Green synthesis, characterization and potential antimicrobial activity of Nb_2O_5 nanoparticles from *Carya illinoinensis* nutshell extract under irradiation.

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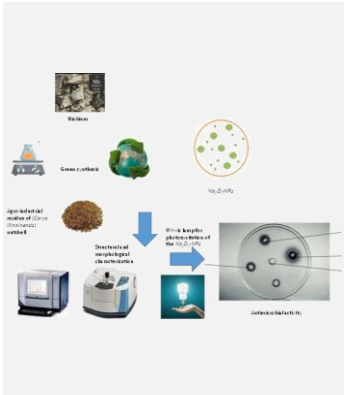

Water contamination by pathological microrganisms resulting from the inadequate treatment is considered a public health problem. In this sense, the development of nanomaterials for application in desinfection of water is a promising alternative for the treatment. In this context, the present work aims to synthesize and characterize Nb_2O_5-NPs by green synthesis from *C. illinoinensis* shell extract and evaluate the potential antimicrobial activity against *Escherichia coli*. The results showed that green Nb_2O_5-NPs are crystalline with average crystallite sizes of 21.6 nm. The surface was negatively charged (-22.7 mV), with a zero charge point of 6.77 and band gap energy of 3.25 eV. The antimicrobial activity tests showed 93.5 % reduction after 60 minutes and 89.8 % reduction after 150 minutes of *E. coli* count under irradiation. Therefore, the green Nb_2O_5-NPs showed potetial for application in desinfection of water under visible light.

Introduction

The growing interest in niobium is related to its multiple uses for the production of high-technology materials. Niobium is one of the strongest Lewis acids known, allowing its use in nanomaterials for catalysis in the form of niobium oxide [1]. The number of studies on the properties of NPs in biological environments is increasing, due to the different physical and chemical characteristics in relation to their micrometric compounds [2,3]. Nb_2O_5 is n-type transition metal oxide that exhibits good chemical stability, non-toxicity and commercial availability [4].

Nb_2O_5-NPs systems have been previously employed for the inactivation of *Escherichia coli* [4], being found significant differences between different processes. In this context, the present work aims to develop and characterize structurally and morphologically Nb_2O_5-NPs using an extract from (agro)industrial residue of (*Carya illinoinensis*) nutshells extract and evaluate their potential antimicrobial activity.

Material and Methods

Synthesis of Nb_2O_5-NPs

Firstly, the green synthesis of Nb_2O_5-NPs were prepared using the *C. Illinoiensis* nutshells extract (10 $g.L^{-1}$, 80 - 90 °C, 200 - 300 rpm, and 10 - 15 min). Then, 50 mL of extract were mixed with 50 mL of solution of 0.1 mol. L^{-1} $NbCl_5$ in 10% v v^{-1} of HCl and 5 mL of NH_4OH under magnetic stirring (200 - 300 rpm, 60 min) for the reduction and nucleation steps. After, the solutions were decanted and dried (70 - 80 °C) for 12 h, followed by calcination at 600 °C for 2 h to stabilize the nanoparticles.

Characterization

X-Ray Diffraction (XRD) was carried out to evaluate the crystallinities of heterogeneous catalysts in a Bruker D2 PHASER diffractometer (10 – 70° and $\lambda_{Cu-\alpha}$ = 0.1532 nm). The crystallite size (dc) and the interplanar distance (d) of the nanoparticles were calculated by the Debye-Scherrer and the Bragg equations [5], respectively. The zeta potential (ZP) was determined in a Malvern-Zetasizer® (ZEN3600, UK), and the zero charge point (pH_{ZPC}) was determined by 10 point assay [6] ranging from pH 2 to 12. The band gap energy (Eg) was determined by diffuse reflectance spectroscopy (in a Jasco V-670 absorption spectrometer ranging from 200 – 800 nm) and calculated by the Kubelka-Munk method.

Bacterial strains

Escherichia coli (ATCC 25922) was used as the indicator organism for antimicrobial activity assays. Strains were maintained on BHI agar plates at 4 ± 2 °C, and subcultured periodically.

Antibacterial activity

The bacterial inactivation test was performed in duplicate in a 96 well plate under irradiation (50 W lamp, 21 cm distance) using a concentration of Nb_2O_5-NPs of 16.5 mg. L^{-1} . The activity microbiological was measured in 30, 60, 90 and 150 min by the determination of colony forming unit (CFU mL^{-1}) following the literature [7].

Results and Discussion

The Nb_2O_5-NPs XRD diffractogram is presented in Figure 1(a). The Nb_2O_5-NPs are crystalline with the presence of characteristics peaks of the pseudohexagonal phase at

22.72° (001, dc = 31.9 nm, d = 3.92 A), 28.60° (100, dc = 15.5 nm, 3.12 A), 36.75° (101, dc = 17.6 nm, d = 2.44 A), 46.22° (002, dc = 28.7 nm, d = 1.96 A) and 55.29° (102, dc = 14.5 nm, d = 1.65 A) (JCPDS nº 28-0317). The average crystallite size of the Nb_2O_5-NPs was 21.6 nm.

Figure 1(b) presents the pH_{ZPC} where it was possible to verify that pH_{ZPC} = 6.77. Thus, when the pH < 6.77 the surface is protonated and pH > 6.77 the surface is deprotonated. The bang gap energy of the Nb_2O_5-NPs was 3.25 eV according to the Tauc-plot displayed in Figure 1(c). Thus, the surface of the green Nb_2O_5-NPs was negatively charged (ZP = -22.7 ± 1.7 mV).

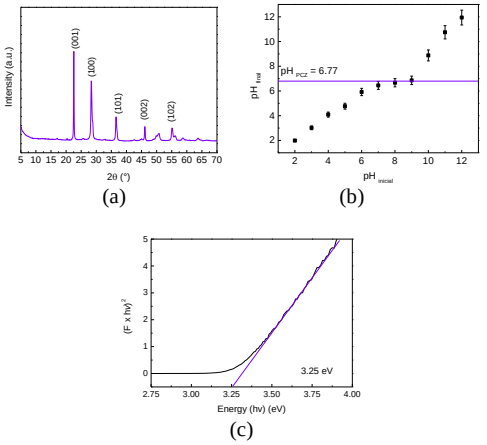

Figure 1. (a) XRD diffractograms; (b) pH_{ZPC} and (c) Tauc plot of Nb_2O_5-NPs.

The antimicrobial activity is represented in Figure 2. The exposure of the plates to UV-vis lamp containing the Nb_2O_5-NPs samples in contact with the *E. coli* was evaluated on a scale of 1000 for times of 0, 30, 60, 90 and 150 minutes after 24 hours plating.

Samples exposed to UV-vis lamp demonstrated a reduction in the number of microorganisms in the initial phase of exposure, indicating that there is an increase in antimicrobial activity within 60 minutes (93.5% reduction in microorganism counts). Samples exposed for a longer time indicated stability in the number of colonies, indicating that within 150 minutes there was an 89.8% reduction in microorganisms, showing a potential antimicrobial activity using UV-vis lamp as a way of photoexcitation of the nanoparticles.

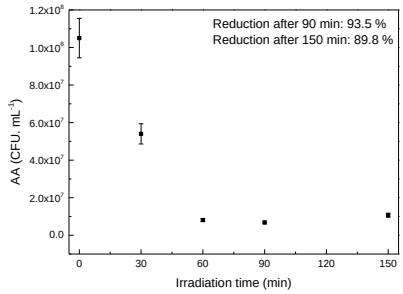

Figure 2. Antimicrobial activity of the nanoparticles on a scale of 1000 for times of 0, 30, 60, 90 and 150 minutes.

Therefore, the green Nb_2O_5-NPs showed potential for application in water treatment through antimicrobial activity. Further studies are needed to evaluate the process variables for optimization of the parameters as well as the other applications of these nanoparticles.

Conclusions

Nb_2O_5-NPs were produced by green synthesis using *C. Illinoiensis* nutshells extract. XRD diffractogram showed that the Nb_2O_5-NPs were crystalline with the presence of pseudohexagonal phase and an average crystallite size of 21.6 nm. The green Nb_2O_5-NPs surface was negatively charged (- 22.7 mV), the zero charge point was 6.77, and the band gap energy was 3.25 eV. Nb_2O_5-NPs showed a potential antimicrobial activity under UV-vis lamp as a way of photoexcitation of the nanoparticles with 93.5 % after 60 minutes time-reduction and 89.8% after 150 minutes time-reduction of *E. coli* count.

Acknowledgments

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References

[1] P.F.O. Cordeiro, Geology Reviews, 41 (2011) 112.
[2] D. Bayot, M. Devillers, Coordination Chemistry Reviews, 250 (2006) 2610.
[3] D. Malyshev, F. Boscá, C-O.L. Crites, G.L. Hallet-Tapley, J.C. Netto-Ferreira, E.I. Alarcon, J.C. Scaiano, Dalton Transactions, 42 (2013), 14049.
[4] L.A. Morais, C. Adán, A.S. Araújo, A.P.M.A. Guedes, J. Marugán, Science & Technology Network, Inc., 2 (2016), 256.
[5] C. C. Silva, M. P. F. Graça, M. A. Valente, A. S. B. Sombra, Journal fof Materials Science, 42 (2007) 3851.
[6] F. I. A. El-Fadl, A. M. Elbarbary, Separation and Purification Technology, 272 (2021), 118972.
[7] R. van Grieken, J. Marugán, C. Sordo, C. Pablos. Catalysis Today, 144 (2009), 48.