

# A Study on the Reduction Behavior of the Ferric Ion From Phosphoric Electrolyte by Hydrazine

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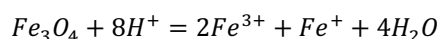
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## 1. Introduction

In the phosphoric acid electrolytic decontamination process, accumulation of metal ions including Fe(2+), Fe(3+), Ni(2+), Cr(3+) takes place in the electrolyte. Regeneration of the electrolyte by removing these metal ions has been carried out in the study. The precipitation of metal ions in the form of ferrous oxalate is the process of removing the metal ions in the waste solution.

On the other hand, electrolytic decontamination of the magnetite formed by the internal of the nuclear reactor generates Fe<sup>3+</sup> and Fe<sup>2+</sup>. The decomposition process of magnetite is as flows.



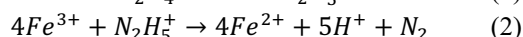
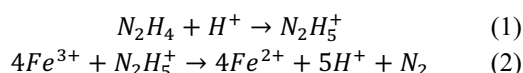
Since Fe<sup>3+</sup> reacts with oxalic acid used in the precipitation process to form chelate, a reduction process using hydrazine is necessary.

In this study, the reduction behavior of hydrazine, which is a typical material for reducing Fe<sup>3+</sup> to Fe<sup>2+</sup> was researched.

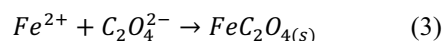
## 2. Experimental details

### 2.1 Experimental theory

The Fe<sup>3+</sup> reduction reaction of hydrazine (N<sub>2</sub>H<sub>4</sub>) is as follows.



The precipitation reaction of reduced Fe<sup>2+</sup> is as follows.



### 2.2 Hydrazine reduction experiment

At first, set the FeCl<sub>3</sub> 0.15 M/L, N<sub>2</sub>H<sub>4</sub>-dehydrate 0.075 M/L (double concentration of a standard capacity). Then, progress reduction reaction 1hr at 25 °C and 90 °C with N<sub>2</sub>H<sub>4</sub>. Cool down the temperature until 25 °C, and add the oxalic acid 0.3 M/L and react the precipitate reaction for 5hr at 25 °C.

The Fe ions were analyzed by ICP-OES after filtering the supernatant and initial solution of the precipitate with 0.2 nm filter. And the components of the sediment were analyzed by FE-EPMA and PSA.

## 3. Results and discussion

In the case of Fe, the removal rate in the case of using hydrazine was 29.12% in RT, and the removal rate was not significantly increased as compared with the case of not using hydrazine. It can be interpreted that hydrazine does not sufficiently reduce Fe<sup>3+</sup> to Fe<sup>2+</sup> at RT. However, when hydrazine was used at 90°C, the removal rate reached almost 80%, indicating that the reduction process using hydrazine

requires a heating process

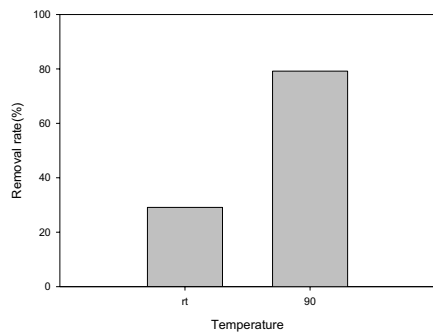


Fig. 1. Fe Removal rate depending on temperature at 4 M/L  $H_3PO_4$ , 0.075 M/L hydrazine.

The analysis of the precipitation is as follows.

Table 1. Result of the precipitation of FE-EPMA

Element	Atom (%)
C	18.2300
O	69.9433
Fe	9.2847
P	2.5421

Since the particles are formed in the phosphoric acid solution, P is the impurity due to this, and the composition for the remaining elements except P is recalculated as follows. C : 18.7055%, O : 71.7676%, Fe : 9.5268%. From this, Fe: C: O = 1: 1.96: 7.5332 ratio can be obtained. It indicate that the precipitate formed  $FeC_2O_4 \cdot 2H_2O$ .

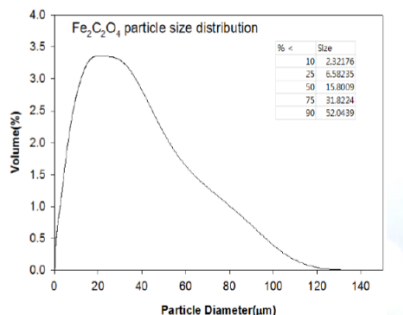


Fig. 2. PSA result of precipitate Fe-Oxalate.

Fig. 2 is the PSA result for sediment. As a result, the Fe oxalate precipitate was more than 25  $\mu m$

above 91.2%, which suggests that the precipitate can be removed by filtration and centrifugation.

#### 4. Conclusion

Through the study on regeneration of phosphate electrolyte decontamination solution, we obtained the following conclusion in the experimental range:

1. The hydrazine reduction rate of ferric ions to ferrous increases with temperature..
2. The phosphate electrolyte decontamination solution can be successfully regenerated by hydrazine reduction and oxalate precipitation

#### ACKNOWLEDGEMENT

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#### REFERENCES

- [1] Andre Angermann et al., "Synthesis of magnetite nanoparticles by thermal decomposition of ferrous oxalate dihydrate", Journal of Materials Science, pp 5123–5130 (2008)