

## Upgrading of Commercial Potassium Chloride by Crystallization: Study of Parameters Affecting the Process

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**Abstract:** The production of high purity KCl from commercial potassium chloride (96% KCl) produced at the Arab Potash Company in Jordan is investigated using Mixed-Suspension Mixed-Product-Removal crystallizer. The effect of both the residence time and the level of supersaturation on the purity of the crystalline product and on the kinetic parameters of crystallization were investigated. It was found that the purity of the produced KCl salt was adversely affected by both parameters. Nucleation rate was found to increase as the level of supersaturation increases while the growth rate was found to decrease. Both nucleation and growth rates were found to decrease as the residence time increases.

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### 1. Introduction

Crystallization process is one of the oldest unit operations which has a widespread use in the production of pure chemicals, pharmaceuticals, fertilizers, etc.

Crystallization in the presence of impurities is generally encountered in the chemical industry. For example, in fertilizer industry, crystallization of KCl from saturated solution is, in general, performed in the presence of ionic impurities such as  $\text{Na}^+$ ,  $\text{Ca}^{+2}$ ,  $\text{Cl}^-$ ,  $\text{SO}_4^{-2}$ . These impurities and many others, have great effect on the crystallization kinetics, crystal habit, purity and yield.

The effect of ionic impurities on the crystallization process has been studied by many investigators, and no general theory has been yet formulated to explain their effects. Dash *et. al.* indicated that the effects of impurities are due to their ionic properties [1]. Bostaris *et. al.* studied the growth of single seed crystals of KCl under constant conditions of temperature, supersaturation, and impurity concentration in the presence of  $\text{Pb}^{+2}$  ions [2]. They suggested a mechanism of non-equilibrium capture of the impurity and indicated to reject the first crystals grown in order to produce KCl crystals free of  $\text{Pb}^{+2}$ . Sarig *et. al.* studied the effect of NaCl,  $\text{MgCl}_2$  and  $\text{AlCl}_3$  on the crystallization of KCl in a batch cooling crystallizer with either linear or non-linear profiles of cooling [3]. No significant effect of these impurities on the crystal habit, median crystal size, or coefficient of variation was observed.

Two mechanisms on the influence of impurity on crystallization were proposed by Mullin, the first one is due to the electric field of the ions; while the second is due to the chemical interaction between the impurities

and crystals [4]. Sayed and Larson studied the effect of the addition of NaCl and  $\text{MgCl}_2$  on the crystallization kinetics of KCl and on the crystal habit [5]. They found that the presence of these salts in low concentrations reduced markedly the tendency toward polycrystal formation. They also found that the presence of  $\text{Mg}^{+2}$  ions reduced the amount of NaCl in KCl crystalline product. Koenig and Emons and Koenig *et. al.* studied the effect of NaCl impurity concentration on the kinetics of KCl crystallization [6, 7]. They found that both nucleation and growth rates decreased as NaCl concentration increased. Akl *et. al.* studied the effect of the addition of NaCl and  $\text{MgCl}_2$ , separately and combined, on the crystallization kinetics of KCl [8]. They developed correlations for growth and nucleation rates and found that the addition of these salts decreased the growth rate and accelerated the nucleation rate. They also found that  $\text{MgCl}_2$  had more pronounced effect on the crystallization kinetics than NaCl. Rohani and NG studied the crystallization of KCl from an aqueous solution co-saturated with NaCl and KCl in the presence of small amounts of  $\text{MgSO}_4$ ,  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ,  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{CrNO}_3 \cdot 9\text{H}_2\text{O}$  and insoluble silica powder in a 750 ml cooling batch crystallizer [9]. They investigated the effect of the admixtures on the crystallization kinetics, crystal habit, and the roughness of the faces of the crystals, the shape of the corners, the polycrystal formation and the crystal yield. Dash *et. al.* investigated the effect of magnesium and sulfate ionic impurities on the crystallization of KCl using MSMPR crystallizer [1]. Their results exhibited that  $\text{Mg}^{+2}$  did not have any significant influence on the crystallization of KCl; while the sulfate ion enhanced the aggregation rate. The nucleation rate was slightly affected by the presence of impurities. The role of the studied

impurities upon KCl crystallization was found to be a function of the chemical character of the type of impurity. They were also able to identify two mechanisms for size enlargement: the first was due to crystal growth by solid deposition and the second was due to aggregation which was found to be the governing mechanism for producing large crystals.

In this work, MSMMPR crystallizer is used to study the effect of the degree of supersaturation and the residence time on the crystallization of KCl from an aqueous solution saturated with commercial grade KCl salt produced by Arab Potash Company (APC). The purity of the crystalline product and crystallization kinetics are investigated.

## 2. Theory

In common crystallization processes solid particles are formed within a homogeneous saturated liquid phase. This process is very important industrially because it provides a practical method to produce pure chemical materials from solutions of various salts.

The crystals are produced from supersaturated solutions for which supersaturation is often expressed as an equivalent temperature difference and it may be generated by one at the following methods [10]:

- Simple cooling and temperature reduction. This method is used when the solubility of the solute increases strongly with increase in temperature.
- Evaporating a portion of the solvent. This method is common when the solubility is relatively independent of temperature.

c) Salting out by adding a third component which acts physically with the original solvent to produce a mixed solvent in which the solubility of the solute is sharply reduced. This method is used when the solubility is very high and neither of the above methods is desirable.

d) Precipitation by adding a third component that will react with the original solute to produce a new insoluble solute. Using this method, which is used when nearly complete precipitation is required, rapid creation of very large supersaturation is possible.

Crystal formation is a two-step process [10]:

a) The birth of a new particle (nucleation) at a microscopic size.

b) The growth to a macroscopic size.

The driving potential for both rates is supersaturation. Nucleation is a consequence of rapid fluctuations on a molecular scale in a homogeneous phase that is in a state of metastable equilibrium. Crystal growth is a diffusional process modified by the effect of the solid surfaces on which the growth occurs.

A crystallization system can be fully described by material and energy balances together with the Crystal Size Distribution (CSD) of the crystallizer product. The CSD is the relationship between crystal numbers and

size and it is determined by the interaction of the nucleation and growth rates in a crystallizer.

An idealized crystallizer model called the Mixed - Suspension Mixed-Product-Removal model (MSMPR) is used as a basis for identifying the kinetic parameters, namely, the nucleation and growth rates, and hence for estimating the CSD of the crystallizer product. This crystallizer is assumed to consist of a well agitated vessel supplied with a solid-free feed solution at a continuous steady rate from which a product stream is removed at steady continuous rate having a CSD and solids concentration exactly the same as that in the crystallizer. Also in this crystallizer, which is operated at steady state, the McCabe's  $\Delta L$  Law is assumed to hold; that is the crystals of all sizes grow at the same rate, ie crystal growth is independent of crystal size; and also they have the same shape and no gross crystal breakage occurs. The basic quantity in the theory of the CSD is the population density  $n$ . Graphically, the population density is the slope of the cumulative numbers vs. size curve:

$$n \equiv \frac{d(N/V)}{dL} = \frac{1}{V} \frac{dN}{dL} \quad (1)$$

where:

L: crystal size

V: volume of mother liquor in the magma. It is considered to be constant since the MSMPR crystallizer is operated at steady state.

N: number of crystals of size  $L$  and smaller in the magma.

Applying the steady state population balance for the size range  $L_1$  to  $L_2$  with population densities  $n_1$  and  $n_2$  respectively, the balance is:

$$\text{Number of crystals in} = \text{Number of crystals out} \quad (2)$$

The crystals may enter or leave the size range via either growth or flow. If  $G$  is the growth rate and  $Q$  is the volumetric flow rate then [11]:

$$Vn_1G_1 + Q_1\bar{n}_1\Delta L = Vn_2G_2 + Q\bar{n}\Delta L \quad (3)$$

upon rearrangement, this equation becomes:

$$V \frac{n_2G_2 - n_1G_1}{\Delta L} = Q_1\bar{n}_1 - Q\bar{n} \quad (4)$$

taking the limit as  $\Delta L$  tends to zero, we have:

$$V \frac{d(Gn)}{dL} - Q_1n_1 - Qn \quad (5)$$

If we let  $\tau = V/Q$ , MSMPR model applies and  $G \neq f(L)$ , and equation (5) becomes:

$$\frac{dn}{dL} - - \frac{n}{G\tau} \quad (6)$$

Integration of equation (6) gives the function of cumulative population vs. length:

$$\ln \frac{n}{n^0} = - \frac{L}{G\tau} \quad (7)$$

where  $n^0$  is the population density at  $L=0$  and is assumed to represent the nuclei.

Thus

$$n = n^0 \exp\left(-\frac{L}{G\tau}\right) \quad (8)$$

The second kinetic parameter that can be deduced from CSD is the nucleation rate. If the nucleation rate,  $B^0$ , is the rate appearance of near zero-sized particles, an expression can be derived

$$B^0 = n^0 G \quad (9)$$

Therefore, a semi logarithmic plot of the population density vs. size yields  $n^0$  from the intercept and  $G$  from the slope as illustrated in eqn. (8).

Since residence time ( $\tau$ ) is fixed, by the choice of the experimental conditions, the nucleation and growth rates can be determined simultaneously by a single measurement of the CSD at steady state conditions.

### 3. Material and Methods

#### Apparatus

A cooled mixed suspension mixed product removal crystallizer (MSMPR) was used. The crystallizer is a jacketed vessel of one liter operating volume. It is constructed from stainless steel. The suspension inside the crystallizer is mixed using a 3-blade-turbine impeller driven by an electric motor. Four baffles are installed to enhance the mixing. Cooling water is circulated through the jacket to keep the crystallization temperature at the desired value. The feed to the crystallizer is supplied from a constant level tank of four liters capacity. This tank is fed from a feed solution tank of about 70 liters capacity and is constructed from Plexiglas. Continuous mixing is maintained inside the two tanks by stirrers driven by electric motors. Figure 1 shows a schematic diagram of the crystallization system.

#### Experimental Method

The saturated feed solution at 40°C was prepared by dissolving the required amount of KCl salt in the feed solution tank. The solution was filtered using a whatman 41 filter paper to remove the excess salt and any other undissolved solid inerts. The filtered solution was then poured into the feed solution tank and its temperature was kept at 5°C above the saturation temperature to prevent crystallization through pipes. When the desired temperature of the feed solution was reached, the constant level tank was filled with feed solution by opening the valve between the feed solution tank and the constant level tank. The temperature of the solution in the constant level tank was the same as that in the feed solution tank. The crystallizer was filled with the solution via the valve between the constant level tank and the crystallizer itself. The stirrer and the cooling water pump were switched on. The flow rate was measured by measuring cylinder and a stopwatch and adjusted to the required value by the valve between the constant level tank and the crystallizer. As the crystallization temperature was achieved, the crystallizer was run for ten residence times. After that, the crystallizer output solution was filtered directly using a whatman 42 filter paper. The collected crystals

were washed using a solution consisting of 50% ethanol and 50% saturated KCl solution. The crystals were then put in the drier overnight and 70°C and allowed to cool to ambient temperature in a desiccator. The dried crystals were subjected to sieve analysis using a stack of sieves. The sieve sizes were 1400, 1000, 710, 500, 355, 250, 180, 125, 90, 75 $\mu$ m and a pan.

The crystals at each size cut were weighed and subjected to chemical analysis using Pye Unicam Sp8 atomic absorption spectrophotometer. The concentration of the most important impurities (NaCl, MgCl<sub>2</sub>, and CaCl<sub>2</sub>) were calculated after measuring the concentrations of (Na<sup>+</sup>, Mg<sup>++</sup>).

### 4. Results and Discussion

Commercial grade KCl salt (wt. % KCl, wt. % NaCl, Wt. % CaCl<sub>2</sub>) produced by the APC was used to prepare the feed solution for all the experiments performed in this study. Two different sets of experiments were conducted: the first one aimed to study the effect of the level of supersaturation on the purity of the product, while the second one aimed to study the effect of the residence time. The wt. % of KCl salt and that of other impurities were plotted against either the degree of supersaturation or the residence time. A semi log plot of population density ( $n$ ) versus crystal size  $L$  for each one of the experiments was used to determine the kinetic parameters. Finally, the growth and nucleation rates were plotted against the degree of supersaturation and the residence time.

#### Effect of the level of supersaturation on the purity of the crystalline product:

Six experiments at six different levels of supersaturation ( $\Delta T=3, 6, 9, 12, 15$  and 18°C) at a constant residence time (=10min.) were performed to study the effect of this parameter. Figure 2 shows that as the degree of supersaturation,  $\Delta T$ , increases, the wt. % of KCl, NaCl and MgCl<sub>2</sub> in the crystalline product decreases; while that of CaCl<sub>2</sub> increases. The solubility data given in Tables 1 and 2 show that the solubility of CaCl<sub>2</sub> > KCl > MgCl<sub>2</sub> > NaCl. Moreover, Table 1 shows that the solubility of CaCl<sub>2</sub> in the temperature range (20-40°C), which is the range of concern in this study, increases more dramatically than the others. Therefore, as the level of supersaturation  $\Delta T$  increases, more amount of CaCl<sub>2</sub> crystals will be produced leading to fewer amounts of other salts since the amount of Cl<sup>-</sup> ions present in the feed solution which are needed to produce the crystals of other salts is constant as the feed solution is prepared to be saturated at 40°C.

It can be deduced that the presence of Ca<sup>+2</sup> ions in the solution acts as crystallization inhibitor for crystallization of KCl, NaCl and MgCl<sub>2</sub>.

### Effect of supersaturation on the crystallization kinetic parameters:

Figure 3 shows that the growth rate decreases while the nucleation rate increases as the level of supersaturation increases. The magma density  $M$  increases with the level of supersaturation. Its increase is very dramatic for  $\Delta T > 9^\circ \text{C}$  as shown in Table 3. This leads to high attrition between the suspended crystals and between the crystals and solid surfaces in the system leading to high rate of secondary nucleation and to low rate of crystal growth due to crystal breakage. Dash *et. al.* mentioned that for identical residence times, higher suspension densities lead to higher secondary nucleation rates and lower growth rates [1].

### Effect of residence time on the purity of the crystalline product:

Six experiments at six different residence times ( $=2.5, 5, 7.5, 10, 17.5$  and  $15\text{min}$ ) and at constant level of supersaturation ( $\Delta T=12^\circ\text{C}$ ) were performed to study the effect of this parameter. Figure 4 shows that as  $\tau$  increases, the wt.% of KCl decreases, those of NaCl and  $\text{CaCl}_2$  increase; while that of  $\text{MgCl}_2$  is nearly independent. It seems that the positively charged ions suffer from high competition for the chloride ions which are present in nearly constant amounts in the feed solution. Table 4 shows the ionic radii of the ionic species present in the crystallization solution. It is known that, for a given identical charge, a smaller ion would produce a more intense electric field. A stronger field implies stronger ion-ion interactions and bonding, therefore  $\text{Na}^+$ ,  $\text{Ca}^{+2}$  ions will attract the chloride ion more than the  $\text{K}^+$  ion. This means that less amount of  $\text{Cl}^-$  ions will be available for  $\text{K}^+$  ions to produce KCl salt. Increasing the residence time increases the possibility of the reactions between  $\text{Na}^+$ ,  $\text{Ca}^{+2}$  and  $\text{Cl}^-$  to reach equilibrium and hence to increase the consumption of  $\text{Cl}^-$  ions and in effect leading to less amount of KCl salt.

### Effect of the residence time on the crystallization kinetic parameters:

Figure 5 shows that both the nucleation and growth rates decrease as the residence time increases. This was also obtained by Dash *et. al.* [1]. It is believed that increasing the residence time increases the dissolution rate due to the high mobility of the ions and hence leading to lower growth and nucleation rates.

### Comparison between the experimental kinetic parameters and the predicted ones:

Equations 10 and 11 show the correlation proposed by Dash *et. al.* [1] for the growth rate and that given by Qian *et. al.* [12] for the nucleation rate, and they are used in the present study for comparison.

$$G = 5.1 \times 10^{-6} \tau^{-0.61} \quad (10)$$

$$B^0 = 3.58 \times 10^8 \tau^{-1.089} \quad (11)$$

Table 5 shows the experimental values of both  $G$  and  $B^0$  correlated from data shown in Figure 6, and the predicted values for both  $G$  and  $B^0$  calculated from Equations 10 and 11. Accordingly, there is compatibility in the general trend between the experimental and predicted values of  $G$  and  $B^0$ .

Figure 6 shows a strong independence relationship between the population density of the crystals and the residence time. On the other hand, Figure 7 shows that the population density of the crystals does not depend on the level of supersaturation,  $\Delta T$ .

**Table (1):** Solubility of NaCl,  $\text{MgCl}_2$ , and  $\text{CaCl}_2$ . [12]

Temperature, °C	Salt Solubility g salt/ 100g H <sub>2</sub> O		
	NaCl	MgCl <sub>2</sub>	CaCl <sub>2</sub>
0.0	35.6	52.8	59.5
10.0	35.7	53.5	65.0
20.0	35.8	54.5	74.5
30.0	36.1	56.0	102.0
40.0	36.4	57.5	-
60.0	37.1	61.0	137.0

**Table (2):** Solubility of KCl [13]

Temperature, °C	20	24.95	30.65	34.85	41.15
Solubility g KCl/ 100 g H <sub>2</sub> O	34.218	35.599	37.298	38.506	40.386

**Table (3):** The variation of magma density with supersaturation

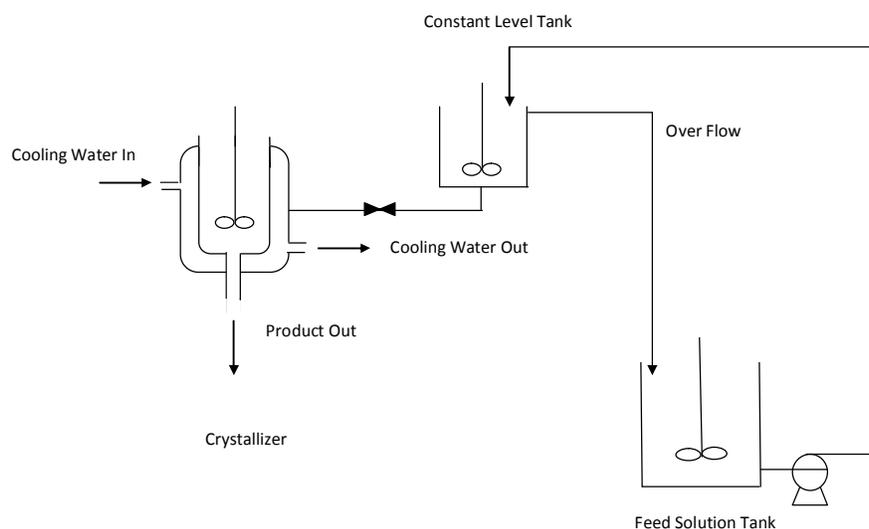
$\Delta T$ , °C	3	6	9	12	15	18
$M_T$ , g crystal/g H <sub>2</sub> O	0.0042	0.0074	0.0054	0.0317	0.0330	0.0392

**Table (4):** Ionic radii of different ionic species [14]

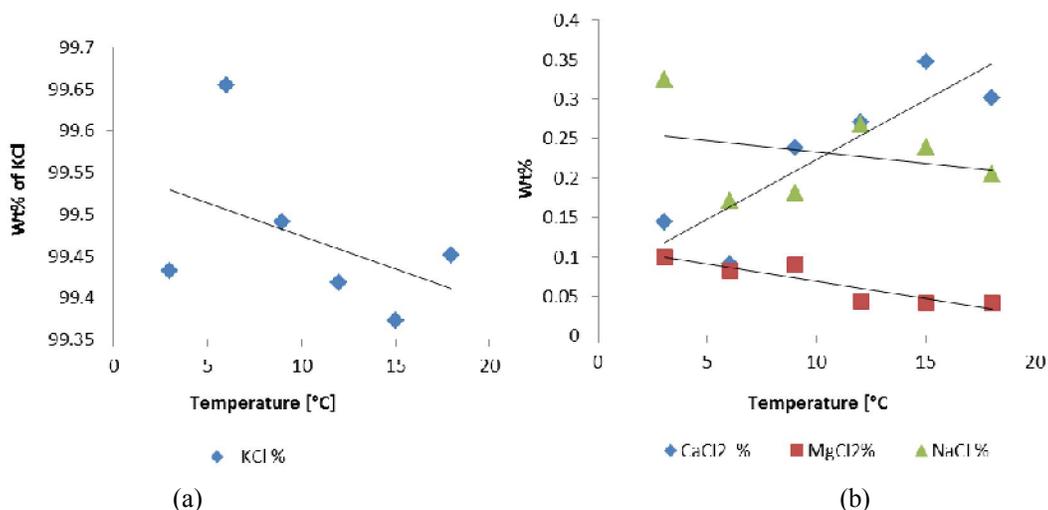
Ionic Species	$\text{K}^+$	$\text{Na}^+$	$\text{Ca}^{+2}$	$\text{Mg}^{+2}$	$\text{Cl}^-$
Ionic Radius(nm)	1.33	0.95	0.99	0.65	1.81

**Table (5):** Experimental and Predicted values of kinetic parameters

$\tau$ [min]	Experimental	Predicted
2.5min (150s)	$G=5.111 \times 10^{-7}$ (m/s) $B^0=4.198 \times 10^4$ (no./S.L)	$G=2.39 \times 10^{-7}$ $B^0=20761 \times 10^4$
5min (300s)	$G=2.48 \times 10^{-7}$ $B^0=1.345 \times 10^4$	$G=1.57 \times 10^{-7}$ $B^0=0.745 \times 10^4$
7.5min (450s)	$G=1.919 \times 10^{-7}$ $B^0=5.356 \times 10^3$	$G=1.22 \times 10^{-7}$ $B^0=3.462 \times 10^3$
10min (600s)	$G=1.556 \times 10^{-7}$ $B^0=4.192 \times 10^3$	$G=1.03 \times 10^{-7}$ $B^0=2.01 \times 10^3$
12.5min (750s)	$G=1.26 \times 10^{-7}$ $B^0=1.911 \times 10^3$	$G=0.89 \times 10^{-7}$ $B^0=1.32 \times 10^3$
15min (900s)	$G=0.983 \times 10^{-7}$ $B^0=6.315 \times 10^2$	$G=0.8 \times 10^{-7}$ $B^0=9034 \times 10^2$



**Figure 1.** Schematic diagram of the crystallization system



**Figure 2.** (a) Wt. % of KCl vs. degree of supersaturation, (b) Wt. % of CaCl<sub>2</sub>, MgCl<sub>2</sub>, NaCl vs. degree of supersaturation

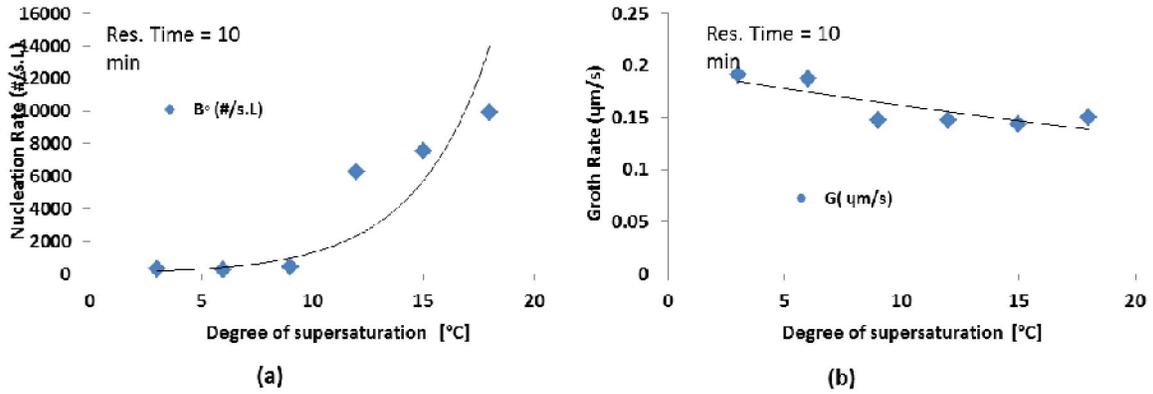


Figure 3. (a) Nucleation rate vs. degree of supersaturation, (b) Growth rate vs. degree of supersaturation

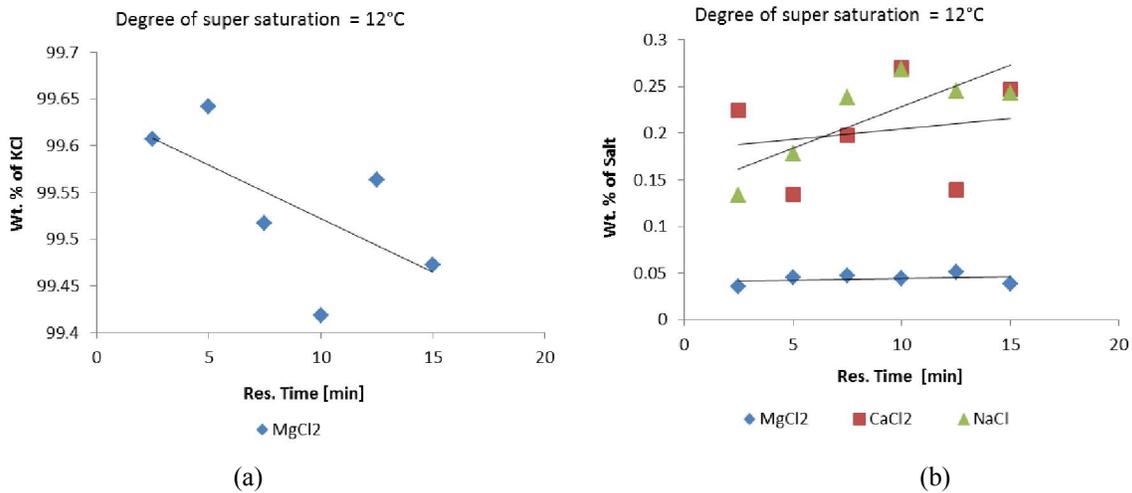


Figure 4. (a) Wt% of NaCl salt vs. residence Time, (b) Wt% of MgCl<sub>2</sub>, CaCl<sub>2</sub>, NaCl salts vs. residence time

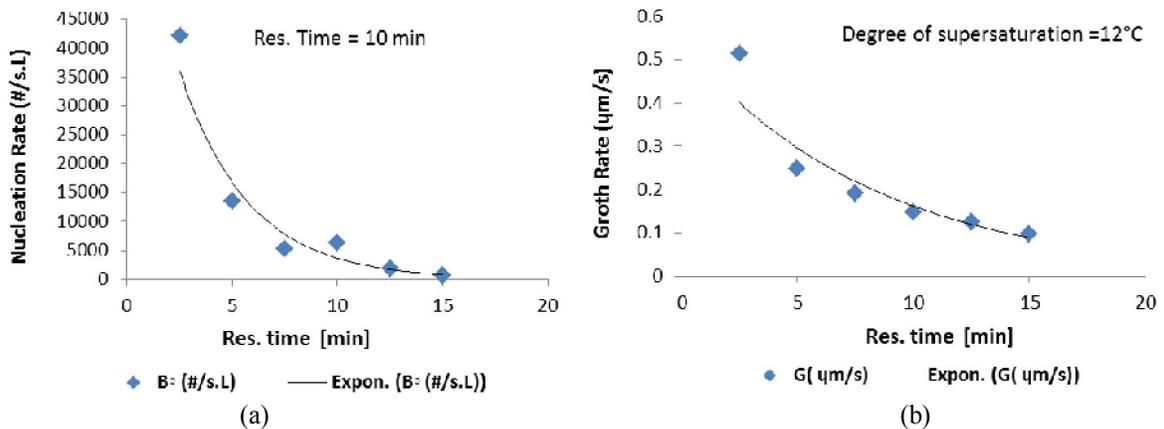


Figure 5. (a) Nucleation rate vs. Residence Time, (b) Growth rate vs. residence time

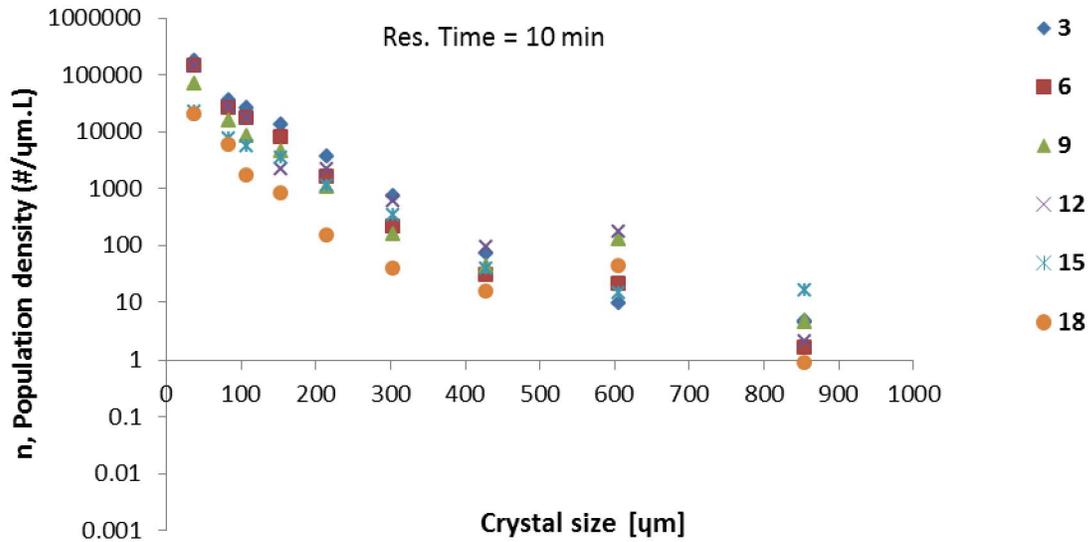


Figure 6. Population density vs. average crystal size at different degree of super saturation

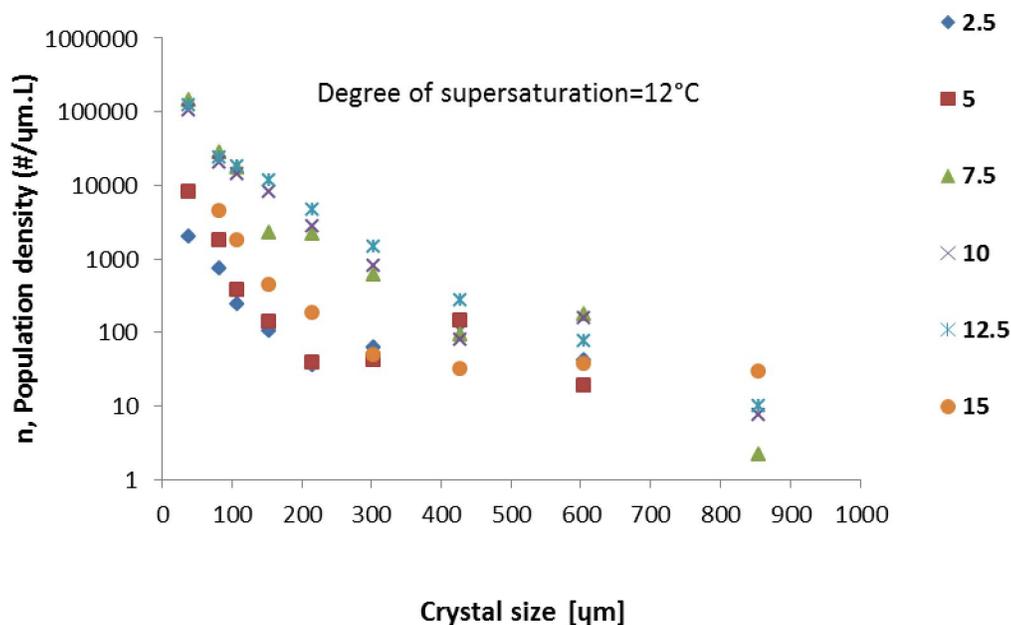


Figure 7. Population density vs. average crystal size at different residence time

## 5. Conclusion

In this study, it has been shown that the ionic properties of the impurity species have significant effect on the purity of the crystalline product and on the kinetic parameters of the crystallization. The purity of KCl salt produced is affected adversely as the level of supersaturation increases as well as the residence time also increases. It has been noticed that the nucleation rate increases while the growth rate decreases as the level of supersaturation was increased. Both rates were found to decrease as the residence time was increased.

## Nomenclature

$B^0$  : nucleation rate (no./L. s)  
 $G$  : crystal growth rate ( m/s)  
 $L$  : characteristic crystal size ( $\mu\text{m}$ )  
 $M_T$  : magma density ( g crystal/ g water)  
 $N$  : no. of crystals of size  $L$  and smaller in the magma  
 $n$  : crystal population density function ( no. / L. $\mu\text{m}$ )  
 $n_0$  : crystal population density function at  $L=0$   
 $Q$  : volumetric flow rate (L/s)  
 $t$  : time (s)

$\Delta T$ : temperature difference between feed tank and crystallizer ( $^{\circ}\text{C}$ )

$V$  : volume of mother liquid in magma (L)

$\tau$  : residence time (s)

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