1. INTRODUCTION

Inorganics escaping from the solid matrix to the gas phase during thermal conversion of solid fuels, incineration of solid wastes, and metallurgical treatment of iron ores often lead to fouling of heat exchangers, particulate emission, and corrosion of refractories. Evaporation is one of the main routes of this inorganics release, especially for alkali compounds. Quantification of the evaporation rate, in which the saturated vapor pressure of the salt is the most important data, plays a great role in determination of the optimal operation condition.

Vapor pressure \( P_s \) of pure salt is often calculated according to the Antoine equation \( \log P_s = \frac{-52.23B}{T} + C \), where \( P_s \) is in millimeter of mercury; \( T \) is temperature in kelvin; \( B \) and \( C \) are constants, and the related constants are often calculated or measured. In many industrial systems, mixtures of salts instead of pure salts are often present. For example, potassium chloride (KCl) and potassium sulfate (K\(_2\)SO\(_4\)) are the main components in the solid phase during combustion of agro stalks with many other salts and eutectic melting often occurring. In the mixture of molten salts, the vapor pressure of each salt often differs significantly from that of pure salt. For an ideal mixture, it is described according to Raoult’s law. For a real molten mixture, reliable data on vapor pressure of each salt can only be obtained by experiments and the database is far from complete. Lack of data makes it difficult to predict the evaporation rate of salts in real industrial systems. Many methods have been developed to measure this vapor pressure; some are quite complex and time consuming. According to our previous studies and pre-experiments, the mass loss rate and heat effect of pure salts and mixtures can be determined in a simultaneous thermogravimetric analyzer (STA). Vapor pressure of salt in mixtures might be obtained via well-designed experiments.

The objective of this paper is to examine the vapor pressure of KCl in its mixture with K\(_2\)SO\(_4\) at different molar fractions and different temperatures using an STA.

2. MATERIALS AND METHODS

2.1. Materials. Pure KCl (>99.5%) and K\(_2\)SO\(_4\) (>99.0%) were acquired from Tianjin Fengchuan Chemical Reagent Co. Ltd. and then crushed into powders with a particle size of <120 mesh using an agate mortar. The powders were dried at 105 °C for 24 h in an electric oven with an air blower. After cooling, eight types of mixtures of KCl + K\(_2\)SO\(_4\) with molar fractions (KCl versus K\(_2\)SO\(_4\)) of 0.37, 0.54, 0.70, 0.73, 0.78, 0.82, 0.90, and 0.95 were prepared. Powders of pure salts and mixtures were sealed in plastic bags and stored in a desiccator for the experiments.

2.2. Measurements of the Evaporation Process. Mass loss and heat effect of pure salts and their mixtures were examined using an STA (TGA/DSC 1/1600, weighing
Potassium salts are reactive at high temperature and could probably damage the instrument. Two measures were taken to avoid corrosion of the instrument by the sample as shown in Figure 1.

The first is to use a pair of crucibles (inner: 50 \( \mu \)L of alumina, outer: 70 \( \mu \)L of platinum) in all runs. Normally, alkali salts could leak from an alumina crucible to pollute/damage the sensor model of the instrument and could injure a platinum crucible at high temperature. By using a pair of crucibles, the outer platinum crucible can prevent direct contact of a little leaked salts with the sensor model of the instrument. The inner alumina crucible can reduce the direct contact of the sample with the platinum crucible and consequently prevent the damage. In expanding of experiments to solid waste, such as biomass ash, it can also prevent the sintering of samples on a platinum crucible, which is much more expensive than an alumina crucible.

The second is to use only a small amount of the samples. According to our previous experiments shown in the right side of Figure 1 and many pre-experiments, small size samples can also get a stable evaporation rate, although it is not as good as big samples (the detailed analysis is discussed later).

After each blank run, a sample of approx. 1.6 mg of KCl was put into the inner crucible and heated from 100 °C to the set temperatures (875, 900, 925, and 950 °C) at a heating rate of 50 °C/min. Nitrogen with a flow rate of 150 mL/min was used as purge gas and nitrogen with a flow rate of 20 mL/min was used as protect gas. It should be noted that the maximum temperatures of the samples in each run are 871, 896, 921, and 946 °C, although the temperatures of the references are 875, 900, 925, and 950 °C, which are the same as the set temperatures.

2.3. Quantification of Vapor Pressure of KCl in Mixtures on the Basis of Relative Pressure. Pre-experiments showed that evaporation of K\(_2\)SO\(_4\) below 950 °C is negligible and the mass loss rate in each run of mixtures of KCl and K\(_2\)SO\(_4\) is the evaporation rate of KCl. Vapor pressure of KCl on the top surface of the condense phase dominates this mass loss rate of KCl from molten salts as shown in Figure 1. The evaporation process is the same with Stephen flow and the mass loss rate (\(m\cdot KCl, \text{kg/s}\)) is given in eq 1:

\[
m_{KCl} = \frac{D_{KCl, gas} P_0}{RT L_F V_0 KCl,1 - KCl,0} \cdot A \cdot M_{KCl} \cdot \ln \left( \frac{p_0 - p_{KCl}}{p_0 - p_{KCl,0}} \right)
\]

where \(A\) denotes the surface area of the condense phase (m\(^2\)); \(D_{KCl, gas}\) denotes the diffusion coefficient of KCl in gas (m\(^2\)/s) (\(D_{KCl, gas} = 1.013 \times 10^{-2} T^{1.75} (\frac{1}{M_{KCl}} + \frac{1}{M_{air}})^{0.5}\)); \(L_F V_0\) denotes the height of the free volume in a crucible (m); \(M_{KCl}\) denotes the molar mass of KCl (kg/mol); \(p_{0}\) and \(p_{KCl,0}\) denote pressure of the atmosphere, vapor pressure of KCl at the top surface of the condense phase and pressure of KCl at the outer surface of a crucible (Pa); \(R\) denotes the universal gas constant (J/(mol K)); \(T\) denotes temperature of the sample (K).

Figure 1. Evaporation of KCl from crucibles in an STA (left) and the characteristics of evaporation rates at different sample sizes (right, data from ref 7).

Figure 2. Reproducibility of experiments and effects of experimental conditions.
Since the flow rate (150 mL/min) of purge gas above the crucible is high, it is assumed that $p_{\text{KCl,1}}$ at the outer surface of the crucible equals 0. Equation 1 transforms into eq 2.

$$m_{\text{KCl}} = -\frac{D_{\text{KCl,gas}}P_0}{RT} \frac{1}{L_{\text{FV}}} \ln(1.0 - p_{\text{KCl,0}}/P_0) A M_{\text{KCl}}$$

(2)

After runs of pure KCl and a mixture of KCl + $K_2SO_4$ in an STA, mass loss rates are obtained. If $T$ and $L_{\text{FV}}$ in eq 2 are well controlled, relationship of the mass loss rate in the mixture and in pure KCl with vapor pressure of KCl in the mixture and pure KCl is given in eq 3.

$$\frac{m_{\text{KCl,mixture}}}{m_{\text{KCl,pure}}} = \ln(1.0 - p_{\text{KCl,0,mixture}}/P_0)/\ln(1.0 - p_{\text{KCl,0,pure}}/P_0)$$

(3)

where $m_{\text{KCl,mixture}}$ and $m_{\text{KCl,pure}}$ denote evaporation rates of KCl in mixtures and in pure KCl (kg/s); $p_{\text{KCl,0,mixture}}$ and $p_{\text{KCl,0,pure}}$ denote vapor pressures of KCl in mixtures and in pure KCl (Pa). When $p_{\text{KCl,0,mixture}}/P_0 \ll 1.0$, which is the case in evaporation of KCl in most combustion and incineration processes, eq 3 can be simplified into eq 4.

$$r = \frac{p_{\text{KCl,0,mixture}}}{p_{\text{KCl,0,pure}}} \approx \frac{m_{\text{KCl,mixture}}}{m_{\text{KCl,pure}}}$$

(4)

where $r$ is the relative pressure, which is the ratio of vapor pressure of KCl in mixtures to that of pure KCl. Since vapor pressure of pure KCl can be accurately calculated using Antoine’s equation ($P_i = 133 \times 10^{-52.218/T+4}^7$) vapor pressure of KCl in the mixture can be quantified using this relationship of relative pressure.

It should be noted that detailed information of variables and constants involved in eqs 1–4 has been reported in references 7 and 14.

3. RESULTS AND DISCUSSION

3.1. Reproducibility of Experiments and Effects of the Heating Rate/Sample Size. Mass loss rates from the experiment are the key parameter in quantifying the vapor pressure as shown in eq 4. In order to check the reproducibility of experiments and the sensitivity of operation conditions, several runs were repeated and runs at different heating rates and different masses of the sample were made. Results of mass loss rates with data on the molar fraction of KCl, mass of KCl, set temperature, and heating rate in the respective legends are shown in Figure 2.

The mass loss rate in all runs remains almost constant in most part of the set temperature and drops at the last stage of evaporation. Reproducibility of the mass loss rate at the constant stage in repeated experiments is high with a relative error of less than 5% as shown in subfigures (a) and (b).

The effect of the heating rate from 10 to 50 °C/min on the evaporation rate at the constant stage is shown in subfigure (c). The mass loss rate increases slightly for about 3 min after the arrival of set temperature at a heating rate of 50 °C/min. More evaporation occurs before set temperature at a heating rate of 10 °C/min than that of 50 °C/min, but the evaporation rate is almost not affected. In addition, the vapor pressure of potassium chloride (KCl) in its mixture is obtained on the basis of relative pressure, which is calculated on the basis of the ratio of the mass loss rate of KCl in mixtures to that of pure KCl according to eq 4. Therefore, a high heating rate has little effect in our experiments, and it was adopted for most runs due to much less time (40 min vs 110 min) required.

The effect of increasing the sample mass of KCl from 1.0 to 1.7 mg on the evaporation rate at most constant stages is shown in subfigures (a), (b), and (d) and it is negligible. It is reasonable that more sample mass has a longer stage of constant mass loss rates. The long stage of constant evaporation rate shows that the process is not sensible to $L_{\text{FV}}$ in certain scope of the sample mass. As a consequence, eq 4 can be applied in determining the vapor pressure.
3.2. Evaporation Behavior of Pure Salts and Mixtures.

Mass loss rates of KCl from pure KCl and mixtures of KCl + K$_2$SO$_4$ at molar fractions of 0.82 and 0.70 (mass ratios of KCl to K$_2$SO$_4$ are 2:1 and 1:1) at different temperatures are shown in Figure 3. Two characteristics can be seen from all the subfigures. The first is that the molar fraction of KCl in the mixture affects the evaporation rate significantly. The evaporation rate of a mixture with a molar fraction of 0.82 is significantly higher while that of 0.70 is significantly lower than that of pure KCl. According to Roult’s law in which partial pressure is assumed to be in linear correlation with its molar fraction (always <1.0), the evaporation of KCl in different molar fraction mixtures should be inhibited by K$_2$SO$_4$ via decreasing vapor pressure of KCl. However, the fact is that vapor pressure of KCl in a mixture is higher than that in pure KCl under some conditions, and this is far away from Roult’s law.

The second observation is that the evaporation rates drop more slowly in all mixtures than that in pure KCl at the last stage of evaporation. More K$_2$SO$_4$ (molar fraction of 0.70 compared to that of 0.82) leads to a longer stage of low evaporation rate. This implies that the existence of K$_2$SO$_4$ affects the vapor pressure of KCl at the last stage (lower molar fraction of KCl due to its evaporation) significantly.

3.3. Vapor Pressure of KCl in the Mixture of KCl + K$_2$SO$_4$.

In order to identify the effect of the molar fraction of KCl on its vapor pressure in the mixture, eq 4 was used in processing of all the data from Figure 3 with the results shown in Figure 4.

![Figure 4. Relative vapor pressure of KCl in the mixture with its molar fraction at different temperatures (legends show the molar fraction of KCl and mass of KCl in the original sample).](image)

![Figure 5. Relationship of the calculated vapor pressure of pure KCl with measured mass loss rates (a) and the phase diagram of KCl and K$_2$SO$_4$ (b).](image)

\[
\begin{align*}
x_{\text{KCl}} &= \frac{(m_{\text{KCl}}/M_{\text{KCl}})}{(m_{\text{KCl}}/M_{\text{KCl}} + m_{\text{K$_2$SO$_4$}}/M_{\text{K$_2$SO$_4$}})} \\
\end{align*}
\] (5)

where $m_{\text{KCl}}$ denotes the mass of KCl remained in the sample (kg), $m_{\text{K$_2$SO$_4$}}$ denotes the mass of K$_2$SO$_4$ in the original sample (kg), $M_{\text{KCl}}$ and $M_{\text{K$_2$SO$_4$}}$ denote the molar mass of KCl and K$_2$SO$_4$ (kg/mol).
Y-axis is the relative pressure \( r \), which is the ratio of vapor pressure of KCl in the mixture to that in pure KCl and is calculated using eq 4.

In eq 4, \( m_{\text{KCl, pure}} \) was chosen 3 min after the maximum temperature has been arrived in the runs of pure KCl. They are 0.086, 0.115, 0.16, and 0.23 at a sample temperature of 871, 896, 921, and 946 °C, respectively. The ratios between these values agree well with ratios of vapor pressure of pure KCl calculated from Antoine’s equation as shown in Figure 5a. This indicates that the measurement is reliable.

Characteristics of relative pressure at different temperatures share similarities as shown in Figure 4. There are three stages (I, II, and III) in all curves—slow change at a low molar fraction (<0.25), significant change at a medium molar fraction (0.25∼0.55), and a stable stage at a high molar fraction (>0.55) of KCl.

In stage I, the relative pressure increases slowly with the increasing fraction of KCl. Higher temperature leads to less deviation among experiments. This is partly due to the high signal/noise ratios in measurement of the mass loss rate at high temperatures. Another reason is the effect of solid in the mixtures. Figure 5b shows the binary phase melting diagram of KCl-K\(_2\)SO\(_4\). In Figure 5b, four groups are classified—the mixture of liquid KCl and K\(_2\)SO\(_4\) (area above the AOB line), the mixture of liquid KCl-K\(_2\)SO\(_4\) and solid K\(_2\)SO\(_4\) (area on the left of the AOE line), the mixture of liquid KCl-K\(_2\)SO\(_4\) and solid KCl (area on the right of the BOF line), the mixture of solid KCl and K\(_2\)SO\(_4\) (area under the EF line). At sample temperatures of 871, 896, 921, and 946 °C, there is a solid phase in the mixture of KCl + K\(_2\)SO\(_4\) when the molar fraction of KCl is very low. It is seen that solid K\(_2\)SO\(_4\) appears when the molar fraction of KCl is less than 0.38 at a temperature of 871 °C. When the molar fraction is lower than 0.24, the solid phase appears in all the above temperatures. If there is solid in the molten mixture, molar fraction calculation using eq 5 cannot represent the state of solution and it will also affect the flow of vapors. In combustion of biomass, some of low-melting eutectic mixtures (such as KCl/NaCl with other components) often appear and salting-out effects on molar fraction calculation using eq 5 and vapor flowing are similar.

In stage II of Figure 4, the relative pressure of KCl increases significantly with an increasing molar fraction and the maximum relative pressure is normally <1.0. It implies that K\(_2\)SO\(_4\) inhibits the evaporation of KCl at this stage. In stage III, relative pressure is almost constant. The value is affected by the original condition of each run, especially the molar fraction of KCl in the original sample. The relative pressure from the mixture with molar fractions of 0.70 and 0.82 is 0.85 and 1.2, respectively.

3.4. Turning Point from Promotion to Inhibition of Evaporation of KCl by K\(_2\)SO\(_4\). Further experiments on evaporation of KCl at 950 °C in an STA from different original samples with the molar fraction of KCl from 0.35 to 0.95 have been performed and the results are shown in Figure 6. Results of all runs show that the evaporation is significantly promoted when the molar fraction of KCl is higher than 0.78. The promotion changes only slightly with the molar fraction of KCl in the scope of 0.78 to 0.95. This implies that adding only a little K\(_2\)SO\(_4\) (its molar fraction from 0.05 to 0.22) into KCl will promote evaporation of KCl significantly. A similar phenomenon was recorded that CsI enhances the evaporation of the cation elements composing FLiNaK.\(^{15}\) The mechanism of promoting evaporation is left for future work.

Figure 6a shows that evaporation of KCl is inhibited when the molar fraction of KCl is lower than 0.73, in which the molar fraction of K\(_2\)SO\(_4\) is higher than 0.27. According to Figure 5b, in a eutectic system of KCl + K\(_2\)SO\(_4\), the molar fraction of KCl is also 0.73. It means the turning point from promotion to inhibition of evaporation of KCl by K\(_2\)SO\(_4\) is the same with its fraction in the eutectic system. Supplement experiments with big samples (0.82, 18.9 mg; 1.0, 19.0 mg) and samples in a platinum crucible have also been performed. The mass loss rates are shown in Figure 6b. For big samples, the evaporation rate of pure KCl is reasonable. However, when the mass drops from 19.0 to 1.83 mg, the evaporation rate is 0.32 mg/min, much larger than 0.23 mg/min observed in experiments with an original
mass of 1.83 mg as shown in Figure 3d. This indicates that the evaporation rate is affected by the original mass. This might be explained by the coffee-ring phenomenon which states that the contact line of a liquid drop is pinned to its original outer surface. The original sample of the surface varies with its mass and the larger sample has a larger outer surface as shown in Figure 7.

Another remarkable effect is that the history of the evaporation rate is a staircase curve for the mixture. Observation shows that K$_2$SO$_4$ climbs quickly out of the inner alumina crucible and sticks to the outer wall of the platinum crucible during the heating of the mixture, even samples that are less than half full in the crucible are loaded. The staircase curve might be related to this.

For the samples in a platinum crucible, characteristics of the evaporation rate are similar to those in crucibles (outer: platinum; inner: alumina) shown in Figure 3. Climbing of K$_2$SO$_4$ also occurs when only a platinum crucible is used. The fleeing of K$_2$SO$_4$ from the condense phase changes the molar fraction of KCl in the mixture. All this shows that there is interaction between salts and some crucibles.

Anyway, the relationship of relative pressure of KCl with its molar fraction is calculated according to eq 4 on the basis of data from supplement experiments of large size and the result is also shown in Figure 6a. It is seen that the promotion and inhibition function are present and the scaling up factor in the promotion stage is also 1.2. Of course, data in stage II is not accurate due to the interaction of the crucible. For samples in a platinum crucible, scaling factors of 1.26 and 0.85 were also obtained at the initial stage when molar fractions of KCl in the mixture are 0.82 and 0.70. It indicates that the evaporation at the initial stage does represent vapor pressure even when there is interaction between the sample and the crucible.

If a type of crucible which has ignorable interaction with salt is chosen, KCl to 1.2 times. The molar fraction of K$_2$SO$_4$ added to the mixture increases the vapor pressure of KCl to 1.2 times. The molar fraction of K$_2$SO$_4$ at the turning point from promotion to inhibition of evaporation of KCl approximates 0.27, which is its fraction in the eutectic system KCl and K$_2$SO$_4$. Vapor pressure of KCl decreases significantly with increasing molar fractions of K$_2$SO$_4$ at the inhibition scope. A rough correlation of vapor pressure with molar fraction is determined and the result can be used as a reference in analyzing the emission of KCl in combustion of solids.

4. CONCLUSIONS

Measurement of the vapor pressure of salt in its mixture with other salts is quite complex. In this study, vapor pressure of KCl in the mixture of KCl + K$_2$SO$_4$ at different molar fractions and different temperatures were analyzed on the basis of relative pressure using an STA. It shows that small amounts of K$_2$SO$_4$ added to the mixture increases the vapor pressure of KCl to 1.2 times. The molar fraction of K$_2$SO$_4$ at the turning point from promotion to inhibition of evaporation of KCl approximates 0.27, which is its fraction in the eutectic system of KCl and K$_2$SO$_4$. Vapor pressure of KCl decreases significantly with increasing molar fractions of K$_2$SO$_4$ at the inhibition scope. A rough correlation of vapor pressure with molar fraction is determined and the result can be used as a reference in analyzing the emission of KCl in combustion of solids.

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