Production and Characterization of Nano-sized Copper particles by Electro deposition

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Abstract

The synthesis of copper NPs many routs ,thermal decrease , chemical decrease and metal vapour synthesis , radiation routes ,removal by laser . Electrolytic precipitation is one of the most suitable, simplest and low cost routes are using for broad range of materials.were put to produce copper nano utilized electrolytic routes . It could be possible to get onnano copper for large scale . They are properties by X-ray diffraction (XRD), scanning electron microscope(SEM).The morphology deposited structure of the nano copper powder prepared by electrolysis has dendritic shape.The particle size was decreased with additives and morphology of copper powder particles was changed from dendritic shape to cluster shape with presence of sodium sulphate and gelatin.

Keywords:-Nano Copper Powder, Sulphate Bath, electro deposition.

إنتاج وتشخيص دقائق نحاس نانوية منتجة بطريقة الترسيب الكهربائي

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الخلاصة

العديد من الطرق تستخدم لإنتاج دقائق نحاس نانوية بطرق مختلفة مثل الاختزال الحراري، الاختزال الكيميائي لبخار المعدن ، الطرق الإشعاعية ، القصف بالليزر ، والترسيب الكهروكيميائي وهو من أكثر الطرق ملائمة لإنتاج النحاس بهيئة مسحوق نانويلأنها طريقه بسيطة وذات كلف قليلة بالإضافة لاستخدامها في إنتاج العديد من المعادن على هيئة مساحيق . وفي هذا

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البحث استخدمت الطريقة الكهروكيميائية لإنتاج مسحوق نحاس نانوي بوجود بعض المضافات مثل كبريتات الصوديوم والجيلاتين ثم تشخيصه بتقنيات مختلفة مثل أشعة اكس و المجهر الالكتروني الماسح . وقد اظهرت النتائج امكانية الحصول على مسحوق نحاس بحجوم نانوية بأستخدام الترسيب الكهربائي على هيئة تجمعات شجيرية وبأضافة كبريتات الصوديوم والجيلاتين تحولت الى تجمعات عنقودية نانوية.

كلمات مفتاحية: مسحوق نانو نحاس،وسط من الكبريتات ،الترسيب الكهربائي

Introduction

Nanotechnology has lately become one of the most important of learning include various science , overall physics, chemistry, biology and engineering. Attention in this discipline is at most due to two cause . Firstly, nano scale materials have many prospects in different technological applications due to most of the time they view functionalities. There is a tremendous field of creating new learning in demonstrate the size rely of the growth of different physical properties , and in demonstrate new and previous unnoticed features. Nanostructured materials can be utilize out put active devices with amended functionalities. For example, one dimensional nanomaterial(tubes, wires, rods, etc.)is an important group of nanostructured with potential applications in electronics ,composite or sensor developments[1] . nanotechnology is the capacity to form nano-sized particles, for example nano powders, which are solid Nano powders can be utilized in most of the mentioned applications; it has been an interesting field [2]. The major interest has been focused on metal nanoparticles due to their bigger applications and special feature in varied fields. Among various non-metal and metal particles, copper nanoparticles have attracted great attention due to of their catalytic, optical, and electrical conducting feature . For the synthesis of copper nanoparticles several routs were developed, thermal reduction, mechanical corrosion , chemical reduction, metal vapour prepare , radiation routs , laser ablation and micro emulsion techniques. In nanoparticle preparation, it is very significant to control the particle size, particle shape and morphology. prepared by mechanical chemical rout have decrease purity and wide particle size distribution. Gasevaporationrout display the costly raw material and complex

equipment[3,6].Nanoparticles prepared electrolytic precipitation is one of the most suitable, simplest and decrease cost routs which are using for wide range of materials and properties [7]. Therefore, in the current search , it was planned to output nano copper powder by utilized electrolytic precipitation from copper sulphate bath under suitable bath conditions[8] and as well as such as sodium sulphate and gelatin[9].

2. practical part

2.1.Electrochemical Synthesis:-

Electrochemical or electro wining method was adapted to recover of the copper powder from commercial electrolyte solution containing more than approximately 25g/L of copper. Most suitable conditions to produce copper powder deposits were used in this work.

2.1.1.Production of Nano Copper Powder:

The recovery of copper powder from copper sulphate with H_2SO_4 solution was performed in the present research by using laboratory apparatus as shown in Figure (2-1). By electrolyzing procedure , copper nanoparticles precipitation on the cathode surface was shown , they removed from the cathode by simple tapping of the cathode electrode. Reversing the current flow every 5-1 min. solves the problem of powder removal. The deposit is collected by filtering the solution through filter paper and washed thoroughly with hot distilled water several times to separate itfrom the electrolyte. The wet powder is then treated with a dilute solution of Na_2CO_3 (0.05 %) to remove traces of acid. The carbonate excess is washed off with distilled water several times. The powder is finally washed with a 6 g/l solution of sodium potassium tartarate as anti-oxidant during drying. This powder dried in oven atabout 110 C0.The powder is removed from the filter paper and stored. Different operating conditions which are required to obtain a deposit of powder can be summarized as shown in Table (2-1).These conditions were used for the production

of electrolytic nano copperpowder.

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Figure Error! No text of specified style in document..1 Apparatus of the electrochemical system

Electrochemical Cell:-

Electrochemical cell as shown in Figure (2.2) consists of a cleaned glass vessel (2L). The volume of the electrolytic solution was only 1L, two lead plate (10cm×10cm×1mm) electrodes as anodes, one copper rode (10cm ×0.8mm) electrode as cathode. The top of the cell (the lid) was made of polymethylacrylate (perspecs) which contained holes through which the electrodes and thermometer were passed and fixed. A low-voltage power supply was used to supply the electrolytic cell with the required direct current density. Circulation of electrolyte was stirred by magnetic stirrer. Surface electrodes were cleaned and connected with positive (Anode) and negative (cathode) of (DC) power supply unit. Electrolysis of this solution was done by crossing constant current inside solution out of an anode and a cathode. Figure 2.3 shows a flowchart lay-out of the electrical circuit.

Figure Error! No text of specified style in document..2 Apparatus of the electrochemical cell

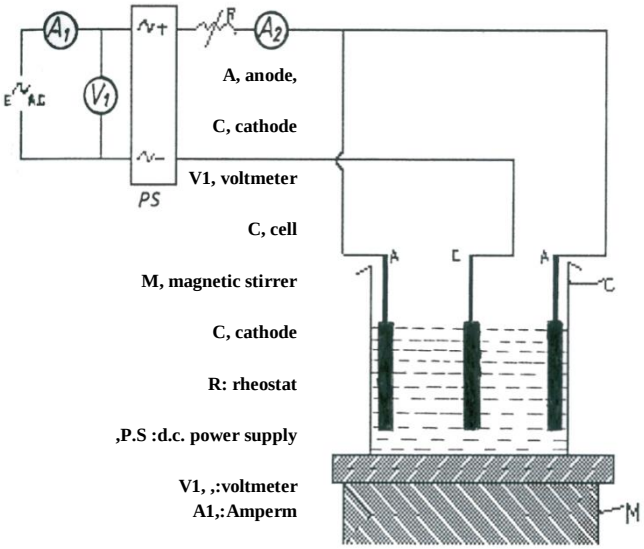

Figure Error! No text of specified style in document..3 Schematic lay-out of the electrical circuit of electrochemical system.

2.1.Characterization of copper powders[10,11]

The morphology and crystal structure of copper powders were examined by SEM (Fig.3.1 ,3.2 ,3.3) and XRD (Fig.3.5). The copper was tested for its purity utilized EDS analysis (Fig.3.4).

Results and Discussion

Table Error! No text of specified style in document..1 Bath conditions for preparation of copper powder

Parameter	Quantity
Copper content in electrolyte	5-30 g/l
Sulfuric acid content in elec.	50-150 g/l
Additive: gelatin	0.5 g/l
Sodium sulphate	3 g/l
Temperature	1 hour
Dimension of anode (lead plate)	(10 cm*10 cm*1mm)
Dimension of cathode (copper rod)	(10 cm *0.8 mm)
Circulation	140 cycl/min
Time of Experiment	2 hours
Current density	14.5 A/dm^2
Bath potential	1.0-1.5v
Distance between elect.	1.5 cm
Reversing current flow	every 5-1min

Dritic morphology is a characteristic of all copper powders at high current density was applied through the bath. model dendritic particles of copper powder are shown in Fig.3.1..

1. Characterization of copper powders:-

The XRD patterns of copper powders are shown inFig.(3.5). From SEM images (Fig.(3.1,3.2,3.3) of copper powderswere highly branched dendrites underconditions and the dendrites consisted of agglomerates ofcopper grains.

1.1 Scanning Electron Microscopy (SEM)

SEM images gave additionally more clear shape of . Dendritic morphology is a characteristic of micro copper powder obtained from electro-deposition (using copper sheet as anode, low current density8.7 A/dm^2, distance between electrodes was 2cm) with diameter from 179 to 434 nm (Fig.3-1).

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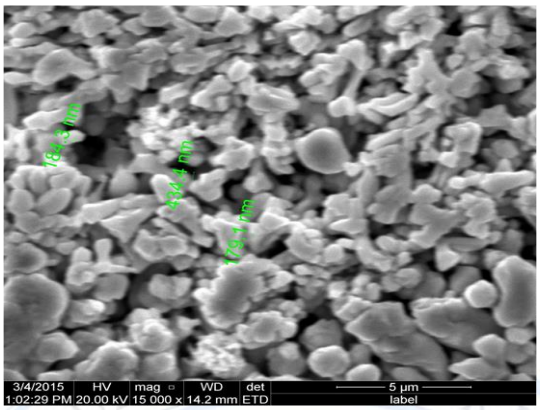

Figure 3.1 SEM images of micro copper powder by electrolysis

From figure (3-2) showed SEM images of nano copper powder obtained from electro-deposition (using lead sheet as anodes, current density at 14.7 A/dm^2, distance between electrodes was 1.5cm). The images showed dendritic and cluster shape (diameter from 103 to 144 nm) as a characteristic of copper powder obtained from electro chemical method.

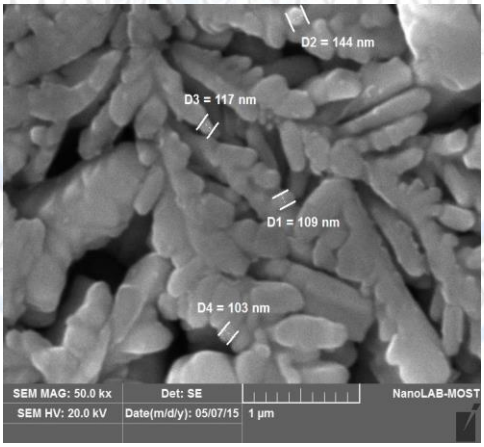

Figure 3.2 SEM images of nano copper powder by electrolysis using lead as anodes, without additives

The addition of agents such as sodium sulfate (3g/l) and gelatin (0.05g/l) to the sample produced more fine particles with average diameters of 45 to 62 nm (Fig.3-3).

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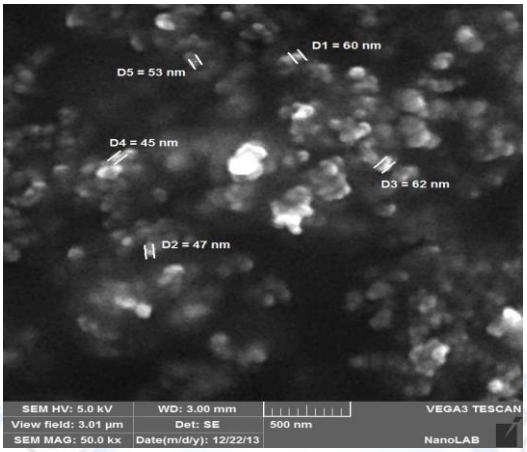
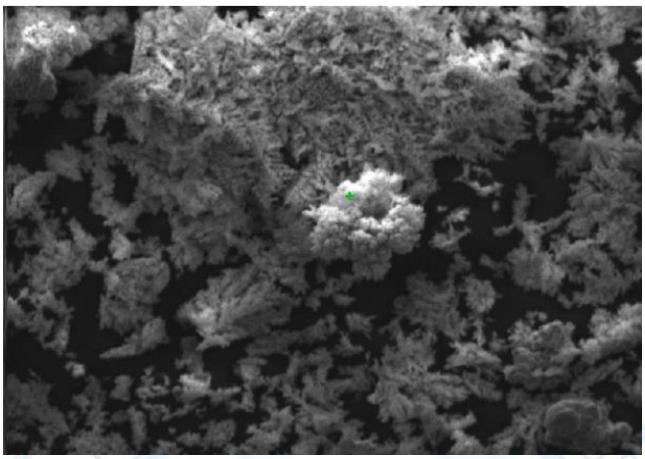


Figure3.3 SEM images of nano copper powder by electrolysis with (3g/l) Na_2SO_4 and (0.05g/l) of gelatin

Energy Dispersive Spectroscopy (EDS)

EDS was used to establish the elemental composition of the nano copper powder obtained from electrolysis, the EDS spectrums of the nano copper powder revealed that the major chemical elements including Cu and O, no impurity was detected,(Fig.3.4).



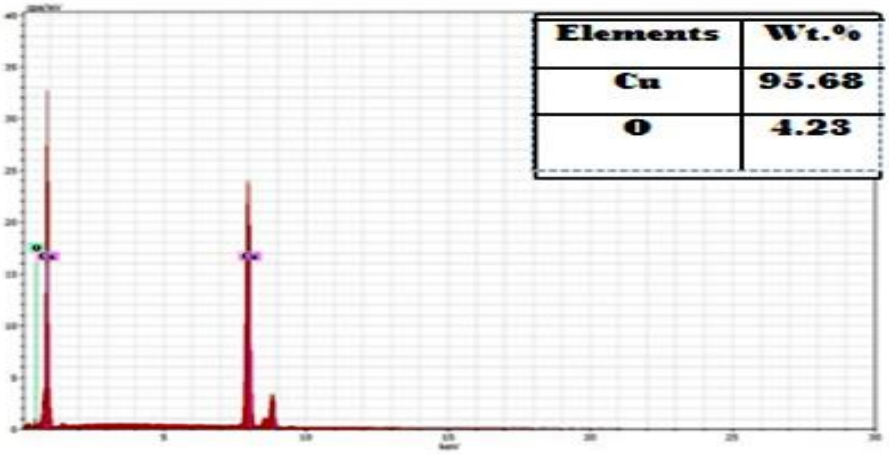


Figure3.4 EDS image and spectrum of Cu nano powder

X-Ray Diffraction (XRD)

XRD types of the properties nano copper powder sample were show at (Fig.3.5). The nano copper model has the main characteristic peaks for pure metallic Cu at 2θ amount of 43.33, 50.45 and 74.13with no other peaks as being offer . The result specific that there were no copper oxides or other crystalline materials formed and only pure Cu powders were under given practical conditions. The density of peaks reflects the high degree of crystallinity of copper nano particles. However, the different peaks are broad which specific that the crystallite size is low [12].

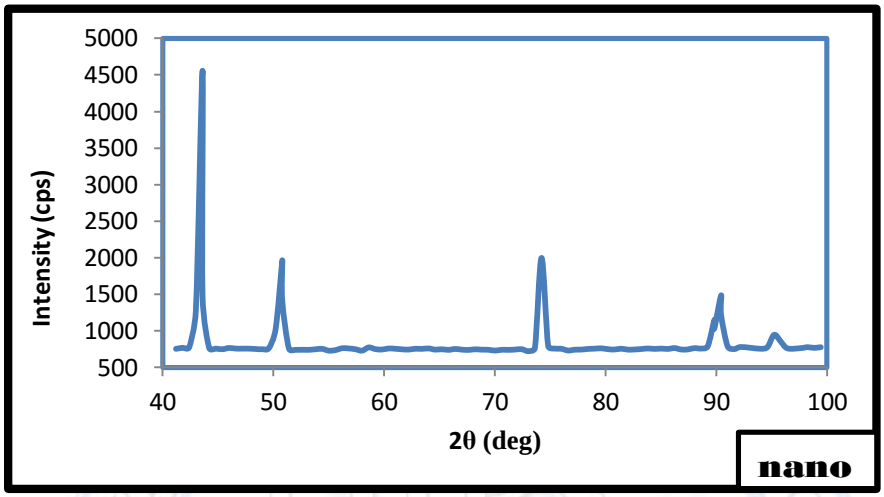


Figure 3.5 XRD pattern of nano, copper powder

Conclusion

The results of this paper showed thattheelectro deposition method was useful to production of copper powder with nano size . At the same time the recovery of copper powder from copper sulphate with H_2SO_4solution could be possible to obtain nano copper powder. The morphology deposited structure of the nano copper powder prepared by electrolysis has dendritic shape.The particle size was decreased with additives and morphology of copper powder particles was changed from dendritic shape to cluster shape with presence of sodium sulphate and gelatin.

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