Salt Effect on the Isobaric Vapor–Liquid Equilibrium Study of Binary Mixtures H₂O–NMP (N-Methyl-2-pyrrolidone)

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ABSTRACT: To study the salt effect of recovering N-methyl-2-pyrrolidone (NMP) from the waste liquid produced in the polyphenylene sulfide (PPS) synthesis process, this study presents vapor–liquid equilibrium (VLE) measurement and correlation for water + NMP, water + NMP + lithium chloride, and water + NMP + sodium chloride at p = 101.3 kPa. The salt effect is discussed and the salts follow the order of lithium chloride > sodium chloride. The NRTL model was used for the correlation with binary parameters of water + NMP, water + NMP + lithium chloride, and water + NMP + sodium chloride. The correlation showed good agreement with experimental data; root-mean-square deviations are less than 0.48 K for the equilibrium temperature and 0.005 for the vapor-phase mole fraction of water.

1. INTRODUCTION

N-Methyl-2-pyrrolidone (NMP), a nitrogen heterocyclic compound, is a colorless liquid with slight ammonia flavor. The boiling point, flash point, and pH of NMP are 204 °C, 95 °C, and 7–9, respectively, which show its weak alkalinity. It can be mixed with water in any ratio and can be completely mixed with most solvents (ethanol, acetaldehyde, ketone, aromatic hydrocarbon, etc.). NMP is also a kind of nonproton transfer solvent, which has low viscosity, strong polarity, low volatility, little toxicity, almost no corrosion, strong biodegradation, good chemical stability, and thermal stability. Therefore, NMP with the above excellent properties is a very widely used organic solvent.

NMP is mainly used in many industries, such as petrochemical industry, pharmaceutical industry, pesticide, dye, and lithium-ion battery. It is widely used in extraction of aromatics, purification of olefins, etc., and also used in the production of polyphenylene sulfide, polyamide, and other polymer engineering plastics, as well as insulation materials, pigment and detergent, etc. Polyphenylene sulfide (PPS) is a special engineering plastic with high added value and application prospect in the world, known as the sixth largest engineering plastic, with ultrahigh cost performance. At present, the synthetic routes of PPS on the market mainly include the sodium sulfide method (Phillips method), sulfur solution method, and hydrogen sulfide method, among which the sodium sulfide method is widely used. In this method, p-dichlorobenzene and sodium sulfide containing crystal water are used as raw materials, and sodium hydroxide, sodium chloride, lithium chloride, and other catalysts and additives are added in the reactor. NMP, which can promote nucleophilic reaction, is often used as a solvent to obtain a high-molecular-weight polymer. Although the yield of this method is high, the industrial wastewater often contains a lot of lithium chloride, sodium chloride, and solvent NMP, and NMP and lithium chloride are expensive as a solvent and catalyst. Therefore, the recovery of NMP and lithium chloride is an important factor restricting the economic efficiency of the industrial process of PPS. Generally, for the high-salt waste liquid produced in the PPS synthesis process, the effect of recovering NMP by the distillation method is better, but after the waste liquid is recovered by distillation, some viscous liquid remains in the tower bottom, which greatly affects the recovery efficiency and also causes pollution to the tower kettle.

Salt effect is a phenomenon where the composition of the vapor phase in equilibrium in a binary solution usually changes by adding a salt with volatile components to the system due to interactions between the salt and solvent components. To recover solvent NMP from industrial wastewater of special engineering plastics PPS, vapor–liquid equilibrium data for the

Received: August 21, 2020
Accepted: October 22, 2020
Published: November 23, 2020

https://dx.doi.org/10.1021/acsomega.0c04056
ACS Omega 2020, 5, 31036–31043
systems water + NMP containing salts are necessary. Li et al. described the measurement of the equilibrium liquid composition and boiling points of an NMP–water binary system at 760 mmHg with a modified Washburn ebulliometer. Gupta and Rawat studied isobaric binary and ternary vapor–liquid equilibria of N-methylpyrrolidone with water and toluene at 760 mmHg. The liquid-phase splitting for a ternary system of water + NMP + 1-pentanol can be enhanced by adding the same percentage of salts (sodium chloride, potassium chloride, or potassium acetate) and the influence follows the order of sodium chloride > potassium chloride > potassium acetate. As far as we know, the salt (lithium chloride and sodium chloride) effect on isobaric VLE for the system of NMP + water has not been reported.

In this study, the isobaric vapor–liquid equilibrium data for the systems water + NMP, water + NMP + lithium chloride, and water + NMP + sodium chloride were determined at 101.3 kPa, and the thermodynamic consistency of the measured VLE data was checked by the van Ness test. The influence of different salts on the vapor–liquid equilibrium of the system is explored. Meantime, the VLE data were correlated by the nonrandom two-liquid model (NRTL).

2. EXPERIMENTAL SECTION

2.1. Chemicals. N-Methylpyrrolidone, sodium chloride, and lithium chloride were all purchased from Sinopharm Chemical Reagent Co., Ltd. The water content of the salts was checked by Karl Fisher titration. NMP was used without further purification. Salts were desiccated under a vacuum for at least 24 h. The deionized water (conductivity, <1.0 μS·cm⁻¹) was made in our laboratory by the ultrapure water machine (Nanjing Miaozhiyi Electronic Technology Co., Ltd). Ethanol, acetone, and ether were all purchased from Sinopharm Chemical Reagent Co., Ltd., and used without further purification. Helium was provided by Nanjing Tianze Gas Co., Ltd. All reagents are of analytical grade except helium (industrial grade). Specifications of the chemicals are listed in Table 1.

2.2. Apparatus and Procedures. Accurate measurements of VLE data for (NMP + H₂O), (NMP + H₂O + LiCl), and (NMP + H₂O + NaCl) were implemented in a modified Rose-type recirculating equilibrium still (CE-3, Tianjin Beiyang Tongchuang Distillation Equipment Co., Ltd.) under atmospheric pressure. The structure of the modified Rose-type recirculating equilibrium still is shown in Figure 1, and the composition of the whole of vapor–liquid equilibrium data analyzer is listed in Table 2. The liquid raw material added in the vapor–liquid equilibrium still was heated slowly by the thermocouple to boiling, and the return flow rate of vapor-phase condensation was controlled at about 30–40 drops/min. The evaporated gas phase rose through the vapor-phase circulating pipe and flowed downward after condensation in the spherical condenser pipe, and there is a certain amount of liquid at the gas-phase sample connection. The rest of the condensate flowed into the kettle through the liquid-phase circulating pipe, forming a vapor–liquid two-phase double circulating system. After boiling for 1.5–2 h, the temperature remained unchanged. It is considered that the vapor–liquid phase has reached equilibrium, and the equilibrium temperature was recorded, and gas and liquid sample connection shall be sampled by syringes. The closed chromatographic bottle was placed in the center of the condensed water and cooled down to the normal temperature quickly, and then the collected samples were analyzed by gas chromatography. By changing the composition of raw materials and repeating the above steps, a series of experimental data of vapor–liquid two-phase equilibrium can be obtained.

2.3. Analysis. Gas chromatography (GC-2014, Shimadzu) was adopted to measure the compositions of NMP and H₂O.
The GC-2014 was equipped with a capillary column of Porapak Q and a hydrogen flame ionization detector (FID), with helium as a carrier gas (>99.999% purity, 100 mL·min⁻¹). The detection conditions for the system containing NMP and H₂O were set as follows: the temperature of the injector was set as 523.15 K, the column of the GC was maintained at 483.15 K, and the detector was set at 523.15 K. At least three measurements were taken for all the samples to guarantee the reliability of the data. The mean value was adopted when the deviation of three times was not more than 0.001.

An external standard method via a GC method was established. As shown in Figure 2, the Pearson correlation coefficient $r$ is 0.99998 for the corrected H₂O + NMP system standard curve, which shows high accuracy. Through the standard curve, the mass relation of each component can be calculated by the relation of peak area of each component.

### Table 3. Experimental VLE Data of Binary System of H₂O and NMP

| $T$ (°C) | liquid (mole fraction) | gas (mole fraction) | $T$ (°C) | liquid (mole fraction) | gas (mole fraction) |
|---------|-----------------------|---------------------|---------|-----------------------|---------------------|
|         | $x_1$ | $x_2$ | $y_1$ | $y_2$ | $x_1$ | $x_2$ | $y_1$ | $y_2$ |
| 100.02  | 1.000 | 0.000 | 1.000 | 0.000 | 112.80 | 0.634 | 0.366 | 0.985 | 0.015 |
| 100.82  | 0.965 | 0.035 | 0.998 | 0.002 | 116.00 | 0.531 | 0.469 | 0.976 | 0.024 |
| 101.60  | 0.924 | 0.076 | 0.997 | 0.003 | 122.20 | 0.477 | 0.523 | 0.961 | 0.039 |
| 101.80  | 0.923 | 0.077 | 0.998 | 0.002 | 126.40 | 0.390 | 0.610 | 0.954 | 0.046 |
| 101.85  | 0.928 | 0.072 | 0.999 | 0.001 | 133.10 | 0.311 | 0.689 | 0.930 | 0.070 |
| 102.10  | 0.902 | 0.098 | 0.997 | 0.003 | 140.00 | 0.243 | 0.757 | 0.909 | 0.091 |
| 102.70  | 0.908 | 0.092 | 0.998 | 0.002 | 144.85 | 0.197 | 0.803 | 0.895 | 0.105 |
| 104.20  | 0.864 | 0.137 | 0.996 | 0.004 | 150.00 | 0.163 | 0.837 | 0.857 | 0.143 |
| 104.68  | 0.804 | 0.196 | 0.993 | 0.007 | 156.10 | 0.114 | 0.886 | 0.827 | 0.173 |
| 104.69  | 0.994 | 0.006 | 0.995 | 0.005 | 163.20 | 0.093 | 0.907 | 0.775 | 0.225 |
| 105.10  | 0.802 | 0.198 | 0.992 | 0.008 | 170.50 | 0.057 | 0.943 | 0.687 | 0.313 |
| 105.85  | 0.785 | 0.215 | 0.994 | 0.006 | 175.50 | 0.035 | 0.965 | 0.611 | 0.389 |
| 106.25  | 0.755 | 0.245 | 0.991 | 0.009 | 180.80 | 0.023 | 0.977 | 0.543 | 0.457 |
| 106.70  | 0.787 | 0.213 | 0.995 | 0.005 | 189.50 | 0.012 | 0.988 | 0.402 | 0.598 |
| 107.12  | 0.747 | 0.253 | 0.990 | 0.010 | 196.60 | 0.002 | 0.998 | 0.252 | 0.748 |
| 107.92  | 0.724 | 0.276 | 0.994 | 0.006 | 198.50 | 0.002 | 0.998 | 0.129 | 0.871 |
| 109.20  | 0.714 | 0.286 | 0.988 | 0.012 | 199.30 | 0.000 | 1.000 | 0.000 | 1.000 |
| 110.00  | 0.698 | 0.302 | 0.988 | 0.012 | | | | |

Note: Standard uncertainties $u$ are $u(x_1) = u(y_1) = 0.006$, $u(T) = 0.1$ K, and $u(P) = 1$ kPa.
NMP (2) + lithium chloride were determined at 101.3 kPa by keeping the mass fractions of lithium chloride nearly constant at 1, 3, and 5%, respectively, which are listed in Table 4.

3.2.2. VLE Data of H2O (1) + NMP (2) + Sodium Chloride. The isobaric VLE data for the systems H2O (1) + NMP (2) + sodium chloride were determined at 101.3 kPa by keeping the mass fractions of sodium chloride nearly constant at 1, 3, and 5%, respectively, which are listed in Table 5.

3.3. Consistency Check. In this study, the reliability of experimental data was judged by the method of van Ness tests, which is based on the Gibbs–Duhem theory. To verify whether the experimental data can pass the van Ness thermodynamic test, the predicted values of mole fraction of the vapor phase calculated by the NRTL model, and the experimental data for the systems of (NMP + water + lithium chloride) at salt concentrations of 1, 3, and 5% (mass fraction), respectively. All the values of  are less than 1, confirming that the measured data passed the thermodynamic consistency of the point-by-point test.

3.4. Salt Effect on Vapor–Liquid Equilibria. The trend of VLE data of the water and NMP system with different LiCl mass ratios was studied and is shown in Figure 4. The vapor–liquid equilibrium T–y–x diagram is shown in Figure 4. It can be found that the addition of LiCl has a certain impact on the T–y and T–x curves in the vapor–liquid equilibrium of water and NMP; that is, the T–y and T–x curves show an upward shift phenomenon, and the upward shift of both increases with the increase in the addition amount of LiCl. When the water content of vapor phase or liquid phase is fixed, the vapor–liquid equilibrium temperature increases with the increase in LiCl content. In terms of the separation degree of the system, the addition of LiCl will reduce the two-phase area of the vapor–liquid equilibrium of the water and NMP system, making the separation of the two components of the system more difficult.
because the complex formed in the process has specific structural characteristics, that is, its thermal stability is strong, it may also have a certain impact on the vapor–liquid equilibrium of the system.

The trend of VLE data of the water and NMP system with different NaCl mass ratios was studied and is shown in Figure 5.

As can be seen from Figure 5, the addition of NaCl has a certain impact on the vapor–liquid equilibrium of water and NMP system, and it is more obvious when the equilibrium temperature is between 140 and 200 °C. Comparing Figures 4 and 5, it can be found that the effect of NaCl addition on the system is less obvious than that of LiCl, and according to Figure 5, the vapor–liquid equilibrium curve of the system does not show a trend of obvious change with the increase in NaCl addition; that is, the vapor–liquid equilibrium of the system is almost not affected by the amount of NaCl addition. This also reflects the particularity of the influence of LiCl on the vapor–liquid equilibrium of NMP + H2O system and further explains the effect of the formation of complexes by adding salt on the vapor–liquid equilibrium of the system. Therefore, to recover solvent NMP from industrial wastewater.

Table 5. Experimental VLE Data of Different NaCl Mass Ratios

| % NaCl | T (°C) | x1 | y1 | ΔT | Δy1 |
|--------|--------|----|----|----|-----|
| 1% NaCl | 100.12 | 1.000 | 1.000 | 0.33 | 0.001 |
| 3% NaCl | 100.12 | 1.000 | 1.000 | 0.32 | 0.000 |
| 5% NaCl | 100.12 | 1.000 | 1.000 | 0.34 | 0.000 |

| % NaCl | T (°C) | x1 | y1 | ΔT | Δy1 |
|--------|--------|----|----|----|-----|
| 1% NaCl | 100.62 | 0.969 | 0.999 | 0.21 | 0.003 |
| 3% NaCl | 100.62 | 0.969 | 0.999 | 0.22 | 0.004 |
| 5% NaCl | 100.62 | 0.969 | 0.999 | 0.24 | 0.005 |

Standard uncertainties are

| Standard uncertainties |
|------------------------|
| u(x1) = 0.006, u(T) = 0.1 K, and u(P) = 1 kPa.

Figure 4. VLE data of different LiCl mass ratios (circle, 0% LiCl; up-pointing triangle, 1% LiCl; down-pointing triangle, 3% LiCl; square, 5% LiCl).
containing lithium chloride of special engineering plastics PPS, it is necessary to change the vapor–liquid equilibrium of water + NMP containing lithium chloride by the decomplexation of the complexes caused by NMP and lithium chloride.

3.5. Calculation. The vapor–liquid phase equilibrium can be expressed as follows

\[ \gamma_i \phi_i = \gamma_i \phi_i \exp \left( \frac{V_i^v (P - P_i^s)}{RT} \right) \]  

(2)

where the Poynting factor \( \exp \left( \frac{V_i^v (P - P_i^s)}{RT} \right) \), \( \phi_i \), and \( \gamma_i \) associated with nonideality were all close to 1 since the pressure was low. \( x_i \) and \( y_i \) represent the mole fraction of component \( i \) in the liquid phase and vapor phase, respectively. \( P_i^s \) is the saturation vapor pressure of pure component \( i \), which was estimated by the Antoine expression.\(^{27} \)

Considering the nonideality of the liquid phase, eq 2 can be simplified as

\[ \gamma_i = x_i \phi_i \]  

(3)

The saturation vapor pressure of pure component is calculated by the Antoine equation, which is given as

\[ \ln(P_i^s / \text{kPa}) = A - \frac{B}{T/K + C} \quad T_{\text{min}} \leq T \leq T_{\text{max}} \]  

(4)

where \( A, B, \) and \( C \) are the parameters for each component \( i \), and \( T_{\text{min}} \) and \( T_{\text{max}} \) are the limits of the temperature range, which are listed in Table 6.

3.6. Correlations of the VLE Data. For the description of vapor–liquid equilibrium state of the nonideal system in this study, excess Gibbs function should be mentioned. This function is closely related to the nonideality of liquid, which can be expressed by eq 5.

\[ \frac{G^E}{RT} = \sum_{i=1}^{n} x_i \ln \gamma_i \]  

(5)

where \( G^E \) represents excess Gibbs function, and \( x_i \) and \( \gamma_i \) represent the mole fraction and activity coefficient of component \( i \) in the liquid phase, respectively.

On the basis of Wilson’s model, Renon and Prausnitz introduced Scott’s two-liquid model to treat the mixed solution as a two fluid and then established the NRTL (nonrandom two-liquid) model, namely, the ordered two-fluid model. Derivation of formula can be found anywhere in the literature,\(^{30} \) and the expression of activity coefficient of binary system is shown as follows

\[ \ln \gamma_{12} = x_2 \left[ \tau_{21} \exp(-2\alpha_{12}\tau_{21}) + \tau_{21} \exp(-\alpha_{12}\tau_{21}) \right] \quad \alpha_{12} = \frac{\tau_{12}}{\tau_{21}} \]  

(6)

\[ \ln \gamma_{21} = x_1 \left[ \tau_{12} \exp(-2\alpha_{21}\tau_{12}) + \tau_{12} \exp(-\alpha_{21}\tau_{12}) \right] \quad \alpha_{21} = \frac{\tau_{21}}{\tau_{12}} \]  

(7)

where \( \alpha_{12} = \alpha_{21} \) \(^{31} \)

\[ \tau_{12} = \left( \gamma_{12} - \gamma_{22} \right) / RT \]  

(8)

\[ \tau_{21} = \left( \gamma_{21} - \gamma_{11} \right) / RT \]  

(9)

Since the NRTL model is frequently used to correlate the VLE data of the salt-containing systems,\(^{31} \) in this work, the NRTL model is adopted to correlate the VLE data. The parameters are regressed from the VLE data by minimizing the following objective function

\[ \sum_{i=1}^{n} \left( \gamma_{i,i+1} - \hat{\gamma}_{i,i+1} \right)^2 \]
\[ F = \sum_{i=1}^{N} \left( \gamma_{i}^{\text{cal}} - \gamma_{i}^{\text{exp}} \right)^{2} + \left( \gamma_{2}^{\text{cal}} - \gamma_{2}^{\text{exp}} \right)^{2} \]  

(11)

where \( \gamma_{i}^{\text{cal}} \) and \( \gamma_{i}^{\text{exp}} \) are the calculated and experimental activity coefficient of component \( i \) in the salt-containing system. The regressed parameters for all the systems are listed in Table 7.

### Table 7. Regressed Parameters of NRTL

| system                          | \( \tau_{12} \) | \( \tau_{21} \) |
|---------------------------------|-----------------|-----------------|
| H₂O(1)–NMP(2)                  | –5519.074       | 1128.327        |
| H₂O–NMP (1% LiCl)              | –9075.977       | 3187.593        |
| H₂O–NMP (3% LiCl)              | –6325.647       | 2161.272        |
| H₂O–NMP (5% LiCl)              | –3985.238       | 2015.275        |
| H₂O–NMP (1% NaCl)              | –9489.609       | 4145.941        |
| H₂O–NMP (3% NaCl)              | –10089.067      | 3371.964        |
| H₂O–NMP (5% NaCl)              | –9768.230       | 3309.599        |

The root-mean-square deviation (RMSD) for the temperature \( (T) \) and the mole fraction of the vapor phase \( (y_{1}) \) are expressed as follows

\[ \text{RMSD}(T) = \left( \frac{\sum_{i=1}^{N} (T_{i}^{\text{cal}} - T_{i}^{\text{exp}})^{2}}{N} \right)^{0.5} \]  

(12)

\[ \text{RMSD}(y) = \left( \frac{\sum_{i=1}^{N} (y_{i}^{\text{cal}} - y_{i}^{\text{exp}})^{2}}{N} \right)^{0.5} \]  

(13)

The values of RMSD \( (y_{1}) \) and RMSD \( (T) \) are listed in Table 8, which are less than 0.005 and 0.48 K, respectively.

### Table 8. RMSD for the Equilibrium Temperature \( (T) \) and Mole Fractions of the Vapor Phase \( (y_{1}) \) of the NRTL Model

| salt                | \( w \) (%) | \( y_{1} \) | T (K) |
|---------------------|-------------|-------------|-------|
| lithium chloride    | 1           | 0.005       | 0.27  |
|                     | 3           | 0.003       | 0.28  |
| sodium chloride     | 1           | 0.000       | 0.26  |
|                     | 3           | 0.003       | 0.32  |
|                     | 5           | 0.003       | 0.28  |

According to the calculated values of RMSD \( (y_{1}) \) and RMSD \( (T) \), the NRTL model is suitable for the VLE calculation for the systems of water + NMP + salts.

### 4. CONCLUSIONS

The isobaric vapor–liquid equilibrium data for the systems NMP + water + lithium chloride and NMP + water + sodium chloride were determined at a pressure of 101.3 kPa. The consistency of the measured VLE data was checked by the van Ness test. Meanwhile, the NRTL model was adopted to correlate the VLE experimental data of the systems, and the interaction parameters of the NRTL model were also regressed. The correlated results were in agreement with the measured data. The salting-out effect of the salts follow the order of lithium chloride > sodium chloride, Therefore, to recover solvent NMP from industrial wastewater containing lithium chloride of special engineering plastics PPS, it is necessary to change the vapor–liquid equilibrium of water + NMP containing lithium chloride by the decomplexation of the complexes caused by NMP and lithium chloride.

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### Notes

The authors declare no competing financial interest.

### ACKNOWLEDGMENTS

The authors are grateful to the financial support of National Natural Science Foundation of China (nos. 91634104 and 2018YFB0604605), Jiangsu Science and Technology Plan Project (no. BM2018007), and the Fundamental Research Funds for the Central Universities.

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