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Experimental Study for the Influence of Operating Parameters on Copper Electrorefining Process

Hayder M. Abbas and Sarmad T. Najim

Chemical Engineering Department – College of Engineering – Al-Nahrain University

Abstract

Copper electrodeposition by electrorefining process in acidic sulfate media contains 40 g/l of cupric ions and 160 g/l of sulfuric acid was achieved to study the influence of the operating parameters on cathode purity, surface morphology, deposition rate, current efficiency and power consumption. These operating parameters and there ranges are: current density 200, 300 and 400 A/m^2, electrolyte temperature 35, 50 and 65 ^{o}C, electrodes spacing 15, 30 and 45 mm and electrolyte residence time 6, 4 and 2 h were utilized. XRF, SEM and EDX analyses were attained to clarify the properties of the produced cathode.

Keywords: Copper electrorefining process, Purity, Morphology, Deposition rate, Current efficiency, Power consumption, Electrolyte residence time.

1- Introduction

Metals are absolutely necessary to reinforce economic development and the work of modern civilization. A metal demand is growing beside the worldwide inhabitance increases and metals are utilized in a larger range of industries, especially connected with the spread of novel technologies [1].

Copper market is one of the biggest of all metals backwards iron and aluminum. Copper acting a considerable role in the manufacturing production because it is simple to work with, effective electricity and heat conductivity and it has a good corrosion resistance [2]. Pyrometallurgical process was achieved 80% of global copper production [3].

In the electrorefining cell the casting anodes were dissolved in the electrolyte solution and the electrical current was applied to deposit the copper as a pure metal on the cathode [4]. Additional benefit was gained from process by the recovery of cells slime to produce valuable metals like selenium, gold, tellurium and silver [3].

The continuously huge demand leads to improve the copper electrorefining process, for example in the last years in order to raise the production rate, the current density was raised to high values with keeping of copper purity level, also improvement was employed by modified the design of the electrorefining cell, the electrolyte movement control at the electrodes surface, optimized the quantities of the additive regents and advanced of substitute additives [5].

The electrorefining process occurs in a cell in which the comparatively impure casting anodes are hanging in an acidic electrolyte every other among soft pure copper starter sheets which will come to be the cathodes.

On applied copper electrorefining processes, acidic electrolyte is used exclusively, the usage of acidic electrolyte for the reason that power and chemicals costs are low and on account of the control of these solutions are simple and forward straight [6,7].

Anode must be connecting to the positive pole of the electrical supply, whereas the pure copper starter sheet must be connecting to the negative pole of the electrical supply. When the electrical supply is turn on, the atoms of copper will lose electrons and diffused to the cathode through the electrolyte, then it will deposit as pure copper, for every ion deposited onto the cathode, else ion passes in the electrolyte from the anode. Finally the anode is spent and its impurities either stay dissolved in the electrolyte or drops to the bottom of the cell creating the slime, by this process pure copper cathode is yield and other expensive metals existing in the anode like platinum, silver, gold, or palladium, can be recovered from the slime [6].

The major electrical factor that can influence the purity of yield cathode is the current density. The universal direction in copper electrorefining process is to work with highly level of current densities; this decreases the size of plant for a new system and growths the production rate for old plant [8].The electrolyte heating is costly but its valuable influence in copper electrorefining process [9]. Electrodes spacing play an important role in the copper electrorefining process, so it has to be set to improve the specifications of produced cathodes [10]. The rate of circulation must realize a minimal change of cell electrolyte every 4-6 h.

Corresponding Authors: Hayder M. Abbas, Email: hayder_mahdi@yahoo.com, Sarmad T. Najim, Email: dr_sarmadalani@yahoo.com

A steady slow electrolyte movement in the cell is needful for increase the mass transport, settle down of anode slimes at the cell bottom, make a good control to electrolyte temperature and carry off the dissolving contaminations from the cell [11].

The main objectives of present work are: design, construct and install of a laboratory electrorefining cell, study the influence of several operating parameters on cathode purity, surface morphology, deposition rate, current efficiency and power consumption, also optimizing the operational conditions of process by obtain the optimum operating parameters that provide the finest quality and quantity of the produced copper.

2- Theory

The fundamentals of electrorefining process based on electrochemical kinetics philosophies [12]. Like every electrodeposition processes, the electrodeposition of copper is an electrolytic process that is created from electrochemical reduction reaction on cathode, so the reducing copper produce is deposited on the cathode surface. If other (undesired) reactions are do not happening in the same time, at that point it can assume that the reaction at cathode in the electrolyte solution is only the desired copper reduction.

$$\mathrm{Cu} + 2 + 2\mathrm{e} - \qquad \mathrm{Cuo} \quad \mathrm{Eo} = 0.337\ \mathrm{V} \tag{1}$$

The copper electrodeposition process contains many steps. Principally the steps are movements of reactive ions from electrolyte to the surface of cathode, adsorption of the ions, transfer of the charge at the cathode surface, cathode surface diffusion and metal deposition, nucleation occurs first and then developing into crystal structure [13,14].

Copper electrodeposition process is a complex heterogeneous system, all electrochemical events take place in three thin layers whose properties dissimilar from those of the bulk electrolyte. The Helmholtz double layer consist of the inner and outer Helmholtz layer, the double layer consist of specially adsorbed cations and molecules of water. Proceeding from the Helmholtz double layer in the direction of the bulk electrolyte are the diffusion layer followed by the hydrodynamic layer. Inside the diffusion layer, the cations concentration variations from that of the electrode surface to that of the bulk electrolyte. The diffusion layer does not change position but with increasing the flow rate of bulk electrolyte, its thickness will reduced to permit greater rates of the electrochemical reaction, theoretically the thickness of the diffusion layer is inversely proportional to the square root of the flow rate.

The hydrodynamic layer or Prandtl layer has the similar composition like the bulk electrolyte, but the movement of the electrolyte decreases from that of the bulk electrolyte to the motionless diffusion layer [15].

The theoretical deposit cathode weight $W_{theo.}$ can be calculated from Faraday's law;

$$W_{theo.}\,(g) = \frac{I.t.A_w}{Z.F} \tag{2}$$

where I is the applied current, t is deposition time, A_w is copper atomic weight, Z is electron valency and F is Faraday's constant.

The actual cathode deposition rate ACDR can be calculated by divided the actual deposit weight of cathode over the deposition time and the deposition area [16].

$$\mathrm{ACDR}\ (g/h.m2) = \frac{W_{acut.}}{t\,.\,A} \tag{3}$$

The current efficiency CE% represented the proportion of current transient over an electrolytic cell in order to achieve the wanted electrochemical reaction. It can be calculated by the following equation [8]:

$$\mathrm{CE}\ (\%) = \frac{W_{acut.}}{W_{theo.}} * 100 \tag{4}$$

However, there is a continuous need to minimizing power consumption by reducing cell voltage, by increasing current efficiency and keeping good electrical connections during the electrorefining process [17]. The power consumption can be calculated from the following equation:

$$P\ (kW.h/kg) = \frac{V_{cell}.I.t}{W_{acut.}} \tag{5}$$

where V_{cell} is the cell voltage.

3- Experimental Work

Two parts consist of 33 experiment were achieved. Electrolyte was prepared by using annular sulfuric acid supplied by BAKER company with specific gravity 1.84 and purity 97%, diluted with distilled water to obtain an electrolyte solution of 160 g/l, then 160 g of copper sulfate pentahydrate supplied by Shanghai Mintchem Company with purity 98%, added to one liter of this electrolyte solution and stirred to gain 40 g/l of cupric ions concentration.

Figure1. represents the experimental apparatus were utilized in the present work. A rectangle lab scale cell constructed from Pyrex glass with active volume of 720 ml was used. The starting cathode sheet was made of pure copper with total area of (100*80*mm) and immersed area of (70*70 mm) with thickness 0.6 mm.

Anode was used with a copper content of (98.48 % wt. Cu) and with total area of (100*80*mm) and immersed area of (75*80 mm) with thickness 6.5 mm.

Before every single experiment, three steps was done for electrodes preparation, the first step was polishing with (220, 320 and 800) grades of sandpaper in order to eliminate film oxides & any contaminations on the surface, in the second step ethanol was used to remove any grease, in the third step electrodes were immersed in (1/5) volume ratio of sulphuric acid to distilled water mixture, after finishing of each steps the electrodes were washed by distilled water and dried by gauze. The electrodes saved inside the desiccator over-night before utilized. The electrical circuit contains a digital DC power supply connected with the cell and ammeter in series, while the voltmeter connected in parallel with the cathode and the anode respectively.

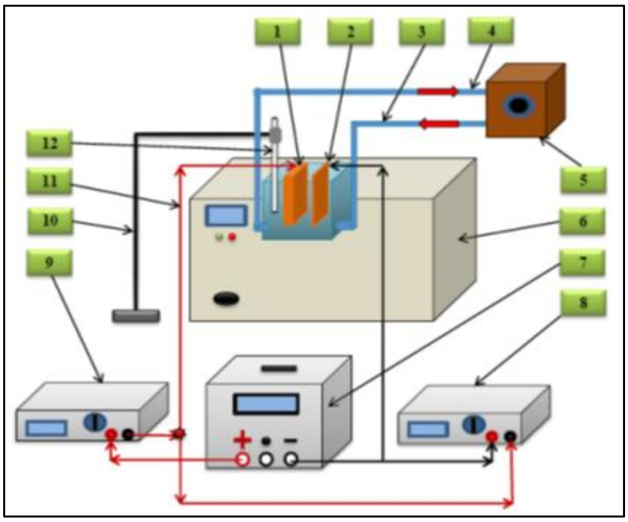

Fig. 1. Schematic diagram of experimental apparatus: 1- Anode, 2- Cathode, 3- Electrolyte input line, 4- Electrolyte output line, 5- Dosing pump, 6- Water bath, 7- Power supply, 8- Voltmeter, 9- Ammeter, 10- Stand, 11- Electrical wires, 12- Thermometer.

Experimental program in part one was to study the effect of current density (CD) 200, 300 and 400 A/m^2, electrolyte temperature (T) 35, 50 and 65 ^{o}C and electrodes spacing (S) 15, 30 and 45 mm with stationary electrolyte on cathode purity, surface morphology, deposition rate, current efficiency and power consumption and obtained the optimum operating parameters, while the second part deals with studying the effect of electrolyte residence time ERT 6, 4 and 2 h on optimum operating parameters obtained from part one. Tables 1 and 2 are summarizing the operating parameters of parts 1 and 2.

Table 1. Summary of the operating parameters for part one

Exp No.	CD A/m^2	T °C	S mm	Exp No.	CD A/m^2	T °C	S mm
1	200	35	15	15	300	50	45
2	200	35	30	16	300	65	15
3	200	35	45	17	300	65	30
4	200	50	15	18	300	65	45
5	200	50	30	19	400	35	15
6	200	50	45	20	400	35	30
7	200	65	15	21	400	35	45
8	200	65	30	22	400	50	15
9	200	65	45	23	400	50	30
10	300	35	15	24	400	50	45
11	300	35	30	25	400	65	15
12	300	35	45	26	400	65	30
13	300	50	15	27	400	65	45
14	300	50	30				

Table 2. Summary of the operating parameters in part two

Exp. No.	CD A/m^2	T °C	S mm	ERT h
28	200	65	15	6
29	200	65	15	4
30	200	65	15	2
31	200	65	30	6
32	200	65	30	4
33	200	65	30	2

4- Results and Discussion

4.1. Part One

Table 3 shows the actual cathode deposition rate, current efficiency and power consumption values resulted from this part.

a. Effect of the Current Density (CD)

Increase the current density would exceedingly decrease the cathode purity, the maximum copper purity of 99.59% Cu was obtained in Exp. No. 7. It can be clarified that by increasing the current density leads to increase the anode dissolution rate which increased the amount of impurities, that gives more chances to deposit of the impurities on the cathode surface caused the deterioration of cathode purity.

Table 3. ACDR, CE% and P of part one

Exp. No.	ACDR g / h.m2	CE %	P kWh/kg
1	232.9	98.23	0.6536
2	232.5	98.04	0.8562
3	231.4	97.61	1.0607
4	234.1	98.73	0.6045
5	233.6	98.52	0.8194
6	232.6	98.11	0.9874
7	234.9	99.07	0.5425
8	234.3	98.82	0.6064
9	233.7	98.56	0.7798
10	346.1	97.30	0.8944
11	345.1	97.03	0.9814
12	344.1	96.74	1.1377
13	346.7	97.49	0.7340
14	346.1	97.32	0.8576
15	345.5	97.15	1.0227
16	348.0	97.84	0.6174
17	347.4	97.68	0.8073
18	346.2	97.33	0.9639
19	448.0	94.47	1.1156
20	446.9	94.25	1.1919
21	445.8	94.01	1.2475
22	450.9	95.09	0.9533
23	449.2	94.72	1.0004
24	446.8	94.23	1.0817
25	452.5	95.43	0.6927
26	451.5	95.21	0.8526
27	450.0	94.90	1.0126

As the current density increase the cathode surface morphology extremely decline and becomes rougher, best surface morphology was obtained in Exp. No. 7 as shown in Fig.2 (a, b and c), the probable cause that by increasing current density would promote the nucleation on active sites, and that would leads to creation of a higher number of small grains which gradually agglomerate in higher local current density active sites at the surface of cathode formed a unequal copper deposition thickness.

It can be seen from Fig. 3 that the CE% decreases with increase current density, the maximum CE% obtained in Exp. No. 7, the possible explanation is that by increasing current density the charged spices will increases also and that will increase the probability to initiate side electrochemical reactions.

A positive influence of the current density increasing on ACDR, the desired maximum ACDR obtained in Exp. No. 25, it can be interpreting that increase the surface of cathode formed a unequal copper deposition thickness.

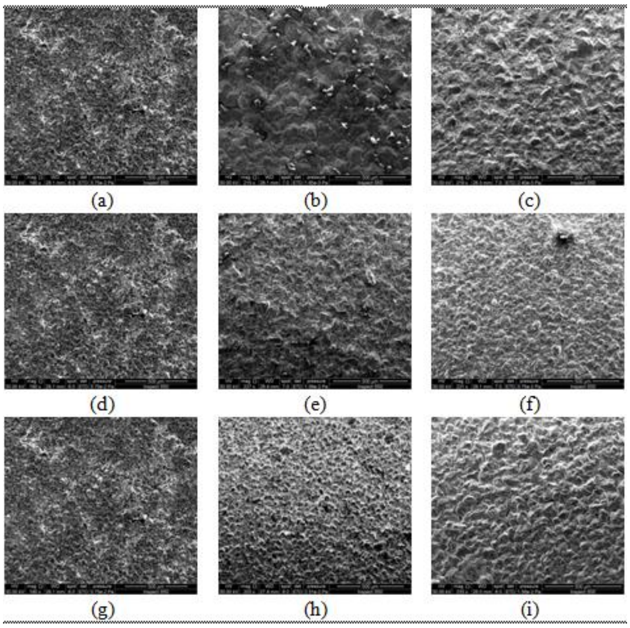

Fig. 2. SEM micrographs show surface morphology effecting by (a, b and c) current density Exp. No. 7, 16 and 25 respectively, (d, e and f) electrolyte temperature Exp. No. 7, 4 and 1 respectively, (g, h and i) electrodes spacing Exp. No. 7, 8 and 9 respectively

It can be seen from Fig. 3 that the CE% decreases with increase current density, the maximum CE% obtained in Exp. No. 7, the possible explanation is that by increasing current density the charged spices will increases also and that will increase the probability to initiate side electrochemical reactions.

A positive influence of the current density increasing on ACDR, the desired maximum ACDR obtained in Exp. No. 25, it can be interpreting that increase of current density would increase intensity of the generating electric field and that would increase the migration rate of the charged spices, beside that the diffusion rate would increasing due to increases of cupric ions concentration on the adjacent anode layer and decrease of cupric ions concentration on the adjacent cathode layer and that is in a good agreement with Najim [18].

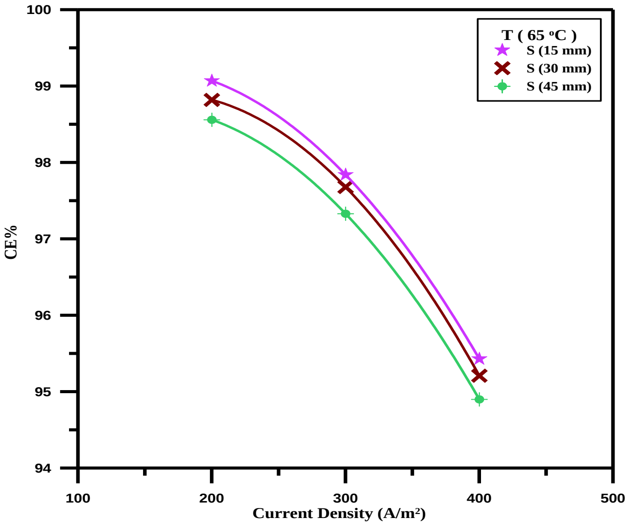

Fig. 3. Effect of current density on current efficiency with constant T 65 ^{o}C and S 15, 30 and 45 mm

The increasing of the current density caused a significant increase for power consumption, the desired minimum P value is 0.5425 kW.h/kg which was obtained in Exp. No. 7, it can clarified that increase of current density leads to increase the cell voltage that causing increase of the power consumption.

b. Effect of the Electrolyte Temperature

As electrolyte temperature increases cathode purity will increase. It can be clarified that increase of electrolyte temperature leads to decrease its density and viscosity, this impact make the bigger contamination particles have a greater settling velocity and will be precipitate to the cell bottom more quickly, so be less probability to deposited on the cathode surface, while the finer contamination particles have a lesser settling velocity will be suspended in the electrolyte, so be more probability to deposited on the cathode surface.

Fig. 2(d, e and f) shows that when electrolyte temperature decreases cathode surface morphology declined and become rougher. The possible explanation that decrease electrolyte temperature will decrease the electrical conductivity of electrolyte and that will hinder the current density distribution in the cell causing unequal spreading of deposit grains over cathode surface producing a rougher deposit.

The increasing of the electrolyte temperature caused reduction in the power consumption as shown in Fig. 4, the increasing of the electrolyte temperature gives rise to minimize polarization of cathode and anode and increase the electrolyte conductivity.

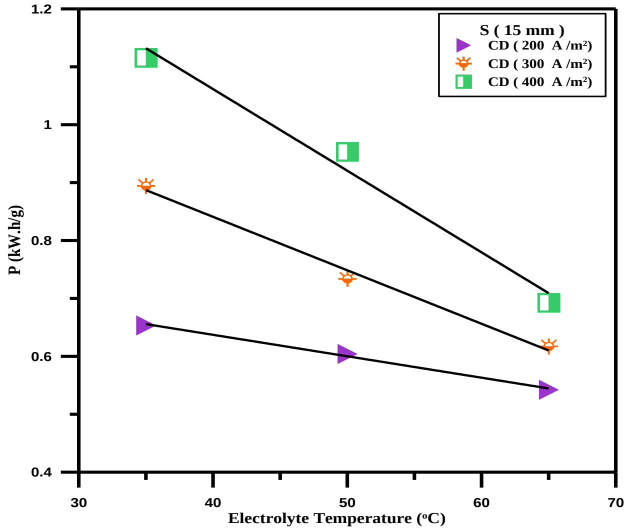

Fig. 4. Effect of electrolyte temperature on power consumption with constant S 15 mm and CD 200, 300 and 400 A/m^2

When the electrolyte temperature increase current efficiency will increase, it can be referred to Arrhenius equation which states that increasing of electrolyte temperature leads to increase the reaction rate of copper deposition, therefore the current efficiency will increase.

Decrease electrolyte temperature decreases ACDR. It can be interpreting that the diffusion coefficient of the cupric ions will decrease with decreasing of the electrolyte temperature, so hindering mass transportation that is in a good agreement with Ibrahim [19].

c. Effect of the Electrodes Spacing

As the electrodes spacing decrease the cathode purity increased very slightly, the possible explanation that the variation in electrodes spacing does not have a significant influence on the natural convection which affect the impurities motion during the process.

When electrodes spacing increase cathode surface morphology become rougher as shown in Fig. 2 (g, h and i), it can be interpreting that the electrolyte resistivity increase with increasing of the electrodes spacing, and that will causing a bad current density distribution through the cell producing unsatisfactory distribution of the deposit grains on the surface creating a rougher deposit form.

It can be observed easily that increase electrodes spacing decrease ACDR as shown by Fig. 5, it can be explained that increase electrodes spacing would increase the electrolyte resistivity and that would reduce the electrical field intensity which drive the charging spices to deposit on the cathode surface.

A negative influence of electrodes spacing increasing on current efficiency, the probable explanation that the unwanted reactions rates may be maximized.

Increase electrodes spacing would increase power consumption, it can be elucidated that electrodes spacing increasing would increase the electrolyte resistivity and that can be maximized the cell voltage due to highly hindering to electrical current passing through the electrolyte. The optimum operating parameters were obtained in Exp. No. 7, the current density 200 A/m^2, the electrolyte temperature 65 ^{o}C and the electrodes spacing 15 mm.

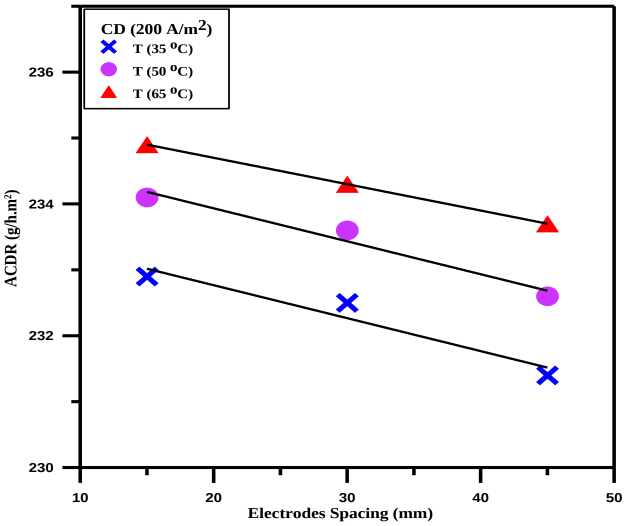

Fig. 5. Effect of electrodes spacing on ACDR with constant CD 200 A/m^2 and T 35, 50 and 65 ^{o}C

4.2. Part Two

Table 4 shows the actual cathode deposition rate, current efficiency and power consumption data resulted in this part.

Table 4. ACDR, CE% and P of part two

Exp. No.	ACDR g / h.m2	CE %	P kWh/kg
28	235.1	99.15	0.4590
29	235.4	99.27	0.4138
30	235.7	99.40	0.3707
31	234.7	98.97	0.5060
32	235.0	99.12	0.4436
33	235.4	99.28	0.4000

Based on logical expectation that the electrolyte circulation with short electrodes spacing 15 mm can deteriorate the cathode purity due to high forced convection of charged contaminations species that may deposit on cathode surface, also short electrodes spacing can increased the chances of short circuit and reduced the current efficiency, hence another electrodes spacing of 30 mm was employed because it gave an acceptable results in part one.

a. Effect of the Electrolyte Residence Time (ERT)

In general, electrolyte circulation increase cathode purity compared with stationary electrolyte state, for the same operating parameters purity was increased from 99.54% in Exp. No. 8 to 99.87% in Exp. No. 31.

Besides that in electrolyte circulation state, decrease ERT more than 4 hours leads to decrease cathode purity, it can be interpreting that the density gradient make an electrolyte loop between the cathode and the anode (natural convection), the electrolyte close the anode is denser than that in facade of the cathode, because the anode is the source of cupric ions by dissolution operation, while it is deposited on the cathode surface, the electrolyte loop is moving with a downward trend over the anode surface and an upward trend over the cathode surface.

When the electrolyte is circulate, a flow pattern with a trend from the bottom to the top will be created, hence the upward electrolyte current close the cathode will be strengthened, upward flow has important influence on contaminations particle conduct, so more probability with lesser ERT to contaminations deposited on cathode surface and deteriorate the purity.

Surface morphology in case of electrolyte circulation is better than electrolyte stationary situations. In addition, as the ERT decrease the cathode surface morphology becomes slightly finer as shown in Fig. 6, it can be clear up that ERT decreases would increase the forced convection and that would decrease the Nernst diffusion layer thickness and permits to produce a smooth copper deposition.

In comparison, the Electrolyte circulation slightly increased mass transport than stationary conditions, ACDR increase from 234.3 in Exp. No. 8 to 235.4 g / h.m^2 in Exp. No. 33.

Also when ERT decrease the ACDR would increase, the feasible analysis that decreasing ERT would increase the forced convection, and that would decreases copper ions near the anode and increases the copper ions near cathode, subsequently the natural convection from anode to cathode would increase and makes a quicker moving of copper ions.

Compare with stationary state, the circulation of electrolyte increase the current efficiency, CE% increase from 98.82% in Exp. No. 8 to 99.28% in Exp. No. 33. Furthermore, It can be observed from table 4 that increasing of ERT would decrease the current efficiency, it can be make clear that increase ERT would decrease the forced convection and that would slows down the speed of copper ions from anode to cathode leads to minimize the reaction rate of copper deposition, therefore the current efficiency will decrease.

As a rule, electrolyte circulation minimizes the power consumption, P decrease from 0.6064 in Exp. No. 8 to 0.4000 (kWh/kg) in Exp. No. 33.

Decrease ERT would decrease power consumption, the possible explanation that the decreasing of ERT would increase the forced convection which enhanced the removing of slimes adhered on anode surface and that would permits to easily passing of current and reduces the cell voltage beside the minimization of electrodes polarization.

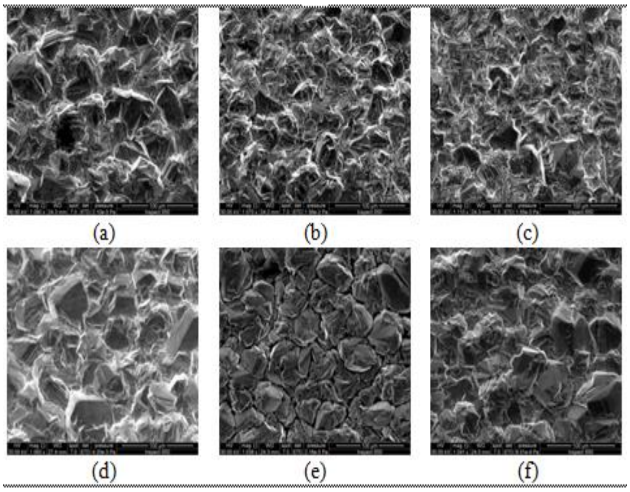

Fig. 6. SEM micrographs show the effect of ERT and electrodes spacing on cathode morphology with CD 200 A/m^2, T 65 ^{o}C, (a, b and c) S 15 with ERT 6, 4 and 2 h respectively, (d, e and f) S 30 with ERT 6, 4 and 2 h respectively

b. Effect of the Electrodes Spacing

When the electrodes spacing increased from 15 to 30 mm with electrolyte circulation the cathode purity would slightly increase, as it happened in experiments 28 and 31when the purity was slightly increased from 99.85% to 99.87%, the possible explanation that the short electrodes spacing with electrolyte circulation will deteriorate the cathode purity due to high forced convection of the charged impurities species that may deposit on cathode surface.

It can be observed that increase of electrodes spacing will decline the cathode surface morphology and become rougher as shown in Fig. 6, it can be clarified that the electrolyte resistivity increase with increasing of the electrodes spacing, and that would generate a bad current density distribution through the cell producing unacceptable distribution of the deposit grains on cathode surface creating a rougher deposit.

It can be notes from table 4 that when electrodes spacing increase ACDR would decrease slightly, ACDR decrease from 235.1 in Exp. No. 28 to 234.7 g / h.m^2 in Exp. No. 31, it can be clarified that increase electrodes spacing would increase the electrolyte resistivity and that would minimize the intensity of electrical field which drives the charging dissolution spices to deposit on the cathode surface.

Increase electrodes spacing would decrease the current efficiency as shown in table 4, CE% decrease from 99.15% in Exp. No. 29 to 98.97% in Exp. No. 31, may be the rates of unfavorable reactions will be maximized.

As electrodes spacing increases the power consumption would decrease as shown in table 4, P decrease from 0.4590 in Exp. No. 28 to 0.5060 kWh/kg in Exp. No. 31, it can be interpreting that the electrodes spacing increasing would decrease the electrolyte conductivity and that can be maximized the cell voltage due to greatly hindering to passing the electrical current through the electrolyte.

Finally, it can be considered that the optimum operating parameters were obtained in Exp. No. 31, with current density 200 A/m^2, electrolyte temperature 65 ^{o}C, electrodes spacing 30 mm and the electrolyte residence time 6 h, all these operating parameters produce a cathode copper with purity of 99.87% wt. Cu, smooth and compact deposit morphology, actual cathode deposit rate 234.7 g / h.m^2, current efficiency 98.97% and power consumption 0.5060 kWh/kg.

5- Conclusions

1- Increase current density will extremely deteriorate cathode purity and morphology, reduce the current efficiency and maximize the power consumption, while it increased ACDR.
2- Increase electrolyte temperature will improve cathode purity and morphology, increase ACDR and current efficiency and minimize the power consumption.
3- Increase electrodes spacing in stationary electrolyte will slightly weaken cathode purity and morphology, faintly reduce ACDR and current efficiency and maximize the power consumption. While increase electrodes spacing with electrolyte circulation from 15 to 30 mm will slightly improve cathode purity only. ;
4- In general, electrolyte circulation improves cathode purity and morphology compared with stationary electrolyte state, furthermore decrease ERT will enhance cathode morphology, rise ACDR and current efficiency values and minimize power consumption, while ERT less than 4 hours leads to drop cathode purity.

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