such that if $R_L>1$, the adsorption of the system is unfavorable; if $R_L=1$, the adsorption is favorable and linear; values of $0<R_L<1$ indicate favorable adsorption, while values of $R_L=0$ indicate irreversible adsorption [8].

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_0} + \frac{C_e}{Q_0} \quad (2)$$

$$R_L = \frac{1}{1+K_L C_0} \quad (3)$$

Table 2 summarizes the data obtained for fitting the experimental data to the Langmuir model; the R^2 was greater than 0.90 in all cases. The calculated R_L values indicate favorable adsorption systems (R_L of 0.015-0.002). On the other hand, pH significantly modifies the maximum adsorption load, favoring it at pH 5, where a Q_o of 109 mgg^{-1} was obtained, while at pH 1 it was 42.55 mgg^{-1}.

	pH 1	pH 3	pH 5
R^2	0.999	0.900	0.990
Q_o (mg/g)	42.553	60.976	**108.696**
K_L (L/g)	0.015	0.003	0.002

Tabla 2 Ajuste de los datos obtenidos experimentales al modelo de adsorción de Langmuir

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Conclusions

According to the results obtained, the SM-silicate presents an excellent capacity to remove Cr^{3+} in aqueous solutions, removing about 80% of the metal at high chromium concentrations of 1633 mgL^{-1}, allowing an efficient removal of chromium present in tannery effluents. This material presents a maximum metal removal capacity of 108.69 mgg^{-1} when the adsorption was carried out at pH 5.

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