

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

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Abstract—Ammonium salt crystallization corrosion failure is an essential problem in the failure of high-pressure hydrogenation heat exchangers. The crystallization equilibrium curves of NH_4Cl and NH_4HS under different pressures were established according to the ammonium salt crystallization process in the high-pressure hydrogenation heat exchange unit, and the crystallization equilibrium temperatures of NH_4Cl and NH_4HS 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_4Cl and NH_4HS 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_4Cl and NH_4HS occur at the initial crystallization temperature, respectively 0.104kg/h and 29.23kg/h. The crystallization rate of NH_4HS is three orders of magnitude higher than that of NH_4Cl .

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_3 , HCl , and H_2S 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_3 in the gas phase will react reversibly with HCl and H_2S , thus generating NH_4Cl and NH_4HS 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_4Cl and NH_4HS 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_4Cl and NH_4HS 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_4Cl in the high-pressure air cooler system is 170°C and the crystallization

location is at the outlet of the heat exchanger. Zhu et al. [14] studied the influence of injection water and chlorine content on crystallization temperature, and calculated the changing trend of crystallization temperature. Ou et al. [15] 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 under-deposited corrosion, and elaborated the crystallization principle of ammonium salts [18,19].

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_4Cl and NH_4HS . In addition, based on the temperature prediction model, the crystallization rates of NH_4Cl and NH_4HS 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

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crystallization failure in the hydrogenation unit.

2. Simulation analysis and verification of ammonium salt crystallization temperature

2.1 Thermodynamic calculation of ammonium salt crystallization reaction

The chemical equations of NH_4Cl and NH_4HS crystallization reactions are shown in Eqs. (1) and (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

reaction 20:

$$K_{p1} = P_{\text{NH}_3} \times P_{\text{HCl}} \quad (3)$$

$$K_{p2} = P_{\text{NH}_3} \times P_{\text{H}_2\text{S}} \quad (4)$$

Taking NH_4Cl as an example, according to the Gibbs function criterion of chemical reaction, when the product of partial pressures of NH_3 and HCl in the system is higher than the theoretical crystallization equilibrium constant at the same temperature, NH_3 and HCl in the gas phase can be considered as reacting chemically to form solid NH_4Cl . The partial pressures of NH_3 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_4Cl in the system. According to the actual operation process of hydrogenation, ten typical working conditions are taken to build the crystallization equilibrium curves of NH_4Cl and NH_4HS , 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.

Table1 Thermodynamic data of each part of the reaction system

	NH_3	HCl	H_2S	NH_4Cl	NH_4HS
P_c/MPa	11.280	8.309	8.937	/	/
T_c/K	405.6	324.6	373.2	/	/
Ω	0.25	0.12	0.10	/	/
$\Delta_f H_m^\ominus/(\text{KJ}/\text{mol})$	-46.11	-92.307	-20.146	-314.43	-156.90
$\Delta_f S_m^\ominus/(\text{KJ}/\text{mol}\cdot\text{K})$	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 (φ_i) can be obtained. When Gibbs function is equal to 0, the chemical reaction reaches equilibrium, which can be expressed as follows:

$$\Delta_r G_m = \Delta_r G_m^\ominus + RT \ln \prod_i \left(\frac{P_i \times \varphi_i}{P_0} \right)^{\nu_i} = 0 \quad (5)$$

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

$$\Delta_r G_m^\ominus = \Delta_r H_m^\ominus - T \Delta_r S_m^\ominus = \sum \Delta_f H_m^\ominus - T \sum \Delta_f S_m^\ominus \quad (6)$$

According to the above principle, the relationship between the temperature of NH_4Cl and NH_4HS and the partial pressure of each component can be obtained as

follows:

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

$$-90.644 + 0.2848T - RT \ln(bK_{p2}) = 0 \quad (8)$$

R is the Gas constant which is 8.314 J/(mol·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_4Cl and NH_4HS 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_4HS , the K_p of NH_4Cl changes more sharply, and the initial crystallization temperature of NH_4Cl is significantly higher than that of

NH₄HS, indicating that NH₄Cl 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 NH₄Cl and NH₄HS will be

higher with the increased system pressure. Therefore, the system pressure should be appropriately reduced in the hydrogenation process to prevent ammonium salt crystallization.

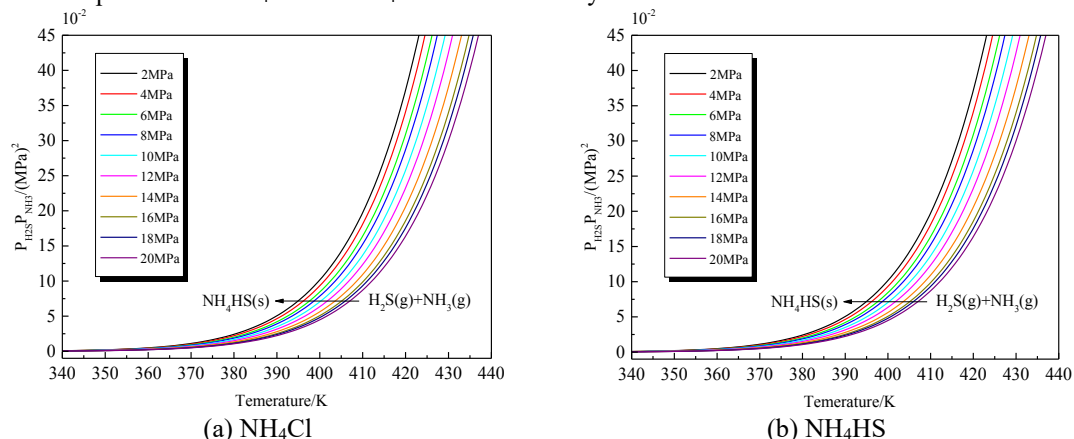
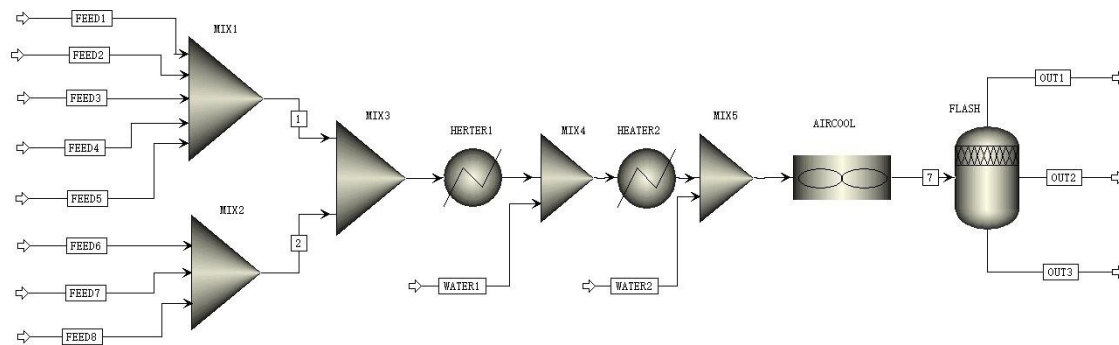


Fig.1 The crystallization balance curves of NH₄Cl and NH₄HS in 10 different pressures

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, denitrication, 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-gas-water 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-NH₃, FEED7-HCl, FEED8-H₂S, OUT1-Gas phase, OUT2-Oil phase, OUT3-Water phase

Fig.1 The simulation model of the hydrocarbon reaction outflow

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

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

Table 2 Distillation data

Project	Distillation range/°C								
	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 ₃	i-C ₄	n-C ₄	i-C ₅	n-C ₅
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)	NH ₃ (ug/g)	HCl (ug/g)	H ₂ S (ug/g)
Flow rate	265.7	86.7	4493.5	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 NH₃, HCl, and H₂S 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 NH₄Cl and NH₄HS 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 NH₄Cl and NH₄HS 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, NH₄Cl starts to crystallize when the temperature drops to 198.95°C in the hydrogenation cooling separation system, and NH₄HS starts to crystallize when the temperature reaches 134.45°C.

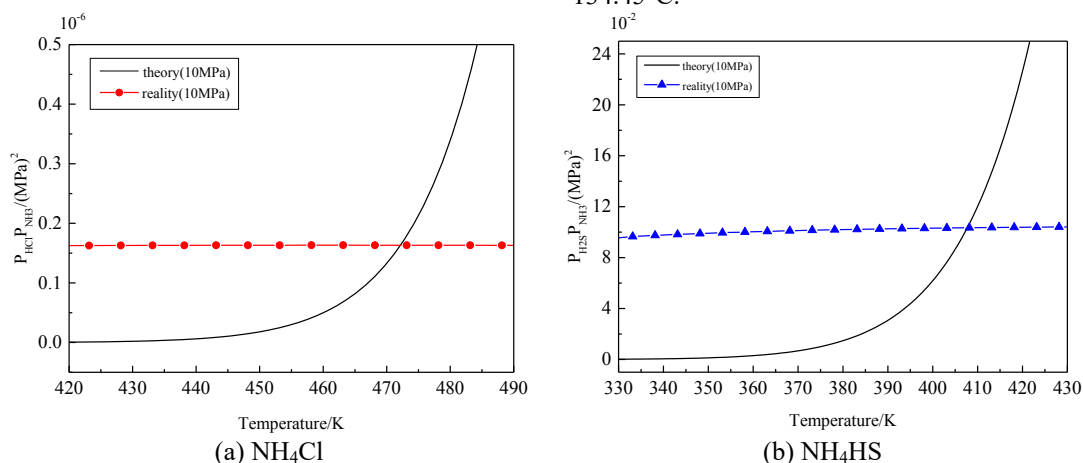


Fig.3 Ammonium salt crystallization temperature of hydrogenation reaction outflow system

2.3 Result verification

To verify the feasibility of the calculation method, a crystallization sample was collected from the high-pressure 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 NH₄Cl crystallization, and the white deposit of heat exchanger E103 is mainly NH₄HS. Thus, the simulation results can be considered reliable.

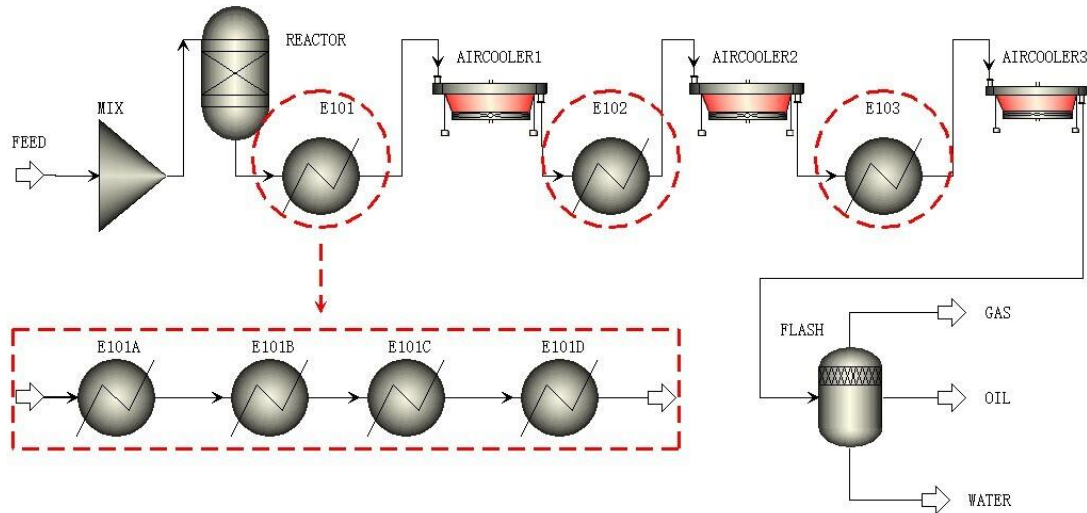


Fig.4 High-pressure hydrogenation heat exchanger system process

3. Prediction model of ammonium salt crystallization rate

3.1 NH₄Cl 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 NH₄Cl 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 NH₄Cl crystallization temperature (point A in Fig.5), NH₄Cl ammonium salt crystallization will be generated in the NH₃ 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 re-separation and cooling of the air cooler, the temperature decreases again. Under the new state of NH₄Cl, the critical point of crystallization temperature is reached again, and NH₄Cl 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 NH₄Cl in this state can be approximated to the mass of NH₄Cl 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 NH₄Cl ammonium salt crystallization equals the mass of NH₃ and HCl reduced in the gas phase. According to the above principle, the expression of the amount of NH₄Cl can be obtained.

$$\Delta n = \left[P_{\text{NH}_3}^A + P_{\text{HCl}}^A - \sqrt{(P_{\text{NH}_3}^A + P_{\text{HCl}}^A)^2 - 4(P_{\text{NH}_3}^A \times P_{\text{HCl}}^A - K_p^B)} \right] \times \frac{n_{\text{total}}}{2P_{\text{tot}}} \quad (9)$$

According to the amount of substance and molar mass, the mass of NH₃ and HCl at any point can be obtained, which is also the crystallization rate of NH₄Cl ammonium

salt.

$$c_{\text{NH}_4\text{Cl}} = \left[P_{\text{NH}_3} + P_{\text{HCl}} - \sqrt{(P_{\text{NH}_3} + P_{\text{HCl}})^2 - 4(P_{\text{NH}_3} \times P_{\text{HCl}} - K_p)} \right] \times \frac{n_{\text{total}} M_{\text{NH}_4\text{Cl}}}{2P_{\text{total}}} \quad (10)$$

K_p of NH₄Cl at 10MPa can be obtained from Eq. (17).

$$-176.013 + 0.284758T - RT \ln(0.604K_p) = 0 \quad (11)$$

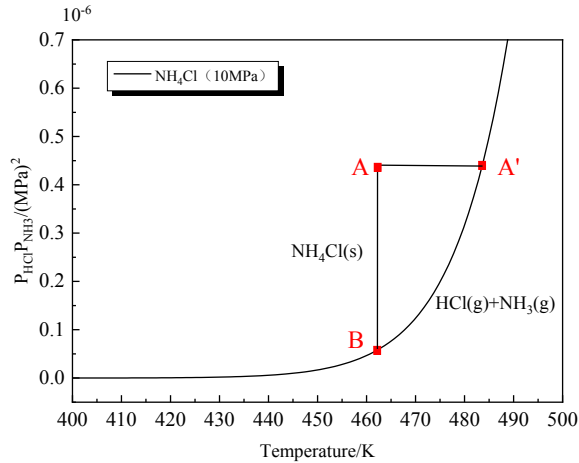


Fig.5 The calculation method of NH₄Cl crystallization rate

According to the calculation method of the NH₄Cl crystallization rate, the curve of the NH₄Cl 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 NH₄Cl), 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 NH₄Cl gradually decreases. When the temperature drops to about 140°C, the crystallization rate of NH₄Cl is 0. Within the range of NH₄Cl crystallization temperature, the crystallization rate of NH₄Cl changes nonlinearly with thermodynamic temperature.

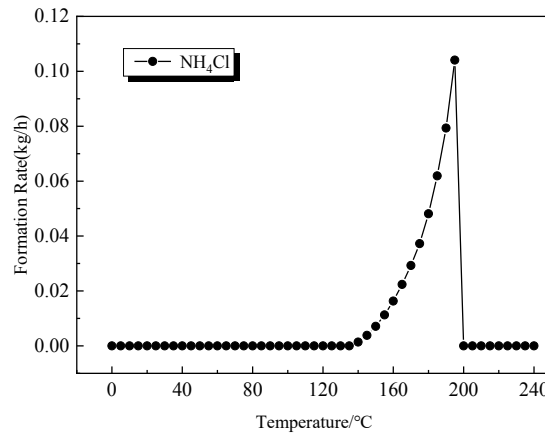


Fig.6 The formation rate of NH₄Cl crystallization

The data with temperature below 198.95°C simulated above are fitted nonlinearly in Origin, and the fitting relationships between the formation rate of NH₄Cl crystallization and crystallization temperature are obtained as follows. The correlation coefficient is 0.99817, and the fitting accuracy is reasonable.

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

3.2 NH₄HS crystallization rate prediction model

Similarly, the calculation formula of the NH₄HS crystallization rate is obtained.

$$c_{\text{NH}_4\text{HS}} = \left[P_{\text{NH}_3} + P_{\text{H}_2\text{S}} - \sqrt{(P_{\text{NH}_3} + P_{\text{H}_2\text{S}})^2 - 4(P_{\text{NH}_3} \times P_{\text{H}_2\text{S}} - K_p)} \right] \times \frac{n_{\text{total}} M_{\text{NH}_4\text{HS}}}{2P_{\text{total}}} \quad (13)$$

K_p of NH₄HS at 10MPa can be obtained from Eq. (20).

$$-90.644 + 0.2848T - RT \ln(0.643K_p) = 0 \quad (14)$$

According to the calculation method of the NH₄HS crystallization rate, the curve of the NH₄HS crystallization rate is shown in Fig.7. When the temperature drops to 134.45°C (the crystallization temperature of NH₄HS), NH₄HS 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 NH₄HS gradually decreases. When the temperature drops to about 70°C, the crystallization rate of NH₄HS is 0. Similarly, within the range of NH₄HS crystallization temperature, the formation rate of NH₄HS changes nonlinearly with thermodynamic temperature.

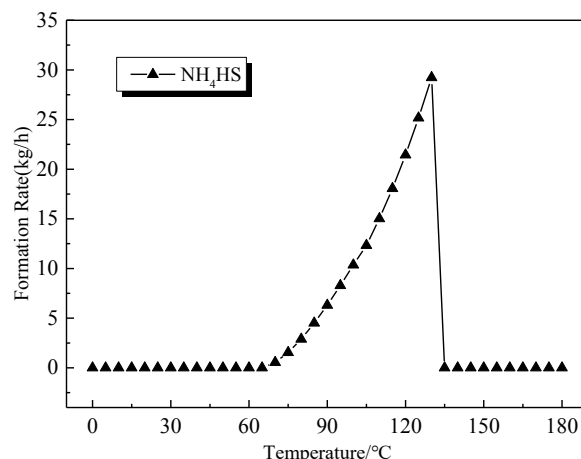


Fig.7 The formation rate of NH₄HS crystallization

The data with temperature below 130°C simulated above are fitted nonlinearly in Origin, and the fitting relationships between the formation rate of NH₄HS 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 NH₄HS is three orders of magnitude higher than that of NH₄Cl, indicating that the crystallization formation amount of NH₄HS is much more than that of NH₄Cl in the high-pressure hydrogenation heat exchanger at the same time. As a result, even though the initial crystallization temperature of NH₄HS in the hydrogenation reaction outflow system is lower than that of NH₄Cl, the risk of NH₄HS deposition and blockage is much higher than that of NH₄Cl. In the high-pressure hydrogenation heat exchanger, the risk of NH₄HS crystallization must be prevented first.

4. Conclusions

In the present work, the crystallization temperature and rate of NH₄Cl and NH₄HS in high-pressure hydrogenation heat exchangers are studied. The main conclusions are as follows.

It is found that the crystallization temperature of NH₄Cl in the hydrogenation reaction outflow system is significantly higher than that of NH₄HS. The crystallization temperature corresponding to NH₄Cl and NH₄HS 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 NH₄Cl first occurred and then of NH₄HS.

The crystallization rate of NH₄Cl and NH₄HS shows a nonlinear trend with the thermodynamic temperature. The maximum crystallization rate of NH₄Cl is 0.104kg/h when the temperature is 198.95°C, and the maximum crystallization rate of NH₄HS is 29.23kg/h when the temperature is 134.45°C. The crystallization rate of

NH₄HS is three orders of magnitude higher than that of NH₄Cl. Therefore, NH₄HS crystallization should be paid more attention to.

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