

# Residual Salt Separation of Reaction Products in Pyroprocessing

Sung-Wook Kim,\* Jin-Mok Hur, Dong Hyun Heo, Sun-Seok Hong, and Eun-Young Choi

Korea Atomic Energy Research Institute, 989-111 Daedeok-daero, Yuseong-gu, Daejeon, Korea

\*swkim818@kaeri.re.kr

## 1. Introduction

Molten salts such as LiCl and LiCl-KCl are used as electrolytes of electrochemical processes (i.e., oxide reduction, electrorefining, electrowinning) in pyroprocessing [1]. Hence, the reaction products after each electrochemical process should contain residual salts. Distillation is the common method to remove the residual salts [2]. The distillation process, however, requires a complex apparatus because it is operated in high temperature condition in vacuum environment to ensure sufficient vapor pressure for evaporating the residual salts. In case of the oxide reduction, such high temperature process should be prohibited because the metal products may be reconverted to the original oxide states owing to chemical reaction at the elevated temperature (e.g.,  $U + 2Li_2O = UO_2 + 4Li$ ) [3].

Here, we proposed simple technique using heating gun without any vacuum components to separate the residual salts from the reaction products. Injecting heated gas with sufficiently high flux seems to be promising for the removal of the residual salts.

## 2. Experimental Setup

Fig. 1 shows photograph of the residual salt separation reactor used in this study. Heating gun is the key component of this reactor, which provides two driving forces of the separation: one is heat and the other is momentum. The heating temperature should be higher than the melting point of the target salts. When the heated gas is injected into the reactor, the residual salts is gradually melted and, at the same time, high momentum of the injection gas particles derives the physical separation of the liquid salt from the reaction products. Gravity is also helpful for the separation, but the momentum of the injection gas should be dominant. The reactor was placed on vertical-type furnace installed inside an Ar-filled glove box. Ar gas inside the glove box was used as

the injection gas and off-gas was also exhausted inside the glove box for circulation.

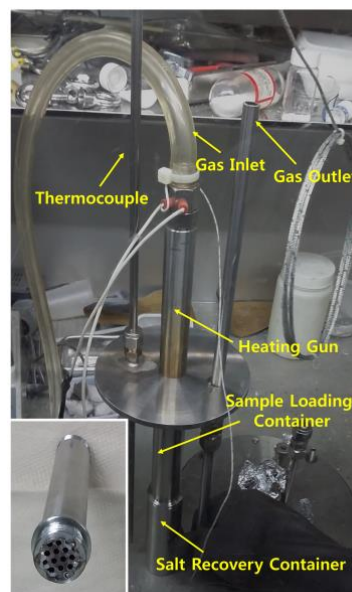


Fig. 1. Configuration of the residual salt separation reactor (inset: sample loading container).

## 3. Results

LiCl, the electrolyte of the oxide reduction process, was chosen as the salt material. 4 pellets of TiO (6.160 g in total) were introduced into molten LiCl at 650°C and then taken out to form the thin salt layer in their surface. Weight of the TiO pellets after the immersion was 6.371 g, indicating that the salt weight is 0.211 g. The TiO pellets became dark as shown in Fig. 2, suggesting that the chemical and/or physical states of the TiO pellets were somehow changed even after the simple immersion. TiO is known to have yellow, brown, or black color depending on its form. The salt-containing TiO pellets were loaded into the reactor. 1.401 g of additional bulk LiCl (total LiCl weight = 1.612 g) was loaded together into the reactor to clearly determine the salt separation capability.

Ar gas, with a flow rate of  $30 \text{ l min}^{-1}$ , was heated to  $700\text{-}720^\circ\text{C}$  inside the heating gun to melt LiCl (melting point:  $\sim 610^\circ\text{C}$ ). The reactor was heated to  $660^\circ\text{C}$  during the experiment to minimize the heat loss of the heated Ar gas. Ideally the reactor temperature needs to be reduced below the melting point to increase the salt recovery efficiency and thus further optimization is required. The heated gas was injected to the reactor for 9 h.

After the injection, the final weight of the TiO pellets was 6.180 g, which is almost identical to the pristine state. No noticeable frozen LiCl existed on the surface of the TiO pellets and the bulk LiCl was completely removed. These indicate that this technique using the heating gun is viable for the effective removal of LiCl. On the other hand, thin white layer was formed on the surface of the TiO pellets as shown in Fig. 2, which would be due to oxidation of TiO to  $\text{TiO}_2$  by residual  $\text{H}_2\text{O}$  and  $\text{O}_2$  in the glove box. Indeed, the reactor was severely oxidized after the reaction. It is worth noting that the process temperature for the residual salt separation can be remarkably lowered compared to the common distillation process (e.g.,  $\sim 800\text{-}900^\circ\text{C}$  for LiCl) [2, 3] using this simple technique.



Fig. 2. (a) Pristine TiO pellets, (b) TiO pellets containing the LiCl salt, and (c) TiO pellets after the salt separation.

#### 4. Conclusion

It was reported that injecting the heated Ar gas with adequate momentum is effective to remove the residual LiCl in the surface of TiO pellets. It is considered that this simple technique can be used in pyroprocessing to separate the residual salts from the reaction products of the electrochemical processes. Scale-up study should be followed for the application in engineering-scale equipment for pyroprocessing.

#### REFERENCES

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