Chemometric Analysis to Predict the Formation of Interfacial Solids

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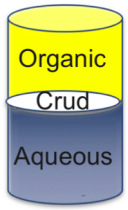
Background

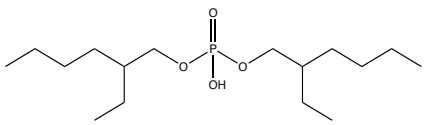
The separation of individual lanthanides at an industrial scale is frequently done using solvent (or liquid-liquid) extraction. A common organic-soluble, metal extractant is bis(2-ethylhexyl)-phosphoric acid (HDEHP)[1]. There are certain aqueous phase conditions, like high pH or metal content, can encourage the formation of interfacial solids (crud). These solids will affect the recovery of additional metal by blocking the interface between the aqueous and organic phase, and have the potential for the density of the solid to cause phase inversion which complicates the recovery of the phases[2]. We are working on determining the spectroscopic signals arising from precursors of interfacial solids formation in the system of using HDEHP to extract neodymium metal (Nd) with Chemometric analysis in an effort to prevent the formation of interfacial solids in an industrial scale system.



Testing Condition

[HDEHP]	[Nd]	Solids?
50mM	10mM	✗
	10.1-50mM	✓
100mM	30mM	✗
	40,50mM	✓

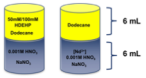
Fig 1. The interfacial solids formation of 50mM [Nd] in both 50mM (left) and 100mM (right) [HDEHP].



Bis(2-ethylhexyl) hydrogen phosphate (HDEHP)

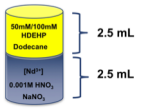
Procedure

Step 1



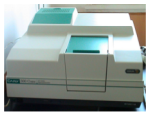
Pre-equilibration :
Saturating the aqueous/organic phase with the component of the other phase except for the metal and extractant.

Step 2



Contact :
Extracting Nd into organic phase.

Step 3



UV-vis Spectroscopy:
Collecting the visible spectra of both aqueous phase and organic before and after contact.

Step 4

Data Analysis(PLS):
Applying Chemometric analysis to the visible spectra taken previously.

Post-Contact Spectra

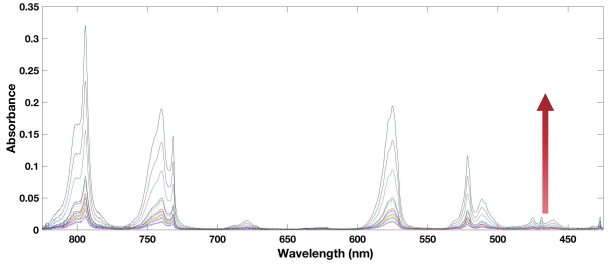


Fig 2. The post-contact aqueous spectra. The absorbance increases with the concentration of Nd.

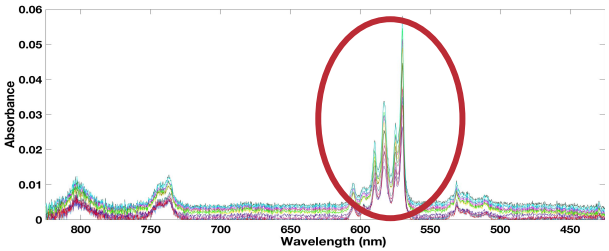


Fig 3. The post-contact organic spectra. The Nd metal complex with HDEHP to form a unique peak.

Result

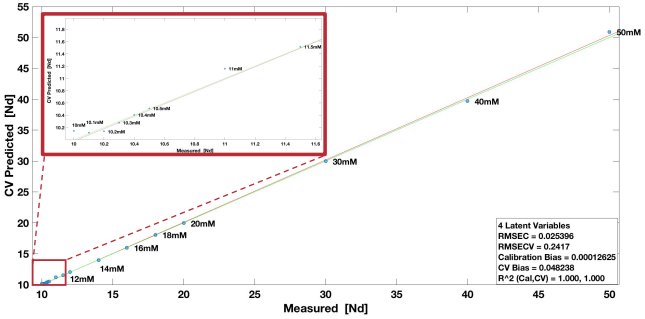


Fig 4. The cross-validation result of the calibration model of aqueous phase.

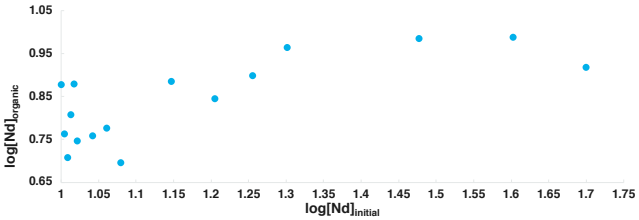


Fig 5. The relationship between the initial aqueous [Nd] and the organic [Nd]. The organic [Nd] is calculated based on the calibration model built from the aqueous spectra before the contact.

Conclusion

The conditions that lead to interfacial solids formation were identified and PLS was applied to build a calibration model based on the aqueous spectra before the contact. The cross-validation results show this model is good for both calibration and prediction. This calibration model was used to predict the concentration of Nd after the contact. This calibration model enabled the relationship between the initial aqueous Nd concentration and the equilibrium organic phase Nd concentration to be determined. As expected, when the initial concentration of Nd increases, the final concentration of Nd should reach a constant value corresponding to a saturation of the organic phase.

Future Work

More samples and variables should be used in the PLS model (eg. pH of the aqueous phase before and after the contact, concentration of HDEHP, etc.) to enable a more complete calibration model for better assessment of interfacial solid precursors. In addition, the spectra of the organic phase after contact should be further examined to find spectral indicators of interfacial solids formation.

Acknowledgement

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Reference

1. Yoshizuka, K., SaKamoto, Y., Baba, Y., Inoue, K., & Nakashio, F. Solvent-Extraction of Holmium and Yttrium with Bis(2-ethylhexyl)phosphoric acid. *INDUSTRIAL & ENGINEERING CHEMISTRY RESEARCH, 31*(5), 1372-1378.

2.Yurtov, E. V., Murashova, N. M., & Datsenko, A. M. Gel formation in extraction of terbium by bis(2-ethylhexyl) hydrogen phosphate. *RUSSIAN JOURNAL OF INORGANIC CHEMISTRY, 51*(4), 670-675.