Influence of Cryolite Content on the Thermal Properties and Coalescence Efficiency of NaCl-KCl salt flux

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Abstract

Salt fluxes with fluoride additions are necessary for the treatment and recycling of contaminated or oxidized aluminium scrap. This study aims to investigate the effect of cryolite additions on the thermal properties and the coalescence efficiency of a NaCl-KCl salt flux mixture. Thermodynamic calculations were carried out to examine the phase diagram of the salt mixture as the cryolite content increases. The study of the melting properties of the salt was carried out by means of differential scanning calorimetry. The coalescence efficiency of the salts was assessed by re-melting coated aluminium chips. The experimental results show a decrease in the liquidus temperature as the cryolite content increases; this differs from the thermodynamical calculations. For cryolite contents up to 3% wt., higher cryolite content in the salt leads to higher coalescence of re-melted chips. However, the coalescence differences observed between the recycling products for cryolite contents of 3% and higher were minor.

Keywords

Salt flux; aluminium recycling; coalescence; melting point; cryolite

Introduction

Aluminium is the second most used metal in the world. Its high specific strength, good corrosion resistance, high electrical and thermal conductivity, and the versatility of its manufacturing processes have made it a competitive material for several applications: packaging, construction, automotive and aerospace, among others. Despite the benefits arising from fuel and energy savings due to the low weight of aluminium products, aluminium requires a substantial amount of energy for its primary production [1]. Due to its strong affinity with oxygen, a great amount of energy is required for the electrolytic conversion of alumina into pure aluminium metal. The Hall-Héroult process is one of the most energy-intensive industrial processes, and it accounts for approximately 2/3 of the total 3% of global GHGs emissions attributed to aluminium production [2]. Other environmental concerns of aluminium's primary production include the air pollutants and the solid waste arising from the Bayer process, also known as red mud. The demand for aluminium products is predicted to increase to 81 % by 2050 [3]. Given the current energy and climate crisis, aluminium recycling is crucial for sustainability since the recycling process requires much less energy and produces significantly fewer waste products and emissions than primary production [1]. The secondary production of aluminium (namely recycling) starts with scrap collection. The scrap is generally categorized according to its origin: *new scrap* originates from the waste of metal-processing industries and is usually characterized by low content of impurities and known composition. *Old scrap* arises from post-consumer utilization, often contains higher concentrations of impurities, and its composition is usually unknown. The scrap collection is followed by comminution and sorting to confer the scrap charge its proper size and eliminate contaminants such as plastic, rubber, glass, and other metals. Some routes of scrap preparation also involve thermal or chemical processes to de-coat the scrap and eliminate paints, paper, moisture, and other contaminants from the scrap surface. The de-coating process may be followed by the compaction of scrap pieces of small size, which tend to oxidate more easily [4]. After the scrap has been prepared, the next step is to re-melt it. Different furnaces are available to melt the scrap according to several parameters such as production volume, energy requirements and cost, scrap characteristics, and final product composition. Rotary furnaces are a suitable choice for processing heavily oxidized and contaminated scrap.

Compared to other furnaces used for re-melting aluminium scrap, such as reverberatory furnaces, rotary furnaces have lower emissions, fuel consumption, and higher melting efficiencies. Rotary furnaces consist of a rotating refractory-lined steel cylinder with a flame burner to melt the aluminium scrap. Melting contaminated scrap in rotary furnaces usually requires the addition of salt fluxes to refine the molten metal [5]. Salt fluxes, which generally consist of a NaCl-KCl mixture with small additions of fluorides, perform three main functions: to cover the molten metal to prevent its oxidation, to collect impurities and oxides from the metal bath, and to promote the coalescence of the metal droplets by freeing them from the surrounding oxide layer [6]. Different percentages of NaCl and KCl can be used as a salt flux. NaCl salt is typically cheaper [7] than KCl, but KCl additions decrease the melting point, reducing energy requirements and the cost of production. An equimolar mixture of NaCl-KCl forms a eutectic point with a melting temperature of approximately 657 °C [5]. During the melting process, the salt flux collects oxides and impurities from the molten scrap and forms a slag layer on top of the bath, which sometimes entraps molten metal droplets. It has been discussed in the literature [8] [9] [6] that the small fluoride additions to the salt flux promote the coalescence of the molten aluminium droplets and reduce the metal losses by entrapment in the salt-slag. The fluorides, which often consist of CaF_2 or Na_3AlF_6, strip the oxide layer surrounding the molten metal droplet, which is then free to coagulate with other droplets and coalesce. The mechanism by which fluorides act is not completely understood in the literature. According to Peterson, the rupture of the oxide layer is related to the fluoride's ability to dissolve aluminium oxide [6]. Tenorio and Espinosa suggested that fluoride additions enhance the rupture of the oxide layer according to a mechanism similar to the hot corrosion process, which helps release the molten metal from the surrounding oxide layer [10]. Interfacial tensions between the salt and the metal also play a crucial role in enhancing the coalescence and the coagulation of metal droplets, as suggested by Roy and Sahai [11]. The treatment and disposal of the waste product from secondary aluminium production, also known as *salt cake*, must be addressed properly due to its potential environmental impact. Usually, salt cakes are either treated for recovery of their components or stored in controlled landfills. The latter is forbidden in most of Europe due to the high reactivity of salt cakes to form pollutants and toxic compounds, so its recovery is the most ecological choice [12].

Although the recycling performance and thermodynamical properties of salts with varying NaCl-KCl ratios have been extensively studied [7, 13], there is little available data regarding how varying cryolite additions affect these properties. In this work, a 95% wt NaCl – 5% wt KCl salt flux with cryolite (Na_3AlF_6) additions ranging from 1 to 15% has been investigated in terms of their melting behaviour and Al coalescence efficiency. The melting behaviour of 70% wt NaCl – 30% wt KCl and 50% wt NaCl – 50% wt KCl mixes, both with 2% cryolite content, was also investigated.

Materials and Methods

The salt fluxes studied in this work were composed of sodium chloride, NaCl (99.5% purity, Fisher Scientific), potassium chloride, KCl, (99.5% purity, Sigma-Aldrich), and cryolite, Na_3AlF_6 (97% purity, Sigma-Aldrich). The composition of the analyzed salts mixtures is summarized in *Table 1*.

Table 1- Chemical composition of the analyzed salt mixtures.

Mixture	wt. % NaCl/KCl	wt. % Na₃AlF₆
95-5-0	95/5	0
95-5-2	95/5	2
95-5-3	95/5	3
95-5-5	95/5	5
95-5-7	95/5	7
95-5-10	95/5	10
95-5-10	95/5	15
70-30-2	70/30	2
50-50-2	50/50	2

The salt mixes with 95/5 ratio NaCl/KCl, and cryolite additions of 0, 2, 3, 5, 7 and 10 *wt*.% were used for the coalescence study. Chips from shredding an aluminium sheet of 8111 alloy and 600 µm thickness, provided by Speira Holmestrand, were re-melted to test the influence of increasing cryolite contents on the degree of coalescence of the recovered

aluminium. The aluminium was coated by a polymeric layer of approximately 25 µm on one side and 5 µ on the other. The exact composition of the coating is unknown but based on analysis from a previous study [14], it consists of a polymer resin with oxide particles of TiO_2, SiO_2 and $BaSO_4$.

Sample Preparation

The salt samples were prepared by melting 300 g of each mixture composition in graphite crucibles by means of an induction furnace with Argon flushing. The molten salt mixtures were cast in a rectangular water-cooled copper mould and let solidify. The aluminium sheet was first shredded into chips with a Getecha RS 1600-A1.1.1 with a grate of 8 mm diameter and then sieved using two sieves of square mesh 5 and 2 mm^2 to unify their size. The mean weight of the chips was calculated in a previous study as 48 mg [14].

Thermodynamic analysis

A preliminary thermodynamic analysis by means of the thermodynamic software *Factsage 8.2 ™* [15] was carried out. Since the NaCl/KCl ratio in each mixture is constant, it was possible to obtain binary phase diagrams in which the considered variables were the temperature and the amount of cryolite. NaCl/KCl mixtures with 95/5, 70/30, and 50/50 ratios were used in the equilibrium and phase diagram calculations. For each NaCl/KCl ratio, a binary phase diagram was created, by setting the amount of cryolite (Na3AlF6) and the temperature as variables. The FTsalt database (SALTA and solid solutions) was used to describe the salt mixture. The formation of bath and cryolite solutions are ignored in the present calculations to avoid compatibility issues. Thermodynamic description of the pure substances was taken from FactPS database.

Differential Scanning Calorimetry (DSC)

A heat-flux DSC apparatus (Linseis STA PT 1600) was utilized to perform the thermal analysis on samples weighing approximately 60 mg. The DSC tests were carried out with an empty graphite reference crucible as heating and cooling scans (5°C/min 820; 10 minutes dwelling at 820 °C; 5°C/min to 25 °C) in an Argon atmosphere with a gas flow of 0.2 l/min. An empty graphite crucible with a 6.8 mm diameter was utilized for the zero line. The samples were obtained by crushing the previously molten salt mixtures. No calibration was carried out for the experiments. By analyzing the DSC curves, the peak extrapolated onset temperature (T_{onset}) and the peak maximum temperature (T_{peak}) were obtained as the intersection between the ascending peak slope and the baseline and as the intersection point between the fitted lines of the descending and ascending slopes of the peak, respectively. The extrapolated baseline was obtained as a straight line between each peak region's initial and final temperature. The initial and final temperatures of the peak region were identified as deviations from the baseline of the DSC curve. The extrapolated peak onset temperature was determined as the liquidus temperature of the samples. When two peaks were present, the second peak maximum temperature was determined as the eutectic temperature of the salt mixtures.

Coalescence study

The re-melting experiments were conducted using ceramic crucibles (Al_2O_3-SiO_2) of 20 cl volume and a Nabertherm™ resistance furnace. For each trial, 80 g of salt-flux were used to re-melt 20 g of chips (salt/scrap ratio 4). This ratio is higher than the typical industrial ones, but it was chosen so that the molten salt would completely cover the scrap and prevent oxidation reactions. The crucibles containing the mixes of salts were placed into the furnace at 830 °C. After approximately 40 minutes (once the salts were completely molten) the chips were charged. Instantly, the combustion of the organic components of the coating generated flames and a dark smoke, and the lid of the furnace was kept open until the end of this combustion (approx. 30 seconds). Then the crucibles were held inside the closed furnace for 7 min, removed and naturally cooled in air. There was no stirring applied. Once the crucibles were at room temperature, the salt was separated from the metal by washing it away with water on a 0.8 mm^2 mesh size sieve. As mentioned in the introduction, the coalescence describes the ability of the individual aluminium pieces to merge, which is critical for a successful re-melting operation without too many metal losses to small metal droplets dispersed in the salt slag. In previous studies, Peterson [6] and Vallejo-Olivares [14] [16] calculated the coalescence efficiency as the percentage of mass recovered which had merged into the largest metal bead per trial. Gökelma [17] and Thoraval [9], as the percentage of mass merged into pieces larger than a specific threshold set depending on the initial size or mass of the scrap. Capuzzi [8] considered in addition the roundness and the diameter of the recovered pieces, and Xiao assessed the coalescence visually in [18]

and quantified the size distributions of the metal beads in the salt [19]. In the present study, the average mass of a shredding was just below 50 mg. Thus, it was assumed that the pieces weighting over 0.5 g originated from the coalescence of at least two chips, and the coalescence efficiency calculated according to Eq 1:

$$\% \ Coalescence = \frac{m_{sum > 0.5 \ g}}{m_{recovered}} * 100$$

where $m_{recovered}$ is the mass sum of all pieces recovered after leaching the salts, and $m_{sum > 0.5 \ g}$ is the mass sum of the pieces heavier than 0.5 g.

Results and discussion

Thermodynamic analysis

The phase diagrams obtained with the thermodynamic software FactSage show the presence of a liquid phase above 757 °C for the 95 *wt.* % NaCl – 5 *wt.* % KCl, regardless of the cryolite content. In the temperature interval 757-788 °C, a solid rocksalt phase is stable with sodium and aluminum-fluoride solid compounds. For temperatures above 788 °C, the rocksalt phase is not present. At temperatures higher than 788 °C, an increase in the cryolite content leads to the disappearance of AlF_3, which is gradually replaced by $Na_5Al_3F_{14}$ and, for cryolite additions greater than 4%, also by Na_3AlF_6. The phase diagram for 95 *wt.* % NaCl – 5 *wt.* % KCl with cryolite additions up to 15 *wt.* % is reported in *Figure 1*.
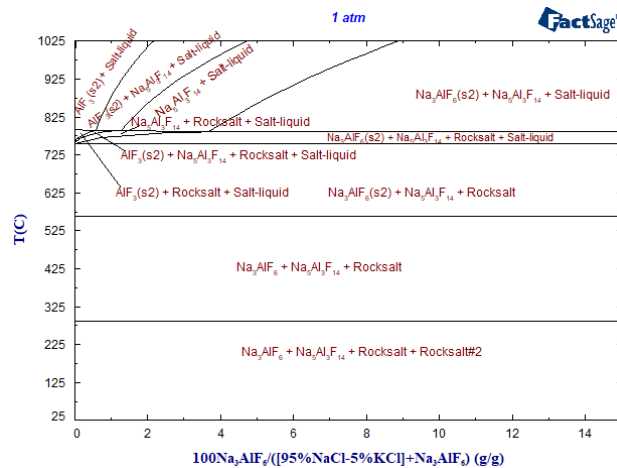

Figure 1 – Phase diagram for the 95% NaCl – 5% KCl salt flux with cryolite additions up to 15% weight.

From the thermodynamic analysis of the 70 *wt.* % NaCl – 30 *wt.* % KCl, a liquid phase is present above 800°C, regardless of the cryolite content. At temperatures higher than 800 °C, an increasing cryolite content leads to the stabilization of sodium-aluminum-fluoride solid compounds (Na_3AlF_6 and $Na_5Al_3F_{14}$), which gradually substitute AlF_3. The phase diagram for the 70 *wt.* % NaCl – 30 *wt.* % KCl mixture with cryolite concentrations up to 15 *wt.* % is shown in *Figure 2*.
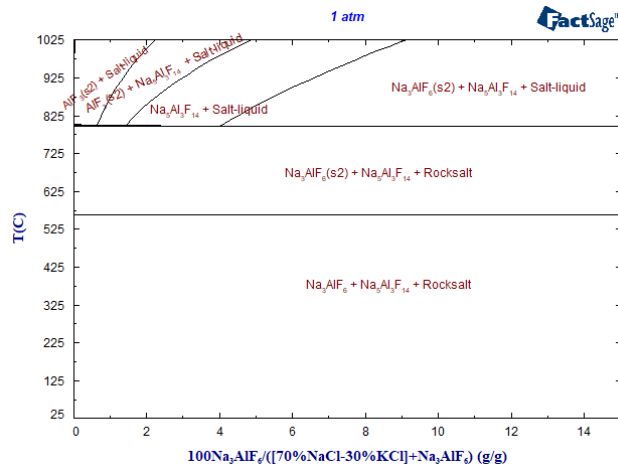
Figure 2- Phase diagram for the 70% NaCl – 30% KCl salt flux with cryolite additions up to 15%.

The investigation of the 50 *wt*.% NaCl – 50 *wt*. % KCl phase diagram at varying cryolite content reveals the presence of a liquid phase above 650 °C, as can be seen in *Figure 3*. The addition of cryolite to the salt mixture for temperature above 662 °C leads to the stabilization of solid Na_3AlF_6 and $Na_5Al_3F_{14}$. However, when cryolite is present below concentrations of 1% at temperatures higher than 775 °C, the presence of a solid potassium-aluminum fluoride compound (KAl_4F_{14}) is also noted, in contrast with the previous phase diagrams.
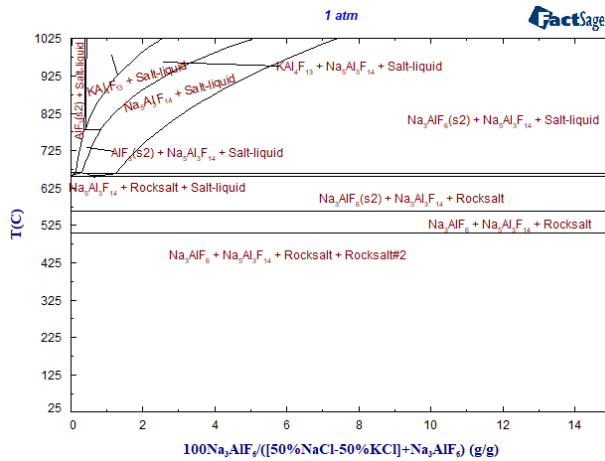

Figure 3 – Phase diagram for the 50% NaCl – 50% KCl with cryolite additions up to 15%.

DSC Analysis

To avoid effects due to superheating, the cooling scans from the DSC were used to determine the characteristic temperatures of the samples. The cooling curves for each mixture containing cryolite showed two main events: a peak due to the solidification of the eutectic and a peak due to the solidification of the remaining phase, as can be seen in *Figure 4* for the 10% cryolite-containing salt. For cryolite concentrations in the range of 2-10 *wt*. %, minor peaks in the temperature range of 631-619 °C were noted and attributed to crystallization phenomena, since these peaks were not found in the heating curves.
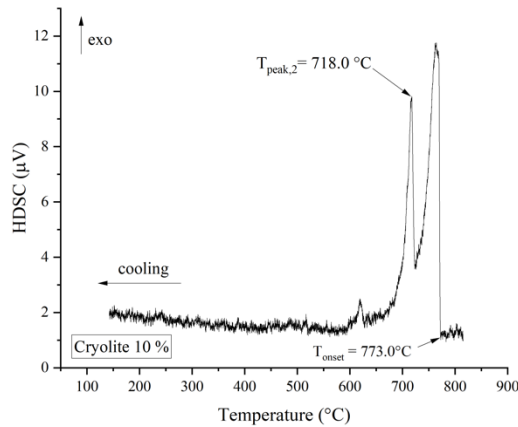
Figure 4 – DSC curve obtained for the 95% NaCl – 5% KCl salt flux with 10% wt. cryolite addition.

The extrapolated onset temperature of the first peak represents the liquidus temperature, whereas the second peak's maximum temperature indicates the eutectic temperature. The mixtures with cryolite contents in the range of 0-3 *wt. %* did not show the presence of the eutectic. The results are shown in *Figure 5*.
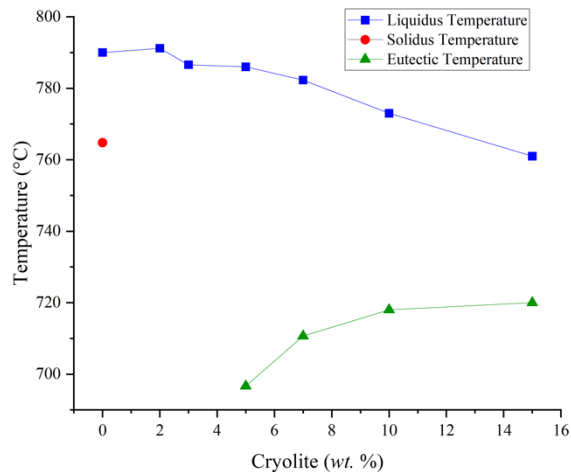

Figure 5 – Plot of the liquidus and eutectic temperatures obtained from the analysis of the DSC curves of the 95% NaCl – 5% KCl salt mixtures. The solidus temperature for the mixture without cryolite is also displayed.

The 95% NaCl – 5% KCl salt without cryolite shows a liquidus temperature of 790 °C, well in agreement with the NaCl – KCl phase diagram from Bolivar and Friedrich [7], whereas the experimental results find the solidus temperature at 765 °C, approximately 10 °C higher than the phase diagram. The addition of cryolite to the 95% NaCl – 5% KCl salt mixture noticeably affects the liquidus temperature of the salt fluxes, especially for cryolite contents above 5 *wt. %*. The liquidus temperature decreases from 790 °C for the salt without cryolite to 761 °C for the 15% cryolite-containing salt. From an industrial point of view, the liquidus temperature is the most relevant: a decrease in the melting point of the salt fluxes is beneficial as it implies lower emissions and reduced energy-related costs. The eutectic temperature for cryolite contents in the range 5-15 *wt. %* increases from 697 to 720 °C.

The liquidus temperatures for the 50% NaCl – 50% KCl and 70% NaCl – 30% KCl mixtures with 2% cryolite are 661 and 725 °C, respectively. According to the NaCl – KCl phase diagram from Bolivar and Friedrich [7], the liquidus temperatures for 50% and 70% NaCl are 660 and 725 °C, respectively: this implies that 2% cryolite additions to the salt mixtures do not affect significantly their liquidus temperature. This is also true for the 95% NaCl – 5% KCl. The liquidus temperatures for the 50% NaCl – 50% KCl and 70% NaCl – 30% KCl mixtures with 2% cryolite are lower when compared with the 95% NaCl – 5% KCl mixtures, regardless of their cryolite content. It is accepted that increasing the KCl/NaCl ratio up to an equimolar mixture decreases the salts melting temperature. [13] This is also seen in the present study and implies higher energy requirements to melt salt fluxes with higher NaCl content. However, it may be economically viable to decrease the KCl content due to its higher cost compared to NaCl [7]. An additional positive effect by lowering the

KCl- content is the decrease of the vapour pressure of the slag and of the salt loss due to evaporation. [20] Furthermore, although the results provide valuable information of the fundamental properties of various salt-flux mixtures, they may not be directly related to their performance during recycling. For instance, neither Pirker's [21] nor Bolivar's [7] recycling studies showed significant differences between the metal yield obtained by salts containing KCl between 10 and 30% when testing in a 160 kg scale rotary furnace and 30kg resistance furnace.

The experimental results from the DSC analysis differ from the preliminary thermodynamic analysis. This may be explained by the fact that the experimental conditions are not those of equilibrium, or to missing information in the used databases. The solubility of $AlCl_3$ and AlF_3 species is not described in the thermodynamic behaviour of the selected liquid salt solution. As such, in the case of solubility of these species in the liquid salt, the overall thermodynamic behaviour of the system might be influenced. As mentioned, the formation of bath and cryolite solutions was ignored due to the compatibility issues with other solutions, which introduces further limitations to describe the system accurately.

Coalescence ability of salt fluxes

According to the coalescence results, presented in Figures 6a and 6b, small additions of 2% or 3 *wt*.% cryolite significantly improve the coalescence ability of the salt fluxes, but further additions have a small effect.
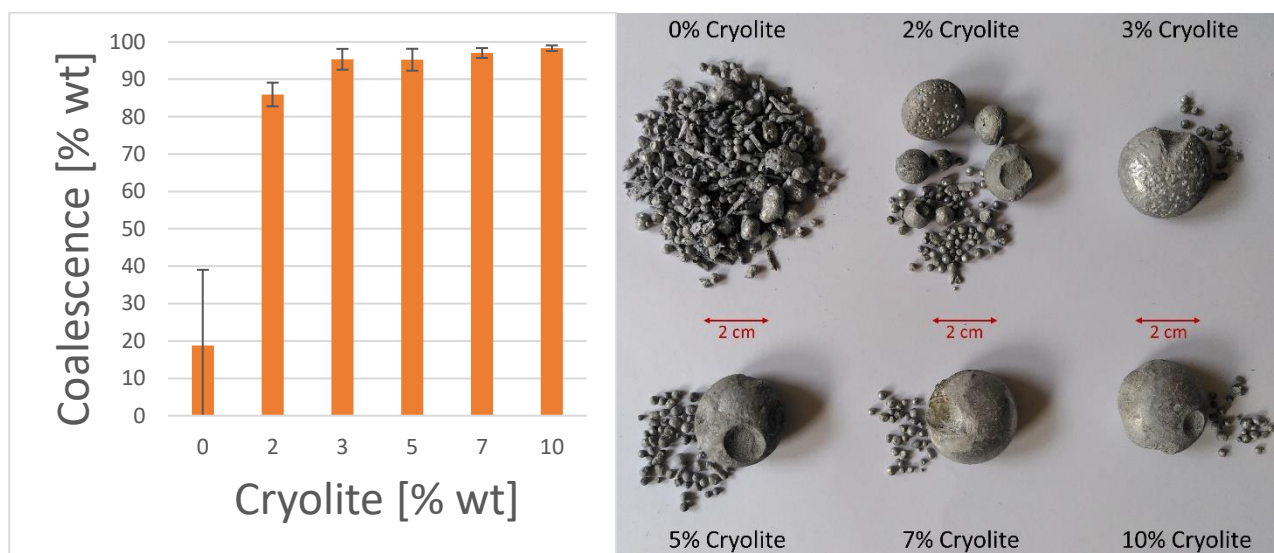

Figure 6 –a) Left: Plot of the degree of coalescence of the metallic pieces recovered after re-melting and washing away the salts. Average and STD from 3 repetitions. b) Right: pictures of the recovered metal products.

Figures 6a and 6b show the remarkable difference between re-meltings without cryolite, on average 19% coalescence, and the re-melting with salt containing cryolite, which led to coalescences over 83% in all cases. There was a large deviation between the three re-melting trials without cryolite, and the exact values of the three trials were of 4, 5 and 47%. When increasing the cryolite content from 2 to 3% there was an improvement from 85% to 95% average coalescence, but higher cryolite additions increased the coalescence just slightly. Therefore, for the current set up increasing the cryolite additions over 2 or 3% would not pay off considering the extra cost and the environmental risks associated with cryolite. Thoraval [9] also reached the optimal coalescence efficiencies with additions of 2% cryolite for clean and recycled salts contaminated with less than 30 *wt.* % oxides. Similarly, Bolivar concluded that adding 1% CaF_2 increased the metal yield if the NaCl/KCl ratio is 0.85 or higher, but further additions were not necessary. However, comparing the coalescence or recyclability results with the literature is not straightforward, as it was demonstrated that they are influenced by many factors, such as scrap size and composition (e.g. oxides, organic contamination) [17] [19] [18], the application or not of thermal or compaction pre-treatments [14] [8] [16], the holding time and temperature [9], the salt/scrap ratio [8] and finally furnace set-up and stirring. For instance, Thoraval [9] used a laboratory scale rotary furnace to re-melt oxidized chips, Gökelma [17] applied stirring manually to recycle oxidized bottom ash, while Capuzzi [8], Vallejo-Olivares [14], and the present study used a static resistance furnace without stirring to re-melt coated scrap. The present coalescence results for 2% cryolite are higher than those obtained for similar coated chips by Vallejo-Olivares using a salt composition of 68.6/29.4/2.0 NaCl/KCl/CaF_2, but lower than those obtained for the de-coated chips. Since CaF_2 is less costly both economically and environmentally, applying a thermal de-coating or mechanical stirring may be more advisable than substituting CaF_2 by cryolite additions to the salt-fluxes to optimize the recycling of aluminium scrap.

Conclusions

This study aimed to investigate the thermal behavior of the 95% NaCl – 5% KCl salt flux, as well as its ability in promoting aluminium droplets' coalescence. The following conclusions were drawn:

Thermal Behavior

According to the preliminary thermodynamic analysis performed with FactSage, an increasing cryolite content does not affect the temperature at which the liquid phases appear in the phase diagrams.

The experimental results from the Differential Scanning Calorimetry show a decrease in the liquidus temperature as the cryolite content in the 95% NaCl – 5% KCl salt flux increases, especially for cryolite contents above 5 *wt. %* For cryolite additions in the range 5-15 *wt.* %, the presence of a eutectic was noted in the temperature range of 697-720 °C.

The liquidus temperatures of the 95% NaCl – 5% KCl salt mixtures are higher when compared with the 70% NaCl – 30% KCl and the 50% NaCl – 50% KCl, regardless of their cryolite content.

Based on the discrepancies between experimental and modelled results, it is suggested that the salt-cryolite system is thermodynamically re-assessed.

Coalescence ability of salt fluxes

While small additions of 2% or 3% wt. cryolite greatly improved the coalescence ability of the salt fluxes, further cryolite additions did not have a significant effect on the coalescence of the recovered aluminium.

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