THE EFFECT OF CONCENTRATION OF GLYCEROL AND ELECTRIC CURRENT ON THE MORPHOLOGY AND PARTICLE SIZE OF ELECTRODEPOSITED CADMIUM POWDER

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Abstract

Cadmium powder was obtained by electrodeposition of cadmium from glycerol and sulphuric acid. The morphology and particle size of these powders were studied. Broken dendrites, intermingled with spongy and irregular particles were observed in the powder. Around 60% of particles were below 100 μm. XRD studies showed that particles with sizes between 212.2 and 303.2 nm were present in the powder. The apparent density of cadmium powder decreased with increase in concentration of glycerol. The stability of the powder and current efficiency were also studied

Keywords: Electrodeposition, Cadmium, Stability , SEM, XRD, Particle size

Introduction

Powder metallurgy method is the oldest method with many advantages. Powder can be produced from pure raw material with good characteristics, and purity can be retained by controlling fabricating steps. This method involves cleaner and quieter operation with longer life of product. In certain applications powder metallurgy process has advantage over other methods. Conventional melting and other standard methods cannot produce sophisticated mechanical parts. Such parts can be produced by powder metallurgy process. A number of patents have been filed on powder fabrication since the early decades of the twentieth century.

Cadmium is a soft ductile metal with a bluish-silvery white crystalline appearance and has many industrial applications. Cadmium is implemented as an alloying element in soft solders and in fusible alloys. It is mixed with copper to produce alloys with increased strength, hardness and wear resistance characters. It is employed

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in alloys which are used in production of bearing materials. It has good resistance for corrosion and is used as resistant plating metal.

Electrolytic preparation of powders of iron, copper, cadmium, nickel, and zinc are among the most extensively studied metals [1-6]. Osborne et al. [7] and Smith [8] studied the electrolytic preparation of copper powder. Hothersall and Gardam [9] reported a method for the commercial preparation of copper powder by electrodeposition. Electrolytic nickel powder properties were studied by Levin [10]. The preparation of finely dispersed nickel was presented by Lashkarev [11]. Electrolytic preparation of zinc dust was studied by Morgan and Raston [12, 13] and Eckardt [14]. Hanseland and Passer [15, 16] performed experiments on electrolytic preparation of zinc powder and described the influence of the variation of the conditions of the electrolysis on the form of deposits. Dutra et al. [17] studied cadmium removal from a diluted aqueous solution by electrorefining technique in a flow cell. Elsherief [18] developed an efficient removal process of cadmium from a dilute acid solution in a flow cell, using rotating glassy carbon and spiral wound steel electrodes. Borikar, Umare and Viswanath [19, 20] studied the effect of acetone on the electrowinning process to obtain copper [19], nickel [20] and cadmium [21] powders.

Viswanath and George reported preparation of cadmium [22] and copper [23] powders in sulphuric acid and glycerol medium. Recently Viswanth and Jachak [24] studied electrodeposition of copper in sulphuric acid and glycerol medium. They found that electrodeposition is combination of electrodissolution of anode by electrocatalytic effect and electrowinning of metal ion. They also reported that copper ion concentration decreased during electrodeposition.

In the present investigation an attempt has been made to study the morphology and particle size of electrodeposited cadmium powder from glycerol and sulphuric acid where the glycerol percentage and electric current were varied.

Eexperimental

Instrumentation

The bath solution ($CdSO_4$, H_2SO_4 and glycerol) is placed into a single compartment of two-electrode cell. The electrodeposition experiments are performed using cadmium plate (15x2.3x0.2 cm) as cathode and anode. Current is supplied by regulated DC power supplier model 34 B DCPS (Testronix). All the experiments are carried out at $(25\pm1)^oC$. The temperature of the bath remained almost constant during the experiments.The current of 0.5, 1, 1.5 and 2 A was applied to the bath. The current variation was $\pm$ 0.01A. The particle size analysis was performed on Saturn Digitizer II 5205 V1.01 and X-ray diffraction (XRD) analysis on Bruker AXSD8 Advance. Morphological analysis was carried out on scanning electron microscope (SEM) JEOL Model JSM - 6390LV, operated at 1 pA to 1mA, at Sophisticated Test and Instrumentation Center, Kochi, India.

Method of cadmium estimation

The concentration of glycerol (taken in volume percent) was varied from 5 to 20%. The cadmium sulphate and sulfuric acid concentration were 0.2 N and 0.5 N, respectively, for all experiments. The total volume of the electrolytic bath solution was made up to 200 ml. Concentration of cadmium sulphate before and after experiment was estimated by titrating bath solution with 0.01 N sodium salt of

ethylenediaminetetraacetic acid (EDTA) using Eriochrome Black-T (EBT) indicator. Volume of bath solution was measured after experiment. The electrodeposition was carried out for 3h to get sufficient quantity of powder for different types of analysis. The deposit was removed by simply scratching the cathode with glass rod. The deposit collected after solution was subjected to decantation. The deposit was ground for about 30 min by adding 10 ml of distilled water. The cadmium powder was separated by decantation and filtration. The powder is dried in a hot air oven at about 90-100°C and stored in air tight bottle.

Symbols abbreviation used in the text

A: Current in amperes,

C_a: Mass of anode dissolved in g atoms

C_c: Mass of metal powder deposited on cathode in g atoms (yield) or electrowinning process,

C_f: Final concentration of Cadmium sulphate in m (molarity),

C_g: Percent by glycerol in volume,

C_i: initial concentration of Cadmium sulphate in m (molarity),

Δc: C_f - C_i,

d_a : Apparent density g ml^{-1},

d_w: Density of water at experimental temperature,

E_f: Electrocatalytic effect efficiency,

f: Number of Faradays = A×t /F,

F: Faraday = 96500 coulombs,

Os: Oxidative stability of cadmium powder,

t: Time in s,

w: Equivalent mass of cadmium,

w_r: relative mass loss of anode,

PFD: Percent of Faraday required for deposition,

PFE: Percent of Faraday required for electrolysis of water.

Results

Physical properties

Determination of apparent density of cadmium powder

The apparent density is given by the following equation:

$$d_a = d_w(W_3 - W_1)/(W_2 - W_1 + W_4 - W_3) \qquad (1)$$

where: W_1 - the mass of dry empty density bottle, W_2 - mass of density bottle and distilled water;

W_3 - the mass of dry empty density bottle and cadmium powder (approximately from 1 to 1.5 g); W_4 - the mass of density bottle with cadmium powder and distilled water.

The apparent density of cadmium powder, obtained at different current levels and percent of glycerol is presented in Table 1. The apparent density may be expressed by the following equation:

$$(0.014A^2 - 0.013A - 0.0935)C_g - (0.856A - 7) \qquad (2)$$

Table1. Apparent density of cadmium powder obtained at different current and percent of glycerol.

Current (A)	Percent of glycerol and apparent density of cadmium ($g\ ml^{-1}$)				
	0%	5%	10%	15 %	20%
0.5	7.2	6.8	6.7	5.8	5.3
1.0	6.7	6.7	6.5	5.9	5.1
1.5	6.4	6.3	6.3	5.2	4.7
2.0	6.4	5.7	5.6	5.2	5.1

Determination of oxidative stability of cadmium powder

Cadmium powder was stored for around fifty days in an air tight sealed bottle then it is studied for its stability. Accurately weighed (0.7 ± 0.3) grams of cadmium powder was taken in 100 ml 0.5 N H_2SO_4.The solution was stirred with a magnetic stirrer for one hour. The solution is filtered and the dissolved cadmium in the filtrate was estimated by titrating the filtrate against standard EDTA 0.01N using EBT indicator. Cadmium powder is susceptible to oxidation and forms cadmium oxide (CdO).This oxide reacts with sulfuric acid and forms cadmium sulphate. However, cadmium does not react with sulfuric acid. The oxidative stability (Os) of cadmium was estimated using the expression:

$$\frac{\text{Weight of metal powder undissolved}}{\text{Weight metal powder taken}} \times 100 \qquad (3)$$

The data is presented in Table 2 represent oxidative stability of cadmium powder obtained at different current and percent of glycerol.

Table 2. Oxidative stability of cadmium powder obtained at different current and percent of glycerol.

Current (A)	Percent of glycerol and oxidative stability of copper (%)				
	0%	5%	10%	15 %	20%
0.5	92.0	88.1	82.9	78.7	73.2
1.0	89.3	83.5	82.4	74.5	72.1
1.5	89.2	82.6	79.1	72.0	67.0
2.0	87.0	81.5	76.1	70.0	66.9

The oxidative stability may expressed by the following equation:

$$Os = (0.8203A^3 - 3.0934A^2 + 3.3732A - 1.9681)\ C_g +$$
$$(-7.7333x^3 + 30x^2 - 38.267x + 105) \qquad (4)$$

Electrochemical studies

Percent of Faraday distribution for the various processes

When the current is passed through cadmium sulphate solution, electrowinning of cadmium, electrolysis of water and warming of the solution (increase of entropy) occur. The current required for warming of solution is very small and so it may be ignored. The percent of Faraday used for electrodeposition (PFD) was calculated from the number of grams deposited on the cathode:

$$PFD = (C_c/w \times f) \times 100 \tag{5}$$

The percent of Faraday (PFE) required for electrolysis of water is:

$$PFE = 100 - PFD \tag{6}$$

The data on percent of Faraday distribution for cadmium deposition at different current and percent of glycerol are presented in Table 3.
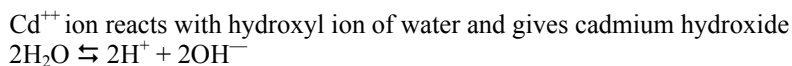
Table 3. Percent Faraday distribution for cadmium deposition and electrolysis of water at different current and percent of glycerol.

Current (A)	Percent of glycerol and percent of Faraday for electrodeposition (PFD) Percent of Faraday of electrolysis (PFE)				
	0%	5%	10%	15 %	20%
0.5	93.6	102	88.4	89.3	103
	6.4	----	**11.6**	**10.7**	----
1.0	94.2	98.4	88.3	89.8	104
	5.8	**1.6**	**15.7**	**10.2**	----
1.5	93.6	85.8	78.4	78.5	95.9
	6.4	**14.2**	**21.6**	**21.5**	**4.1**
2.0	81.6	80.2	71.2	75.0	97.9
	18.4	**19.8**	**28.8**	**25.0**	**2.1**

Reaction mechanism of electrodeposition of cadmium

The PFD value is very high and sometime greater than 100. This may be due to autodeposition of cadmium. However, PFE is very small in number of cases. This may be explained by the following elecrochemical reactions.

The anode is positively charged.

$$Cd = Cd^{++} + 2e \tag{7}$$

Cd^{++} ion reacts with hydroxyl ion of water and gives cadmium hydroxide

$$2H_2O \leftrightarrows 2H^+ + 2OH^- \tag{8}$$

$$Cd^{++} + OH^- = Cd(OH)_2 \tag{9}$$

$$Cd(OH)_2 + H_2SO_4 = Cd\ SO_4 + 2H_2O \tag{10}$$

$$2H^+ + 2e = H_2 \tag{11}$$

Cadmium ion is deposited on the cathode.

$$Cd\ SO_4 = Cd^{++} + SO_4^{--} \tag{12}$$

$$2H_2O \leftrightharpoons 2H^+ + 2OH^- \tag{13}$$

$$2H^+ + SO_4^{--} = H_2SO_4 \tag{14}$$

$$2OH^- = H_2 + O_2 + 2e \tag{15}$$

$$Cd^{++} + 2e = Cd \tag{16}$$

Two gram moles of water are eliminated from the solution when one gram atom of cadmium is dissolved. Two gram moles of water are eliminated from the solution when one gram atom of cadmium is deposited. Also one gram mole of hydrogen is removed from the solution from eq.11. Even though two gram moles of water are formed from eq. 8, effectively three gram moles of water are eliminated from the solution during the electrodissolution and electrodeposition process. 2-14% volume loss of the solution is noticed. The volume loss increases with increasing electrodissolution and electrodeposition process or current. Based on these theoretical calculations, the above mechanism is suggested.

Oxidation of glycerol

While liberated oxygen at anode reacts with glycerol and forms several products. These reactions are discussed by Viswanath and George [22, 23]. The formed product contains different organic acids like glyceric, tartonic and mesoxalic acids which cause the increase of acid concentration.

Elecrocatlytic effect and autodeposition studies

It is observed that in absence of cadmium sulphate, and under similar conditions there is some cadmium deposited on cathode, whereas anode lost some mass. At the same time, almost black-grayish shade loose powder deposit is found on the anode. This type of black-grayish powder is not found on the anode in the original experiments. This powder was removed by scratching and analyzed. The powder is found to be cadmium metal. On the other side, analysis of solution indicated that cadmium sulphate is formed in the solution. If the anode is left for some time in the solution, the anode powder turned into hard layer and could not be removed by simple scratching. If acid concentration is kept very low the black-grayish powder is found on the anode along with small white powder. A white precipitate is found in solution which settled later at the bottom of the cell. If sufficient acid is not present and only glycerol is present in the solution, no precipitate is found. When similar experiment is carried in 0.1N ammonium hydroxide a large amount of white precipitate is observed. The powder was found to be cadmium hydroxide, $Cd(OH)_2$. The hydroxyl ions obtained from dissociation of water attack the positively charged cadmium anode and form cadmium hydroxide which in turn reacts with acid and forms cadmium sulphate. Eqs 7-11 suggest that this is all due to the electrocatalytic effect. This cadmium sulphate formed in the solution acts as medium for electrodeposition and electrowinning. Eqs 12-16 suggest that this is the autodeposition process. The relative mass loss of anode is given by the equation as:

$$w_r = \frac{\text{Mass loss of anode in absence of electrolyte}}{\text{Mass loss of anode in presence of electrolyte}} \tag{17}$$

The data on the relative mass of cadmium obtained at different current and percent of glycerol is presented in Table 4.

Table4. Relative mass of anode and electrocatalytic effect efficiency obtained at different current and percent of glycerol.

Current (A)	Percent of glycerol and relative mass of anode (w_r) Electrocatalytic effect efficiency (E_f)				
	0%	5%	10%	15 %	20%
0.5	1.09	1.03	1.16	1.14	0.97
	1.02	**1.05**	**1.03**	**1.01**	**0.94**
1.0	1.09	0.99	1.18	1.23	1.23
	1.03	**0.98**	**0.98**	**0.99**	**0.99**

Threshold current theory of electrode dissolution

It appears that a critical value of current must be applied to electrolytic cell to start electrodissolution of anode and auto deposition on cathode. In case of copper more than 0.02A (0.81-0.82V) and 0.01A (0.72-0.74V) current must be applied when the sulphuric acid concentration is 0.5N and 1N, respectively, to initiate these processes. In the case when the acid concentration is 0.5N and current applied to electrolytic cell is greater than 0.005A (0.1V), cadmium will slowly react with acid and then only electrodissolution of anode and autodeposition on cathode will occur. The electrocatalytic effect efficiency is given by the equation as:

$$E_f = \frac{\text{Mass loss of anode in absence of electrolyte}}{f} \tag{18}$$

The electrocatalytic effect efficiency obtained at different current and percent of glycerol is also illustrated in Table 4. The charge, coulombs per s, on the anode is numerically equal to the number of amperes applied to the electrolytic cell. Therefore Aw/F is equal to number of grams of anode metal dissolving per sec. E_f values conform this theory.

Mathematical concept of combined study of electrocatalytic and electrowinning process

The electrocatalytic and electrowinning are combined processes occurring simultaneously during electrodeposition of metals. The result is either increase or decrease in the concentration of electrolyte or metal ions in the solution. Δc is a function of applied current, glycerol percent, initial concentration, time and acid concentration. If C_i and t are maintained constant and sufficient acid is available, then Δc is a function of A and C_g, as:

$$\Delta c = f(A, C_g) \tag{19}$$

Differentiating the equation 19:
$$d(\Delta c) = (\delta(\Delta c)/\delta A)_{C_g} dI + (\delta(\Delta c)/\delta C_g)_I dC_g \tag{20}$$

where**:** $(\delta(\Delta c)/\delta A)_{C_g}$ and $(\delta(\Delta c)/\delta C_g)_A$ are partial derivatives.

If C_g is kept constant, then the second term is equal to zero and above equation may be written as complete derivative:

$$d(\Delta c)/dA = (\delta(\Delta c)/\delta A)_{C_g} \tag{21}$$

Δc may be expressed in power series of I as a polynomial:

$$\Delta c = a_1 A^x + a_2 A^{x-1} + \ldots \ldots \tag{22}$$

where: a_1, a_2 *etc* are coefficients of the polynomial and x, x-1, are indices of the polynomial.

This polynomial is differentiated with respective I and derivative $d(\Delta c)/dI$ is calculated. This derivative is function of Δc and may be written as:

$$d(\Delta c)/dA = k(\Delta c)^n \tag{23}$$

where: k is the constant of process similar to specific reaction rate constant and n is order of process. The logarithmic expression for the equation may be expressed as:

$$\ln[d(\Delta c)/dA] = n \ln[(\Delta c)] + \ln(k) \tag{24}$$

The plot of $\ln[d(\Delta c)/dA]$ *vs.* $\ln[(\Delta c)]$ gives a straight line. n is slope and $\ln(k)$ is intercept of the line. The values of n and $\ln(k)$ are presented in the Table 5. The unit of k is mol A^{-1} When glycerol percent is zero the order of process is zero which indicates the process is independent of electrolyte concentration. n and $\ln(k)$ are expressed by following equations:

$$n = -0.609C_g + 0.3488 \text{ and } \ln(k) = 0.0888C_g - 1.3765 \tag{25}$$

These equations are valid for all glycerol percent.

Table 5. The constants for the combined process of electrocatalytic and elecrtowinning.

% of glycerol	n	$\ln(k)$ m A^{-1}	Inflection point values I(A)	Δc(m)
0	0.363	-1.471	2.1	0.405
5	0.699	-0.751	1.58	0.122
10	0.923	-0.541	0.83	0.063
15	1.138	-0.107	0.53	0.11
20	1.666	-0.426	0.35	0.027

When Δc is constant the term $d(\Delta c)/dI$ becomes zero. This is known as inflection point of the curve. A and Δc values of the inflection point are also presented in Table 5. At this point, rate of electrowinning process is equal to rate of electrocatalytic processes. In other words rate of electrodissolution of cadmium is equal to rate of removal of cadmium ions from the solution. The mass loss of cadmium anode (in g at. mass) may be related to current and glycerol percent by the following equations:

$$C_a = (10^{-04} C_g{}^2 - 0.0028C_g + 0.0634)C - 2\times10^{-05} C_g{}^2 + 0.0006 C_g + 0.0056 \tag{26}$$

Valid between 5 to 15% glycerol

$$C_a = 0.0535x + 0.0041 \text{valid for 0\% glycerol} \tag{27}$$

$$C_a = 0.0528x + 0.0033 \text{ valid for 20\% glycerol} \tag{28}$$

Similarly, the mass of cadmium deposited on cathode (in g at. mass) given by following equation:
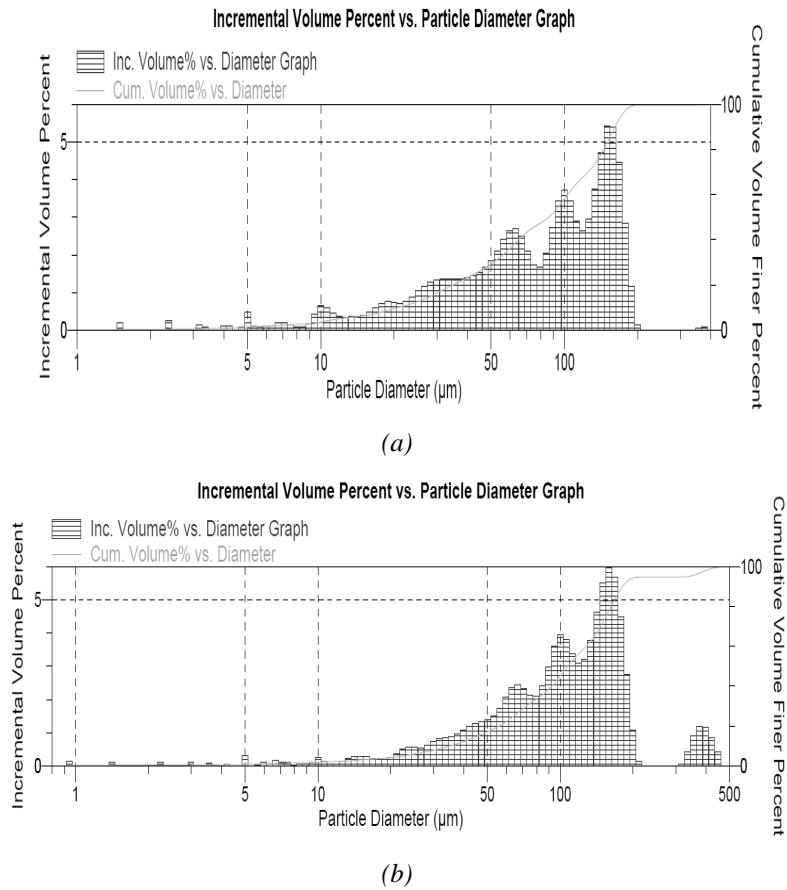
$$\ln(C_c) = (-0.0009C_g + 0.0478) \ln(C) + (9 \times 10^{-05} C_g{}^2 - 0.0028 C_g + 0.0686) \tag{29}$$

Particle size analysis

Cadmium powder is analyzed for particle size by two methods. First one is particle size distribution curve analysis which gives particles in 1μm size. The second one is XRD analysis which may be used for calculating particles in nm (i.e. below 1μm) size by Debye Sharer formula.

Particle size distribution

The distribution of cadmium particle size is shown in Figure 1 and the data of distribution are given in Table 6. Sulfuric acid 0.5 N and 5% glycerol with 0.5A current give satisfactory small particle size. Around 60% of particles were below 100 μm. 10% glycerol with 1.5A current also gives desirable smaller particle size and shows some nano-character. This is evident from SEM and XRD studies also.
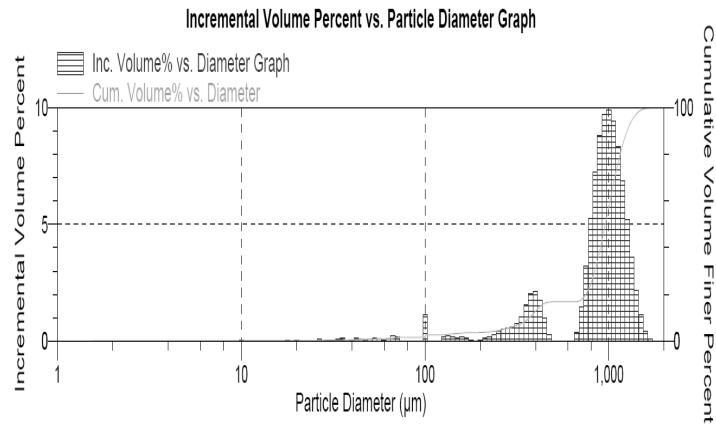

(a)

(b)

Incremental Volume Percent vs. Particle Diameter Graph

(c)

Incremental Volume Percent vs. Particle Diameter Graph

(d)
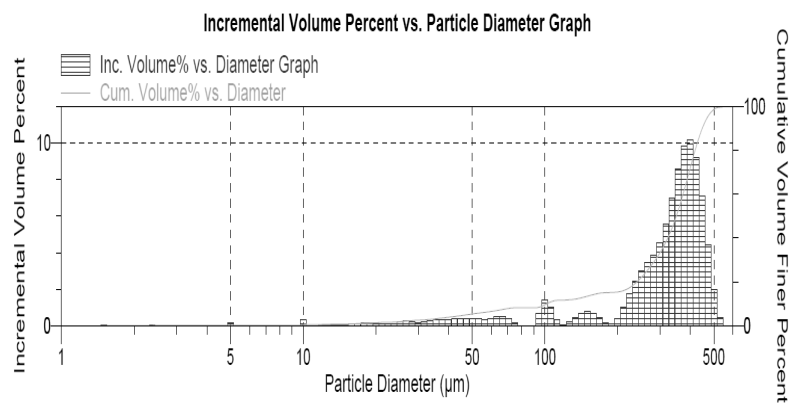
Figure: 1 Particle distribution (a) 5% glycerol and 0.5A (b) 10%glycerol and 1.5A, (c) 20 % glycerol and 1.5 A and (d) 15% glycerol and 2 A.

Table 6. Particle size of cadmium powder obtained at different current and percent of glycerol.

| Current and % of glycerol | Percent of particles | | | | | | |
| | 1-15 | 15-30 | 30-45 | 45-60 | 60-75 | 75-85 | 85-100 |
				Size of particles in μm			
0.5 A	4.93	28.24	51.19	73.60	102.99	136.08	152.77
and 5	28.24	51.19	73.60	102.99	136.08	152.77	185.92
1 .5 A	6.05	45.84	72.55	99.520	127.71	154.47	170.36
and 10	45.84	72.547	99.52	127.71	154.47	170.36	416.89
1.5A	62.79	426.26	839.35	928.85	1014.12	1111.87	1198.54
and 20	426.26	839.35	928.85	1014.12	1111.87	1198.54	1495.20
2 A	17.90	174.63	286.02	336.86	372.68	406.1	431.65
and15	174.63	286.02	336.86	372.68	406.1	431.65	503.89

XRD particle size analysis

The particle size is calculated from XRD data of peaks from graph and by using Debye-Sharer formula**:**

$$D = k \, \lambda / \beta \, \cos\theta \tag{30}$$

where: k - crystalline shape factor and λ - wave length in angstrom (Å)

$$\beta \text{ (radian)} = FWHM \times \pi / 180 \tag{31}$$

where: Full-Width Half-Maximum (FWHM) - from instrument data, π is 22.7 and θ is angle. Values of particle size are shown in Table 7. XRD pattern of cadmium powder particles of some samples are shown in Figure.2. A peak height expressed in 2θ values is related to grain size of the particle in nm. The height of the peak indicates intensity of diffraction or may be related to number of particles.

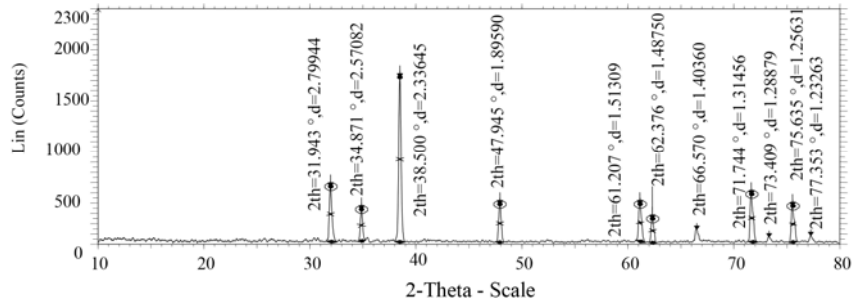*Table 7. Particle size of cadmium powder obtained at different current and percent of glycerol.*

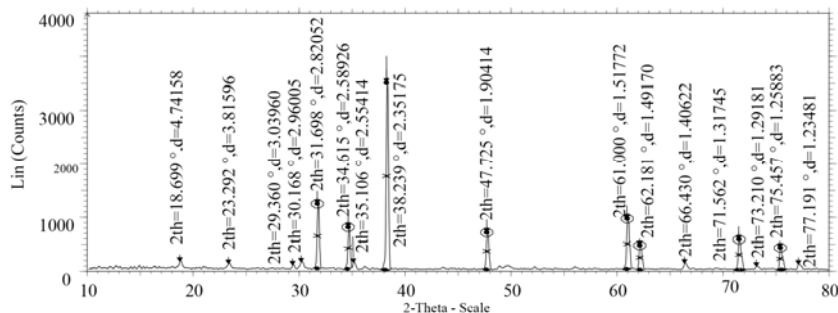| Current and % of glycerol	Particle size in nm
0.5 A and 5 %	212.2, 224.5, 231.3, 234.8, 234.8, 241.4, 243.7, 250.9,
1.5 A and 10 %	230.2, 244.5, 248.4, 251.7, 253.3, 264.7, 291.7, 303.2
1.5A and 20 %	212.7, 213.9, 224.5, 232, 232, 243.7, 259.3, 272.1
2 A and15 %	234.1, 246.8, 250, 255.9, 266.5, 267.4, 271.2, 274.1
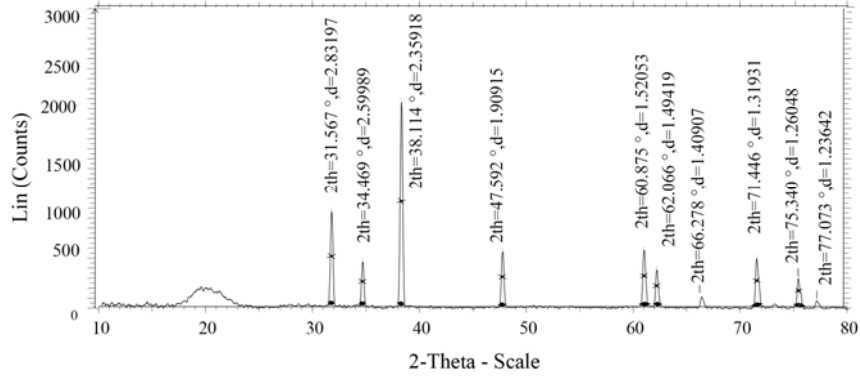
a)
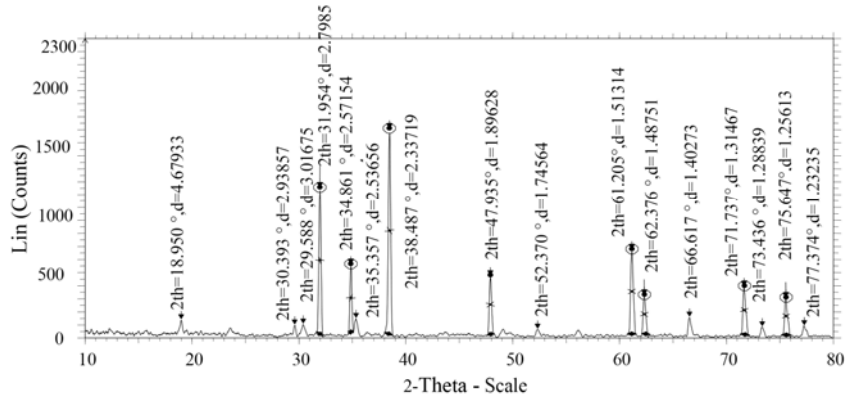
b)

c)

d)

Figure: 2 XRD graphs (a) 5% glycerol and 0.5A (b) 10%glycerol and 1.5A, (c) 20 % glycerol and 1.5 A and (d)15% glycerol and 2 A
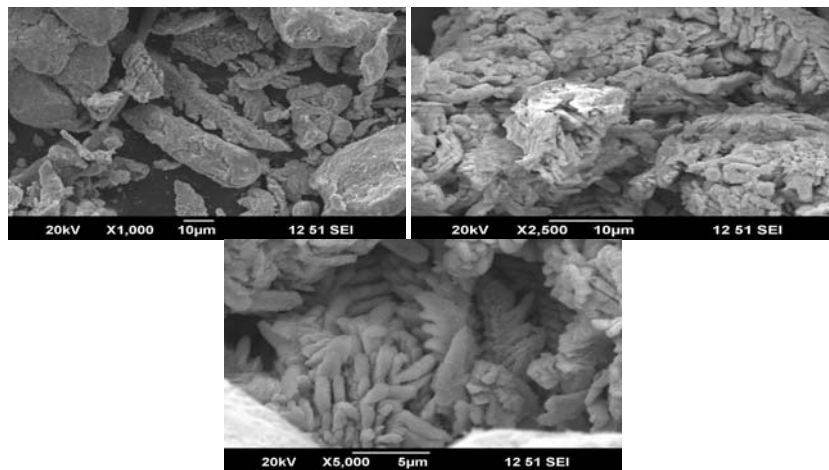
<u>Morphological studies</u>

SEM microphotographs of cadmium powder are shown in Figure.3. The relevant physical data of respective powders are presented in Table 8. Some particles are broken pieces of dendrites that are amalgamate or intermingled with spongy, irregular, acicular particles, or spongy, aggregate particles and look like lumps.
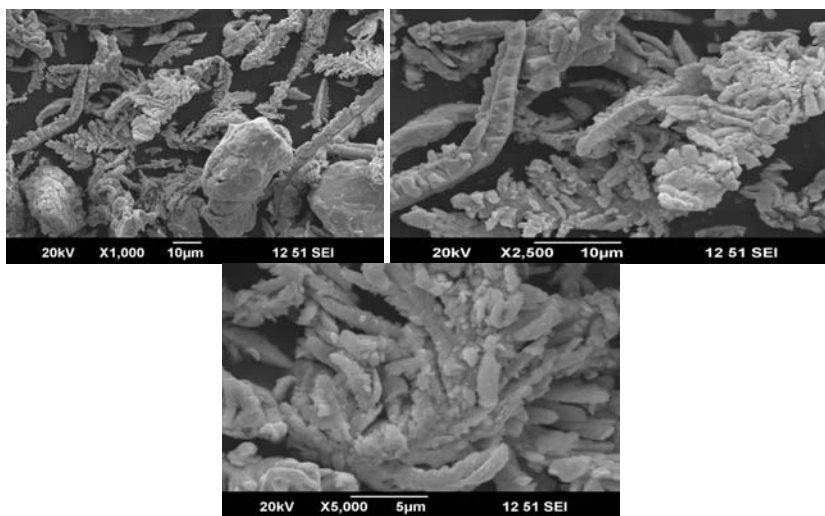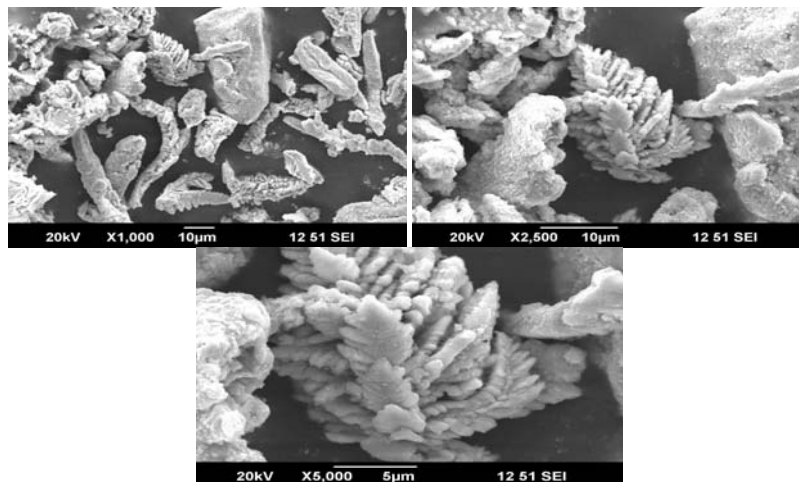

(a)
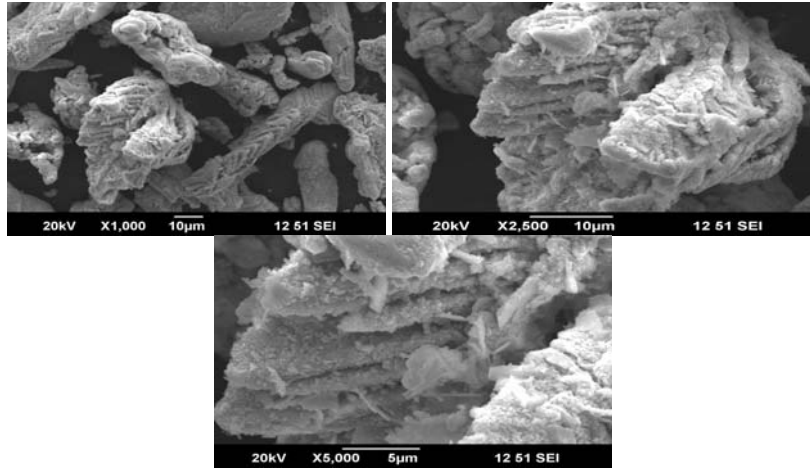
(b)

(c)

(d)

Figure.3 SEM microphotographs of Cadmium powder (a) 5% glycerol and 0.5A:(b)10%glycerol and 1.5A:(c)20 % glycerol and 1.5 A: (d)15% glycerol and 2 A:

Table 8. Morphology of cadmium powders and related data.

Current (A) % glycerol	Apparent density	Stability	Morphology of the powder and % particle size below 100 μm
0.5 A and 5%	6.8	88.1	mostly broken dendrites acicular, spongy, aggregate, 60
1.5 A and 10%	6.3	79.1	broken dendrites, intermingled with spongy and irregular particles, 45
1.5A and 20%	4.7	67	angular, acicular or nearly cylinderical broken dendrites, 15
2A and 15%	5.2	70	broken into long branches of dendrites, spongy and unbroken dendrites, 15

Discussion

10% glycerol and 1.5A of current produce powder particles which are intermingled, spongy and irregular in shape with broken dendrites. These particles look like lumps. It is quite possible that the grinding of powder may have resulted in the formation of these lumps because of soft, malleable and ductile nature of metal. Glycerol concentration between 10 to 20% and current 1 to 1.5A produce powders which contain broken long branches of dendrites, spongy and unbroken dendrites particles. For the passage 0.5A current through 5% glycerol all produced particles are below 185 μm. XRD studies show that smaller particles are in the range between 212 and 250 nm. The particles are mostly broken dendrites, acicular, spongy aggregates.

During electrolysis cadmium anode dissolves into solution, all positive ions align and migrate towards the cathode, while negative ions migrate towards the anode. The metal ion is discharged at the cathode. The solid metal atoms deposited on the cathode should have strong metallic bond. A large amount of hydrogen liberated at the cathode makes the deposit porous. The solution containing organic solvent which is covalent in nature also disturbs this ion environment particularly at the cathode causing loose bonding between metal-metal atoms. The effect of these parameters results in the formation of metallic powder.

When the electrodeposition process was completed the acid concentration is increased. This increase of concentration is due to formation of sulphuric acid from sulphate ions, and different carboxylic acid by the electrolytic oxidation of glycerol molecules and elimination of water molecules from the solution by electrolysis. The increase in the acid concentration was found to be in the range from 0.12 to 0.15N. It is found that cadmium ion concentration increased during electrodeposition process. This increase in the cadmium ion concentration may be attributed to slower electrowinning process than electrodissolution process.

The oxidative stability of the powder decreases with increases current as well as with percent glycerol. If the powder has large surface area, lesser is its oxidative stability. This may be due to the fact that more number of cadmium atoms at the surface has free valences. In the present case oxidative stability of powder is due to smaller size of the particles having small surface area.

According to results of Tables 3 and 4 abnormalities in PFD, PFE, w_r and E_f values at low glycerol percent may be seen. These values decrease with increase in glycerol percent. These high values may be due the fact that cadmium reacts slowly with the acid. The increase in percent of glycerol reduces the cadmium reaction with the acid. Yield of the powder is high at low glycerol percent and decreases with increase in glycerol percent.

Conclusions

It may be concluded that 5% glycerol and 0.5A current produce powder having good properties. Under these conditions, 60% of particles are below 100 μm with very few dendrites particle

Acknowledgement

References

[1]   D.Primavesi, Production of Electrolyte Iron Powder, French Patent, 976,453 (1951).
[2]   C.F. Burgress , C.Hambucehn, Trans.Am.Elecrtochem.Soc., 5 (1904) 201-210.
[3]   T.Kikuchi and K.Yamaguchi, (To Nippon mining Co.) Powdered Electrolytic Copper without Reducing Operation. Japan Patent, 1514/50 (1950).

[4]   H.R.Copson,A.Wesley,T.H.Wickenden (to International nickel co. of Canada Ltd.)Electrolytic Production of Nickel Powder, Canada Patent No.475 /886 (1951).

[5]   M.Kurodea, G.Ito (to Scientific Research Institute Ltd.)Electrolytic Preparation of Nickel Powder, Japan Patent no. 5166/53 (1953).

[6]   N.Fujise,Electrolytic Production of Iron, Nickel and Cobalt Powders by use of Magnetic Cathode, Japan Patent, 2363/57 (1957).

[7]   W.H. Osborne , S.B.Tuviner, Trans.Electro.Chem.Soc., 85 (1944) 107-117.

[8]   G.B. Smith, Iron Age, 153No.22 (1944) 55-56.

[9]   A.W.Hothersall , G.E.Gardam, J Electropositors Tech.Soc.20(1945)61-68, Abstract In Metal Ind. Progress, 66 No.15 (1945) 234-236.

[10]   A.J.Levin, J.Applied.Chem.(USSR) 19 No.8 (1946) 779-787.

[11]   M.Loshkarev, A.Krukova , O.Gornostaleva, J. Appleid Chem., USSR 19No.8 (1946) 46-50.

[12]   H.J. Morgan, O.C. Ratson,Trans. Am.Electrochem.Soc.,30 (1916) 229-239.

[13]   H.J. Morgan , O.C. Ratson, Met.Chem. Eng.,15 (1916) 465-468.

[14]   W.Eckardt, US.Bur,Mines Inform Circ., 7466 (1948) 1-6.

[15]   G.Hanesland , M.Passer, Wiss.Veroffentl Siemens-Werk, Weerstoff Sanderheft,9 (1940)124-137.

[16]   G.Hanesland , M.Passer, Chem.Zentr, (II)112 (1941) 1252.

[17]   A.J.B. Dutra, E. Espinola , P.P. Borges, Mineral. Engg. 13No.10-11(2000) 1139-1148.

[18]   A.E. Elsherief, Electrochimica. Acta , 48 (2003) 2667-2673.

[19]   D.K Borikar, S.S. Umare , S.G.Viswanath, Trans.SAEST 39 (2004) 9-12 & 41(2006)30-34,

[20]   D .K Borikar,S.S. Umare ,S.G.Viswanath, Metalurgija J. Metallurgy 45 (2006) 3-8.

[21]   D.K.Borikar,S.S. Umare ,S.G.Viswanath,.Bull Electro. Chem., 21(8) (2005) 423-431.

[22]   S.G.Viswanath , Sajimol George, Metallurgia J. Metallurgy, 16 (1) (2010) 25-38.

[23]   S.G. Viswanath, Sajimol George, Ind. J Chem. Tech., 18 (2011) 37-44.

[24]   S.G. Viswanath, M. M. Jachak, Metall. Mater. Eng., 19(2) (2013) 119-134.