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Pyrometallurgical recovery of Aluminum from Salt slag

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Abstract: Slag of aluminum is a residue which results during the melting process of primary and secondary aluminum production. Salt slag of aluminum is hazardous solid wastes due to the presence of the European Catalogue for Hazardous Wastes. Hence, recovery of aluminum not only save the environment, but also has advantages of financial and economic returns. In this research, aluminum was recovered and purified from the industrial wastes generated as waste as a result of General Company for Electrical and Electronic Industries (Iraq/Baghdad) works and waste from the General Company for Mechanical Industries (Iraq/ Babel) works. It was found that these contain tiny proportions of other elements such as iron, copper, nickel, titanium, lead, and potassium. Wastes were recovered for green sustainability, saving energy and cost effectiveness. The method applied for recovering aluminum was pyro-metallurgical method by smelting and refining. X- Ray fluorescence spectroscopy and X- Ray diffraction techniques of the slag sample were used to determine the chemical analysis and phases, respectively. Melting experiments were conducted by using different types of fluxes (KAlF_4 , NaCl , KCl and AlCl_3) at different percentages (0, 5, 10) and different melting temperatures (700, 750, 800°C). Design of experiment (DOE) by Taguchi method, orthogonal array L9, was used in melting experiments. Melting efficiency of aluminum was 86%. Electrorefining of aluminum was done by using anhydrous aluminum chloride and NaCl as ionic liquids at low temperature 100 °C in electro-refining method producing aluminum of 99% purity.

Key words: Salt slag of aluminum, %Recovery, Melting process, Design of Experiment (DOE), Electro-refining.

I. Introduction

Aluminum is the most abundant element after the oxygen and silicon in the earth's crust (by weight). It is never been as a free metal in nature and constitutes approximately eight percent of the solid earth surface [1]. Aluminum (Al) is one of the most important metals, since its industrial production, demand for aluminum has been continuously increasing to around 53 million tons in 2014 and its application has expanded to variety of economic sectors. There are three ways for extracting Aluminum, primary source from aluminum ores (bauxite ore), secondary source from aluminum scrap and white dross, tertiary source from black dross and salt slag. Extraction of aluminum starts with bauxite mining, bauxite is processed into alumina, and then alumina is processed into aluminum metal. Extraction of aluminum involves mining of bauxite (open pit), and alumina is produced by Bayer process. Secondary aluminum production is by electrolytic smelting by Hall Heroult technique [2]. All aluminum products can be recycled after use. Recovering of aluminum is very important because of several environmental and economic reasons. Comparison with extraction of aluminum, recycling of aluminum products needs as little as 5% of the energy, and emits only 5% of the greenhouse gas. In the production of aluminum, there is a significant difference in energy between aluminum extraction and recycling compared with other metals; 186 MJ/kg for primary compared to 10–20 MJ/kg for secondary [3].

Table.1. Comparison of Primary and Secondary Aluminum Smelting Processes (data from Gil 2005)

	Primary Smelting	Secondary Smelting
Consumption of energy (GJ/tonne Al produced)	174	10
Atmospheric emissions (kg/tonne Al produced)	204	12
Solid waste (kg/tonne Al produced)	2,100-3,650	400
Consumption of water (kg/tonne Al produced)	57	1.6

Today, about 50% of scrap is old scrap (scrap from “end of life products EOL”). Recycling is fully dependent on scrap as an important source in the aluminum industry, such as used cans, foils, extrusions, commercial scraps, turnings, and old rolled or cast metal. Aluminum can also be recovered from skimming, dross and slag [4]. The

main general categories of operations included in the secondary aluminum production are scrap pretreatment, smelting and refining. Pretreatment operations of scrap involve sorting, processing, and cleaning scrap. Smelting and refining operations involve “cleaning, melting, refining, alloying, and pouring of aluminum recovered from scrap”. Some steps may be combined or reordered, depending on the quality of scrap, scrap sources, design of the furnace, availability of auxiliary equipment and product specifications. Factory configuration, the type of scrap usage, and the output product vary over the secondary aluminum industry. During melting process for aluminum scrap, a layer of oxide is formed on the surface of the molten in large quantities, and the oxide layer must be removed from the molten product for a high quality finished product. In order to avoid the formation of more oxidation phases and enhance an easier separation during smelting, the flux must be used to protect the molten. The flux works on docking with the impurities to be removed from the molten and separating the metal from the impurities as oxide. The molten surface layer is made of oxides, gases, slag eliminators and some metals with a quantity of metal additive. Its common name is aluminum slag [5]. A large number of experiments and studies indicate that the bulk of undesirable metals is a real and increasing problem, at every metal recycling processes. In status of aluminum, the schedule of problematic impurities is very large, involving but not limited to Fe, Si, Cu, Mg, Ni, Zn, Pb, Cr, Mn and V [6]. The production of secondary Al annually generates worldwide a considerable amount of slag (more than 3.5 million tons dross and salt slag), which presents the variability of chemical and mineralogical composition. Generally, the Aluminum slag is a mixture of Al oxide, metals, chloride, fluoride, alloying elements and salts from the fluxing agent, nitride, carbide, sulfides, and other substances, which are the result of molten Al reactions with other elements present in the melting system. In contact with rain water, they can react emanating inflammable or toxic gases, such as methane and ammonia, or may become soluble, infiltrating the soil and finally reaching the groundwater; therefore they are considered dangerous for the environment [7]. The main purpose of this project is to understand the fundamental knowledge of recovery of aluminum from industrial waste in Iraq and the limitation of recovery. The aims of the study was designed to explain the different parameters affecting on recovery of aluminum from industrial waste (slag) by using melting technique, Recovery of aluminum from the industrial waste (slag) by collecting the slag and then crushing, mixing, smelting and refining to obtain aluminum with high purity and Investigating the effect of major factors that may affect the efficiency of melting, such as temperature, types of fluxes, stirring and using inert gas (argon).

II. Experimental work

To prepare data-base of characterization of slag produced in private sector factories, samples of slag have been taken from different regions in Iraq and then tested. The chemical composition of slag's sample is shown in Table.2. These tests involved chemical composition, microstructure, XRF and scanning electron microscope. Effect of additives of the flux has been studied with different fluxes and different percentages of fluxes through melting process. In order to study the effect of re-melting recovering parameters on the salt slag of aluminum, the designs of experiment and optimization should be performed. This study is designed to determine the important parameters and the method of characterizations. The different techniques used to evaluate the object of study are also explained. The process planning is summarized in Figure 1.

Table.2. Chemical composition of slag's sample

Fe%	Cu%	Mn%	Cr%	Ni%	Zn%	Ti%	Pb%	V%	Al%
24.85	24.09	4.50	1.192	0.848	17.82	0.0005	1.213	0.0010	15.82

2.1 Melting Procedure

The slag sample of 15 kg received from General Company Electrical and Electronic Industries and General Company for Mechanical Industries was crushed by laboratory jaw crusher. Slag particles size at the end of operation was (1-3 mm) and then slag was mixed in a mechanical mixer.

After crushing and mixing, slag carried out the smelting process. 100 gm of slag samples placed in a crucible made of graphite or alumina and then placed in the electrical furnace for melting the slag. The slag was melted at different temperatures (700 – 750 – 800°C) with using inert gas (argon) to prevent the oxidation that occurs during the melting process, then removing the slag that formed at the top of the molten and then pouring the molten in the mold. The mold used in this process was carbon steel, and the mold was heated to 300°C before pouring the melt into the mold. Smelting process returned again, was conducted at different temperatures (700-750-800°C) using fluxes (KAlF₄ – NaCl, KCl – AlCl₃) with different quantities and using inert gas (argon) and stirring process for the molten. The molten was pouring again into the mold and then tested in XRF.

The efficiency of melting process was determined using two terms, namely

% Recovery and % Yield.

Where:

$$\% \text{ Recovery} = M2/M1 \quad \dots (1)$$

$$\% \text{ Yield} = M2/M1 + M3 \quad \dots (2)$$

Where:

M1: Weight of the slag before melting. M2: Weight of the sample after melting. M3: Weight of the flux.

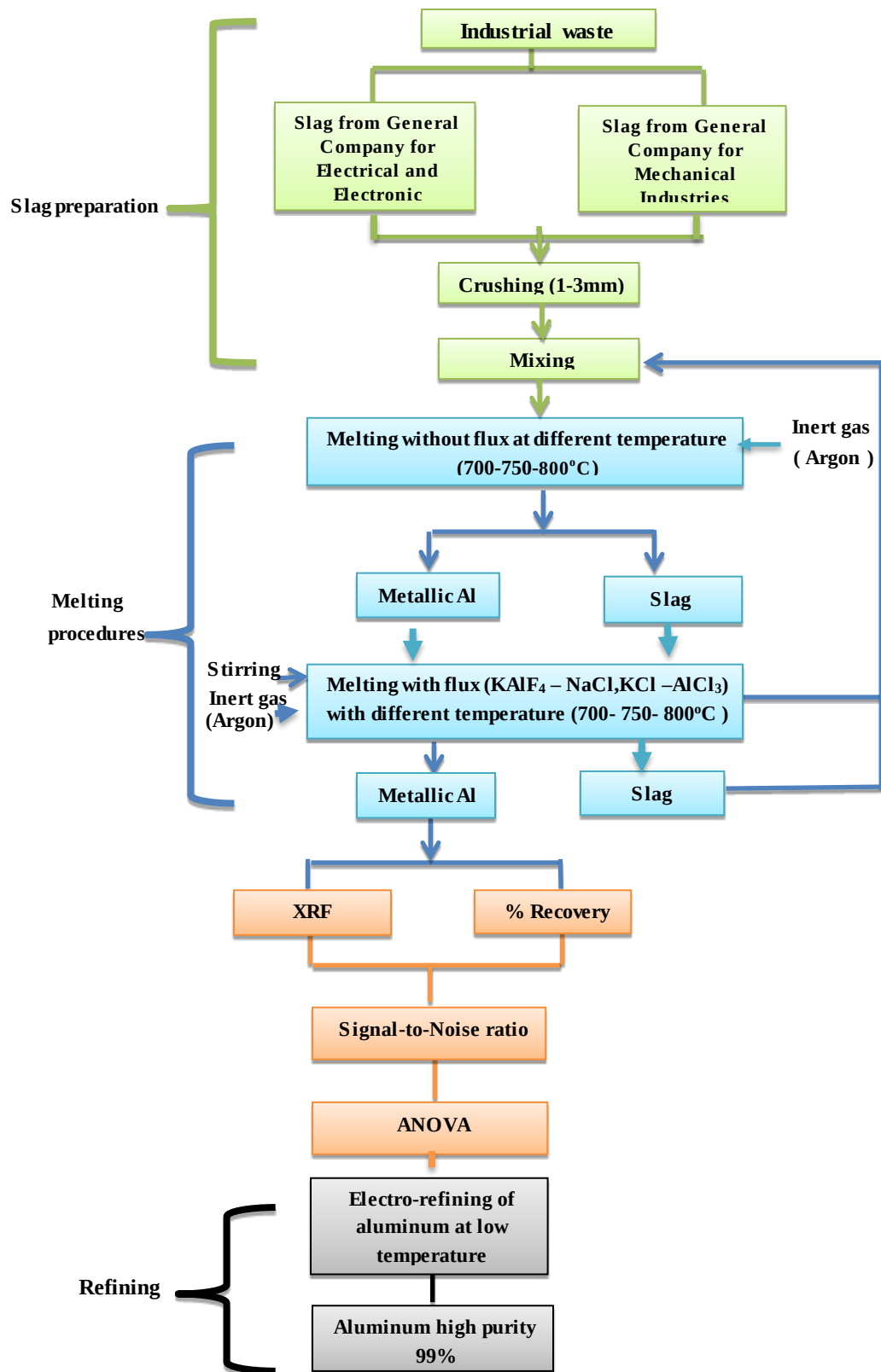


Figure.1. Flow diagram of beneficiation technique

2.2 Electro-refining of Impure Aluminum at Low Temperature

In the electro-refining process, the impure aluminum alloy is used as the anode and it is electro-refined in an ionic liquid prepared from anhydrous AlCl_3 and NaCl. Electro-refining methods at low temperature (100°C) produce aluminum with 99% purity. Electrolysis at low temperature can provide significant energy savings. For this purpose, anhydrous AlCl_3 and NaCl are the ionic liquid. The non-pure aluminum is placed as an anode and a solution of (anhydrous AlCl_3 -NaCl). The cathode is either pure aluminum or pure copper. This electrolysis is capable of removing Mn, Fe, Si, Cu, Zn, Ni, and Pb. The solution can be reused more than once, thus making the process more environmentally friendly. This is because the ionic solution is stable at low temperatures. Aluminum deposited on the cathode can be seen by Light optical microscopy and scanning electron microscope (SEM).

2.3 Process Variables and Design of Experiment (DOE)

Operation variables of temperature, weight of KAlF_4 flux, weight of NaCl, KCl flux and weight of AlCl_3 flux were chosen as main four control factors with their identical three levels to be investigated. Table 3. shows the investigated four controlled factors and their three corresponding levels.

Table 3. Controlled factors and their corresponding levels

Controlled Factors	Correspondence Levels		
	Level 1	Level 2	Level 3
Weight of KAlF_4 flux, gm	0	5	10
Weight of NaCl, KCl flux, gm	0	5	10
Weight of AlCl_3 flux, gm	0	5	10
Melting temperature, $^\circ\text{C}$	700	750	800

III. Results and discussion

3.1 Design of Experiments

Nine experiments were selected according to Taguchi method (Orthogonal array) (Table 4). Using the Taguchi rules with three levels and four processing variables, the nine experiments using L9 array is described in (Table 5). Table 6. represents all nine experiments with their levels. Two trials were made for each experiment to get the average data.

Table 4. Taguchi orthogonal array [8]

Orthogonal Array Selector

Number of Levels

↓

→

Number of Factors

		Number of Parameters (P)																														
		2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	
Number of Levels	2	L4	L4	L8	L8	L8	L8	L12	L12	L12	L12	L16	L16	L16	L16	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	
	3	L9	L9	L9	L18	L18	L18	L18	L27	L27	L27	L27	L27	L36	L36	L36	L36	L36	L36	L36	L36	L36	L36	L36	L36	L36	L36	L36	L36	L36	L36	
	4	L16	L16	L16	L16	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	L32	
	5	L25	L25	L25	L25	L25	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	L50	

Table 6. The experiments used in the melting process

Experiments	Weight KAlF_4 flux, gm	Weight	Weight	Melting temperature, ($^\circ\text{C}$)
1	0	0	0	700
2	0	5	5	750
3	0	10	10	800
4	5	0	5	800
5	5	5	10	700
6	5	10	0	750
7	10	0	10	750
8	10	5	0	800
9	10	10	5	700

Table7. Values and average of melting percentage of Aluminum with and without using flux at different temperature at (TOA) L₉.

Exp.	Weight KAlF ₄ flux, gm	Weight NaCl,KCl flux, gm	Weight AlCl ₃ flux, gm	Melting temperature, °C	Melting1 Al Wt%	Melting2 Al Wt%	Average melting Al Wt%
1	0	0	0	700	83.611	83.8	83.7055
2	0	5	5	750	84.39	84	84.1950
3	0	10	10	800	84.57	84.1	84.3350
4	5	0	5	800	84.66	84.73	84.6950
5	5	5	10	700	85.1	84.9	85.0000
6	5	10	0	750	84.1	83.8	83.9500
7	10	0	10	750	84.34	84.7	84.5200
8	10	5	0	800	84.2	83.6	83.9000
9	10	10	5	700	88.7	88.97	88.8350
Mean weight for all experiments is 84.7							

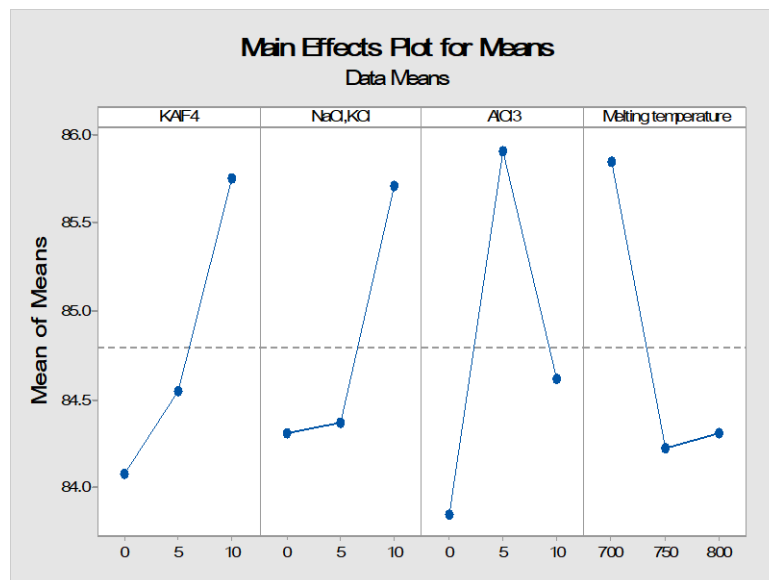


Figure 2. Melting efficiency of Aluminum by using different types of fluxes and different temperatures for each level of the controlled factors

3.2. Signal–Noise(S/N) Ratio

At parameters design, the factors were divided into two kinds: control factors which can be easily controlled by the experimenters and noise factors which can be difficult or expensive to control during operation. To determine (S/N) ratio, there are three types of formulations as shown in table (8). In this study, the second formula is used which represents the larger signal to noise ratio giving the best melting efficiency [9]. In recovery process, the largest % recovery is required for many reasons such as economic and scientific.

Table 8. S/N ratios formulations [10].

1-Lower is the better

2-Higher is the better

3-Nominal is the better

$$\left. \begin{aligned} \frac{S}{N} &= -10 \log \left(\sum_{i=1}^n \frac{y_i^2}{n} \right) \\ \frac{S}{N} &= -10 \log \left(\sum_{i=1}^n \frac{1}{n y_i^2} \right) \\ \frac{S}{N} &= 10 \log \left(\sum_{i=1}^n \frac{y_i^2}{s^2} \right) \end{aligned} \right\} \dots (3)$$

Table 9. Values of S/N ratio for melting aluminum by using different fluxes and different temperatures.

Exp.	Weight KAlF ₄ flux, gm	Weight NaCl,KCl flux, gm	Weight AlCl ₃ flux, gm	Melting temperature, °C	Melting1 Al Wt%	Melting2 Al Wt%	S/N Ratio
1	0	0	0	700	83.611	83.8	38.4551
2	0	5	5	750	84.39	84	38.5057
3	0	10	10	800	84.57	84.1	38.5201
4	5	0	5	800	84.66	84.73	38.5572
5	5	5	10	700	85.1	84.9	38.5884
6	5	10	0	750	84.1	83.8	38.4804
7	10	0	10	750	84.34	84.7	38.5391
8	10	5	0	800	84.2	83.6	38.4751
9	10	10	5	700	88.7	88.97	38.9717
Mean SNs for all experiments is 38.55							

3.3. Analyses of Variances (ANOVA)

This analysis method is used to determine the importance of any processing variable on the overall of the process concern. The general terms described are in % importance [11].

$$SS_T = SS_{(KAlF_4)} + SS_{(NaCl, KCl)} + SS_{(AlCl_3)} + SS_{Temp.} + SS_{error} \quad (4)$$

$$SS_T = \sum_{i=1}^n x_i^2 - \frac{G^2}{N} \quad (5)$$

$$SS_C = \frac{\sum_{i=1}^n x_i^2}{n} - \frac{G^2}{N} \quad (6)$$

Where:

SS_T is total sums of squares parameters and error. G is sum of the results data for all the experiments, N is total number for all the experiments. i is level number. C is one of the tested parameters. $\sum x_i$ is sum of results for all experiments; i.e, at parameter c , and at level i . n is number of parameter

Degree of freedom was calculated by the following equation:

$$DOF_C = C - 1 \quad (7)$$

$$DOF_T = T - 1 \quad (8)$$

Where; C presents the levels for each parameter (controlled factor), T presents the number of experiments. Also, contribution percentage (% ρ) was calculated for each controlled factor and for the error from the following equation

$$\% \rho = \frac{SS_C}{SS_T} \quad (9)$$

When:

$\rho_{error} \leq 15\%$, so the chosen controlled factors are not active.

$\rho_{error} \geq 50\%$, so chosen controlled factors are active.

Table 10. ANOVA for melting aluminum from slag for each controlled factors.

Factors	Degree of freedom (DOF)	Sum of squares (S)	% contribution (% ρ)
Weight KAlF ₄ flux, gm	2	0.037	18.407
Weight NaCl, KCl flux, gm	2	0.037	18.407
Weight AlCl ₃ flux, gm	2	0.057	28.358
Temperature °C	2	0.047	23.382
Error	0	0.023	11.442
Total	8	0.201	100%

3.4. Electro Refining of Aluminum at Low Temperature

Electro-refining methods are shown in figure 3. for produced aluminum of 99% purity at 1.5 volt and low temperature about 100°C by using anhydrous aluminum chloride $AlCl_3$ and NaCl.

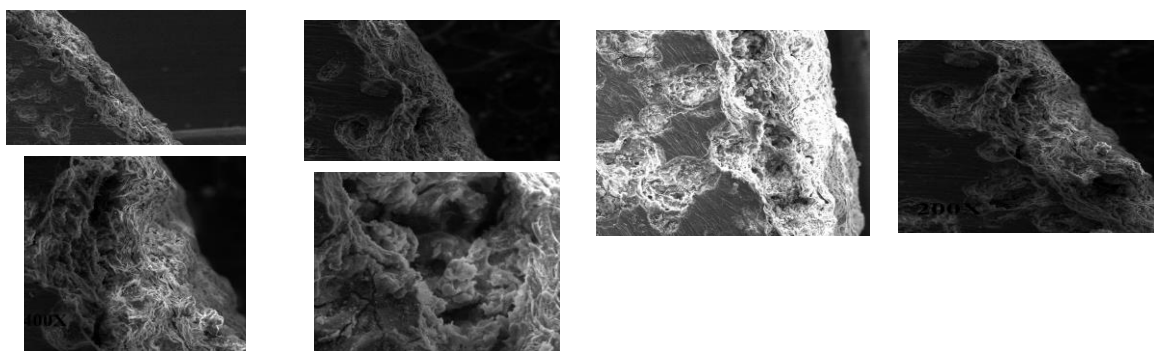


Figure 3. Scanning electron images (SEM) of aluminum deposited

3.5. Discussion of Melting Process

Melting process was effective process for recovering aluminum from industrial waste, so using different fluxes and different temperatures and using inert gas (argon) and stirring the melt were the alternative for aluminum recovery.

3.5.1. Effect of adding $KAlF_4$ flux

The effect of adding $KAlF_4$ flux on the melting of aluminum slag was studied, and the ratio of adding $KAlF_4$ flux is in the range of (0 - 10). The results represented in Fig.2. show the increase in amount of $KAlF_4$ at the melting process leads to increase the melting efficiency of aluminum. The role of $KAlF_4$ flux is to separate metals from slag and oxide and to prevent agitating burning of the aluminum during melting process, and therefore maximizing yields. When $KAlF_4$ is added to a mixture of NaCl and KCl, the resulting flux is more fluid. This increasing fluidity better covers the exposed molten metal and facilitates the release of metals entrapped in the slag. The active fluoride will also remove magnesium (Mg) in the melt and improve the metal purity. It is possible to conclude that the fluoride additions decrease the viscosity of the molten salt and cause an increase in aluminum recovery as the amount of fluoride increases in the molten salt [12]. The best result in adding $KAlF_4$ flux was done at 10wt% with using inert gas during melting process and stirring the molten metal. At fluoride concentrations 10 wt%, the drop in flux-aluminum interfacial tension was roughly. But generally, the salts especially those containing fluorides can destroy and remove the strong and dense oxide skins from the molten aluminum alloys, which can then freely coalesce. Though, that can be provided up to 10% oxide concentrations, at higher concentrations some forms of mechanical agitation are also required because the coalescence is extremely impaired due to the viscosity changes [13].

3.5.2. Effect of adding NaCl- KCl flux

It can be seen clearly from Fig.2. the effect of adding NaCl- KCl fluxes on melting process, the ratio of adding NaCl - KCl fluxes in the range of (0 - 10) , the increasing in amount of NaCl- KCl fluxes at melting process leads to increasing the melting efficiency of aluminum. This may be related to the fact that Li, Ca, Ho, Sr, Dy, Dg, La, Mg and Ce [14]. (NaCl, KCl) fluxes added to separate the surface attached to impurities, to enhance coagulation of drops of melt, and to protect the bath surface against oxidation. All the chloride salts formed are less dense than liquid aluminum, and will float and accumulate at the dross layer on top of the melt. Figure 2. shows that the best result is at 10wt% of NaCl - KCl flux, and the addition was done with using inert gas (Argon) and stirring the molten metal.

3.5.3 Effect of adding $AlCl_3$ flux

The effect of adding $AlCl_3$ during melting process was studied, and the ratio of addition is in the range of (0 - 10) The results represented in Figure 2. shows that increasing the amount of $AlCl_3$ fluxes in melting process to 10wt% had a negative effect on the melting efficiency of aluminum but the good result was at 5wt% with $KAlF_4$ flux and NaCl - KCl fluxes.

3.5.4. The effect of temperature

The temperature effect on the melting of aluminum by using electric furnace was investigated. The results showed that the temperature had a negative effect at 800°C on the melting of aluminum, as shown in Fig.2. No satisfactory explanation is offered for the decrease in the length of the spirals resulting from superheating to 800°C. Gas absorption by the molten aluminum during melting was suggested in the previous work as a possible explanation of this phenomenon. At the present time, it is generally accepted that hydrogen is the gas most readily absorbed by molten aluminum. Methods of removing hydrogen from molten aluminum or aluminum alloys with chlorine, chlorine mixed with nitrogen, or chlorine compounds have been studied by a number of investigators [15].

4. Conclusions

In this work, the main following conclusions can be obtained:

- 1- Melting technique of salt slag that obtained from General Company for Electrical and Electronic Industries and General Company for Mechanical Industries can be successfully applied.
- 2- The effect of fluxes ($KAlF_4$, NaCl, KCl) on the melting process was positive, and increasing the level of these three factors led to increase the melting efficiency of aluminum.
- 3- The effect of $AlCl_3$ flux on the melting process was negative at 10wt% but was good at 5 wt%, and increasing the level of this factor to 10wt% led to reduce the melting efficiency of aluminum.
- 4- The effect of high temperature above 800 °C on the melting process was negative, and increasing the temperature led to reduce the melting efficiency of aluminum.
- 5- Aluminum with high purity was obtained; purity was 99% by electro-refining of impure aluminum by using anhydrous aluminum chloride and NaCl as ionic liquids at low temperature (100°C).
- 6- Taguchi design of experiments (DOE) is proved to be very effective tool to draw a clear picture on the recovering process of aluminum.

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