

Ultrasonic-assisted NH_4VO_3 precipitation from NH_4 -based low-content vanadium solutions

M. Kokko¹, T. Hu¹, U. Lassi¹, J. Pesonen¹

¹University of Oulu, Research Unit of Sustainable Chemistry, Northern Ostrobothnia, Finland

Presenting author email: maria.kokko@oulu.fi

Keywords: Ammonium vanadate precipitation, dilute vanadate solution, impurities' effect, ultrasonic treatment

INTRODUCTION

Vanadium is a critical metal for many industries, including steel and battery industries. It does not occur in concentrated deposits in the earth's crust and therefore its recovery is mainly focused on secondary raw materials or tailings. The steel industry produces a significant amount of solid wastes of which slags can contain variable amounts of vanadium. Vanadium recovery is possible to perform via a roasting-leaching procedure (Li et al., 2016; Wen et al., 2019) or through a completely hydrometallurgical route (Wan et al., 2021). A very common stage after leaching is the purification and/or concentration of the vanadium-containing leachate by liquid extraction (Qin et al., 2020; Yang et al., 2016). Investigations show that vanadium precipitation by ammonium salt from the concentrated solution is viable. The purity of the product can reach high quality due to the purification stage.

A notable part of the research is focused on alkaline or acidic NH_4VO_3 precipitation from vanadium-rich solutions produced by acidic leaching (Muthukumar et al., 2020; Wen et al., 2019). The precipitation studies directly from alkaline ammonium salt leachate have received less attention, although it has been observed to hold significant potential for vanadium recovery (Kokko et al., 2024; Li et al., 2016). To utilize the advantage of ammonium leaching related to direct precipitation, a concentration and purification step after ammonium leaching is not a viable option. However, the precipitation of vanadium as NH_4VO_3 directly from dilute ammonium salt solutions has not been extensively investigated.

To improve the higher utilization potential of industrial solid waste, it is precious to study the precipitation of vanadium directly from ammonium-based dilute vanadium solutions. This research investigates the effects of the factors (pH of the ammonium carbonate solution, vanadium concentration, effect of ultrasonic treatment, and temperature) on the precipitation rate of NH_4VO_3 from the dilute solutions.

MATERIAL AND METHODS

The effects of the factors were studied with synthetic solutions and evaluated with real process waters. Experiment runs at each temperature were designed using MODDE software. Synthetic solutions were prepared by dissolving the desired quantity of extra pure ammonium metavanadate (NH_4VO_3 , Sigma Aldrich, puriss. 99.95%) into the deionized water. The required amount of ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$, Thermo Scientific, USA, extra pure) was added to reach the constant concentration of 200 g/L. The sample volume was 200 ml. The sample was filtered through 1.6 μm filter before pH adjustment which was done by adding nitric acid (HNO_3 , >69%, Honeywell, USA, puriss. p.a.). The sample was treated with an ultrasonic probe () with constant time followed by sufficient agitation time (24 hours) with a paddle mixer. Intermediate samples (2 ml) were taken to verify the progress of precipitation. Formed crystals were separated from the solution by filtering as previously described. NH_4VO_3 was dried in the exicator at room temperature overnight. Crystals were analyzed by a scanning electron microscope- energy dispersive spectrometer (SEM-EDS) and X-ray diffraction (XRD) to observe the crystal shape and analyze the phase composition of the precipitate. Liquid samples were measured by inductively coupled plasma-optical emission spectrometry (ICP-OES).

RESULTS AND DISCUSSION

Based on the preliminary experiments it was observed that NH_4VO_3 precipitation was accelerated by ultrasonic treatment (Figure 1a). Precipitation yield (%) seems to increase along with an increasing concentration (Figure 1b) in ultrasonic-assisted experiments.

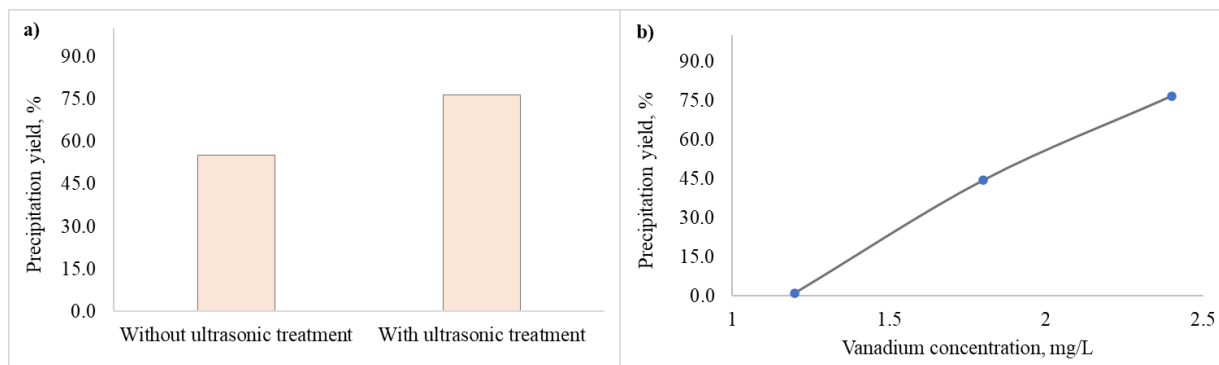


Figure 1. Effects of the ultrasonic treatment (a) and vanadium concentration (b) on the precipitation yield of the NH_4VO_3 (22 ± 2 °C, 24 hours, pH 7.5).

CONCLUSIONS

This study investigated NH_4VO_3 precipitation from $(\text{NH}_4)_2\text{CO}_3$ solution with low vanadium concentrations (1.2-2.4 g/L). The results indicate that NH_4VO_3 can be precipitated from dilute vanadium solutions. The results suggest that it could be possible to recover vanadium from low-vanadium-containing materials by ammonium salt leaching followed by direct NH_4VO_3 precipitation.

REFERENCES

- Kokko, M., Kauppinen, T., Hu, T., Tanskanen, P., Kallio, R., Lassi, U., Pesonen, J., 2024. Two-stage leaching of calcium and vanadium from high-calcium steelmaking slag. *Environ Technol* 1–16. <https://doi.org/10.1080/09593330.2024.2316671>
- Li, H.-Y., Wang, K., Hua, W.-H., Yang, Z., Zhou, W., Xie, B., 2016. Selective leaching of vanadium in calcification-roasted vanadium slag by ammonium carbonate. *Hydrometallurgy* 160, 18–25. <https://doi.org/10.1016/j.hydromet.2015.11.014>
- Muthukumar, K., Patel, K.M., Mohapatra, D., Padh, B., Reddy, B.R., 2020. Selective recovery of vanadium as AMV from calcium vanadate sludge by direct AS leaching process: An industrial approach. *Waste Management* 102, 815–822. <https://doi.org/10.1016/j.wasman.2019.11.040>
- Qin, Z., Zhang, G., Xiong, Y., Luo, D., Li, C., Tang, S., Yue, H., Liang, B., 2020. Recovery of vanadium from leach solutions of vanadium slag using solvent extraction with N235. *Hydrometallurgy* 192, 105259. <https://doi.org/10.1016/j.hydromet.2020.105259>
- Wan, J., Du, H., Gao, F., Wang, S., Gao, M., Liu, B., Zhang, Y., 2021. Direct Leaching of Vanadium from Vanadium-bearing Steel Slag Using NaOH Solutions: A Case Study. *Mineral Processing and Extractive Metallurgy Review* 42, 257–267. <https://doi.org/10.1080/08827508.2020.1762182>
- Wen, J., Jiang, T., Zhou, W., Gao, H., Xue, X., 2019. A cleaner and efficient process for extraction of vanadium from high chromium vanadium slag: Leaching in $(\text{NH}_4)_2\text{SO}_4$ - H_2SO_4 synergistic system and NH_4^+ recycle. *Sep Purif Technol* 216, 126–135. <https://doi.org/https://doi.org/10.1016/j.seppur.2019.01.078>
- Yang, X., Zhang, Y., Bao, S., Shen, C., 2016. Separation and recovery of vanadium from a sulfuric-acid leaching solution of stone coal by solvent extraction using trialkylamine. *Sep Purif Technol* 164, 49–55. <https://doi.org/10.1016/j.seppur.2016.03.021>