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Study on salt effect of NaCl in cyanuric acid wastewater treatment

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ABSTRACT

The pollution of cyanuric acid-contaminated saline wastewater has been a major problem in industrial wastewater treatment in the production of trichloroisocyanuric acid. In this study, the solubility of cyanuric acid in different concentrations of NaCl solution was studied via data fitting and salt effect theory, and a mathematical model was established to analyze the salt effect of NaCl on cyanuric acid wastewater and to predict the solubility of cyanuric acid in different NaCl solution at even lower temperatures. After the experimental verification, the deviation between the data fitting results and the experimental results were analyzed. The optimum salting-out effect of NaCl on cyanuric acid was selected and the fitting equation was modified. It was found that the optimum precipitation point of cyanuric acid is that NaCl was added to the mass concentration of 25%, and the solution temperature was adjusted to 0°C, which caused the concentration of cyanuric acid in the simulated wastewater solution was dropped from 1400 mg/L to 744 mg/L.

Keywords: Salting-out; Cyanuric acid wastewater; Data-fitting; Separation; Setschenow

1. Introduction

Cyanuric acid has acquired a great importance as intermediates in the production of different chemical products, and it is commonly used in the manufacturing of trichloroisocyanuric acid, which is used in swimming pools as a chlorine stabilizer [1–6]. During the industrial production of trichloroisocyanuric acid. High-salt content organic wastewater has always been a big problem in water treatment [7–9]. The wastewater, which is produced during the production of trichloroisocyanuric acid, is difficult to biodegrade due to its high salt content [10–14]. The high-concentration organic matters in the wastewater can also severely contaminate membranes and filtration equipment during the membrane filtration process and cause the membrane treatment difficult to carry out [15–17]. Thus, the high-salt contained cyanuric acid wastewater is considered to be a type of industrial refractory wastewater. But considering the purity of the salt content (mainly 99% of NaCl) in cyanuric acid wastewater, by removing cyanuric acid which is the only organic matter in the wastewater, the remaining saline water can be used as raw material for the chlor-alkali industry.

In general, a high valence salt (such as Na_2CO_3, Na_2SO_4) has a stronger salting-out effect on cyanuric acid in a water solution than that of a monovalent salt (such as NaF, NaCl). A high valence salt, due to its higher ionic strength, is often more capable of snatching solvent water from cyanuric acid. However, the contents in the industrial cyanuric acid wastewater are mainly cyanuric acid and NaCl, therefore, the choose of a higher valence salt as a salting-out agent can cause a secondary contamination, which leads to the remaining salt solution to become non-recyclable.

In this study, the precipitation effect, caused by adding NaCl in simulated cyanuric acid wastewater, was studied by data fitting method and experimental verification, which would provide an effective and feasible treatment for the high-salt content cyanuric acid wastewater.

The addition of electrolytic solute to a non-electrolytic solution changes the activity coefficient of the non-electrolytic solute. This phenomenon is called salt effect [18–20]. After the addition of salt, if the solubility of non-electrolyte increases, the phenomenon is called salting-in and if the

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solubility of non-electrolyte decreases, the phenomenon is called salting-out. To summarize the phenomenon of salt effect, Setschenow [21–25] puts forward the empirical equation describing salt effect:

$$\lg(S^0 / S) = C_S K \tag{1}$$

where S^0 is the non-electrolyte's solubility in pure water, S is the non-electrolyte's solubility in a salt solution of concentration C_S, and K is the salt effect coefficient, which depends largely on ionic strength of the salt which was added in the solution. If K > 0, $S^0 > S$, then the salting-out effect occurs. If $K < 0$, $S^0 > S$, then the salting-in effect occurs. Eq. (1) is suitable for use in a large salt concentration range. The study of salting-out effect in the literature has mainly focused on the low salt concentration range [26–30], which could hardly be applied to cyanuric acid wastewater treatment.

In this study, the solubility of cyanuric acid in NaCl solution, with different NaCl content under different temperatures, was studied via data fitting of existing cyanuric acid solubility data with extrapolation and salting-out effect theoretical analysis.

2. Materials and method

2.1. Data fitting

According to the experimental results of Zhang and Liu [31], the solubility data of cyanuric acid in different concentrations of NaCl solution at different temperature were analyzed via data fitting as follows:

As seen in Fig.1, the solubility of cyanuric acid in water decreases with the increase of NaCl concentration, that is, NaCl plays a role as a salting-out agent for cyanuric acid. Due to the empirical trend of the given data and the features of the solubility of the solute, that is the solubility never goes to 0 (which means solute is insoluble) and the solubility changing was continue with the temperature changing, an exponential function was chosen to be the data fitting equation for the solubility data of the cyanuric acid under different temperatures and salinities, as follows:

$$S = ae^{bT} \tag{2}$$

where S is the solubility of cyanuric acid in the solution at any given NaCl concentration, where a and b are empirical constants. T is the solution temperature. In Table 1, the coefficient of determination R^2 is more than 0.99, which means the solubility of cyanuric acid was in agreement with the data fitting model. Thus, the fitting equation from the graph above is used for further calculations of the solubility of cyanuric acid at even lower temperatures. From the simulation data, the solubility of cyanuric acid in 25% NaCl solution at –20°C was the lowest. Considering the solidification of the solution at lower temperatures, further estimation is not intended. The fitted curve suggests that at 25% NaCl and –20°C the solubility of cyanuric acid is estimated to be 102 mg/L.

2.2. Theoretical analysis of salt effect

The fitting curve is based on the regression of the solubility data of cyanuric acid. The Setchenow equation is then used for theoretical analysis of the salt effect in the cyanuric acid-NaCl solution, and it can be used to test the applicability of the fitted equation. Since the solubility of NaCl in water seldom changes within the temperature range from 30°C to –20°C, therefore, the ionic strength of the NaCl solution is relatively constant.

The salt effect constant K in Eq. (1) is related only to the ionic strength of the salt ions in the solution, thus in temperature range from 30°C to –20°C, K is only related to the concentration of NaCl in a particular solution. Thus, in Eq. (1) K is set to a fixed value for a NaCl solution with a certain NaCl concentration, which does not change with temperature. At a certain NaCl solution, K is calculated from the solubility data of cyanuric acid at 30°C and applied to other temperatures with same NaCl content. $K = \frac{\lg S^o / S}{C}$, e.g. $K = \frac{\lg 2.143 / 1.7858}{64} = 0.00124$. The salt effect constant in the presence of 6% NaCl is calculated to be 0.00124. Sim-

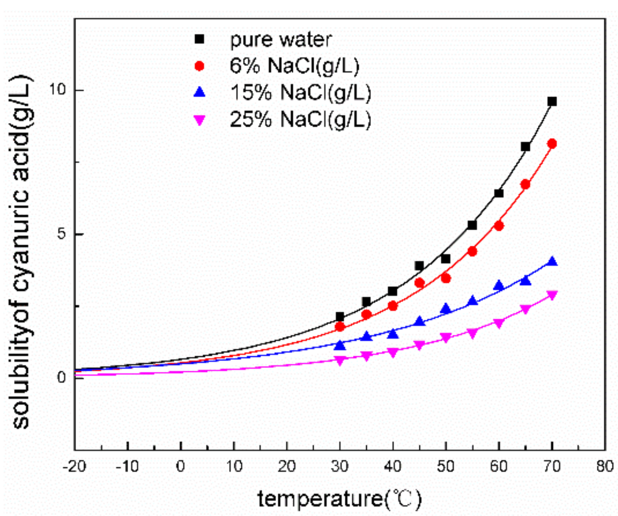

Fig. 1. Solubility of cyanuric acid at different NaCl concentrations.

Table 1
Parameters of data fitting equation

Equation:	y = a*exp(b*x)	R^2:	0.99576
Equation parameters		Value	Standard error
0% NaCl(g/L)	a	0.65706	0.03428
0% NaCl(g/L)	b	0.03827	8.47E-04
6% NaCl(g/L)	a	0.53565	0.03354
6% NaCl(g/L)	b	0.03874	0.00102
15% NaCl(g/L)	a	0.4988	0.05003
15% NaCl(g/L)	b	0.03001	0.00167
25% NaCl(g/L)	a	0.21539	0.0361
25% NaCl(g/L)	b	0.03715	0.00273

ilarly, the salt effect constants in the presence of 15% NaCl and 25% NaCl are 0.00162 and 0.00155, respectively. Then these three calculated salt effect constant *K* at different NaCl content are applied in Eq. (1) for further calculations, and the results are presented in Fig. 2, which compares with actual NaCl data, as follows:

It can be seen from Fig. 2, the calculated results of Eq. (1) are in agreement with the literature data. Thus, the above assumptions about the salting-out constant, were in consistent with the salt effect theory, and were also in consistent with the actual solubility data of cyanuric acid's solubility in NaCl solution with different NaCl content. Therefore, for the cyanuric acid-NaCl-water system, fitting Eq. (2) is consistent with the salt effect theory and conforms to the literature data.

The solubility of cyanuric acid at different NaCl concentrations from 30°C to 70°C are in agreement with Setchenow equation. However, the dissolution process of cyanuric acid is not always the same as the precipitation process. Therefore, in the salting-out experiment, we do not expect the experimental results to be the exact same to the regressed results from the data fitting. Instead we use the data fitting equation as a theoretical guide, which leads to the point where we can get the best precipitation effect of cyanuric acid in water solution.

2.3. Materials and experiments

2.3.1. Materials

The reagents used in the experiments were provided by Beijing Weiss Chemicals without further purification. All chemical reagents were of analytical grade, and distilled water were used during the process of experiments.

2.3.2. Experiments

Through the investigation of the production of trichloroisocyanuric acid, the cyanuric acid content in the

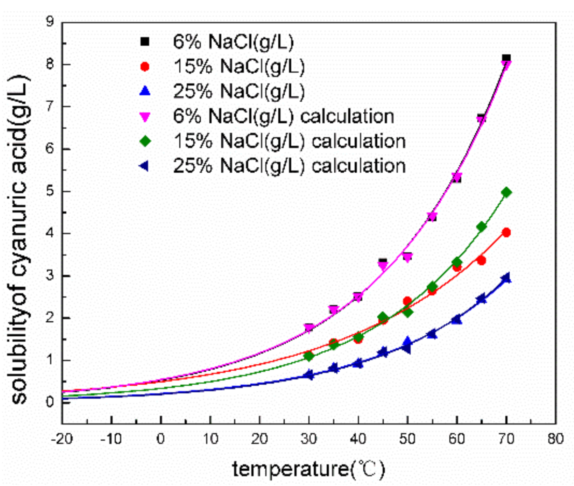

Fig. 2. Comparisons between calculated solubility data of cyanuric acid at different NaCl content to that of the actual data from the literature.

industrial cyanuric acid wastewater was found to be around 1400 mg/L. In this study, an electronic analytical scale, a temperature-adjusted electronic stirrer, seven100 mL beakers, seven 50 mL volumetric flasks and cyanuric acid solid were used to precisely prepare seven 50 mL, 1400 mg/L cyanuric acid simulate solution samples. One simulate solution sample was under room temperature (25°C). After the addition of NaCl to 25%, stirring until NaCl was fully dissolved, then stop stirring. The sample was placed under room temperature for 30 min. Later, a small amount of crystal particles was merged at the surface of the solution.

After the crystals were filtered off, titration method [32,33] was used to test the remaining cyanuric acid content in the solution. First, 0.1 mol/L HCl solution was titrated to the simulate solution by an acid burette, and the PH of the simulate solution was controlled to 4.7 by a pH meter. At this pH, all the cyanuric acid dissolved in the simulate solution turned into cyanuric acid molecules. Second, 2~3 drops of phenolphthalein reagent were added into the simulate solution, and 0.1 mol/L NaOH solution was then titrated into the simulate solution until the color was changed into light pink(pH 8.5). At this PH, all the cyanuric acid in the solution was changed into cyanuric acid monosodium salt. The amount of substance of NaOH added to the solution, from pH 4.7 to 8.5, was the same as the amount of substance of cyanuric acid dissolved in the solution. Therefore, the cyanuric acid concentration in the solution was calculated by the following equation:

$$\omega = \frac{C_{NaOH} V_{NaOH}}{50} M \times 1000 \quad (3)$$

In the above equation, C_{NaOH} is the concentration of NaOH, mol/L; V_{NaOH} is the volume of the NaOH solution that is added into the simulate solution, mL; M is the molar mass of cyanuric acid, g/mol; ω is the mass concentration of cyanuric acid in the sample, mg/L.

The concentration of cyanuric acid simulate solution sample was reduced from 1400 mg/L to 980 mg/L. Thus, adding NaCl into the simulate cyanuric acid wastewater led to cyanuric acid precipitation, that is, there was a salting-out effect of NaCl on cyanuric acid in water solution.

Afterward, seven experiments with the same simulate cyanuric acid wastewater solution samples were conducted by the above mentioned method, in which, NaCl was added to 25%, however, the solution temperature was adjusted differently. After the experiments, the content of cyanuric acid in the solution was analyzed and listed as follows:

From the first four groups at 25°C, 15°C, 5°C and 0°C, the results showed that it was in accordance with the assumption as the trend of data fitting followed, which suggested that cyanuric acid was emerged after NaCl addition and the precipitation effect became stronger by the decrease of the experimental temperature. However, with the experimental temperature dropped below 0°C, the precipitation effect was not in accordance with the temperature anymore. From the last three groups at –5°C, –10°C and –18°C, the results showed that the precipitation effect of cyanuric acid was weakened with the decrease of temperature, which was opposite to the assumption and the results of data fitting.

3. Results and discussions

It can be seen from Table 2 that the experimental results are in agreement with the salting-out theory and the trend of data fitting in the temperature range from 25°C to 0°C. It suggests that the above assumptions about the nature of NaCl solution are in consistent with the experimental results within the temperature range from 25°C to 0°C. But when the temperature goes below 0°C, the experimental results are opposite to the data fitting results. That is, with the decrease of temperature, the precipitation effect weakened. The reasons of this phenomenon are listed as follows:

First, under low temperature conditions, although the solution was not solidified, its viscosity increased. Cyanuric acid particles are dispersed evenly in the viscous gel-like solution, and the increased viscosity impeded precipitation. This phenomenon is illustrated in Fig. 3.

In a beaker, a salted out cyanuric acid particle could be modeled by a homogeneous pellet with same weight and size. The process of a small spherical object precipitate in a stationary viscous fluid environment, and the particle's force analysis during the process would be suitable for a stokes' law model. Assuming the precipitated cyanuric acid to be a spherical particle which is rising steadily in the 25% NaCl solution. The particle is subjected to an excess force F_p due to the difference between the buoyancy and weight of the spherical particle, which is given by:

Table 2
Remaining cyanuric acid content in simulate wastewater samples after precipitation

Temperature (°C)	Cyanuric acid content (mg/L)
25	980
15	893
5	838
0	774
–5	820
–10	923
–18	1100

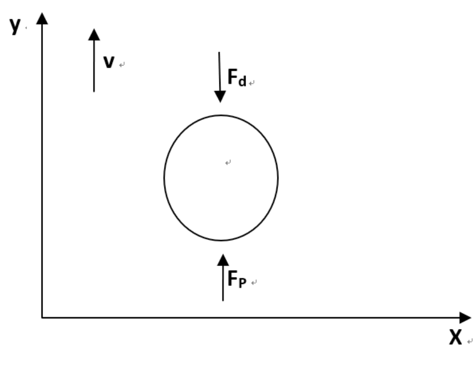

Fig. 3. Free body diagram of cyanuric acid particle.

$$F_P = (\rho_f - \rho_P) g \frac{4}{3} \pi R^3 \quad (4)$$

where F_p is the excess force from the buoyancy and weight of the spherical particle, ρ_f and ρ_p are the densities of the fluid and particle, respectively, and g is the gravitational acceleration. The particle is also subjected to a viscous force F_d opposite to the direction of its movement during the precipitation process, which is illustrated by the equation, as follows:

$$F_d = 6\pi\eta R v \quad (5)$$

where F_d is the viscous force, η is the viscosity of the liquid phase, R is the radius of the particle (which equivalent to a spherical object of same volume), ν is the velocity of the particle relative to the liquid phase. The viscous force to the particle increases with the decrease of the temperature of the liquid phase. When $F_d > F_p$, the cyanuric acid particle's precipitation process begins to slowing down, until cyanuric acid particles suspended in the NaCl solution. When the temperature of the NaCl solution decreases below 0°C, the viscosity of the liquid becomes so large that the cyanuric acid particles which have been salted out cannot precipitate and rise to the surface. Instead, most of cyanuric acid particles dispersed at the NaCl solution evenly without actual precipitation.

Second, as the temperature drops further below 0°C, the nature of the salt solution changed greatly. At low temperature, part of NaCl molecules are no longer ionized into Na^+ and Cl^- ions, but became NaCl $2H_2O$ molecules which dissolved in the water. Thus, at low temperature the ion strength of NaCl solution drops significantly, so that NaCl as a salting-out reagent, losts part of its ability to attract water molecules, and it has caused salting-out effect weakening.

These two phenomena above could not have been shown in the data fitting equation, so the original date fitting equation becomes invalid at temperature range below 0°C. Therefore, when the salt effect analysis of NaCl-non-electrolytes system is carried out below 0°C, the data fitting Eq. (2) should be modified properly.

It can be seen from Fig. 4 that after salting-out effect, from temperature range 0°C to 25°C by adding NaCl to 25%, the remaining cyanuric acid content in the solution still in accordance with the data fitting Eq. (2) .

$$S = ae^{bT} \quad (6)$$

When the temperature ranges from 0°C to –18°C, the equation Eq. (2) is no longer in consistent with experimental data, then a different equation is needed. According to the data from 0°C to –18°C, a new data fitting equation can be given by:

$$S = 0.00143T^3 + 0.441T^2 - 10.6T + 774 \quad (7)$$

where S is the concentration of cyanuric acid in the solution after adding NaCl to 25% at the temperature range from 0°C to –18°C. Where, T is the experimental temperature. Eq. (6) is based on the actual experimental data, so it has certain practical reference value.

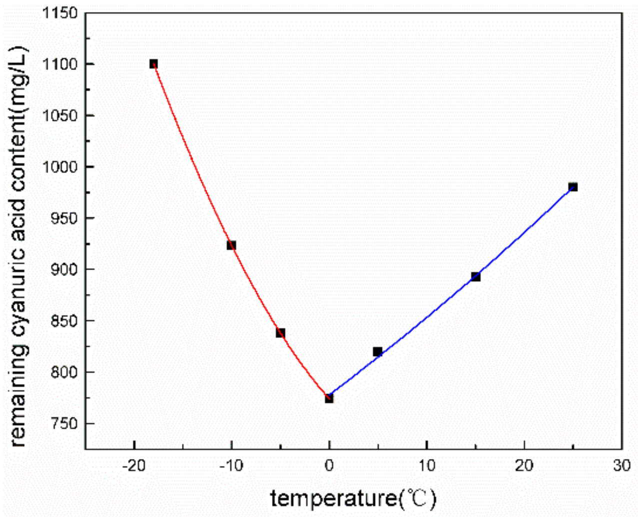

Fig. 4. Modified salt effect data fitting curve.

Table 3
Parameters of salting-out data fitting equation Eq. (2) of cyanuric acid simulate solution by adding NaCl to 25% from temperature range 0°C to 25°C

Equation:	y = a*exp(b*x)	R^2	0.99731
Equation parameters		Value	Standard error
25% NaCl（g/L）	a	778	3.48016
25% NaCl（g/L）	b	0.00925	2.75765E-04

4. Conclusion

In this study, the application of salt effect in cyanuric acid wastewater treatment was studied by data fitting and experimental verification. The best result of salting-out effect on cyanuric acid, with NaCl as a salting-out reagent, was predicted by data fitting. After experimental verification, the results were analyzed and corrected by the salt effect theory. It is found that, when NaCl is added to 25% and the solution temperature is dropped to 0°C, the best condition for cyanuric acid precipitation is reached. Under the best condition, the cyanuric acid content decreased significantly from 1400 mg/L to 774 mg/L. The remaining solution with condensed NaCl content met the requirement of raw material for chlor-alkali industry, which can be recycled.

Based on the experimental results, the original equation was modified as follows:

$$\begin{cases} S = 778e^{0.00925T} & 0 < T \leq 25 \\ S = 0.00143T^3 + 0.441T^2 - 10.6T + 774 & -18 \leq T \leq 0 \end{cases} \quad (8)$$

Eq. (7) can be used to calculate the remaining concentration of cyanuric acid in industrial cyanuric acid wastewater at the temperature from –18°C to 25°C, when NaCl is added to 25% accurately.

Symbols

S^0 — Non-electrolyte solubility in pure water, g/L
S — Non-electrolyte solubility in a salt solution, g/L
C_S — Salt concentration, g/L
K — Salt effect coefficient, l
T — Temperature, °C
C_{NaOH} — Concentration of NaOH, mol/L
V_{NaOH} — Volume of the NaOH solution, mL
M — Molar mass of cyanuric acid, g/mol
ω — Mass concentration of cyanuric acid in the sample, mg/L
F_P — Excess force from the buoyancy and weight of the spherical particle, N
F_d — Viscous force, N
ρ_f — Density of the fluid, g/cm^3
ρ_P — Density of the particle, g/cm^3
g — Gravitational acceleration, m/s^2
η — Viscosity of the liquid phase, Pa·s
R — Radius of the particle, m
ν — Velocity of the particle relative to the liquid phase, m/s

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