Synthesis & Intercalation of Alpha-Zirconium Phosphate Nanoplatelets

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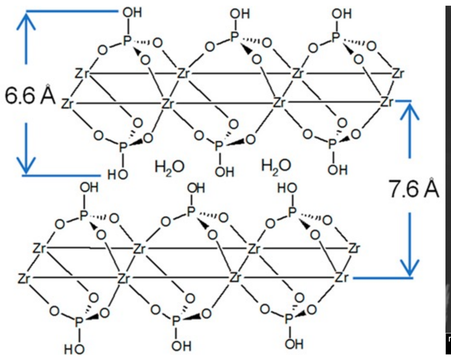
Abstract

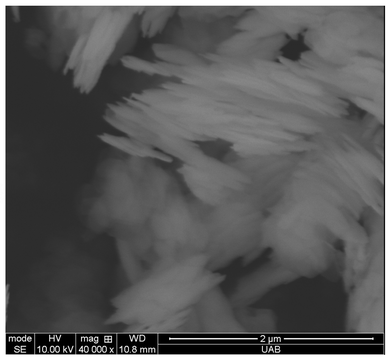
The inorganic ionic material, α-zirconium phosphate (α-ZrP), has been researched to better understand its effectiveness in targeted alpha therapy. The crystalline, layered compound is capable of being a drug-delivery tool due to its ion exchange properties and size. Prior research has shown that the intercalation of various metallic ions into the layers of the material is quick and has high uptake. Additionally, there is minimal leaching of less than 1% present in a human plasma-like environment. Our aim is to intercalate the particles with redox-active materials and investigate if the redox-active material leaches out from the layers. Analysis of the properties of the intercalated α-ZrP was done by characterizing the material with potentiometric titration, thermogravimetric analysis, X-ray photoelectron spectroscopy, UV-Vis spectroscopy, powder X-ray diffraction, scanning electron microscopy imaging, and particle size analysis.

Preparation of α-ZrP

$$ZrOCl_2{\cdot}H_2O + 2H_3PO_4 \xrightarrow{200^oC} Zr(O_3POH)_2{\cdot}H_2O_{(s)} + 8H_2O + 2HCl$$

1.07 g $ZrOCl_2{\cdot}H_2O$ was dissolved in 5 mL of H_2O. 4.11 mL of 4 M H_3PO_4 was added to the solution slowly while constant stirring. This solution was heated for 6 h in a hydrothermal reaction vessel at 200ºC. Once cooled, the product was filtered and washed four times. The α-ZrP was then dried at 65ºC overnight and ground into a fine powder.





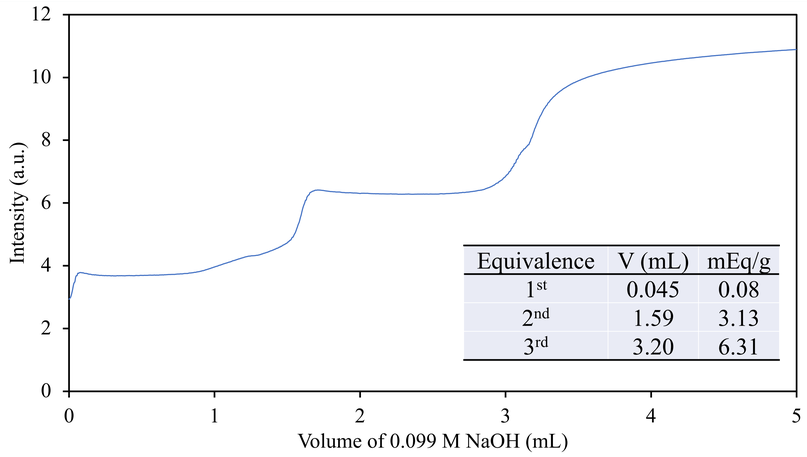
Intercalation of α-ZrP

$$Zr(O_3POH)_2{\cdot}H_2O + Na^+ + {}^-OH \longrightarrow Zr(O_3PONa)_2{\cdot}xH_2O + H_2O$$

$$Zr(O_3PONa)_2{\cdot}xH_2O + M^{2+} \longrightarrow Zr(O_3POM_{0.5})_2{\cdot}xH_2O + 2Na^+$$

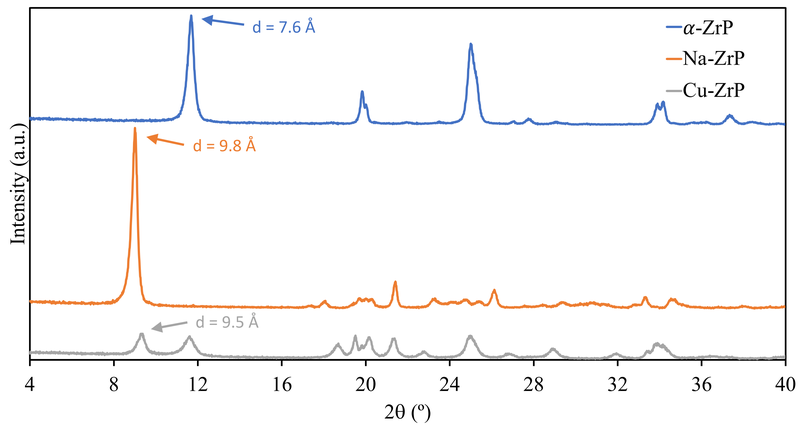
500 mg of α-ZrP was added to 100 mL of H_2O and titrated with 0.1 M NaOH to a pH ≥10.5 to fully convert it to Na-phase (Na-ZrP). The Na-ZrP material was filtered, dried at 20ºC overnight, and ground into a powder. The Na-ZrP was then intercalated with Cu^{2+} (Cu-ZrP) or Co^{2+} (Co-ZrP) by interacting it with a solution of $Cu(NO_3)_2$ or $Co(NO_3)_2$ at a pH of 3.

Ion Exchange Capacity (IEC)

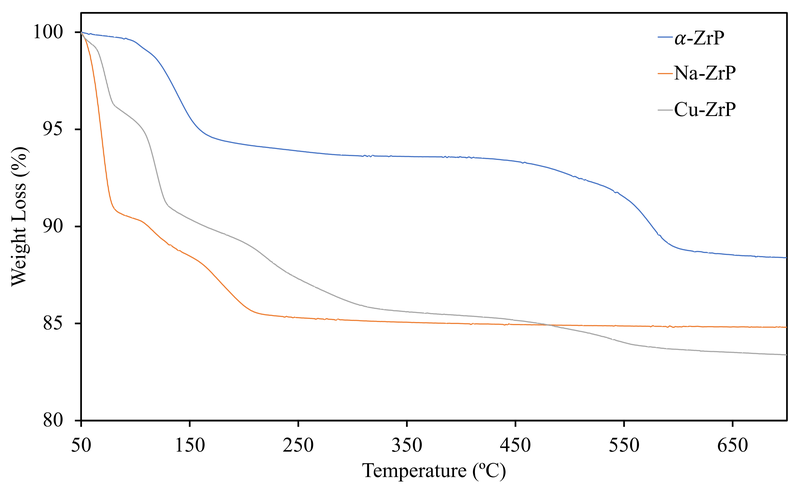


Equivalence	V (mL)	mEq/g
1st	0.045	0.08
2nd	1.59	3.13
3rd	3.20	6.31

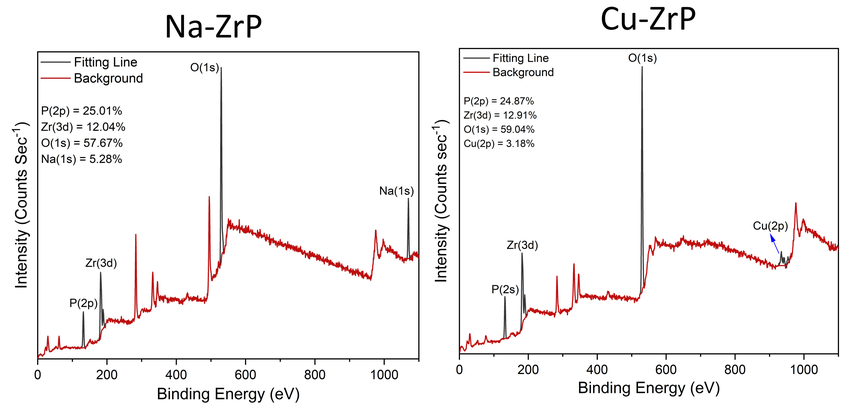
Powder X-ray Diffraction (PXRD



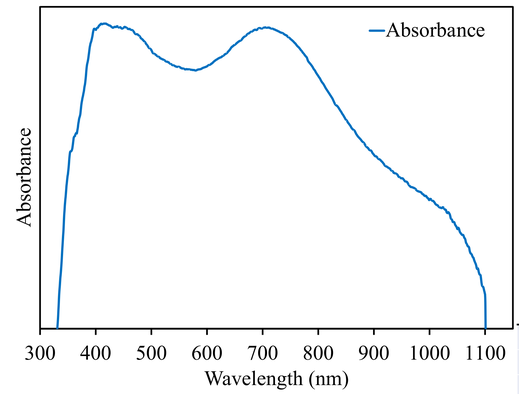
Thermogravimetric Analysis TGA

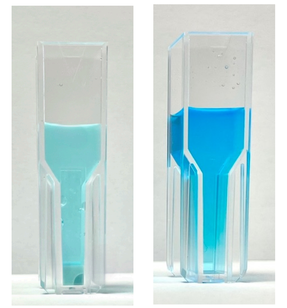


X-ray Photoelectron Spectroscopy (XPS)



Leeching Experiments





Intercalated Cu	0.80 mmol
Cu in Buffer	0.46 mmol
Cu Retained	0.33 mmol

Future Work

- Interact other intercalated particles with reducing and oxidizing agents to observe the leaching properties
- Synthesize α-ZrP using the microwave-assisted technique to check for uptake differences and overall better intercalation
- Conduct leeching experiments in a neutral pH buffer and controlled temperatures

References

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