Extractions of Tin Metal from Electronic Circuit Scrap by Wet Techniques

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Received on: 3/1/2012 & Accepted on: 7/6/2012

ABSTRACT

According to the larger accumulation of electronic equipment, then new environmental techniques (wet extraction process) are studied to treat these scrap and produced a utilized metal such as tin metals that used in different industrial processes.

Different experimental are carried out in this studied in order to optimize the production of Tin metal from scrap circuits (stannous chloride) such as temperature of reaction (30, 35, 45, 55, 60 c°) concentration of scrap introduced (5.0, 7.0, 10.0, 13.0, 15% wt.) and residence time through smelting step as (2.0, 2.5, 3.5, 4.5, 5.0 hrs) respectively.

A characterization and monitoring for production metals occurred by the use of x-ray scanning electron microscope to scan the internal structure of Tin metal, also an atomic absorption to determine the concentrations of Tin agglomerates.

The results show that; optimum conditions that produced high percent of Tin metal from stannous chloride are (53°c temperature of residence, 8.5 wt% concentration of $SnCl_2$ and 4.7 hrs residence time for reduction step at 2°c/min rating ratio). Afterward for smelting stage are (388°c curing temperature and 5 hrs residence time).

Also the characteristic results proved that; agglomerate of Tin metal products appeared at these optimum conditions at x-ray and atomic absorption as 0.25 wt. % for standard blank metal as Cu and clear agglomerate of Tin metal in the electron microscope.

Keywords: Tin Agglomerates, Extraction, Characterization, Wet method

استخلاص معدن القصدير من نفايات الدوائر الالكترونية باستخدام التقنية الرطبة

الخلاصة

بالنظر الى زيادة المتراكم من الاجهزة الالكترونية قادنا ذلك الى دراسة معالجة هذه النفايات باستخدام تقنيات بيئية جديدة (عملية ا لاستخلاص الرطب) وانتاج مواد معدنية مثل معدن القصدير المستخدم في مختلف العمليات الصناعية.

اجري العديد من التجارب المختبرية في هذه الدراسة وذلك للحصول على اكبر كمية منتجة من معدن القصدير من النفايات الالكترونية وذلك وفق المتغيرات التجريبية وهي على

https://doi.org/10.30684/etj.30.20.11
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التوالي درجة الحرارة عند (30 ،35 ،45 ،55 ،60 م°) وتركيز نفايات للمعالجة عند (5 ،7 ، 10 ، 13 ، 15% نسبة وزنية) وزمن ابقاء عند (2، 2.5، 3.5، 4.5 ، 5 ساعة).
وتم تقييم وتصوير المنتج النهائي وذلك باستخدام جهاز الـ x- ray والمجهر الماسح الالكتروني (SEM) وجهاز الامتصاص الذري للعناصر، لتحديد تركيز القصدير النهائي.
ان النتائج التجريبية اثبتت; ان افضل ظروف مثالية تم الحصول عندها على منتوج عالي لمعدن القصدير من كلوريد القصدير هي عند 53°م وتركيز نفايات معالجة عند 8.5 % وزمن بقاء عند 4.7 ساعه وبمعدل اختزال 2 درجة / دقيقة. بعد ذلك مرحلة التقسية للمعدن عند 388 °م وزمن 2 ساعة.
وقد اثبتت نتائج الفحوصات ايضا وجود تكتلات واضحة عند هذه الظروف المثالية وكما اوضحته نتائج الـ x-ray والامتصاص الذري للعناصر وعند تركيز 0.25 نسبة وزنية للمنتج مقارنة بنموذج قياسي اضافة لما اوضحته الصور الملتقطة باستخدام المجهر الماسح الالكتروني (SEM).
الكلمات المرشدة: القصدير، استخلاصة، خصائصة، الطريقة الرطبة.

INTRODUCTION

According to Tin plate mills were manufacturing plants generate a large amount of scrap Tin plate, which is handled in detaining plants by passing chlorine gas through dried Tin scrap, the product Tin tetrachloride was distilled off under controlled temperature to other Tin compounds and returned other metal such as iron steel mills as high- grade scrap for the open- hearth furnaces [1].

Recently a preparation of Tin or its compounds chemically occurred by use of wet extraction by action of hydrogen chloride gases or chlorine gases, the stannic chloride collected as fuming liquid at 100°c mouline and labootte, this new method technique used for recovery of Tin from scraps of Tinned plate, and also for preparing Tin salts from the cutting of Tin-plate sheet or scrap of dye-works. Svedberg [2] obtained colloidal solution of Tin in organic solvents by suspending Tin-foil in the solvent and using iron or aluminum electrode at potential difference 110 volts.

Krajewski [3] have been studied a process for recovering Tin Oxide from Sulfide low-grade, low concentration of Tin are prepared by decomposition KOH at high temperature, then leached with water, then separated electrolytically in one or more stages unpurified Tin is prepared according to this method due to the evaporation of impurities in dehydration stage.

Scott [4] has been investigated the recovery of tin and other metals from scrap printed circuit boards [PCBs] by combination of both leaching and electro deposition. The deposition of Tin was obtained as adherent of the stainless steel cathode at lower current densities, high current densities to prepare a spongy structure of Tin metals at high efficiency of conversion at 95%.

Finally both Ino and Kawamura [5,6] have been studied the preparation of tin metal from sludge during a halogen type electrolytic tin plating, then alkalifying the filtrate to obtain a tin in the first separation step as deposit of Tin-sludge, then re-dissolving in alkali solution and electrolytic reduction to prepare a high purity of metallic tin with high yield.

Maslii A. [7] has been studied a two – stage technological scheme of Copper, nickel, Tin, Zinc and Chromium ion extraction from industrial effluent.

Errol F. [8] who wrote a report of preliminary assessment of the N Z (North Zone) in early 2010, about the production and sale of Tin metal from the N Z due to the recent advances in small scale pyrometalhurigical (smelting) technologies.

Sargar B. [9] has been developed the solvent extraction of Tin (IV) from 8M HCl with 4% N – noctylaniline, and Tin (IV) from the organic phase was determined spectrophotometerically with pyroocatechol violet of 550 nm.

Braz J. [10] presented the experimental results for the leaching of printed circuit boards (PCB) from obsolete computers for extracting and recovering Tin and Copper by means of leaching followed by precipitation were these waste cut in to small pieces and fed in to cylinder mill. The powder obtained was leached by using aqueous sol. 2.18 H_2So_4 2.8 N H_2 So_4 + 3 N HCl, 3 N HCl (2.7% for Sn and lower than 0.01% Cu with extraction percent of 98% for Sn and 93% for Cu.

This work aimed at the Preparation of useful metal from scrap wastes as an environmental solution for this problem. Then optimization of best conditions for preparation method such as temperature of reaction, residences time, concentration of scrap solution, and heating rate in order to get high quantity and high purity of Tin metal. Monitoring the final product to estimate the internal structure of agglomerate Tin metal by the use of electron scanning microscope, x-ray, and atomic absorption.

EXPERIMENTAL WORK

Materials

1- Stannous chloride, $SnCl_2.2H_2O$ from scrap printed circuit of $SnCl_2.2H_2O$, 25.6g/mole, Al 26.9 g/ mol, Sn 118.7 g/ mol and $AlCl_3$ 133.3 g/mol.
2- Aluminum powder of (88.7%) purity and 53 μm particles size by BDH.

Procedure

The preparation of Tin metal from scrap stannous chloride achieved by two stages reduction and smelting process at different reaction properties due to the reaction below:

$$3SnCl_2.2H_2O_{(aq)} + 2Al_s \text{ -----> } 3Sn_s + 2AlCl_3 + 6H_2O \quad \ldots\ldots (1)$$

The first stage of preparation (reduction stage) has been achieved by dissolving (50) g of stannous chloride ($SnCl_2.2H_2O$) with 900ml of $H_2O\ at$ continuous mixing using magnetic stirrer until complete homogeneity of 5% stannous chloride solution was attained. Then a reaction vessel was installed by immersing the thermometer and cooling coil inside reaction vessel and connecting the chiller with the cooling coil afterward filling the reaction vessel with a specified composition of stannous chloride solution (50g) with heating and mixing, then an addition of aluminum powder was incorporated (3-8g) with controlling the temperature of reaction in order to avoid the side reaction by adjustment heating plate of controlling thermostat and cooling the reaction container by chiller cooler in order to fixed the temperature during the experimental period. At the end, the agglomerates that precipitated in to the solution, then separated by washing reactant solution with deionized water. Then agglomerates were transferred inside the drying oven and kept there for 2 hrs at 100°c. The dried agglomerates were

weighted and analyzed by flame atomic absorption to measure the percent of Tin in the final agglomerates differed in size and color at 30mm size and color from dark gray to shying silver as shown in Figure(1).

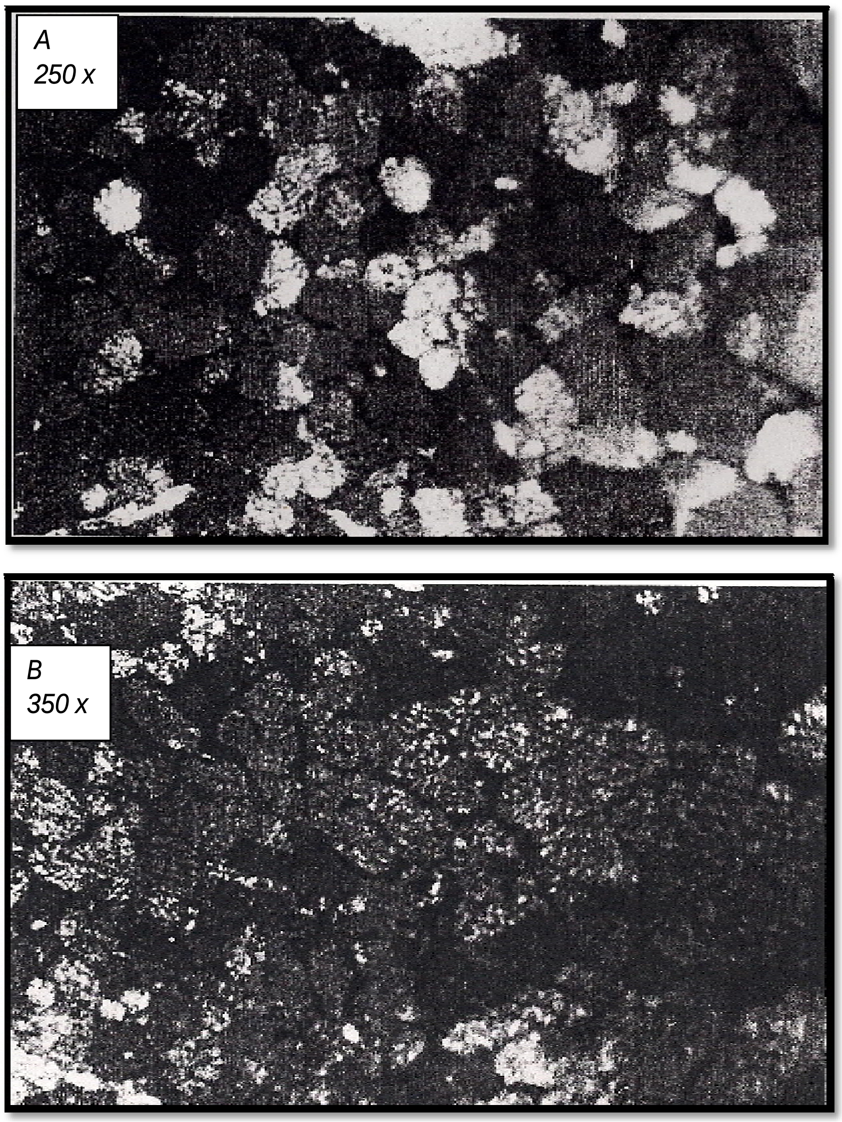

Figure (1) A, B crystalline structure of Tin agglomerate at magnification force 250x, 350 x for reduction and smelting stages (Olympus PMG3, TOYO Japan).

Afterward the second stage has been achieved (smelting step) by cleaning of the crucible and weighting the agglomerates of known Tin composition before placing it inside the electrical furnace Nabertherm type, model L08/14, Germany (under controlling it temperature by electronic program to get a desired rate of heating 2 ̊c/min and suitable residence time (5 hrs) for prepared sample. At the end the content of the crucible was transferred out from the furnace and sieving the content by the use of shaker series Type RETSCH, Germany and sieve of 150 µm size to separate the Tin metal from the slag material with sieving for 2 hrs, eventually the metal was separated and weighted. Then its purity was measured by the use of atomic flame analysis.

In order to optimize the extraction reaction process with final amount of extracted Tin metal therefore different experimental conditions were used as shown in Table(1). The experimental apparatus shown in Figure (2).

Table (1) the experimental condition that studied for the extraction system.

Exp. no.	Conc.(wt.%) of stannous chloride	Temperature of reaction control (°c)	Time of reaction (hr)
1	5	30	5.0
2	7	35	4.5
3	10	45	3.5
4	13	55	2.5
5	15	60	2.0

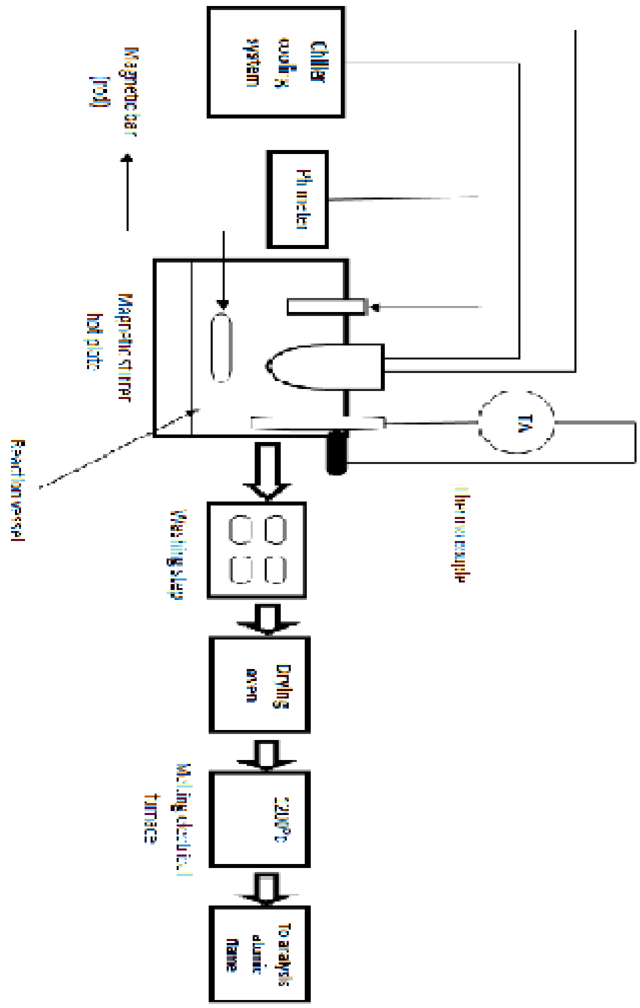

Figure (2) Experimental apparatus of extraction and smelting system for Tin metal from scrap circuits.

Then the final dried products were characterized by using different characterization equipments such as (x-ray Olympus PMG3 TOYO/ Japan, flam atomic absorption Shimadzu model AA- 670/ Japan, SEM Olympus PMG TOYO/ Japan) in order to monitoring the Tin metal and estimate the internal structure at these preparation conditions before application in industrial processes.

RESULTS AND DISCUSSION:

Effect of temperature on the yields of Tin metal:

Figure (3) shows the effect of temperature on yield of extracted Tin.It was notice that raising temperature accommodates to high amount of extracted Tin until

54°c been reached then the temperature reverses its action. This ascribed to retardation of the main reaction, beyond this

optimum temperature results in accelerating other side reactions (reaction of HCl with forming stannous chloride). The characteristic of the reduction reaction in somehow may stimulate as reaction that occurred an electro chemical cell, where stannous ions play as cathode while aluminum powder plays as an anode. Increasing the temperature would accelerate the reaction in both electrodes and higher temperature would accommodate with high hydrogen over potential at the cathode electrode that resulting in decreasing the resulting in decreasing the reduction- extraction reaction [6, 11].

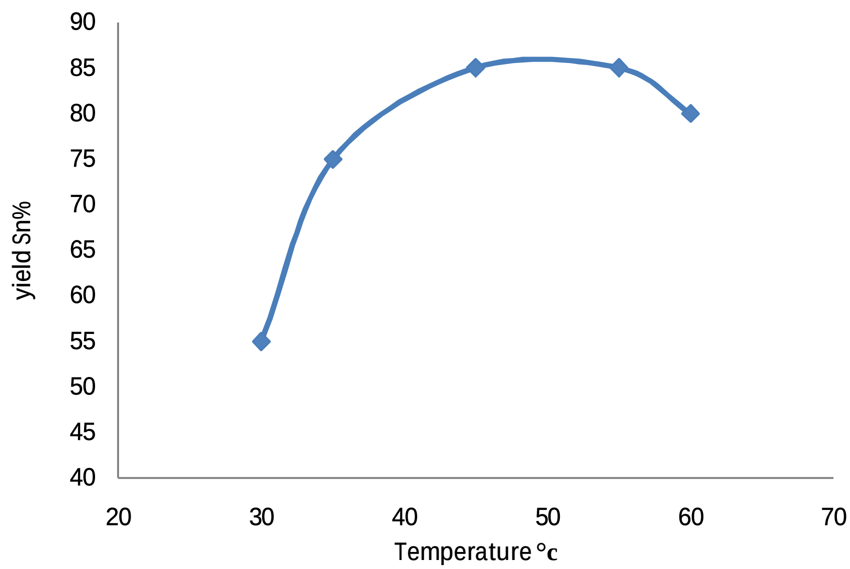

Figure (3) shows the effects of temperature on Sn yields.

Effect of salt concentration on the yield of Tin metal

Figure (4) shows the effect of stannous chloride concentration on the yield of extracted Tin at optimum condition of temperature and time. It is found that highest amount of Tin was resulted at 8.5wt% concentration of stannous chloride afterward the decrease in amount of Tin would accommodate with concentration increase. Higher concentration that 8.5wt% at causes higher concentration of hydrogen ion (lower PH value) which in return decreases the reactivity of aluminum powder according to evolution of hydrogen gas (hydrogen over potential) [11].

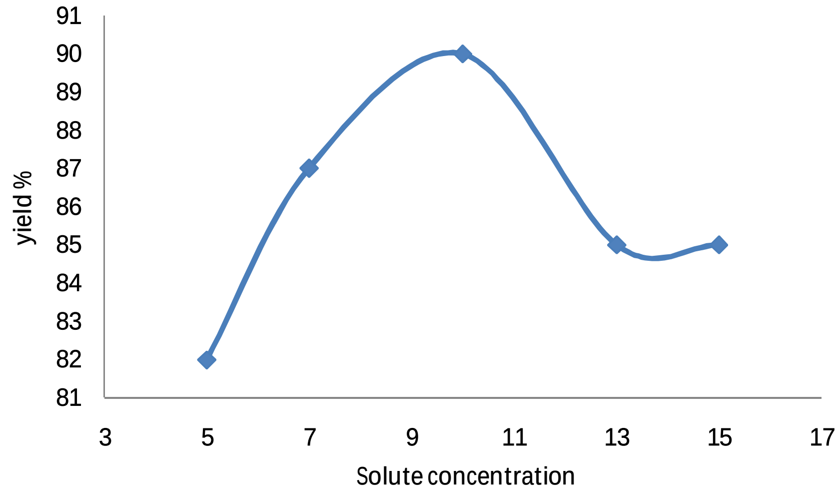

Figure (4) Shows the effects of stannous chloride concentration on Sn yields.

EFFECT OF RESIDENCE TIME ON THE YIELD OF TIN METAL

Figure (5) illustrates the effect of reaction time (estimates by initial addition of aluminum powder) on the production of extracted Tin at optimum conditions of temperature and concentration. It was clearly notified that at 4.7 hrs maximum amount of Tin in the agglomerates was achieved. Higher times show no further improvement. Interaction effects between the operating parameters were studied as well pictorial representational of these interaction were observed in figures (6, 7, 8).

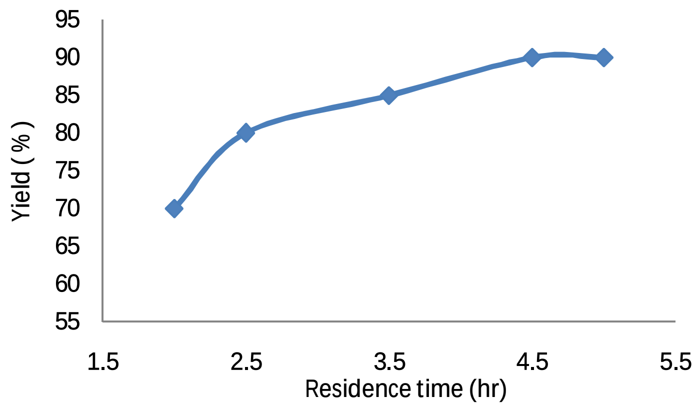

Figure (5)Shows the effects of residence time on Sn yields.

Figure (6) shows the interaction between temperature and stannous chloride concentrations on the production of extracted Tin at optimum reaction time. At low level of temperature 30̊ c this dependence tends to contract the increasing influence of concentration that resulted to decrease the amount of Tin with higher concentration of stannous chloride while at lower temperature the reaction would retard until the net effect become decreasing.

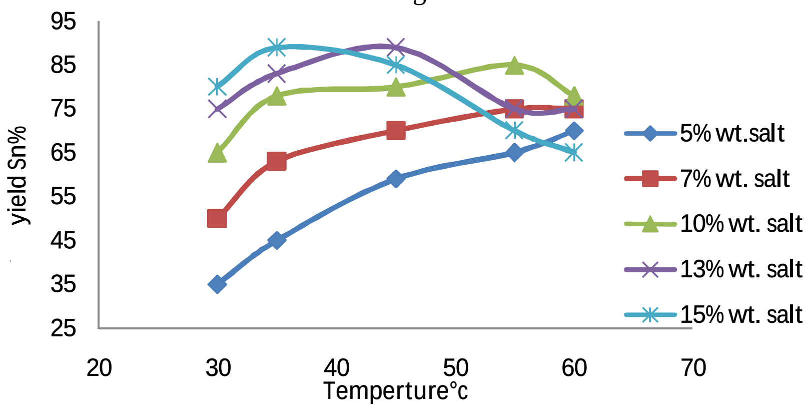

Figure (6) Shows the effects of temperature and salt concentration on Sn yields.

Figure (7) demonstrates the interaction effect between temperature and time on the production of extracted Tin in the agglomerates. It was found that at higher temperature an increase in amount of Tin with time occurred while at lower temperatures a decrease in amount was attributed to slower the rate main reaction side reactions and figure (8) shows the interaction effect between stannous chloride concentration and reaction time on tin conversion, It was found that slight increase in Tin amount occurs with increasing time at different concentrations.

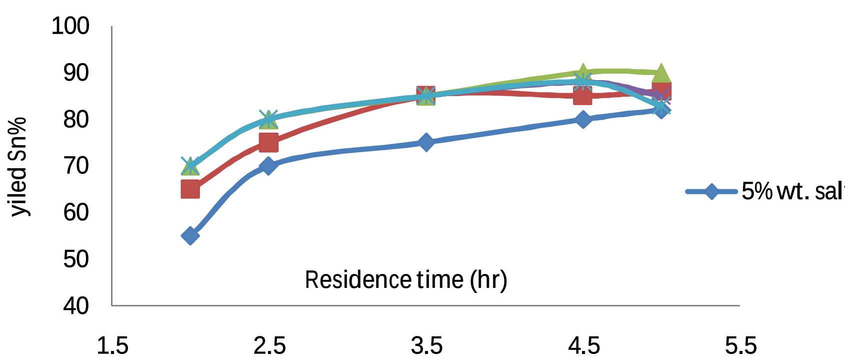

Figure (7) Shows the effects of temperature and residence time on Sn yields.

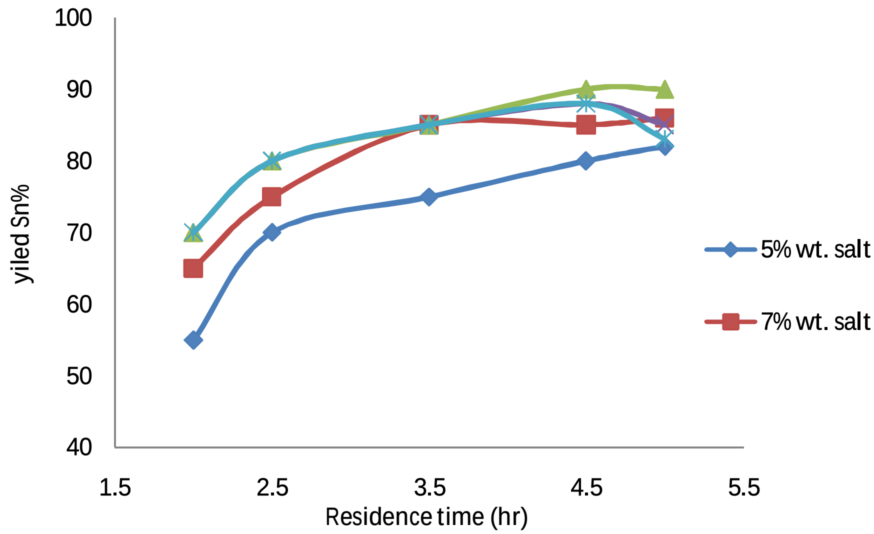

Figure (8) Shows the effects of salt concentration and residence time on Sn yields.

ANALYSIS OF EXTRACTED TIN PRODUCTS

Atomic flame analysis

A flame atomic absorption spectrophotometer has been used for determination of Tin products after reduction extraction stage, also for determination of impurities in the remaining slag where Tables (2, 3) show the products and slag analysis at optimum conditions.

Table (2) shows the Sn extract analysis:

Sample no.	1	2	3	4	5	6	7	8	9	10
Metal wt.% Sn	64	61	72	65	65	69	74	80	92	98

Table (3) shows the slag analysis for optimum sample no. (10):

Sample no. 10	Zn%	Cu%	Fe %	Ni%	Co %	Ag %	Cr %	Mn %	Pb %	Cd %
Wt. %	0.03	0.25	0.07	0.011	nil	0.08	nil	nil	nil	0.11

The above Table (2) shows high percentage and concentration accurate optimum conditions (53°c, 8.5 % wt. and 4.7 hrs) of reduction extraction process.

X-RAY ANALYSIS

This analysis has been achieved by use of x-ray spectro-photometer, to identify the phases of Tin product. The results show that clear phases of Tin metal appeared at length angle "x" (30.8, 32.2, 34.7, 43.5, 44.0, 45.1, and 55.5) as shown in Figure (9). And Figure (10) shows the impurities slag analysis for phases that results appeared no metal found in remain slag at length angle "x" (26.7, 30.6, 32, 33.2, 34, 38, 51, 54.9, 58) respectively.

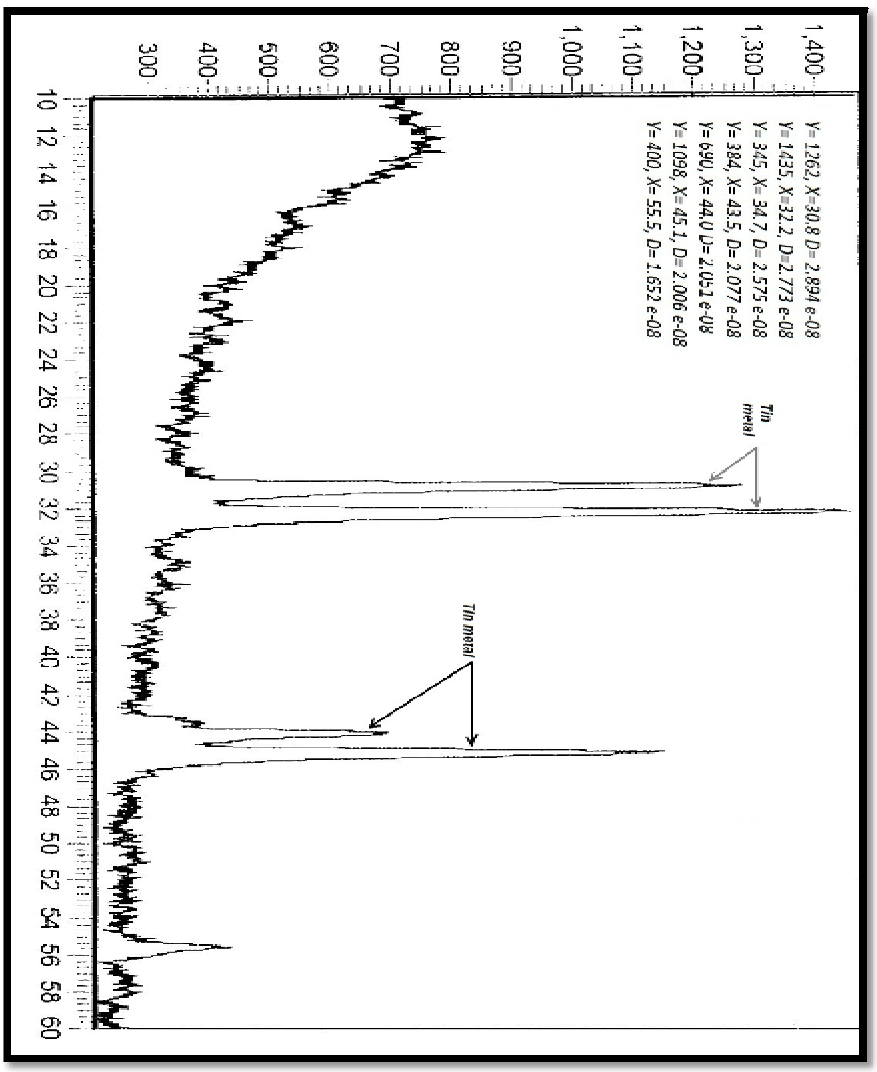

Figure (9) Agglomerates after reduction stage.

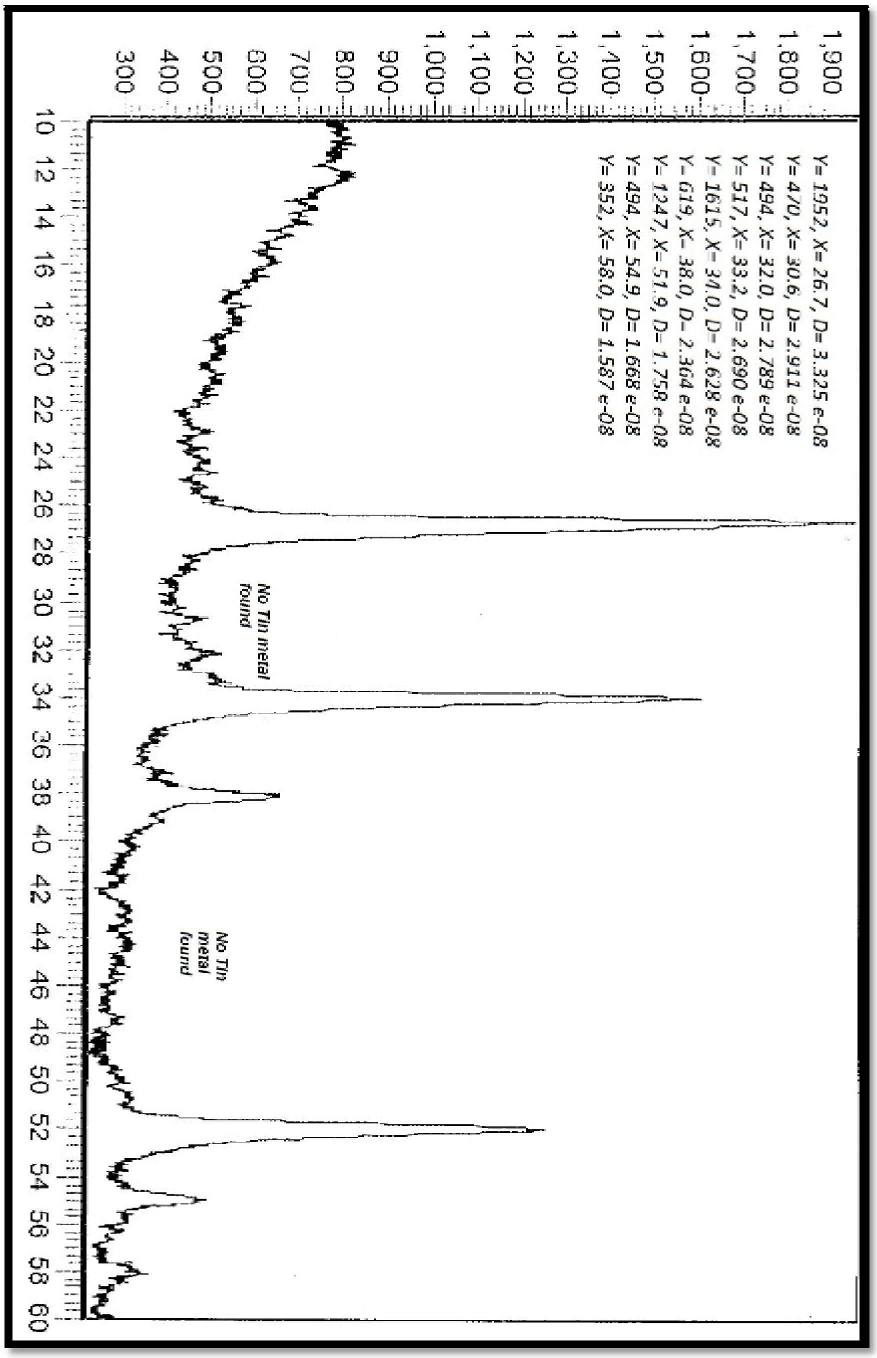

Figure (10) x-ray slag after the smelting stage.

CONCLUSIONS

From the above studied it could conclude that:

1- Optimum conditions were predicted and found equal to Tin the reduction Extraction process to temperature of 53°c concentration of stannous chloride of 8.5% wt and reduction time of 4.7 hrs which produced a Tin metal of 98% wt.

2- Higher yield of Tin metal related to lower rate of heating 2°c/min and higher temperature 388°c and residence time 5 hrs for smelting stage.

3- Higher yield of Tin in the smelting step was matched with lower rate of heating 2°c/min and higher temperature 54°c and residence time 4.7 hrs. Although temperature of smelting has the significant attributable to the productivity of Tin in comparison to the other studied variables.

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