

Rigorous Simulation Model of a KCl Food Production Plant to Address Impurity Precipitation

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Potassium chloride (KCl) is widely used in many fields of the processing industry. The application in the food industry, as well as the one in the pharmaceutical sector, requires very pure KCl crystals (not less than 99.0%), therefore the elimination of impurities in the production process is a mandatory task. The case study analyzed in this work is a real pilot plant for producing high-purity KCl developed by an Italian chemical company. The major issue of this process is given by the occasional presence of potassium sulfate (K_2SO_4) in the final product which invalidates the stringent purity specifications required. The actual socio-economic situation linked to the energy and raw materials markets makes it very difficult for the industries to spend efforts for experimental investigations that are normally often long and expensive. Therefore, a rigorous simulation model of the plant has been built using UniSim Design[®] with the thermodynamic package OLI[®] that enables the possibility to consider different liquid-solid equilibria. The developed model has been used to verify the formation of the impurities in three different ways: checking the process normal operation, verifying the fluid dynamics design of the crystallization unit, and investigating the start-up conditions. The analysis shows a potential source of formation of K_2SO_4 nucleus under the actual brine concentration recirculated in the crystallizer during the start-up phase, albeit further investigations have to be performed on the pilot plant to confirm such a hypothesis.

1. Introduction

The importance and use of potassium chloride (KCl) are well known in different fields of the processing industry, e.g. as a fertilizer, in medicine, and food processing. The large global demand for KCl causes different production processes to be developed. Mansour and Takroui (2007) used the power of ultrasound waves during a crystallization process to enhance KCl precipitation and to improve its purity which reached a maximum value of 97.31%. A pyrometallurgical process of microcline chlorination was demonstrated to be efficient in producing KCl with a 93% potassium extraction yield at 900°C, but a purity of the final product that is only above 60% (Orosco and Ruiz, 2015). An extraction process of potassium from feldspar through a pyro-hydrometallurgical route produced 99.5% pure KCl crystals using different chemicals (Samantray et al., 2019). A novel membrane-promoted crystallization process seems to be a promising approach for the sustainable production of KCl from brines, albeit the purity of the produced crystal is to be well analyzed (Ji et al., 2022). Anyway, the application in food and pharmaceutical sectors requires at least 99.0% KCl crystals (Younes et al., 2019). The elimination of impurities in the production process has been studied by different authors, particularly at the nucleation and crystallization level (Rawajfeh et al., 2014). A thermodynamic model that predicts the composition of crystallized salts after water removal and cooling from multicomponent solutions or brines was proposed to aid the process design and improve the KCl purity (Silva et al., 2017). Understanding how the crystal nucleation and growth process work can help control the crystal size and quality of the final KCl product (Zheng et al., 2023).

A recent pilot plant at a semi-industrial scale for producing KCl *Food* grade developed in an Italian chemical company (later named Altair) registered the occasional presence of potassium sulfate (K_2SO_4) in the final product which invalidates the required purity specifications. Altair uses an already existing system to purify the brine fed to the plant. Hence to investigate such a problem and avoid expenses linked to experimental tests a rigorous simulation model seems the optimal trade-off between field analysis and simplified chemistry equilibrium models. Nevertheless, rigorous simulators have proven to be efficient in different industrial fields,

e.g. to predict pollutant emission in geothermal power plants (Vaccari et al., 2020), to evaluate risk assessment (Marroni et al., 2022), or examining reactive distillation columns (Brunazzi et al., 2018). Therefore, this work aims to develop a rigorous simulation model of the Altair pilot plant and use it to analyze and possibly understand which are the operational conditions that induce the K_2SO_4 impurities precipitation/formation.

2. Altair process description and problematics

The production process analyzed in this paper treats KCl extracted from mines that although already of high quality it cannot be classified directly as high purity (food/pharma grade). Hence, Altair adopts a purification system to obtain an extremely pure brine to feed an electrolysis circuit. The KCl from mines is dissolved in water, creating a saturated solution to be suitably purified from impurities (Ca^{2+} , Mg^{2+} , Fe^{3+} , Ni^{2+} , SO_4^{2-}) through a two-step purification process. Calcium, magnesium, and corpuscles in suspension are removed with a decanter and a subsequent gravity sand filtration, while ionic exchange resins are used to eliminate ionic components (SO_4^{2-} , Na^+ , Mg^{2+}). After purification metal ions concentrations have significantly decreased (<20 ppb), while the presence of SO_4^{2-} is tolerated in the order of 4-7 g/L. ClO_3^- are formed in the electrolysis and then recirculated back to the purification plant, implying an acceptable range of 1-15 g/L. At this stage the brine is named "super-pure", and a portion of it is sent to the KCl food production plant according to the scheme shown in Figure 1. The very low content of impurities allows a recrystallized salt that potentially meets pharma specifications. Another advantage of the Altair scheme is the possibility of reusing the mother liquor from the KCl plant to guarantee a continuous exchange and limit the accumulation of elements such as sulfates and chlorates.

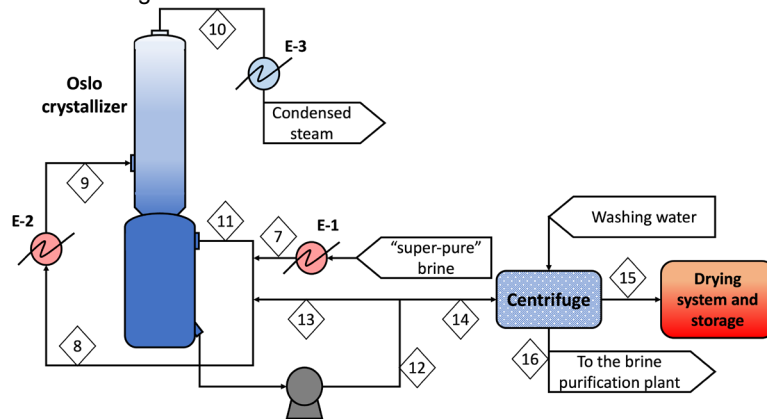


Figure 1: KCl Food production process scheme

The incoming stream (Stream 7 in Figure 1) is $1.7 \text{ m}^3/\text{h}$ of "super-pure" brine preheated up to 52.6°C . The heart of the process is the Oslo-type evaporative crystallizer: an evaporation chamber is placed above a fluidized bed section where the liquor rises through a classified suspension, progressively decreasing its grade of oversaturation. The crystallizer is characterized by three outlets: steam (Stream 10), mother liquor (Stream 11), and magma (Stream 12). The operating conditions are regulated to obtain magma coming out of the bottom of the crystallizer with a density equal to $1280 \text{ kg}/\text{m}^3$ and an average size of the crystals equal to 1 mm. The mother liquors are completely recycled forming $\approx 93\%$ of the crystallizer feed (Stream 8), while the magma is extracted and divided between recycling (Stream 13) and centrifuge feeding (Stream 14) where the crystals are separated, washed, and sent to the drying stage. The two recycles and the fresh feed enter the Oslo crystallizer with a total flow rate of $300 \text{ m}^3/\text{h}$, which is preheated to $\approx 60^\circ\text{C}$ in the exchanger E-2 powered by steam at 3 bar. The flow rate of magma sent to the centrifugation phase (Stream 14) is $\approx 1 \text{ m}^3/\text{h}$. During washing in the pusher centrifuge crystals smaller than $400 \mu\text{m}$ come out together with the saturated solution (Stream 16) and are recycled to the saturation tank. The crystals, separated by the centrifuge, are sent to the drying system. The presence of SO_4^{2-} ions in the processed brine can cause the precipitation of K_2SO_4 . From the literature (John et al., 2018) we know that for binary systems (water+salt) KCl is more soluble than K_2SO_4 , and for both the solubility increases with increasing temperature. Furthermore, the presence of KCl influences the precipitation of K_2SO_4 , i.e. the saturation limit of K_2SO_4 decreases from $115 \text{ g}/\text{L}$ to $11.7 \text{ g}/\text{L}$ at 30°C (Liu et al., 2015). Although the concentration of SO_4^{2-} ions in the "super-pure" brine is kept at low values 4-7 g/L, in some production lots 5 to 10 g/Kg of K_2SO_4 have been detected which exceed the limits of the pharmaceutical market (300 ppm). K_2SO_4 particles are also the only impurities that invalidate the process and causes of their precipitation can be: an accumulation during the continuous process, malfunctions associated with the fluid dynamics of the crystallizer, or even an excessive concentration of the solution during the start-up phase of the Oslo equipment. In the next section, such different hypotheses are examined through design procedures and simulations.

3. Material and method: rigorous simulation model description and validation

To answer the doubts about the K_2SO_4 precipitation, a rigorous simulation model of the process has been built on UniSim Design® R470 using the OLI Engine® 10.0 package suitable for electrolytic equilibria. After verifying the correct replication of the solubility trend of KCl in the binary system, validation tests are performed for the ternary system KCl–H₂O–K₂SO₄, comparing literature data with the values calculated by the simulator. The experimental data in (Liu et al., 2015) were simulated at 303.15 K and 100 kPa, with compositions as reported in the literature. Figure 2 shows that comparing such results, the software with the selected thermodynamic package can satisfactorily predict the behavior of the ternary system and is therefore suitable for studying the process dynamics reliably. The saturation mass fraction of K_2SO_4 in water is 0.115 and as KCl is added, its solubility limit decreases to the value of 0.0117 (Liu et al., 2015) when the solution is saturated for both salts.

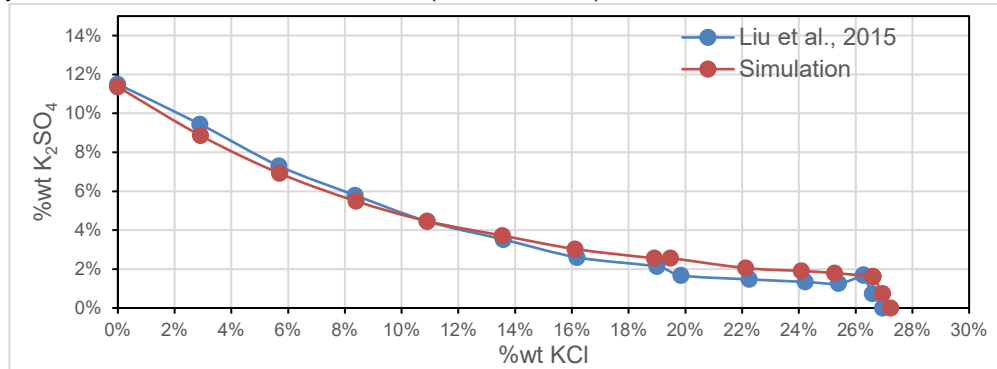


Figure 2: Solubility data for the ternary system KCl–H₂O–K₂SO₄: experimental (Liu et al., 2015) vs simulation

After validation, the KCl pilot plant was simulated; the steps modeled are the salt crystallization, the recycling of mother liquor and magma, and the separation by centrifuge. The simulation of the continuous process is taking as reference 120 kg/h of salt as it was the maximum production of food grade KCl obtained from the pilot plant. Pressure is kept constant throughout the process at 18 kPa since its variations do not influence the salts solubility, i.e. pressure drops have been neglected. Not all data needed for the simulation were available, so the system (1) for solving material balances has been set in the developed iterative procedure shown in Figure 3.

$$\left\{ \begin{array}{ll}
 \text{Global balance on KCl: } F_{7,KCl} = F_{15,KCl} & (1a) \\
 \text{Balance on magma: } F_{12} = F_{13} + F_{14} & (1b) \\
 \text{Balance on crystallizer: } F_8 = F_7 + F_{11} + F_{13} & (1c) \\
 \text{Global balance on water: } F_{7,H_2O} = F_{10,H_2O} + F_{14,H_2O} & (1e) \\
 \text{Balance on the centrifuge: } F_{14,KCl_s} = F_{15,KCl_s} + F_{16,KCl_s} & (1f)
 \end{array} \right. \quad (1)$$

With reference to Figure 1, $F_{i,j}$ is the mass flow of stream i and component j . The fresh feed (Stream 7) is at 56.2 °C and has a volume flow of 1.7 m³/h; to determine its composition, the mass fraction of the two components (KCl and H₂O) were changed to match the stream density to the reference value (1191.6 kg/m³). F_{13} was calculated from the volumetric flow of Stream 12 (20 m³/h) and of the centrifuge feed (1 m³/h) using Eq. (1b) since the streams involved have the same density (1280 kg/m³). This value was also used to fix the composition of the magma knowing its temperature (62.4 °C). The balance on the crystallizer Eq. (1c) was used to calculate the mother liquor flow rate (F_{11}) knowing the feed to the crystallizer is 300 m³/h and setting its density. The mix of these three streams (7, 11 and 13) forms the feed to the crystallizer. The thermal power of the exchanger $E-2$ is fixed to obtain the mass flow of steam in Stream 9 calculated via Eq (1e) (605 kg/h). The crystallizer is simulated with a Simple solid separator as it has the three typical outputs of the Oslo equipment: top outlet (steam), bottom outlet (magma), and side outlet (mother liquor). Splitting parameters are set to not have crystals in the vapor stream (Stream 10) nor the mother liquor (Stream 11), while to tune the amount of solution in Stream 12 the density of the magma is checked to be 1280 kg/m³. The centrifuge is also simulated with the simple solid separator, but only two outputs are now needed: the crystals (Stream 15) and the saturated solution recirculated to the brine pre-treatment process (Stream 16). Once the system is solved, it is possible to conclude that the simulation model gives a complete description of the process. Indeed, despite the solved system in Eq (1), the water and salt quantities were defined by checking the resulting densities for the various streams. An interesting aspect deduced from the simulation is the loss of crystals during washing in the centrifuge. To obtain the same production as Altair it is necessary to set that 45% of the crystals are dragged by the mother liquor (Stream 16). This loss is probably due to the the size of the crystals: particles with a characteristic diameter <400 μm are filtered together with the saturated solution during washing and recycled to the purification cycle.

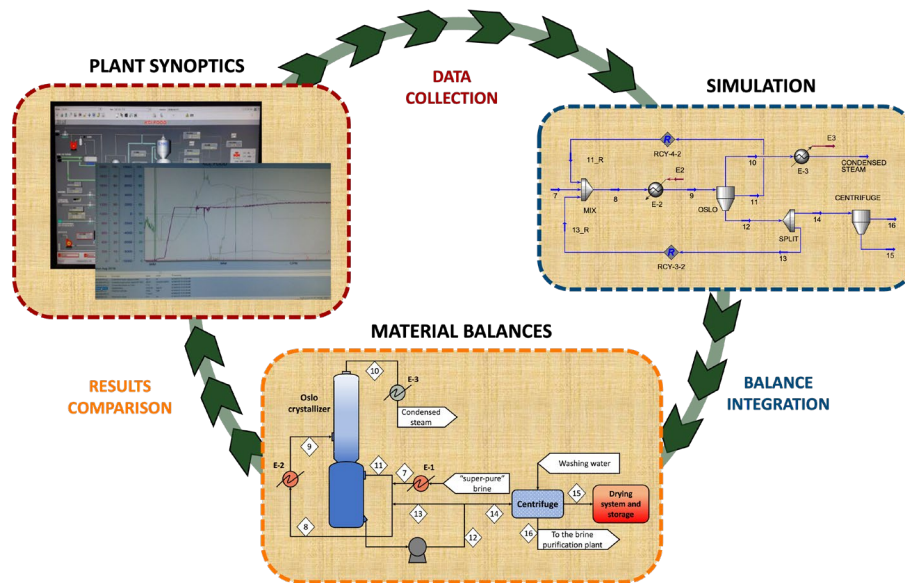


Figure 3 Iterative procedure to build the simulation model of the pilot plant to produce high-purity KCl

4. Results: impurities precipitation verification

4.1 Rigorous simulation model SS

To analyze the precipitation of K_2SO_4 and have a more truthful simulation, further assumptions are needed. Being an impurity, the mass flow rate values of K_2SO_4 in the individual streams represent a further unknown factor. As mentioned, sulfate ions are tolerated in fresh feed in quantity 4-7 g/L, therefore, to analyze the worst-case scenario, we consider the largest concentration to test the most critical conditions of solubilities. Hence, the mass flow rate of K_2SO_4 in Stream 7 is calculated by converting the ions concentration into salt mass. This addition of K_2SO_4 modifies the density of the fresh feed stream, so to maintain the value of 1191.6 kg/m^3 the amount of KCl is changed iteratively to match the density value. Once this is done, the simulation model spreads the K_2SO_4 in each stream automatically; a further check on the coherence of the simulation parameters with the values previously discussed is needed. The concentration of sulfates equal to 7 g/L in the "super-pure" brine does not give rise to precipitation of K_2SO_4 . The first crystal of impurities is obtained for an input concentration value not less than 15 g/L. Hence the causes that lead to the precipitation of sulfate during operation of the pilot plant are to be found elsewhere, not in the continuous process. One possible option to consider is cooling due to the centrifugation of the crystals which may involve further salt precipitation. Nonetheless, by simulating a cooling down to $15 \text{ }^\circ\text{C}$, a product increase of 33 kg of KCl is obtained but still no deposits of impurities.

4.2 Fluid dynamics verification

The incorrect design of the Oslo could be another cause of the presence of impurities in the final product despite the quantities being below the solubility limit since several factors can influence the precipitation of sulfates. Hence, following the fluidized bed crystallizer design procedure (Guarise, 2006), the following aspects were considered: the ascent speed of the solution in the fluidized bed, the suspension void fraction, and the crystal-solution contact time. To carry out the verification, the operating conditions obtained from the simulation model (described in Section 3) have been adopted together with the dimensions of the equipment to compare the results with standard values for correct operation (Guarise, 2006).

Ascent rate of the solution in the fluidized bed

The surface velocity v' of the solution refers to the empty section; thus, it remains constant from the base to the top, with a consequent decrease of the liquid-crystal relative velocity along the bed. The relative velocity v is evaluated from the balance between the friction and the gravity forces for a spherical particle of dimension l (see Eq. (2)).

$$\frac{C_a \pi l^2}{4} \frac{1}{2} \rho v^2 = \frac{\pi l^3}{6} (\rho_s - \rho) g \quad (2)$$

where C_a is the effective friction coefficient which considers the non-sphericity of the crystal. Exploiting the experimental results in (Guarise, 2006), the Reynolds number is determined as a function of C_a , and so the

relative velocity v . Subsequently, the surface speed v' is calculated, using the empirical relation (3), and verified to match the one evaluated by referring the simulated flow rate to the crystallizer cross-section.

$$v' = v\epsilon^z \text{ with } \begin{cases} z = 4.4 Re^{-0.1} \text{ for } 1 < Re < 500 \\ z = 2.4 \text{ for } Re > 500 \end{cases} \quad (3)$$

Where ϵ is the suspension void fraction. Assuming $\epsilon_e=0.91$, $l_e=0.02$ mm, and $l_u=2.1$ mm, where the subscripts e and u identify the suspension top and bottom conditions, respectively, results are obtained with equation (4).

$$\bar{\epsilon} = \epsilon_u + \frac{\epsilon_e - \epsilon_u}{l_u - l_e} (l_u - \bar{l}) \text{ with } \epsilon_u = \left(\frac{v}{v'}\right)^{1/z} \text{ and } \bar{l} = \left[\frac{l_u^4 - l_e^4}{4(l_u - l_e)}\right]^{1/3} \quad (4)$$

The obtained values ($\bar{\epsilon} = 0.87$, $\epsilon_u = 0.88$, $\bar{l} = 1.33$ mm) fall within the recommended limits for correct usage.

Crystal-solution contact time

Inside the fluid bed, crystals shift from the top to the base, in fact, the crystal when growing moves downwards, in equilibrium with the rising current of the solution. The number of crystals of size l_e entering the bed must be equal to the one of characteristic size l_u extracted. Given the scarcity of data at the desired operating conditions, we used the simulation model to extract the quantity of precipitated salt and documents provided by Altair for the crystallizer volume. So, knowing the volume of the fluidized bed (V), the density of the salt (ρ_s), and the average void fraction ($\bar{\epsilon}$) it is possible to determine the solid hold-up in the bed via Eq. (5).

$$W_s = V\rho_s(1 - \bar{\epsilon}) \quad (5)$$

With the dimensions of the crystals previously considered and the mass flow rate of crystals formed (G_s), we calculate the crystal-solution contact time, t_s as in Eq.(6).

$$t_s = 4W_s(l_u - l_e)/(G_s l_u) \quad (6)$$

The resulting crystal residence time value of 4.7 h is probable. Furthermore, another useful parameter to verify the correct sizing of the equipment is the separation intensity $Sl = G_s l_u / V \approx 87$ mm kg/h m³. Typical values are between 50-300 mm kg/h m³, consequently, the resulting Sl is also within the desired range.

Check on re-entrainment

The last check concerns the speed of the vapor exiting the crystallizer. This must be lower than a threshold beyond which significant re-entrainment of drops of saline solution is observed in the vapor phase. The velocity v_{vap} is determined by knowing the volumetric flow rate from the simulation model and the area of the top section of the Oslo crystallizer. The densities of the liquid and the vapor phase ρ_{vap} are taken from the simulation, therefore at operating temperature (62°C). Then the K factor is determined using the relation (6).

$$K = v_{vap} \sqrt{\rho_{vap}/(\rho - \rho_{vap})} \quad (8)$$

Since $K=0.0106$ m/s falls within the range 0.01-0.05 m/s in which liquid droplets are not dragged, the re-entrainment check can be considered satisfied.

4.3 Start-up simulation

Since neither the continuous process nor the crystallizer fluid dynamics verifications failed, we investigate the start-up phase to check if the precipitation of K₂SO₄ occurs. During the start-up phase, the crystallizer powers up gradually evaporating the feeding solution, and concentrating it more than when in continuous operation. Fresh feeding flow (F_7) is much less than the total one which includes the recycling of magma and mother liquor (F_9). To test this hypothesis, and as done in Section 4.2, we consider an input SO₄²⁻ concentration of 7 g/L. Then the fresh feed composition is determined: $F_{7,H_2O} = 1441.6$ kg/h, $F_{7,KCl} = 561.4$ kg/h, $F_{7,K_2SO_4} = 21.6$ kg/h. Through the synoptics provided by Altair, the following information about the Oslo crystallizer start-up is extracted: crystallizer liquid level (76.7%), charging time (6h), pressure (0.165 bar), temperature (71.9 °C), solution density (1191 kg/m³), steam flow rate (1062 kg/h). The cumulative quantities of the components at the end of the start-up phase are determined by multiplying the feed flow rate by the charging time, i.e. $M_7 = F_7 \times 6$ h, resulting in: $M_{7,H_2O} = 8649.6$ kg, $M_{7,KCl} = 3368.4$ kg, $M_{7,K_2SO_4} = 129.5$ kg. Since water is the only stream extracted during the start-up phase, the amount of water evaporated at the same time is subtracted ($m_{H_2O,vap} = 950$ kg/h). Hence, using these values on the simulated model, i.e. $[F_{7,H_2O} - m_{H_2O,vap}, F_{7,KCl}, F_{7,K_2SO_4}]$, the resulting precipitate is composed of 2005 kg of KCl and 30 kg of K₂SO₄, and the concentration of K₂SO₄ in the mother liquor is 19.5 g/L. The K₂SO₄ crystals precipitated during start-up probably do not immediately affect the product as most remain in the process via the recycling of the magma. Consequently, re-entering the crystallizer they also may act as seeds influencing the crystallization of KCl and K₂SO₄. Furthermore, it must be considered that there may also be problems with stagnant zones inside the Oslo where the nucleation of impurities is easier.

5. Conclusions

The analysis of the KCl production process elaborated in this work has allowed drawing interesting conclusions on the pilot plant of an Italian chemical company (Altair) studied to answer experimental evidence of K_2SO_4 impurities precipitation in the final product. A rigorous simulation model of the pilot plant was built using an iterative process that involved solving mass balance equations, extrapolating data from Altair, and using the information calculated by the software simulator exploiting a specific electrolyte equilibrium model. This process model has been used to analyze different aspects of the KCl production plant to validate hypotheses made for the precipitation of K_2SO_4 . In particular, the plant normal operation (continuous phase), and the Oslo crystallizer design have proven to reflect the standards required to produce KCl food grade. Consequently, evaluating the Oslo start-up phase in the worst-case scenario (7 g/L of sulfate ions in the feed stream), 30 kg of K_2SO_4 crystals were obtained. Using the reverse procedure, it is found that the sulfate concentration in the fresh feed must be limited to 5.4 g/L to avoid precipitation during the start-up phase. Consequently, a first strategy could be to limit the density of the magma which, however, leads to a reduction in the yield of KCl. Another process strategy to overcome these problems is to further purify the “super pure” brine fed to the crystallization and lower the maximum allowable concentration of sulfate ions. Clearly, this is the starting point for possible optimization of the process by exploring different configurations that avoid the impurities precipitation problem. In addition, dedicated system tests should be performed for effective improvement in production continuity.

Acknowledgments

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