

Synthesis, characterization, and bacterial growth inhibition studies of decavanadate associated with organic cations

Juliana M. Missina^{1*}, Kahoana Postal¹, Francielli Santana¹, André L. Rüdiger¹, Emanuel M. de Souza², David L. Hughes³, Giovana G. Nunes¹, Jaísa F. Soares¹

¹Chemistry department, ²Biochemistry and Molecular Biology department, Universidade Federal do Paraná, Curitiba, Brazil. ³School of Chemistry, University of East Anglia, Norwich, UK

*e-mail: missina@globo.com

Decavanadate is an oxoanion made up of ten vanadium(V) centers connected by oxo bridges.¹ It has recently been studied for its antidiabetic, anticancer, antibacterial, among other biological effects. Organic and inorganic cations are used in order to modulate the intended activity of the polyoxoanion $[V_{10}O_{28}]^{6-}$.¹ The objective of the present work was to synthesize decavanadate associated with different organic cations, in order to test the growth inhibition activities of the products against *Escherichia coli* cultures. The decavanadate containing 2-amino-2-hydroxymethyl-propane-1,3-diol (tris, $[(HOCH_2)_3CNH_3]_4[H_2V_{10}O_{28}] \cdot 10H_2O$, **I**) as counterion was prepared in aqueous medium by the addition of the cation solution to a $NaVO_3$ solution (pH = 4).² Product **II** ($((1,3-C_6H_7N_2O)_6[V_{10}O_{28}] \cdot 2H_2O)$, with nicotinamide as counterion, was synthesized by adding an aqueous nicotinamide solution to a V_2O_5 suspension.³ Yellow crystals of **I** and **II** were obtained at 4 °C after layering propan-2-ol onto the reaction mixture. Products were characterized by single-crystal X-ray diffractometry, metal analysis, FTIR, and thermogravimetric analyses (TG), confirming the expected structures. Products were also characterized in aqueous solution by ¹H and ⁵¹V NMR analyses and UV-Vis spectroscopy. ⁵¹V NMR spectrum of **I** showed that the polyoxoanion suffers partial speciation into vanadates of lower nuclearity ($H_2VO_4^-$, $HV_2O_7^{3-}$, $V_4O_{12}^{4-}$, $\delta = -560, -573$ and -578 ppm, respectively). Product **II**, in its turn, remains intact in solution, presenting signals of the protonated oxoanion $[H_3V_{10}O_{28}]^{6-}$ with $\delta = -425, -507$, and -525 ppm, corresponding to the three different vanadium environments. The growth inhibition assays were made by turbidimetry, using *Escherichia coli* DH5 α cultures in LB broth. Compound $[Na_3(H_2O)_9]_{2n}[V_{10}O_{28}]_n$ was used to compare the activity of **I** and **II** to the oxoanion without an organic cation.² Product **I** exhibited growth inhibition of 38% in the highest concentration analyzed (10 mmol L⁻¹), while **II** displayed a more drastic profile, with increasing inhibition from 1 mmol L⁻¹ to 10 mmol L⁻¹ (30 to 79%). These results indicate that contributions to the evaluated activity of the polyoxovanadate depend on the associated counterion; therefore, future studies will involve decavanadate with other organic cations, aiming at correlating their biological activities with commercial antibiotics.

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