

Synthesis and characterization of SiO₂/Ag nanocomposites by chemical reduction for agricultural applications

Síntesis y caracterización de nanocompuestos de SiO₂/Ag mediante reducción química para aplicaciones agrícolas

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ABSTRACT: This study aims to synthesize SiO₂/Ag nanocomposite (NC) by hydrothermal method (Barbosa, A. D. *et al.*, 2012; Morales, S. L. *et al.*, 2014; Madathil, A. N. P. *et al.*, 2007) and chemical reduction using aqueous extracts of *Chenopodium ambrosioides* L. (epazote) as reducing and stabilizing agents to synthesize SiO₂/Ag NC. For the characterization of the nanocomposites, it was used techniques such as transmission electron microscopy (TEM), X-ray diffraction (XRD), FTIR and UV-Vis spectroscopy to analyze the morphology, structure and functional properties of the synthesized NC. The synthesized NC aims to remediate wastewater for agricultural use.

KEYWORDS: nanocomposite NC, silver nanoparticles, wastewater treatment.

RESUMEN: El objetivo de este estudio es sintetizar un nanocomposito (NC) de SiO₂/Ag mediante un método hidrotérmico (Barbosa, A. D. *et al.*, 2012; Morales, S. L. *et al.*, 2014; Madathil, A. N. P. *et al.*, 2007) y reducción química, utilizando extractos acuosos de *Chenopodium ambrosioides* L. (epazote) como agentes reductores y estabilizadores para sintetizar el NC de SiO₂/Ag. Para la caracterización de los nanocompositos se utilizaron técnicas como la microscopía electrónica de transmisión (TEM), la difracción de rayos X (XRD), la espectroscopía FTIR y la espectroscopía UV-Vis para analizar la morfología, la estructura y las propiedades funcionales del NC sintetizado. Los NC sintetizados tienen como objetivo remediar las aguas residuales para su uso agrícola.

PALABRAS CLAVE: nanocompósito NC, nanopartículas de plata, tratamiento de aguas residuales.

Introduction

One significant global issue, including in Mexico, is the increased presence of environmental pollutants. In Mexico, water contamination remains a cha-

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llenge, with diseases transmitted by contaminated water impacting approximately 6% of the rural population (Norma Oficial Mexicana NOM-001-SE-MARNAT-2021). In recent years, several methods for the synthesis of nanostructured systems have been developed due to their enormous technological applications and fundamental scientific interest. Colloidal nanoparticles (Nps) exhibit interesting physicochemical properties, which are mainly due to changes in parameters such as size, shape, composition or crystalline structure (Jaque, D. *et al.*, 2016.). The unique properties exhibited by Nps have a wide range of potential applications in fields such as medicine, biotechnology, catalytic reactions and especially in wastewater remediation. Photocatalytic materials exhibit specific properties and can therefore be used to solve the problem of water pollution. Certain classes of semiconductors such as SiO₂ for heavy metal removal, non-toxicity and excellent surface properties (Liu, J. *et al.*, 2019). In addition, silicon-based nanocomposites are easily surface modified by groups such as amino (-NH₂), or thiol (-SH), etc., or serve as a support for nanocomposites. Previous studies by Kotsyuda, S. S. *et al.* (2017), showed that synthesized silica nanospheres biofunctionalized by 3-aminopropyl and phenyl groups and investigated their removal effects towards Cu(II) and cationic, observed good results in removing these contaminants. Functionalized silica nanospheres had improved adsorption capacities towards Cu(II) and methylene blue compared to amino-functionalized nanosilica. In addition, nanoscale SiO₂ has been shown to be useful in the removal of heavy metals. Najafi, M. *et al.* (2012) have investigated the removal effects for Cd(II), Ni(II) and Pb(II) with functionalized SiO₂, including amino-functionalized silica gel (NH₂-SG), amino-functionalized silica hollow nanosilica (NH₂-SNHS) and non-functionalized silica hollow nanosilica (SNHS) [9]. SNHS and the metal adsorption trend was Pb(II) > Cd(II) > Ni(II). With the previous results performed by these working groups, our hypothesis is that the photocatalytic/antibacterial NCs proposed in this research project could be an alternative for wastewater remediation.

This project presents a method for synthesizing nanocomposites based on SiO₂/Ag nanoparticles intended for wastewater treatment that is both cost-effective and environmentally friendly. These nanocomposites are designed to act as heavy metal adsorbents and antimicrobial agents, with the goal of lowering concentrations of metals and enterobacteria and allowing treated wastewater to meet permissible contaminant limits for agricultural use.

Materials and methods

Preparation of extract from *Chenopodium ambrosioides* L.

Epazote plants (*C. ambrosioides* L.) were grown in a greenhouse located in Texcoco, State of Mexico; seeds were purchased from Kristen Seed. Epazote extracts from *C. ambrosioides* L. were obtained using a variation of the method previously reported by Carrillo, L. M. *et al.* (2014), in which the leaves are

manually separated from the plants, extensively washed with deionized water, and dried in an oven at 65 °C for 72 h. The dried leaves and stems are ground in an agate mortar to pass through a 1.0 mm (20 mesh) sieve for homogeneous particle size. Then, 10 g of the dried and sieved biomass is heated in 100 ml of deionized water for 5 min and allowed to cool at room temperature (25 °C) for 1 h. The next step is centrifugation with a Thermo Scientific Heraeus Megafuge 8 centrifuge (Massachusetts, USA) 3 times at 4000 rpm for 5 min and finally decanting the extract until a green, sediment-free liquid is obtained.

Determination of total phenols in the extract by UV-Vis spectrophotometry in a plate reader

The total phenolic content in the extract was quantified by the colorimetric method using the Folin-Ciocalteu reagent (Attard, E. 2013, Barrón, Y. *et al.*, 2011) In this method, 15 µL of the diluted extract is added to 240 µL of distilled water, 15 µL of Folin-Ciocalteu reagent (1 N) is added and after 3 minutes the reaction is neutralized with 30 µL of 4 N sodium carbonate. The mixture is incubated for 120 minutes. The absorbance at 760 nm was determined using a Thermo Scientific Varioskan Multimode Flash Microplate Reader. Spectral readout modes: absorbance, fluorescence intensity and luminescence (Waltham, Massachusetts, USA). The concentration of total free phenolics is calculated from a standard curve ($y = mx + b$) plotted with gallic acid as standard and expressed as mg gallic acid equivalents (GAE) per g dry matter. The extract with the highest total phenolic content is used for the synthesis of nanocomposites.

The extracts shall be stored in amber flasks and covered to avoid interaction with light at 4 °C.

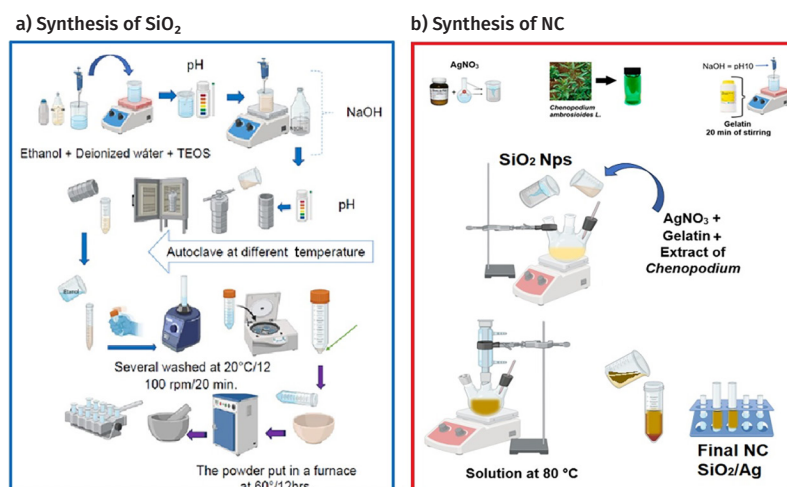
Synthesis of the nanocomposites

Six samples were synthesized at different temperatures (150–210 °C) and reaction times (12–24 h), and were labeled as T1, T2, T3, T4, T5, and T6 for this purpose, tetraethyl orthosilicate (TEOS 99% - Sigma-Aldrich, St. Louis, USA), ammonium hydroxide (NH₄OH 27% - Sigma-Aldrich, St. Louis, USA), absolute ethanol (99.5% - Sigma-Aldrich, St. Louis, USA), silver nitrate (AgNO₃ 99% - Sigma-Aldrich, St. Louis, USA), and gelatin from porcine skin (gel strength 300, Type A, Sigma-Aldrich, St. Louis, USA) were used.

The experiment was conducted in two stages. The first stage involved synthesizing SiO₂ nanoparticles (Nps) using a hydrothermal method (Barbosa, A. D. *et al.*, 2012) (see figure 1, left). The second stage consisted of coating the synthesized SiO₂ Nps with Ag Nps to produce a nanocomposite. Ag Nps were prepared by a chemical reduction method. Specifically, 1.12 mL of tetraethyl orthosilicate and 0.90 mL of sodium hydroxide (NaOH 1M) were dissolved in 20 mL of ethanol and 0.08 mL of water. This mixture was stirred for 5 minutes before being transferred to an autoclave and subjected to hydrothermal treat-

ment at varying temperatures and durations. Following natural cooling to room temperature, the resulting Nps were separated by centrifugation, washed four times with a 1:1 ethanol-water solution, and dried at 60 °C for 12 hours. For the chemical reduction synthesis of SiO₂/Ag Nps, 10 mL of water-dispersible SiO₂ Nps (0.26 g) and AgNO₃ (0.16 g), along with solutions containing a passivating agent (1% gelatin), were combined and heated at 80 °C. When the target temperature was reached, 5 mL of *C. ambrosioides* L. extracts were gradually added. The solution, initially transparent green, changed to a brown colloidal suspension, indicating the formation of Ag Nps.

Figure 1. a) Schematic representation of the hydrothermal method of SiO₂ Nps; b) the chemical reduction method of AgNO₃ to form SiO₂/Ag nanocomposites.



Source: Author's elaboration with Biorender.com.

Characterization of nanoparticles and nanocomposites

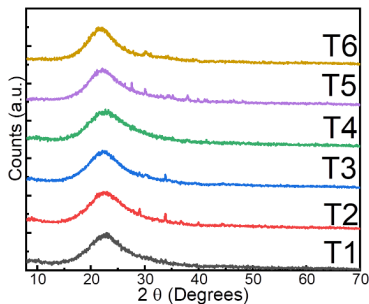
The crystal structure of the SiO₂ nanoparticles was characterized by X-ray diffraction (XRD) using a Bruker D8 ADVANCE ECO diffractometer (Karlsruhe, Germany). The diffraction patterns were recorded in the 2θ range of 8–70°, with Cu Kα radiation ($\lambda = 1.5418 \text{ \AA}$) as the X-ray source. The vibrational functional groups of the samples were analyzed by Fourier-transform infrared spectroscopy (FTIR) using a Bruker Alpha II spectrometer, in the wavenumber range of 400–4000 cm⁻¹ (Germany, Berlin). The UV–Visible spectra of the nanoparticles and nanocomposites (NC) were obtained using a spectrophotometer Thermo Scientific (Waltham, Massachusetts, US) in the absorbance range of 280–800 nm. Raman spectra were recorded with a Thermo Scientific confocal micro-Raman system (Waltham, Massachusetts, USA) using a 532 nm laser with 2.33 eV excitation energy. Measurements were

performed over the 50-3000 cm^{-1} spectral range. Transmission electron microscopy (TEM) observations were carried out using a FEI, Tecnai G2 Spirit microscope operated at 120 kV (FEI, Hillsboro, USA). The samples for TEM analysis were prepared by depositing the nanoparticle powder onto a 300-mesh carbon-coated Cu grid. Finally, HRTEM image was measured with a microscope JEM-ARM200F, JEOL (Akishima, Tokio, Japan) operated at 200 kV.

Results and discussion

X-ray diffraction was used to investigate the crystalline nature of the SiO_2 . The X-ray diffraction patterns confirmed the amorphous nature of the prepared SiO_2 samples as shown by a broad peak observed at 23° of the amorphous structure. We also observed a crystalline structure of SiO_2 in a cristobalite structure. In the case of SiO_2 , it has been observed that amorphous samples exhibit a characteristic diffraction pattern that includes a broad peak, generally around 23° , indicating a lack of long-range order in their structure. This behavior is typical of amorphous materials, where the absence of a well-defined diffraction pattern indicates that the structural units are not organized in a regular crystal lattice.

Figure 2. Comparison of the X-ray diffraction patterns for different samples T1-T6.

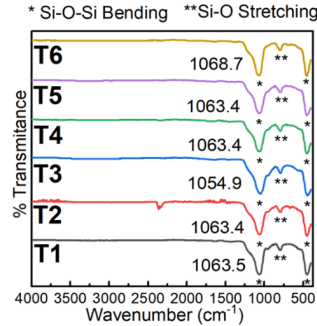


Source: Author's elaboration.

The FTIR spectrum of the nanoparticles showed typical absorption bands of the silicate at 484.5, 797.9, 1063.4 cm^{-1} , which are usually assigned to tosilo-xane bond (797.9), Si-O-Si bending (1063.4 cm^{-1}) and silanol (Si-OH) symmetric stretching (3396.4 cm^{-1}).

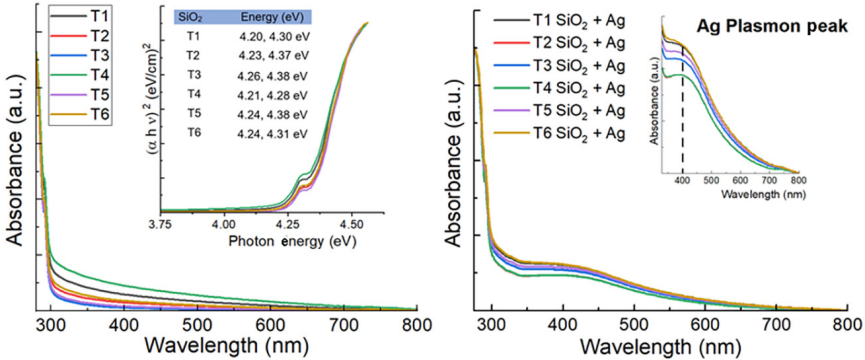
UV-vis absorbance spectra of SiO_2 Nps, according to Tauc's equation the band gap value was calculated, and we show inset in the table, from [4.20-4.38] eV values. SiO_2/Ag nanocomposites were synthesized using the aqueous leaves extract of *C. ambrosioides*, which had higher total polyphenol content (9.89 mg GAE/ g) than the stems aqueous extract (5.68 mg GAE/ g). Shown on figure 4 is the absorbance spectra of the nanocomposite, in which the presence of Ag plasmon near 400 nm is observed.

Figure 3. The Fourier-transform infrared (FTIR) spectra of the samples T1-T6 exhibit the characteristic absorption bands of SiO₂, including the strong Si–O–Si asymmetric stretching vibration.



Source: Author’s elaboration.

Figure 4. a) Absorbance spectra and band gap energy for the different SiO₂ Nps; b) absorbance spectra of the SiO₂/Ag NC samples.



Source: Author’s elaboration.

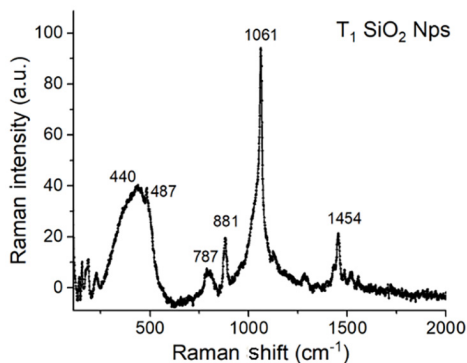
Table 1. Raman frequencies and the assignation for SiO₂ nanoparticles.

Raman frequencies (cm ⁻¹)	Band assignation
1454	Cristobalite
1061	TO
881	W ₃
787	Cristobalite
487	D1 breathing relaxation mode of the 4-atom rings
440	ω ₁ bending mode of oxygen atoms

Source: Author’s elaboration.

A representative Raman spectrum of the sample T1 is shown in which 6 main bands corresponding to SiO₂ are observed, also the cristobalite phase is detected. Table I shows the frequency and band assignment (figure 5).

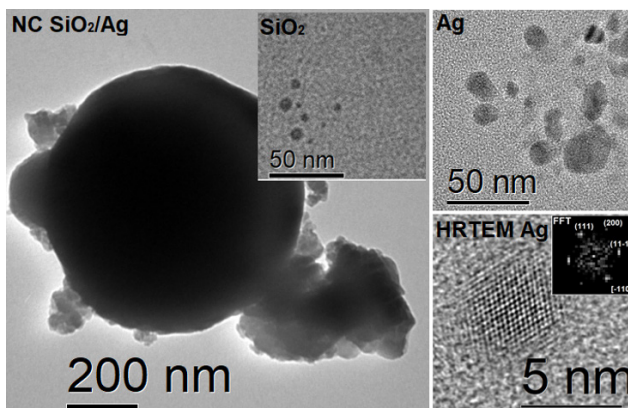
Figure 5. Representative Raman spectrum of the SiO₂ nanoparticles (T1) displays the characteristic band at ~465 cm⁻¹, corresponding to the symmetric stretching vibration of Si–O–Si bonds, confirming the presence of amorphous silica.



Source: Author's elaboration.

Finally, electron microscopy studies were performed to analyze the shape and size of the NCs (figure 6). An image of the NC is shown in which a 400 nm particle can be distinguished in which there are small Ag particles on the surface. An image of small NCs of SiO₂ individually is also presented, as well as a bright field image of only Ag nanoparticles. Finally, high-resolution transmission electron microscopy (HRTEM) image is shown in which it is clearly observed that, in addition to a single crystal structure, there is a hexagonal profile with a size of 5 nm. In the inset of the figure, the FFT are shown the crystallographic planes corresponding to (1, 1, 1), (1, 1, -1) and (2, 0, 0) of FCC Ag- COD 96-500 -0219, with $d_{111} = 0.735$ nm, $d_{1-1-1} =$ and $d_{002} = 0.374$ nm, respectively, this particle is oriented in the [-1, 1, 0] direction.

Figure 6. a) Transmission electron microscopy (TEM) micrographs of the SiO₂/Ag NC, SiO₂ and Ag synthesized at different temperatures.



Source: Author's elaboration.

Future work

The nanocomposites (NCs) will be utilized as agents for wastewater remediation. The planned remediation strategies comprise: (i) removal of heavy metals frequently present in contaminated water (including As, Cd, Cr, Cu, Ni, Pb, Zn, and Hg), and (ii) reduction of bacterial load, with a particular focus on *Escherichia coli*. To evaluate their remediation performance, a cost-effective reactor will be developed, leveraging the inherent physicochemical properties of the NCs. In particular, multifunctional systems such as SiO₂/Ag nanocomposites are anticipated, integrating the antibacterial activity of Ag nanoparticles with the heavy metal adsorption capabilities of SiO₂ (Gankhuyag, S. 2021). This approach is intended to offer a sustainable technological solution addressing global freshwater scarcity by facilitating wastewater treatment for agricultural reuse.

Conclusions

A hydrothermal method is described for synthesizing SiO₂ nanoparticles, which are then combined with *C. ambrosioides* L. extract to form SiO₂/Ag nanocomposites. Characterization of these nanocomposites is essential for assessing their performance in wastewater remediation. The second stage of the study will evaluate the ability of these nanocomposites to remove heavy metals such as As, Cd, Cr, Cu, Ni, Pb, Zn, and Hg, as well as biological contaminants including *Escherichia coli*. Additionally, a mini-reactor is proposed for treating wastewater intended for reuse in agricultural irrigation. Implementing this system in the fields of San Ignacio and San Juan at the Autonomous University of Chapingo may support crop and livestock management and could provide a reference for other institutions and communities.

Authors contribution

Ramos-Mondragón Pedro, Navarro-Cerón Elizabeth, Hernández-Rodríguez Landy, Ascencio-Aguirre Francisco: conceptualization, article design, methodological development.

Navarro-Cerón Elizabeth, Sánchez-Fernández Rosa, Canseco-González Daniel, García-Muñiz José Guadalupe: methodological development, analysis and interpretation, review and final editing of the text.

Navarro-Cerón Elizabeth, Ascencio-Aguirre Francisco: conceptualization, article design, methodological development, data mining, analysis and interpretation, writing of the original draft, and final review and editing of the text.

References

- Attard, E. (2013). A rapid microtitre plate Folin-Ciocalteu method for the assessment of polyphenols. *Open Life Sciences*, 8(1), 48-53. <https://doi.org/10.2478/s11535-012-0107-3>.
- Barbosa, A. D., Santos, E. R., Leite, R. C. & Rodrigues, M. G. (2012). Obtención de la zeolita mcm-22 a través de la síntesis hidrotermal utilizando diferentes métodos. *Avances en Ciencias e Ingeniería*, 3(3), 59-67. <http://www.redalyc.org/articulo.oa?id=323627687007>.
- Barrón-Yáñez, Rosario M., García-Mateos, M. del Rosario, Soto-Hernández, Marcos R., Colinas-León, Teresa, Kite, Geoffrey. (2011). Flavonoides y actividad anti-oxidante de *Calia secundiflora* (Ort.) Yakovlev. *Revista Fitotecnia Mexicana*, 34(3): 151-157, julio-septiembre. <https://doi.org/10.35196/rfm.2011.3.151>.
- Carrillo-López, L. M., Zavaleta-Mancera, H. A., Vilchis-Néstor, A., Soto-Hernández, R. M., Arenas-Alatorre, J., Trejo- Téllez, L. I. & Gómez-Merino, F. (2014). Bio-synthesis of silver nanoparticles using *Chenopodium ambrosioides*. *Journal of Nanomaterials*, 2014, 198-198. <https://doi.org/10.1155/2014/951746>.
- Gankhuyag, S., Bae, D. S., Lee, K. & Lee, S. (2021). One-pot synthesis of SiO₂@ Ag mesoporous nanoparticle coating for inhibition of *Escherichia coli* bacteria on various surfaces. *Nanomaterials*, 11(2): 549. doi.org/10.3390/nano11020549.
- Jaque, D., Richard, C., Viana, B., Soga, K., Liu, X. & García Solé, J. (2016). Inorganic nanoparticles for optical bioimaging. *Advances in Optics and Photonics*, 8(1): 1-103. <https://doi.org/10.1364/AOP.8.000001>.
- Kotsyuda, S. S., Tomina, V. V., Zub, Y. L., Furtat, I. M., Melnyk, I. V. (2017). Bifunctional silica nanospheres with 3-aminopropyl and phenyl groups. Synthesis approach and prospects of their applications. *Applied Surface Science*, 420: 782-791. <https://doi.org/10.1016/j.apsusc.2017.05.150>.
- Liu, J., Chen, Y., Han, T., Cheng, M., Zhang, W., Long, J. & Fu, X. (2019). A biomimetic SiO₂@ chitosan composite as highly efficient adsorbent for removing heavy metal ions in drinking water. *Chemosphere*, 214: 738-742. <https://doi.org/10.1016/j.chemosphere.2018.09.172>.
- Madathil, A. N. P., Vanaja, K. A. & Jayaraj, M. K. (2007). Synthesis of ZnO nanoparticles by hydrothermal method. In *Nanophotonic materials IV*. Proceedings vol. 6639: 47-55. September. SPIE. Digital Library. <https://doi.org/10.1117/12.730364>.
- Morales, S. L., Cerón, E. N. & Sánchez, M. E. (2014). *Síntesis hidrotermal de nanoflowers*. https://scholar.google.com/scholar?hl=es&as_sdt=0%2C5&q=Morales%2C+S.+L.%2C+Cer%C3%B3n%2C+E.+N.%2C+%26+S%C3%A1nchez%2C+M.+E.+S%C3%ADntesis+Hidrotermal+de+Nanoflowers+de+TiO2&btnG.
- Najafi, M., Yousefi, Y., Rafati, A. A. (2012). Synthesis, characterization and adsorption studies of several heavy metal ions on amino-functionalized silica nano hollow sphere and silica gel. *Separation and Purification Technology*, 85: 193-205. <https://doi.org/10.1016/j.seppur.2011.10.011>.
- Pogorilyi, R. P., Melnyk, I. A., Zub, Y. L., Carlson, S., Daniel, G., Svedlindh, P., Seisenbaeva, G. A., Kessler, V. G. (2014). New product from old reaction: uniform

magnetite nanoparticles from iron-mediated synthesis of alkali iodides and their protection from leaching in acidic media. *RSC Advances*, 4: 22606-22612. <https://doi.org/10.1039/C4RA02217C>.

Secretaría de Medio Ambiente y Recursos Naturales. (2022). Norma Oficial Mexicana NOM-001-SEMARNAT-2021, que establece los límites permisibles de contaminantes en las descargas de aguas residuales en cuerpos receptores propiedad de la nación. *Diario Oficial de la Federación*. https://dof.gob.mx/nota_detalle.php?codigo=5678901&fecha=2022-03-11.