## A Study on crystallization temperature and rate of ammonium salt in the high-pressure hydrogenation heat exchanger

Zhixuan Tang<sup>1</sup>, Wenying Ye<sup>1</sup>, Jinguang Wang<sup>2</sup>, Qiang Wang<sup>3</sup>, Guo-Yan Zhou<sup>1\*</sup>

<sup>1</sup>Key Laboratory of Pressure Systems and Safety, MOE, School of Mechanical and Power Engineering, East China University of Science and Technology, Shanghai 200237, PR China.

<sup>2</sup> Sinopec Engineering Incorporation, Beijing 100101, PR China.

<sup>3</sup> Tianjin Petrochemical Equipment and Instrumentation Research Institute, Tianjin 300451, PR China.

**Abstract**—Ammonium salt crystallization corrosion failure is an essential problem in the failure of highpressure hydrogenation heat exchangers. The crystallization equilibrium curves of NH<sub>4</sub>Cl and NH<sub>4</sub>HS under different pressures were established according to the ammonium salt crystallization process in the highpressure hydrogenation heat exchange unit, and the crystallization equilibrium temperatures of NH<sub>4</sub>Cl and NH<sub>4</sub>HS were obtained based on Aspen Plus. In addition, a prediction model for the crystallization temperature of ammonium salt was established, then the relationship between crystallization temperature and rate of ammonium salts was elaborated. The results indicate that the initial crystallization temperatures of NH<sub>4</sub>Cl and NH<sub>4</sub>HS are respectively 198.95°C and 134.45°C when the system pressure is 10MPa, and the temperature is 433.15K. The maximum crystallization rate of NH<sub>4</sub>Cl and NH<sub>4</sub>HS occur at the initial crystallization temperature, respectively 0.104kg/h and 29.23kg/h. The crystallization rate of NH<sub>4</sub>HS is three orders of magnitude higher than that of NH<sub>4</sub>Cl.

#### 1. Introduction

In petrochemical production, the heat exchanger, air cooler, and pipeline system in the high-pressure hydrogenation heat exchange system often suffer from corrosion failure, which brings enormous safety risks and economic losses 14. Many failure cases show that in the hydrogenation process, the elemental compounds of chlorine, nitrogen, and sulfur in cheap crude oil generate NH<sub>3</sub>, HCl, and H<sub>2</sub>S in the hydrogenation reaction outflow system and then enter the high-pressure heat exchanger, low-pressure heat exchanger, air cooler and other hydrogenation reaction effluent devices. When the temperature drops to a certain critical point, NH<sub>3</sub> in the gas phase will react reversibly with HCl and H<sub>2</sub>S, thus generating NH<sub>4</sub>Cl and NH<sub>4</sub>HS crystallization which causes serious pipe plugging and corrosion problems 58.

To solve this problem, scholars have collected many failure cases and proposed measures such as formulating NH<sub>4</sub>Cl and NH<sub>4</sub>HS content standards in hydrogenation units and using partial pressure of hydrogen sulfide as an indicator to evaluate the corrosiveness of sewage to reduce the risk of corrosion failure 910. Alvisi et al. 11 analyzed the corrosion crystallization samples and found that the crystallization corrosion of NH<sub>4</sub>Cl and NH<sub>4</sub>HS was the leading cause of heat exchanger tube failure. Munson et al. 12 deduced the ammonium salt crystallization equation to predict the crystallization temperature of ammonium salt in the hydrogenation unit. Liu et al. 13 found that the initial crystallization temperature of NH<sub>4</sub>Cl in the highpressure air cooler system is 170°C and the crystallization location is at the outlet of the heat exchanger. Zhu et al. 14studied the influence of injection water and chlorine content on crystallization temperature, and calculated the changing trend of crystallization temperature. Ou et al. 1517 numerically analyzed the flow, temperature and concentration fields of the heat exchanger and concluded that the ammonium salt crystallization and deposition process is a coupling of multiple physical fields. In conclusion, the current research on the crystallization of ammonium salts mainly focuses on the establishment of a simplified thermodynamic model. Many scholars have studied the characteristics of ammonium salt underdeposited corrosion, and elaborated the crystallization principle of ammonium salts18,19.

However, there are few studies on the relationship between the crystallization rate and temperature of ammonium salt, so it is difficult to understand the change of risk in the process of ammonium salt crystallization. To further understand the crystallization formation of ammonium salt in the high-pressure hydrogen heat exchanger, it is necessary to find out the variation rule of the crystallization rate of ammonium salt with temperature to comprehend the risk of ammonium salt crystallization. In this study, Aspen Plus was used to establish the process simulation model of the hydrogenation reaction outflow system to predict the crystallization temperature of NH<sub>4</sub>Cl and NH4HS. In addition, based on the temperature prediction model, the crystallization rates of NH<sub>4</sub>Cl and NH<sub>4</sub>HS are further calculated, and the crystallization risks of the two ammonium salts are compared, which is expected to provide systematic theoretical support for effectively solving the problem of ammonium salt

\*Corresponding author. E-mail address: zhougy@ecust.edu.cn

crystallization failure in the hydrogenation unit.

reaction 20:

# 2. Simulation analysis and verification of ammonium salt crystallization temperature

## 2.1 Thermodynamic calculation of ammonium salt crystallization reaction

The chemical equations of NH<sub>4</sub>Cl and NH<sub>4</sub>HS crystallization reactions are shown in Eqs. (1) and (2).  $NH_3(g)+HCl(g) f NH_4Cl(s)$  (1)  $NH_3(g)+HCl(g) f NH_4Cl(s)$  (2)

Since the reaction equilibrium state is mainly affected by each component's gas phase partial pressure, the theoretical crystallization equilibrium constants  $K_{p1}$  and  $K_{p2}$  of two ammonium salts are defined according to the above chemical reaction equation. The theoretical crystallization equilibrium constants are only a function of temperature, which can be calculated from the thermodynamic data of each substance in the chemical

$$K_{p1} = P_{NH_3} \times P_{HC1}$$
(3)  
$$K_{p2} = P_{NH_3} \times P_{H_2S}$$
(4)

Taking NH<sub>4</sub>Cl as an example, according to the Gibbs function criterion of chemical reaction, when the product of partial pressures of NH3 and HCl in the system is higher than the theoretical crystallization equilibrium constant at the same temperature, NH<sub>3</sub> and HCl in the gas phase can be considered as reacting chemically to form solid NH<sub>4</sub>Cl. The partial pressures of NH<sub>3</sub> and HCl at different temperatures can be obtained through the Aspen Plus process simulation. The intersection point of the two curves is the crystallization temperature of NH<sub>4</sub>Cl in the system. According to the actual operation process of hydrogenation, ten typical working conditions are taken to build the crystallization equilibrium curves of NH4Cl and NH<sub>4</sub>HS, where the system pressure is 2 to 20MPa, and the temperature is 433.15K. According to the chemical reaction equation, the thermodynamic data of each gas phase component in the ammonium salt reaction system were obtained by access to Table 1.

<b>TE 1 1 1 T</b>	- 1		0 1	1	
Table1 1	Thermodynan	ne data	of each	part of the	reaction system

	NH3	HCl		H <sub>2</sub> S	NH4Cl	NH4HS
Pc/MPa	11.280		8.309	8.937	/	/
$T_{\mathcal{C}}/\mathrm{K}$	405.6		324.6	373.2	/	/
${\it \Omega}$	0.25	0.12		0.10	/	/
$\Delta_f H_m^{\Theta}/(\mathrm{KJ/mol})$	-46.11		-92.307	-20.146	-314.43	-156.90
$\Delta_f S_m^{\Theta}/(\mathrm{KJ/mol}\cdot\mathrm{K})$	0.1925		0.1869	0.2058	0.0946	0.1134

Taking the working condition with a system pressure of 10MPa and temperature of 433.15K as an example, the comparison temperature  $(T_r)$  and pressure  $(P_r)$  according to the thermodynamic data in Table 1 need be calculated first. The next step is looking up the generalized fugacity coefficient table to get the fugacity coefficients of each component when it exists alone. Then according to Lewis Randall's theory, the fugacity coefficients of each component in the mixed component  $(\varphi_i)$  can be obtained. When Gibbs function is equal to 0, the chemical reaction reaches equilibrium, which can be expressed as follows:

$$\Delta_{\rm r}G_{\rm m} = \Delta_{\rm r}G_{\rm m}^{\Theta} + RT\ln\prod_{\rm i}\left(\frac{p_{\rm i}\times\phi_{\rm i}}{p^0}\right)^{\rm v} = 0 \quad (5)$$

Where  $\Delta_r G_m^{\Theta}$  is the standard Gibbs function of chemical reaction at 25°C and 100KPa, and the expression is as follows:

$$\Delta_{\rm r}G_{\rm m}^{\Theta} = \Delta_{\rm r}H_{\rm m}^{\Theta} - T\Delta_{\rm r}S_{\rm m}^{\Theta} = \sum \Delta_{\rm f}H_{\rm m}^{\Theta} - T\sum \Delta_{\rm f}S_{\rm m}^{\Theta} \tag{6}$$

According to the above principle, the relationship between the temperature of NH<sub>4</sub>Cl and NH<sub>4</sub>HS and the partial pressure of each component can be obtained as follows:

 $-176.013 + 0.284758T - RT \ln(aK_{p1}) = 0$  (7)

$$-90.644 + 0.2848T - RT \ln(bK_{n^2}) = 0 \qquad (8)$$

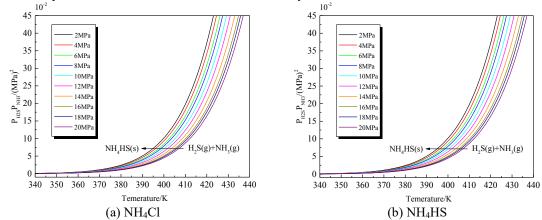
R is the Gas constant which is  $8.314 \text{ J/(mol}\cdot\text{K})$ . *a* and *b* are constants dependent on system pressure and temperature and are taken as 0.604 and 0.643, respectively, when the system pressure is 10MPa and the temperature 433.15K.

The image was drawn in Origin, and the crystallization equilibrium curves of NH<sub>4</sub>Cl and NH<sub>4</sub>HS under ten different pressures were obtained, as shown in Fig.1. It can be seen that when the temperature is high in the hydrogenation process, the ammonium salt will not deposit and crystallization. When the temperature drops to a certain critical value, the product of the gas phase partial pressure in the component is greater than the corresponding  $K_{p1}$  and  $K_{p2}$ , and the ammonium salt starts to crystal. Compared with NH<sub>4</sub>HS, the  $K_p$  of NH<sub>4</sub>Cl changes more sharply, and the initial crystallization temperature of NH<sub>4</sub>Cl is significantly higher than that of

higher with the increased system pressure. Therefore, the

system pressure should be appropriately reduced in the hydrogenation process to prevent ammonium salt

NH<sub>4</sub>HS, indicating that NH<sub>4</sub>Cl crystallizes first when the reaction effluent enters the cooling separation system. When the gas components in the system are the same, the crystallization temperature of NH<sub>4</sub>Cl and NH<sub>4</sub>HS will be

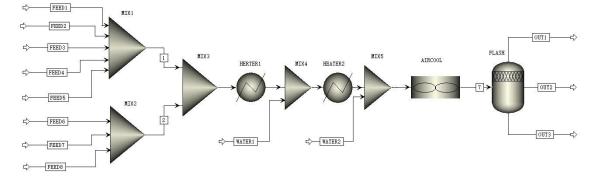


crystallization.

Fig.1 The crystallization balance curves of NH<sub>4</sub>Cl and NH<sub>4</sub>HS in 10 different pressures

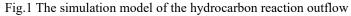
## 2.2 Establishment of the simulation model of ammonium salt crystallization temperature

The feedstock of a hydrogenation unit in a petrochemical refinery is a mixture of processed coking gasoline, coking diesel oil, and catalytic diesel oil. The hydrogen-mixed crude oil is sent from the central tank farm to the hydrotreating reactor through the hydrogenation feed pump in the tank far. Then complex reactions such as hydrodesulfurization, denitrification, deoxidation, olefin saturation, and aromatic ring opening saturation are carried out under the action of the catalyst. The reaction products are cooled to 40°C through the heat exchanger, air cooler, and water cooler in turn, and finally enter the low-pressure and high-pressure separators for oil-gaswater three-phase separation 21. According to the principle of material conservation, combined with the process flow of the high-pressure hydrogenation system, Aspen Plus is used to model and simulate a petrochemical refinery's hydrogenation reaction outflow system under typical working conditions. The simulation model of the high-pressure hydrogenation multiphase equilibrium system is shown in Fig.2.



FEED1-Circulating hydrogen, FEED2-Dry gas, FEED3-Low separation gas, FEED4-Diesel, FEED5-Naphtha, FEED6-NH<sub>3</sub>, FEED7-HCl, FEED8-H<sub>2</sub>S, OUT1-Gas phase, OUT2-Oil phase,

OUT3-Water phase



The gas component, the gas-liquid flow rate of each component feed component, and the distillation data of each oil are sho

component in the gasoline and diesel hydrogenation unit are shown in Tables 2-4.

			Т	able 2 Di	stillation c	lata			
	Distillation range/°C								
Project	0%	5%	10%	30%	50%	70%	90%	95%	100%
Diesel	198	201	207	218	236	245	262	269	273
Naphtha	86	95	98	106	115	128	146	154	161

Table 3 Gas phase composition (%)									
Name		$H_2$	$C_1$	$C_2$	C <sub>3</sub>	i-C4	n-C <sub>4</sub>	i-C <sub>5</sub>	n-C <sub>5</sub>
Circulating hydrogen		78.32	10.49	5.88	3.01	1.23	0.60	0.40	0.07
Low separation gas		67.51	18.80	5.62	3.47	2.67	0.94	0.87	0.12
Dry gas		20.60	20.24	22.14	20.21	12.95	3.04	0.53	0.27
Table 4 Flow rate of feed components									
Name	Diesel (t/h)	Naphtha (t/h)	Circulating hydrogen (t/h)		Dry gas (t/h)	Low separation gas (t/h)	NH3 (ug/g)	HCl (ug/g)	H <sub>2</sub> S (ug/g)
Flow rate	265.7	86.7	449	93.5	25.6	2.3	1000	10	6000

According to the established high-pressure hydrogenation reaction outflow system, the components and the physical parameters in the system's oil, gas, and water phases can be obtained by simulation analysis. The molar fractions of NH<sub>3</sub>, HCl, and H<sub>2</sub>S components in the gas phase are multiplied by the system pressure to get the molar partial pressure of these three components, and then the  $K_p$  of NH<sub>4</sub>Cl and NH<sub>4</sub>HS gas phase are calculated.  $K_p$  of different temperatures can be obtained by setting the

independent temperature variable in the sensitivity analysis part in Aspen Plus, then the crystallization temperature of NH<sub>4</sub>Cl and NH<sub>4</sub>HS in the high-pressure hydrogenation reaction outflow system can be obtained. The above calculation mainly adopts the Peng-Robinson equation. As shown in Fig.3, NH<sub>4</sub>Cl starts to crystallize when the temperature drops to 198.95°C in the hydrogenation cooling separation system, and NH<sub>4</sub>HS starts to crystallize when the temperature reaches 134.45°C.  $10^2$ 

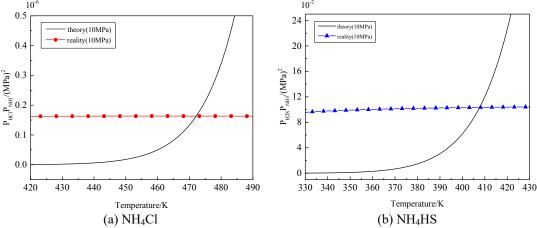


Fig.3 Ammonium salt crystallization temperature of hydrogenation reaction outflow system

#### 2.3 Result verification

To verify the feasibility of the calculation method, a crystallization sample was collected from the highpressure hydrogenation heat exchange system of the petrochemical refinery. The system flow chart is shown in Fig.4. The inlet temperature and outlet temperature of heat exchanger E102 are 250°C and 180°C. The inlet temperature and outlet temperature of heat exchanger E103 are 180°C and 60°C. When Sinopec shut down for maintenance, it checked the failure of the high-pressure hydrogenation heat exchanger. No apparent white deposits were found in the heat exchangers E101A, E101B, E101C, and E101D, while white deposits were discovered in E102, E103, and the air cooler. The analysis results show that the white deposit of heat exchanger E102 is mainly NH<sub>4</sub>Cl crystallization, and the white deposit of heat exchanger E103 is mainly NH<sub>4</sub>HS. Thus, the simulation results can be considered reliable.

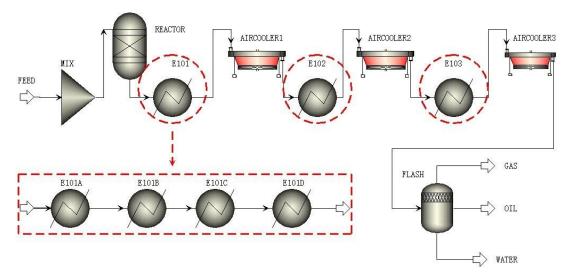


Fig.4 High-pressure hydrogenation heat exchanger system process

#### 3. Prediction model of ammonium salt crystallization rate

#### 3.1 NH<sub>4</sub>Cl crystallization rate prediction model

It can be seen from Eq. (1) that during the cooling and separation process of a high-pressure hydrogenation system, the formation of the NH<sub>4</sub>Cl solid phase is a continuous reversible equilibrium process. After separated cooling through the air cooler, if the thermodynamic temperature in the system decreases to the critical point of NH<sub>4</sub>Cl crystallization temperature (point A in Fig.5), NH<sub>4</sub>Cl ammonium salt crystallization will be generated in the NH<sub>3</sub> and HCl gas phases. At the same time, the equilibrium constant in the gas phase will change and quickly return to the position of crystal equilibrium, and

the system will return to equilibrium again. With the reseparation and cooling of the air cooler, the temperature decreases again. Under the new state of NH<sub>4</sub>Cl, the critical point of crystallization temperature is reached again, and NH<sub>4</sub>Cl ammonium salt crystallization is generated. The above process will be repeated until the temperature of the high-pressure hydrogenation system reaches the minimum. When the temperature decreases each time tends to infinity, the reaction time also tends to infinity. The crystallization rate of NH<sub>4</sub>C1 in this state can be approximated to the mass of NH<sub>4</sub>Cl ammonium salt crystallization generated from the point of supersaturation (point A in Fig.5) to the point of equilibrium (point B in Fig.5). According to the mass conservation law of the chemical reaction in Eq. (1) the mass of NH<sub>4</sub>Cl ammonium salt crystallization equals the mass of NH<sub>3</sub> and HCl reduced in the gas phase. According to the above principle, the expression of the amount of NH<sub>4</sub>Cl can be obtained.

$$\Delta n = \left[ P_{\text{NH}_3}^{\text{A}} + P_{\text{HCl}}^{\text{A}} - \sqrt{(P_{\text{NH}_3}^{\text{A}} + P_{\text{HCl}}^{\text{A}})^2 - 4(P_{\text{NH}_3}^{\text{A}} \times P_{\text{HCl}}^{\text{A}} - K_p^{\text{B}})} \right] \times \frac{n_{\text{total}}}{2P_{\text{tot}}}$$
(9)  
According to the amount of substance and molar mass. salt.

According to the amount of substance and molar mass, the mass of NH<sub>3</sub> and HCl at any point can be obtained, which is also the crystallization rate of NH4Cl ammonium

$$c_{\rm NH_4Cl} = \left[ P_{\rm NH_3} + P_{\rm HCl} - \sqrt{(P_{\rm NH_3} + P_{\rm HCl})^2 - 4(P_{\rm NH_3} \times P_{\rm HCl} - K_{\rm p})} \right] \times \frac{n_{\rm total} M_{\rm NH_4Cl}}{2P_{\rm total}}$$
(10)

 $K_p$  of NH<sub>4</sub>Cl at 10MPa can be obtained from Eq. (17).  $-176.013 + 0.284758T - RT \ln(0.604K_p) = 0$ 

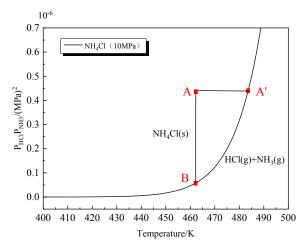


Fig.5 The calculation method of NH<sub>4</sub>Cl crystallization rate

According to the calculation method of the NH<sub>4</sub>Cl crystallization rate, the curve of the NH<sub>4</sub>Cl crystallization rate is calculated as shown in Fig.6. It can be seen that when the temperature drops to 198.95°C (crystallization temperature of NH<sub>4</sub>Cl), NH4Cl crystallization starts to occur in the heat exchanger, and the crystallization rate reaches the maximum, which is 0.104kg/h. As the

temperature continues to decrease, the crystallization rate of NH<sub>4</sub>Cl gradually decreases. When the temperature drops to about 140°C, the crystallization rate of NH<sub>4</sub>Cl is 0. Within the range of NH<sub>4</sub>Cl crystallization temperature, the crystallization rate of NH<sub>4</sub>Cl changes nonlinearly with thermodynamic temperature.

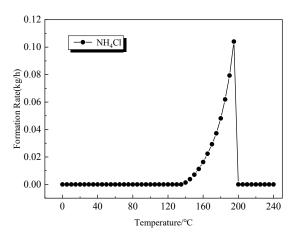


Fig.6 The formation rate of NH<sub>4</sub>Cl crystallization

The data with temperature below  $198.95^{\circ}$ C simulated above are fitted nonlinearly in Origin, and the fitting relationships between the formation rate of NH<sub>4</sub>Cl crystallization and crystallization temperature are obtained as follows. The correlation coefficient is 0.99817, and the fitting accuracy is reasonable.

$$y=7.11064 \times 10^{-24} T^{9.64847}$$
(12)

#### 3.2 NH<sub>4</sub>HS crystallization rate prediction model

Similarly, the calculation formula of the NH<sub>4</sub>HS crystallization rate is obtained.

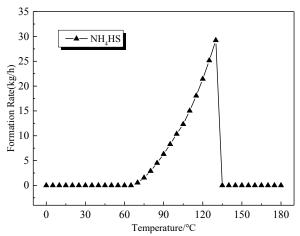
$$c_{\rm NH_4HS} = \left[P_{\rm NH_3} + P_{\rm H_2S} - \sqrt{(P_{\rm NH_3} + P_{\rm H_2S})^2 - 4(P_{\rm NH_3} \times P_{\rm H_2S} - K_{\rm p})}\right] \times \frac{n_{\rm total} M_{\rm NH_4HS}}{2P_{\rm total}}$$
(13)

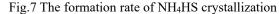
 $K_p$  of NH<sub>4</sub>HS at 10MPa can be obtained from Eq. (20).

$$-90.644 + 0.2848T - RT \ln(0.643K_{p}) = 0$$
<sup>(14)</sup>

According to the calculation method of the NH<sub>4</sub>HS crystallization rate, the curve of the NH<sub>4</sub>HS crystallization rate is shown in Fig.7. When the temperature drops to 134.45°C (the crystallization temperature of NH<sub>4</sub>HS), NH<sub>4</sub>HS crystallization starts to occur in the heat exchanger and the formation rate reaches the maximum of 29.23kg/h.

As the temperature continues to decrease, the crystallization rate of NH<sub>4</sub>HS gradually decreases. When the temperature drops to about 70°C, the crystallization rate of NH<sub>4</sub>HS is 0. Similarly, within the range of NH<sub>4</sub>HS crystallization temperature, the formation rate of NH<sub>4</sub>HS changes nonlinearly with thermodynamic temperature.





The data with temperature below 130°C simulated above are fitted nonlinearly in Origin, and the fitting relationships between the formation rate of NH<sub>4</sub>HS crystallization and crystallization temperature are obtained as follows. The correlation coefficient is 0.99451, and the fitting accuracy is reasonable.

 $y=7.11064 \times 10^{-24} T^{9.64847}$ (15)

Compared with Fig.6 and Fig.7, the crystallization rate of NH<sub>4</sub>HS is three orders of magnitude higher than that of NH<sub>4</sub>Cl, indicating that the crystallization formation amount of NH<sub>4</sub>HS is much more than that of NH<sub>4</sub>Cl in the high-pressure hydrogenation heat exchanger at the same time. As a result, even though the initial crystallization temperature of NH<sub>4</sub>HS in the hydrogenation reaction outflow system is lower than that of NH<sub>4</sub>Cl, the risk of NH<sub>4</sub>HS deposition and blockage is much higher than that of NH<sub>4</sub>Cl. In the high-pressure hydrogenation heat exchanger, the risk of NH<sub>4</sub>HS crystallization must be prevented first.

#### 4. Conclusions

In the present work, the crystallization temperature and rate of NH<sub>4</sub>Cl and NH<sub>4</sub>HS in high-pressure hydrogenation heat exchangers are studied. The main conclusions are as follows.

It is found that the crystallization temperature of NH<sub>4</sub>Cl in the hydrogenation reaction outflow system is significantly higher than that of NH<sub>4</sub>HS. The crystallization temperature corresponding to NH<sub>4</sub>Cl and NH<sub>4</sub>HS at 10MPa and 433.15K is respectively 198.95°C and 134.45°C.

Crystallization samples in the high-pressure hydrogen heat exchange system of the petrochemical refinery were collected. The test results are in agreement with the simulation results that crystallization of  $NH_4Cl$  first occurred and then of  $NH_4HS$ .

The crystallization rate of NH<sub>4</sub>Cl and NH<sub>4</sub>HS shows a nonlinear trend with the thermodynamic temperature. The maximum crystallization rate of NH<sub>4</sub>Cl is 0.104kg/h when the temperature is  $198.95^{\circ}$ C, and the maximum crystallization rate of NH<sub>4</sub>HS is 29.23kg/h when the temperature is  $134.45^{\circ}$ C. The crystallization rate of

NH<sub>4</sub>HS is three orders of magnitude higher than that of NH<sub>4</sub>Cl. Therefore, NH<sub>4</sub>HS crystallization should be paid more attention to.

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