

Ionic Liquids Metal Extraction from Regolith

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Summary

Ionic liquids (ILs) are salts that exist as liquids at around room temperature. For this to be achieved, the ions within the ILs are bulky and asymmetrical, which greatly reduces the chances of an ionic lattice forming.

ILs have a range of attractive properties such as:

- High thermal stability
- Highly solvating
- Generally being suited to extreme environments i.e. a wide range of pressure and temperature, enduring the harsh vacuum of space

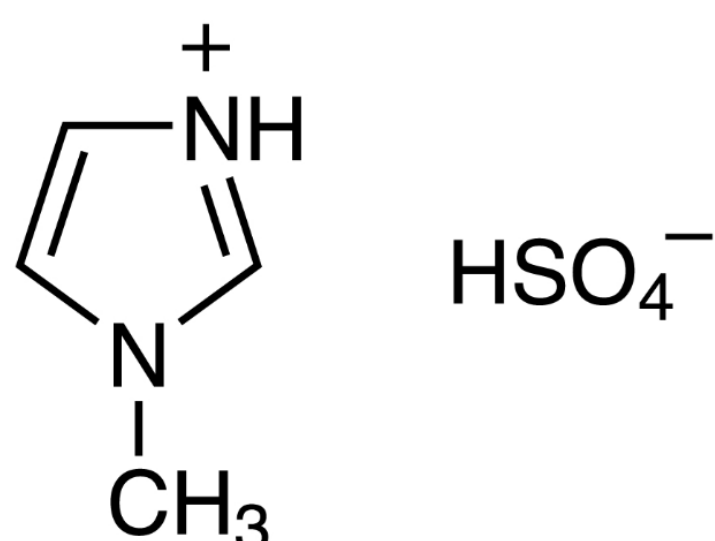
The listed properties make ILs ideal for use in space exploration and more precisely, use in the dissolution of lunar regolith.¹ Regolith is described as a layer of unconsolidated rock, largely comprised of metal oxides, covering the bedrock of a planet. Such material has the potential to provide important resources for in-situ utilisation, like oxygen for breathing air and a range of metals. Using ILs, a process to extract metals from metal oxides found in regolith can begin, providing a useful alternative method in an extra-terrestrial environment.

1. Karr, L.J., Curren, P.A., Thornton, G.S., Depew, K.E., Vankeuren, J.M., Regelman, M., Fox, E.T., Marone, M.J., Donovan, D.N., Paley, M.S., 2018. Ionic liquid facilitated recovery of metals and oxygen from regolith. In: 2018 AIAA SPACE and Astronautics Forum and Exposition. American Institute of Aeronautics and Astronautics Inc, AIAA.

Research aims

- Synthesise 1-methylimidazolium hydrogensulfate
- Assess the use of ILs to extract metal ions

In this research, the IL, 1-methylimidazolium hydrogensulfate was specifically used. This is mainly due to its low costs and easy production method. This research will determine whether this affordable IL can be used to extract metals from their oxides in a setting with limited technology.



Synthesis of 1-methylimidazolium hydrogensulfate

- Measure out the required masses of 1-methylimidazole and concentrated sulfuric acid
- Add the sulfuric acid to a round bottomed flask equipped with a stirring bar
- Place into an ice bath to stir inside of a fume cupboard
- Slowly add the 1-methylimidazole dropwise
- After 1 h remove from the ice bath
- Allow to rise to room temperature as it stirs for a further 48 h



Addition of 1-methylimidazole to conc. sulfuric acid



A vial of 1-methylimidazolium hydrogensulfate

Extraction of metal ions from their oxides

The typical procedure for the dissolution of metal oxides:

- Measure out the required masses of IL, metal oxide and water
- Add the IL to a round bottomed flask equipped with a stirring bar
- Add the solid, insoluble metal oxide into the flask
- Leave the mixture to stir at room temperature for 48 h

M _x O _y	Observation in water	Observation in IL solution
CuO	Black suspension	Blue solution (CuO dissolved)
Fe ₂ O ₃	Brick-red suspension	Brick-red suspension
MnO ₂	Black suspension	Black suspension
ZnO	White suspension	Colourless solution (ZnO dissolved)



CuO in the IL solution compared to in water

Unsuccessful experiments

For the metal oxides that did not dissociate in solution after the initial experiment, further steps in attempt to get positive results were taken. The mixture was heated at reflux, using an oil bath with constant stirring, hoping that this would change the outcome, however this too was unsuccessful. From this it was concluded that for certain metal oxides to go into solution, greater temperatures may be necessary.

Reasoning behind unsuccessful experiments

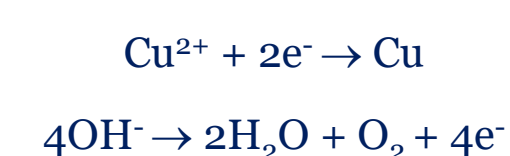
Since ionic compounds were involved in these experiments, it was concluded that lattice enthalpies may be limiting which metals could be extracted. Lattice enthalpies of dissociation for the oxides were calculated using Born-Haber cycles, and it was found that the enthalpy values

Lattice enthalpy of dissociation / kJ mol ⁻¹	
Fe ₂ O ₃	14744
MnO ₂	13115
CuO	4050
ZnO	3971

for the metal oxides from which metals were not extracted were much higher than those of the ones that led to success extraction. Perhaps in order for metal to be extracted from oxides like Fe₂O₃ and MnO₂, a greater energy input is required, one much greater than what can be achieved in a classroom setting.

Electrolysis

Once the IL was used to extract the metal ions from their oxides, electrolysis would be carried out to convert the metal ions to elemental metals by reduction. This is achieved by passing a current through the solution containing the IL, water, and metal ions. The current causes the metal ions to move to the cathode and OH⁻ ions to move to the anode. This was carried out with the Cu²⁺ solution and metal Cu was observed forming at the cathode as well as bubbles forming at the anode, indicating the formation of oxygen gas via oxidation.



Conclusion

- The IL 1-methylimidazolium hydrogensulfate can be used to extract metal ions from their oxides
- The extraction at room temperature and pressure can only extract a limited range of metal ions due to lattice enthalpies of dissociation
- Lattice enthalpies of dissociation can be used to predict which metal oxides can be dissolved by the IL 1-methylimidazolium hydrogensulfate
- For metals less reactive than hydrogen, the extracted metals ions can be reduced using electrolysis.
- There is potential for use of ILs in space exploration where they are used as solvents for the extraction of metals from their oxides in regolith

