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Electrochemical Preparation of Ultrafine Zinc Powder

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In this research work, statistical analysis and optimization of the electrolytic preparation of zinc powder from an alkaline solution containing zinc oxide were carried out using Taguchi methodology. The analysis considered the effect of ZnO concentration (20-80 g L^{-1}), electrolyte temperature (30-70 °C), and current density (150 - 450 $mAcm^{-2}$) on the specific surface area of zinc powder. The effect of organic additives such as D-sorbitol and sucrose on the morphological structure of zinc powder was studied based on the optimum operating conditions of Taguchi design. XRD-diffraction, atomic force microscopy (AFM), BET, and SEM were used to characterize the zinc powder. The results indicate that ZnO concentration has the major effect on the specific surface area of zinc powder followed by current density while the temperature has no significant effect on the specific surface area. The optimum conditions for preparing zinc powder at a higher specific surface area and a nanostructure were a current density of 450 $mAcm^{-2}$, an electrolyte temperature of 30 °C, ZnO at a concentration of 20 g L^{-1}, and D-sorbitol at a concentration of 4 g L^{-1}. The current efficiency and energy consumption were 91.3 %, and 3.0 KWh kg^{-1} respectively. The produced powder has a specific surface area of 6.218 m^2 g^{-1} and an average particle size of 67 nm.

Keywords: Taguchi method, zinc powder, specific surface area, electrodeposition, D-sorbitol, sucrose.

1. INTRODUCTION

In view of valuable features such as its low cost, abundance, and low equilibrium potential, zinc powder has been widely used in various industrial applications [1]. It can be used in both chemical industries such as preparation of benzidine, rongalite, and hydrosulphite, and in metallurgical industries such as production of precious metals like gold and silver by using a cementation process. Moreover, it was used for the preparation of amalgam alloy which is used in dental fillings [2]. Zinc powder can also be used as a paint for a heavy-duty coating for large-scale structures such as sea containers, bridges, and other marine equipment. The most important application of zinc powder is as

an anode in secondary alkaline batteries which are extensively used in aerospace, military, commercial, and many other fields [3, 4]. There are several methods for the preparation of zinc powder including physical vapor deposition, chemical vapor deposition, electrodeposition, and zinc oxide reduction [5]. Electrodeposition is the preferred over other methods because of its low energy consumption and less gaseous emission, in addition to a better control of the particle size and surface area of the powder. Among the possible media, alkaline electrolytes are preferable compared to acid electrolytes. In the alkaline media, hydrogen evolution as a side reaction on the cathode surface is even more obstructed than in the acid media. Thus, a higher current efficiency approaching 100% can be attained when high current densities are applied, which favors the electrodeposition of ultra-fine zinc powder [6]. Several investigators have been studying the electrolytic preparation of zinc powder from alkaline media [7–10]. However, there is a lack of enough understanding about the direct effect of electrodeposition parameters on the specific surface area of zinc powder which plays a vital role in the performance of anodes made from this powder in many alkaline batteries.

Organic additives, such as surfactants are normally used in many areas such as zinc electroplating and alkaline batteries. In battery technology, the most important effect of surfactants was to suppress gas evolution [11]. In zinc electroplating, surfactants have been widely used to reduce the harmful effects of different metallic impurities [12, 13]. However, using organic additives in the electrolytic preparation of zinc powder is limited to a few works [14, 15] and finding a new convenient organic additive remains a significant research. Among of the most organic additives used in zinc electroplating, D-sorbitol and sucrose have been used and given excellent results with respect to the appearance of the film deposited [16,17]. Using of these additives in the electrolytic preparation of zinc powder had not been investigated quantitatively in the literature.

Recently, design of experiment (DoE) has considered the common method used to create numerical relationships between different input factors that affect the electrolytic process variables in order to focus the factors that control the desired electrolytic product. The statistical design of experiments is an effective procedure for planning experiments so that the data obtained can be analyzed to get valid and objective conclusions. The two main applications of experimental design are screening, in which the parameters that effect on the experiment are recognized, and optimization, in which the optimal settings or conditions of an experiment can be determined [18]. Taguchi method is one of the experimental designs that has been shown to be an effective method for enhancing the productivity in the research and development stage so that high quality items can be created rapidly at low cost. It has proved to have multiple applications in a wide range of industrial fields all over the world because of its widespread applicability to all engineering fields [19–21]. To the best of the authors' knowledge, there are no previous works on the using Taguchi experimental design and ANOVA for studying the effect of different process parameters on the performance and properties of the electrolytic zinc powder prepared from alkaline baths.

The current work sets out to investigate the electrochemical preparation of ultrafine zinc powder from an alkaline electrolyte. Current density, ZnO concentration, and temperature were selected as the most controlling parameters on the electrodeposition process. Taguchi experimental design method and analysis of variance (ANOVA) were used to determine the most important parameters as well as the optimum zinc powder preparation conditions for maximizing the specific

surface area of zinc powder. The Effect of D-sorbitol and sucrose on the specific surface area of zinc powder was also studied based on the optimum operating conditions of Taguchi design.

2. EXPERIMENTAL WORK

2.1 Materials

All chemicals were of analytical grade. Electrolytic zinc powders were produced by electrolysis of alkaline solutions containing zinc oxide. These solutions were prepared by dissolving a given amount of ZnO in 6 mol L^{-1} NaOH. After complete dissolution, solutions were stored in 500 ml volumetric flasks. Removing of water from the prepared zinc powder was performed using analytical grade ethanol and acetone mixture.

2.2 Electrolytic cell

The electrochemical cell consists of a 0.5 L Pyrex beaker provided with polytetraflouroethylene (PTFE) cover. A magnesium cathode (2 cm width× 13 cm long) and a stainless steel (316-AISL) anode at the same dimensions were used. The active surface area of the cathode was 10 cm^2. The back face of the cathode was covered with an epoxy to prevent of zinc deposition. On magnesium, the lamellar deposit cannot be formed and only a powdery structure can be electrodeposited, which are easily removed from the cathode [22]. Moreover, the anode to cathode distance was maintained at 3 cm, and all experiments were completed after passing a total charge of 16200 Columns. Power Supply- model UNI-T: UTP3315TF-L was used to run the galvanostatic zinc deposition. To obtain a constant temperature during the electrolysis, the electrolytic cell was placed in a water bath. Fig. 1 shows the electrochemical system.

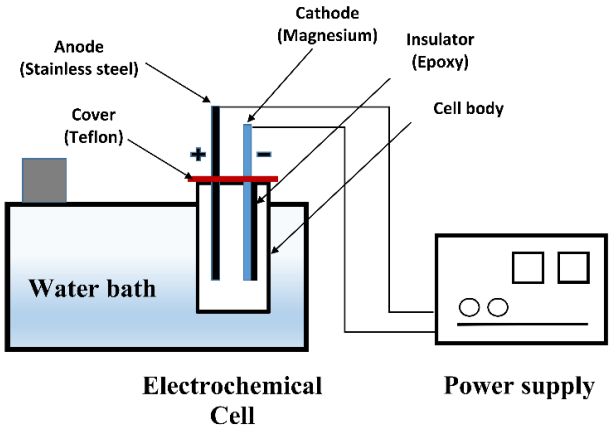


Figure 1. Electrochemical system

The prepared zinc powder could easily be removed from the cathode surface and was washed in distilled water for several times until all the possible existing alkaline solution was removed from the powder particles. This was verified by adding a few droplets of phenolphthalein to the ablution water. After that, the powder was treated with an alcohol-acetone mixture (ethanol-acetone = 1:1) to remove water, then dried for 2 h in 100 °C, weighed, and stored in a polyethylene plastic container to suppress further oxidation. A different weight of Zn powder was obtained in each experiment, the current efficiency (CE) was calculated using Eqs. (1) and (2) as follows [3]:

$$m_{th} = \frac{M_W It}{nF} \quad (1)$$

$$CE = \frac{m_{exp}}{m_{th}} \quad (2)$$

where m_{th} is the theoretical weight of zinc powder (g) ; m_{exp} is measured weight of powder (g) produced in time (t); M_w is atomic weight of Zn (65.37 g mol^{-1}); I is current in ampere; t is time of operation in seconds, n is zinc valence which is 2 and F is Faraday constant (96486 C mol^{-1}).
The energy consumption was calculated using the following formula [23]:

$$EC = \frac{2.778 \times 10^{-4} EIt}{m_{exp}} \quad (3)$$

Where EC is the energy consumption (kWh kg^{-1}) and E is the cell potential in volts.

2.3 Characterization of zinc powder

The prepared zinc powder was analyzed using X-ray diffraction (CuKα radiation as the X-ray source, λ= 1.54056 ˚A). Granularity cumulation distribution chart of zinc powder was obtained using atomic force microscopy (AFM) SPM-AA3000 (Angestrom Advanced Inc., USA) in contact mode. The AFM images were recorded over a scan area (2 µm x 2 µm). The SEM micrographs were obtained using scanning electron microscope (FEI Company –Holland). The apparent density (d_a) of zinc powder was measured by the standard pycnometric method, and calculated based on the following formula: [24]

$$d_a = \frac{d_w(w_3 - w_1)}{(w_2 - w_1) - (w_4 - w_3)} \quad (4)$$

where: w_1 is the mass of the dry empty density bottle**;** w_2 is mass of the density bottle with distilled water**;** w_3 is mass of the dry empty density bottle and zinc powder (about 1 to 1.5 g)**;** w_4 is mass of the density bottle with zinc powder and distilled water**;** d_w is the density of water at that temperature. The specific surface area (SSA) of zinc powder was measured by BET method using (ASAP 2020 surface area analyzer).

2.4 Taguchi design of experiment

Choice of process parameters and their levels is a vital exercise to be performed while designing the experiments. The properties of zinc powder depend on several factors such as ZnO concentration, alkaline (NaOH or KOH) concentration, applied current density, temperature, distances between electrodes, and stirring [3, 6, 8, 10]. In the present research, NaOH concentration and the distance between electrodes were kept constant at 6 mol L^{-1} and 3 cm respectively. These are the

optimum values in which a maximum current efficiency and a lower cell voltage were obtained as reported by Youcai et al. [25] where the current efficiency of the zinc deposition decreased dramatically as the NaOH concentration increased beyond this value due to the dissolution of the zinc produced. Stirring significantly leads to the formation of compact deposits rather than dendrite powdery deposits [9], therefore using a stagnant solution was preferred to ensure the formation of powdery deposits. For optimization the specific surface area, Current density (CD), ZnO concentration and temperature (T) were selected as three main variable parameters. All these factors were tested at three levels as shown in Table 1. The lower and maximum levels were chosen based on the thorough investigation of the conditions of previous works [3, 7-9].

According to the Taguchi design approach, an experimental design should be designated for these control factors. The most suitable method for designing the experimental plan was the orthogonal array experimental design (OA). Ideally, we should select an array that contains three levels and three factors, i.e., L_6 (3^3) orthogonal array. However, no such published array exists, then the next smallest array that will outfit our work is L_9 (3^4) and hence it was chosen for this research. Table 2 shows the L_9 orthogonal array of coded and real values of the control factors.

Table 1. Experimental parameters and their levels

Factors \ Level	1	2	3
CD , mA cm^{-2}	150	300	450
[ZnO], g L^{-1}	20	50	80
T, °C	30	50	70

Table 2. L_9 experimental plan

Exp. No.	Coded value			Real value		
	A	B	C	CD (mA cm^{-2})	[ZnO] (g L^{-1})	T (°C)
1	1	1	1	150	20	30
2	1	2	2	150	50	50
3	1	3	3	150	80	70
4	2	1	2	300	20	50
5	2	2	3	300	50	70
6	2	3	1	300	80	30
7	3	1	3	450	20	70
8	3	2	1	450	50	30
9	3	3	2	450	80	50

Taguchi recommends the use of the loss function to measure the performance characteristic deviating from the desired value. The loss function value is further converted to a signal-to-noise (S/N) ratio, where signal represents the desirable value (i.e. the mean for the output characteristic) and noise represents the undesirable value (i.e. the square deviation for the output characteristic). Therefore, the

S/N ratio is the ratio of mean square deviation. Its unit is dB. The S/N ratio equation depends on the criterion for the quality characteristic to be optimized.

Several different possible S/N ratios were used, however, three of them are considered standard and are usually applicable in most situations: larger is better (LTB), smaller is better (STB), and nominal is better (NTB). In this study, LTB is the specific surface area of zinc powder defined by the following equation [26]:

$$(S/N)_{LTB} = -10\log\left[\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right] \quad (5)$$

Where n is the repetition number of each experiment under the same conditions for design parameters and y_i is the response of each experiment.

3. RESULTS AND DISCUSSION

3.1 Optimum conditions for higher specific surface area of zinc powder

Table 3 shows the results of the experimental design, including specific surface area (SSA), current efficiency (CE), and energy consumption (EC). The corresponding calculated S/N ratio is also reported. The data of the specific surface area were analyzed by the Taguchi method to estimate the effect of each process parameter on the optimization criterion. The analysis of these data was made with the help of software package MINITAB-17 and using a general linear model. The S/N ratio calculation was made to maximize the specific surface area of zinc powder.

Table 3. L_9 experimental results

Exp. No.	CD (mA cm^{-2})	[ZnO] (g L^{-1})	T (°C)	E (V)	m_{exp}* (g)	SSA (m^2 $g^{-1)}$	S/N** Ratio	CE (%)	EC (KWh kg^{-1})
1	150	20	30	2.6	3.512	2.716	8.678	64.00	3.344
2	150	50	50	2.2	5.226	0.354	-9.020	95.23	1.901
3	150	80	70	2.3	5.37	0.092	-20.72	97.85	1.934
4	300	20	50	2.7	4.398	2.897	9.239	80.14	2.773
5	300	50	70	2.6	5.131	0.501	-6.003	93.50	2.288
6	300	80	30	3.2	5.076	0.230	-12.76	92.50	2.847
7	450	20	70	2.8	5.26	4.349	12.768	95.85	2.404
8	450	50	30	3.2	4.95	1.920	5.666	90.20	2.920
9	450	80	50	3.0	5.288	0.360	-8.874	96.36	2.562

*Theoretical amount of zinc= 5.488 g, **Based on specific surface area results, [NaOH] = 6 mol L^{-1}

The statistical analysis of variance (ANOVA) was achieved to see whether the process parameters are statistically significant or not. The *F*-value for each process parameter shows which parameter has a significant effect on the specific surface area value. Usually, the larger the *F*-value, the greater the effect on the value of the specific surface area of zinc powder due to a change in the process parameter. An Optimal combination of process parameters can be predicted using ANOVA statistical

test and performance characteristics [27]. Results of ANOVA for zinc powder preparation are shown in Table 4, where DF is the degree of freedom, Seq SS is the sequential sums of squares, Contrib.% is the percentage contribution of each factor, Adj SS is the adjusted sums of squares , Adj MS is the adjusted mean squares calculated by dividing the adjusted sum of squares by the degrees of freedom, F-Value is a test of the equality of variance between populations or factor levels and it is simply a ratio of the squared deviations to the mean of the squared error, and P-Value is a determination of the appropriateness of rejecting the null hypothesis in a hypothesis test.

Table 4. Results of ANOVA for the electrolytic zinc powder at 95% confidence level.

Source	DF	Seq SS	Contrib. %	Adj SS	Adj MS	F-Value	P-Value
CD, mA cm^{-2}	2	2.3604	12.47%	2.3604	1.1802	5.88	0.145
[ZnO], gL^{-1}	2	15.7947	83.44%	15.7947	7.8973	39.34	0.025
T, °C	2	0.3725	1.97%	0.3725	0.1862	0.93	0.519
Error	2	0.4015	2.12%	0.4015	0.2008		
Total	8	18.9290	100.00%				

The percentage contribution in the ANOVA table shows that ZnO concentration is the most effective factor with the greatest percentage contribution (83.44%). On the other hand, the contribution of the temperature is the smallest (1.97%). The *F*-value for this condition with 95 % confidence level is 19 [27]. Therefore, the results of the *F*-value from Table 4 show that the ZnO concentration has a significant effect on the specific surface area of the prepared zinc powder. Based on ANOVA results, the effect of temperature could be ignored, hence referring to Table 3, it can be seen that increasing CD led to increasing the current efficiency (exp. 1, 4, 7). This behavior was confirmed by Pierce and Piron [22] in NaOH media and Sharifi et al. [3] in KOH media. This behavior can be interrupted as the hydrogen overvoltage is very high on magnesium hence higher CD can be used with no hydrogen evolution leading to attain higher current efficiency [28]. Also increasing ZnO concentration led to increasing the current efficiency and this behavior was diminished as the CD increased (exp. 1, 2, and 3). At higher current densities, the energy consumption is less than 3 KWh kg^{-1} which is lower than the industrial zinc electrowining value (3.25-3.4 KWh kg^{-1}) [6].

In the present work, the experimental results of experiments versus the predicted results of MINITAB-17 were compared as shown in Fig.2 based on the specific surface area of zinc powder. The study describes that the MINITAB-17 predicted about the response is useful. Also, the correlation coefficient for the specific surface area is 0.99 which was near to (1). It means there is accordant fit between data.

Table 5 represents the response table for signal to noise ratio. The response table shows the average of the selected characteristic for each level of the factors. It includes ranks based on delta statistics, which compare the relative magnitude of effects. The delta statistic is the difference between highest average and lowest average for each factor. Ranks are given based on delta values; rank 1 is given to the highest delta value, rank 2 to the second highest delta value, and so on [27]. It is clear that results of response table are in agreement with ANOVA results where ZnO concentration is the most

significant factor. The effect of control factors on the specific surface area of zinc powder based on means of S/N ratio is illustrated in Fig. 3. The highest S/N level that is calculated by Eq. (5) is the optimal level of a process parameter. The best value of each graph is the value of the maximum point that marks a particular parameter in each graph [27].

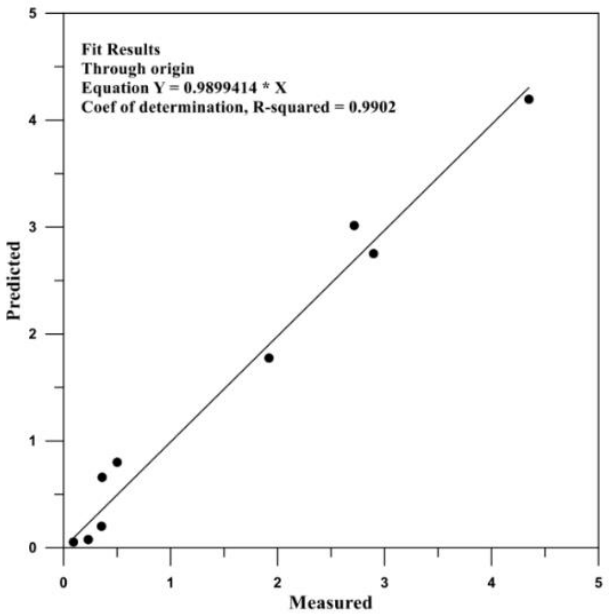


Figure 2. The predicted values verses the measured values of the specific surface area

Table 5. Response Table for Signal to Noise Ratios Larger is better

Level	CD (mA cm^{-2})	[ZnO] (g L^{-1})	T (°C)
1	-7.022	10.229	0.526
2	-3.177	-3.119	-2.885
3	3.187	-14.121	-4.653
Delta	10.209	24.350	5.179
Rank	2	1	3

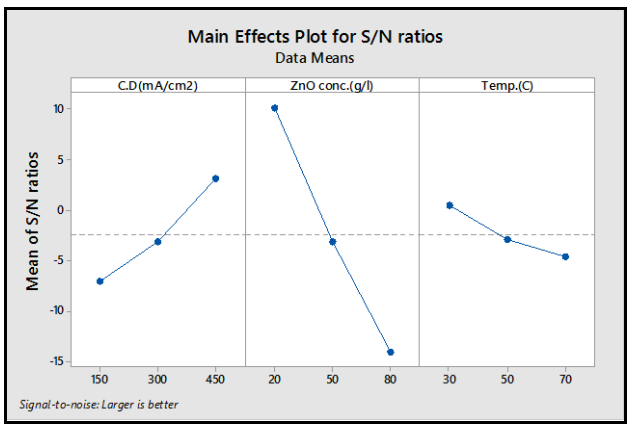


Figure 3. Main effects plot for S/N ratios

According to Fig.3, in terms of maximizing the specific surface area: CD= 450 mA cm^{-2},[ZnO]=20 g L^{-1}, T=30 °C were selected as optimum conditions. Under these conditions, the predicted specific surface area was 4.33 m^2 g^{-1}. It can be seen that the experiment corresponding to the optimum conditions was not achieved during the experiments. Therefore, a confirmation experiment should be performed to verify the conclusions drawn based on Taguchi method. The confirmation experiment was carried out twice at the optimum operating conditions (CD= 450 mA cm^{-2}, [ZnO] =20 g L^{-1}, T=30 °C), and the average of experimental results of the specific surface area under these conditions was 4.534 m^2 g^{-1} with a current efficiency of 92.7 % and an energy consumption of 2.66 KWh kg^{-1}. It is clear that there is a good agreement between the predicted and the experimental results, and these experimental results are within the 95 % significance level confidence intervals confirming the validity of the applied technique for optimizing the electrolytic zinc powder preparation.

3.2 Effect of additives

D-Sorbitol and sucrose were used as additives with a range of concentration (2-8 g L^{-1}). The effect of these additives on the specific surface area is shown in Fig. 4. It can be seen that increasing of D-sorbitol concentration leads to a higher specific surface area up to a concentration of 4 g L^{-1} beyond which the specific surface area decreases with more addition of D-sorbitol. With respect to sucrose, increasing its concentration leads to a higher specific surface area, however, a slight increase in the specific surface area was observed as the concentration of sucrose exceeds 4 g L^{-1}.

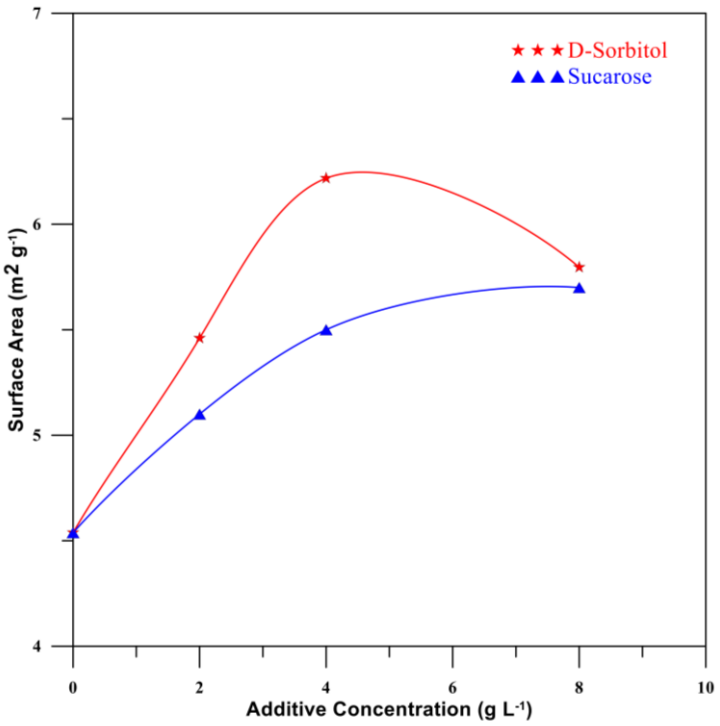


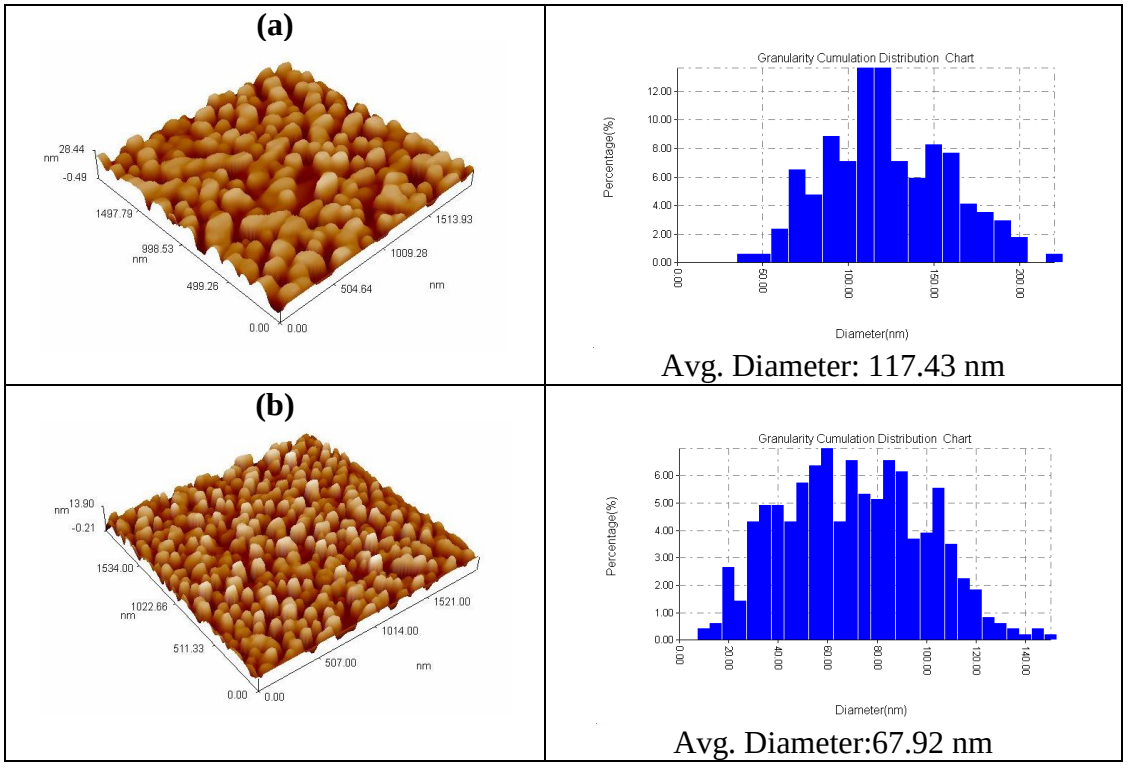
Figure 4. Effect of additives on the specific surface area of zinc powder CD=450mA cm^{-2}, [ZnO] =20 g L^{-1}, [NaOH] = 6 mol L^{-1}, T=30 °C

The results of BET tests proved that the addition of additives offers a greater specific surface area than the case of no additives, which resulted in an optimum of 6.218 $m^2 g^{-1}$ for D-sorbitol addition at 4 g L^{-1} and 5.76 $m^2 g^{-1}$ for a sucrose addition at 8 g L^{-1} in comparison with a value of 4.534 $m^2 g^{-1}$ in the case of no addition. This behavior may be attributed to the diminishing of the nucleation rate and increases the number density of active sites leading to a smaller crystal size. Previous studies indicated that addition of D-sorbitol forms a barrier that controls the metal discharge [16]. Ullah et al. [29] confirmed this behavior when they added sodium dodecyl sulfate (SPS) and Triton x-100 where higher specific surface areas of zinc powders were obtained. The apparent density of zinc powder with and without additives presents in Table 6 where a higher apparent density was observed in the case of the addition of D-sorbitol and sucrose. The AFM results of the zinc powder with and without additives are shown in Fig. 5.

Table 6. Apparent density of zinc powder

Parameter	SSA ($m^2 g^{-1}$)	d_a ($g\ cm^{-3}$)
Without additive	4.534	3.619
D-Sorbitol (4g L^{-1})	6.218	5.881
Sucrose (8g L^{-1})	5.76	6.381

CD=450mA cm^{-2}, [ZnO] =20 g L^{-1}, [NaOH] = 6 mol L^{-1}, T=30 °C



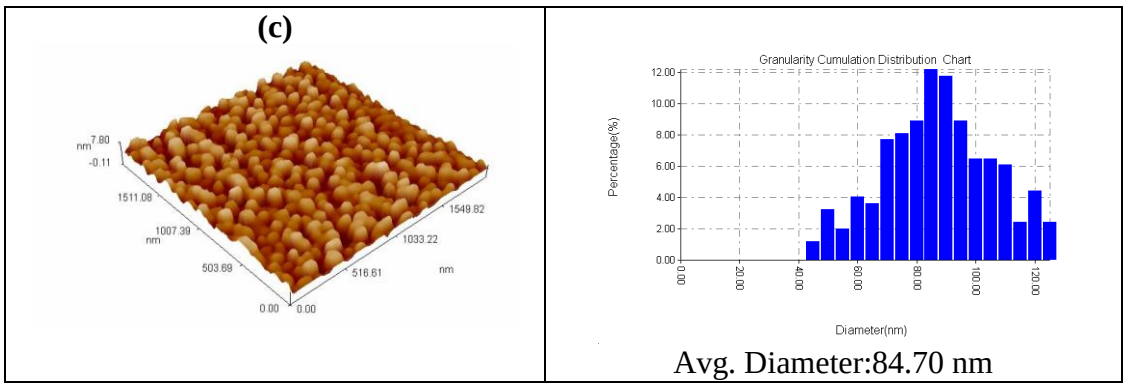


Figure 5. AFM results of zinc powder: a) addition free, b) D-Sorbitol (4 g L^{-1}), c) Sucrose (8 g L^{-1}) CD=450mA cm^{-2}, [ZnO] =20 g L^{-1}, [NaOH] = 6 mol L^{-1}, T=30 °C

It is interesting to note that the addition of D-sorbitol or sucrose gives a nanostructure powder with an average particle size of 67.92 nm in the case of D-sorbitol and 84.7 nm for sucrose. This finding is in agreement with the results of specific surface area. This behavior was also reported by Qiang et al. [14] where zinc powder with a smaller grain size was obtained when cetyltrimethylammonium bromide (CTAB) or sodium laueyl sulfate (SLS) is added in comparison with the addition free. Based on AFM results, the optimum zinc powder that has a higher specific surface area and a smaller particle size can be obtained by adding D-sorbitol at a concentration of 4 g L^{-1} and applying the optimum operating conditions determined by Taguchi method.

Structural characterization of zinc powder is performed by XRD measurement for the sample of the optimum zinc powder as shown in Fig. 6. The characteristic zinc peaks at 2θ values 36 (002), 39 (100), 43 (101), 54 (102), 70 (103), 71 (110), and 77 (004) while smaller peaks of zinc oxide are also shown at 33, 35, 48, 57 and 63. The presence of zinc oxide in the powder results from a trace of an alkaline zinc oxide solution which may be entrapped within the powder in addition to a further oxidation during the drying. However, this zinc oxide is below the recommended value, as the percentage of zinc powder obtained is 99.3%. The surface morphology and shape of the optimum zinc powder sample is shown in Fig.7.

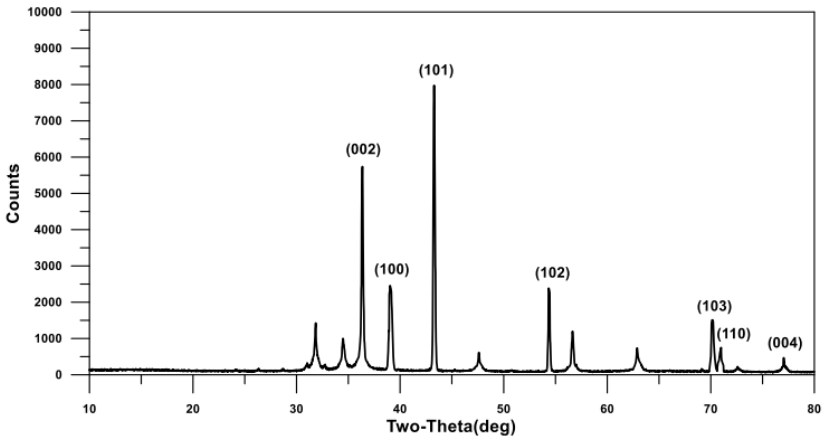


Figure 6. XRD pattern of zinc powder
CD=450mA cm^{-2}, [ZnO] =20 g L^{-1}, [NaOH] = 6 mol L^{-1}, [D-Sorbitol] = 4 g L^{-1}, T=30 °C

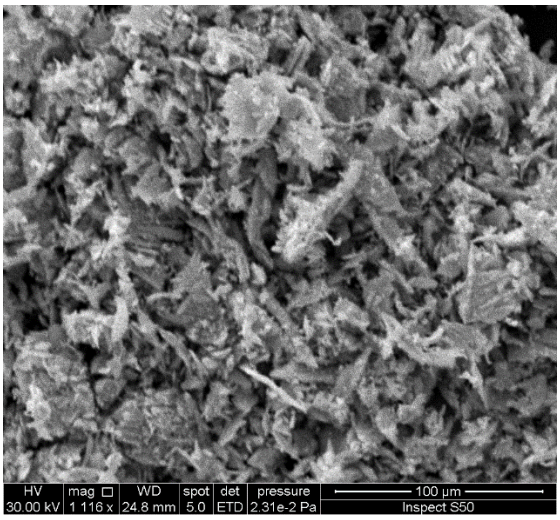


Figure 7. SEM micrograph of the optimum zinc powder.
CD=450mA cm^{-2}, [ZnO] =20 g L^{-1}, [NaOH] = 6 mol L^{-1}, [D-Sorbitol] = 4 g L^{-1}, T=30 °C

From a macroscopic viewpoint, the prepared powder consists of particles having the shape of classical dendrites, which results in a higher specific surface area. This is very essential in zinc powder applications such as extraction of gold and silver, and cementation where a high specific surface area is needed to increase the reaction rate.

4. CONCLUSIONS

The optimum conditions for ultrafine zinc powder preparation by elecrodeposition in an alkaline solution (6 mol L^{-1} NaOH) at higher specific surface area were determined as:

– Current density= 450 mA cm^{-2}

– Electrolyte temperature = 30 °C

– ZnO concentration =20 g L^{-1}

– D-sorbitol concentration= 4 g L^{-1}.

A current efficiency of 91.3 % with an energy consumption of 3.0 KWh kg^{-1} were achieved under the above conditions. The produced powder has a specific surface area of 6.218 m^2 g^{-1} and an average particle size of 67 nm. The addition of D-sorbitol led to increase the energy consumption by 12 %, however, it is still less than the industrial value (3.25-3.4 KWh kg^{-1}) with the advantage of increasing the specific surface area by 40 %. Therefore the present results are considered to be cost-effective due to the higher specific surface area achieved, in addition to simple and easy to be operated and managed the preparation method at a lower cost.

The Most effective parameters on the zinc powder preparation were ZnO concentration followed by current density while the temperature was not a statistically significant parameter. Taguchi method can successfully be applied to the preparation of zinc powder for maximizing the specific surface area. The statistical model developed by using the experimental results was effective in

navigating the design space of the process. The addition of D-sorbitol resulted in finer and more homogeneous zinc powders.

Due to the higher specific surface area and the dendrite nanostructure of the prepared zinc powder, it could be used as an anode material in zinc-MnO_2 and zinc-silver oxide batteries which required a larger specific surface area [7], also it may be suitable for preparation of zinc rich-paints with improved properties. Further investigation in these directions is a promising step.

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