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Synthesis and Applications of Copper Nanopowder – A Review

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Abstract. Copper nanopowder demonstrates excellent conductivity, catalytic behaviour, excellent compatibility and antibacterial properties. Copper nanopowder has been used as an essential component in the future nano-devices, a wide range of industrial applications and used as a replacement for more expensive metals. Copper nanopowder properties are strongly influenced by shape, size and size distribution which are often varied by varying the synthetic methods. Accordingly, this review presents different of synthetic methods of copper nanopowder and applications of this nanopowder in different fields.

1. Introduction

The copper nanopowder is a copper powder that has particle sizes below 100 nm and considered as an alternative material other than precious metals such as gold and silver that are more economical wherein pure form has good electrical and heat conductivity properties and are antibacterial. In addition, the advantages possessed by nano-sized powder particles are the value of the ratio of surface area to high volume. These considerations cause copper nanopowder widely used for electronics, magnetic, optical, catalysts, and pigments [1].

The potential needs of nanomaterial show an increasing trend [2]. The constraints faced during the execution of the production of copper nanopowder are the sensitivity of the copper nanopowder to the aqueous environment and the air, which will aggregate and oxidize. The steps taken to overcome these constraints are the conditioning of inert environments using argon and nitrogen gas when packaging or providing a protective layer of polymer or surfactant before packaging [1]. Another obstacle is hazard [2].

Nanoparticles are synthesized by three different methods: chemical, physical, and biological [3]. Each synthesis method has advantages and disadvantages in producing copper nanopowder. The synthesis method was chosen to be applied consider several things including morphology, size distribution, cost, the scale of production and hazard [3]. In this review will be presented about the method of synthesis according to its classification by displaying its advantages and disadvantages along with the characteristics of copper nanopowder produced. Similarly, about the application of copper nanopowder. This review complements the previous review of copper nanopowder by displaying information about copper nanopowder that has been produced by industry and the methods

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used. Specifications of copper nanopowder that has been produced from several industries will also be delivered.

2. Methods of synthesis

The synthesis methods of copper nanopowder can be done by chemical reduction, microemulsion reduction, sonochemical and sonoelectrochemical, microwave, photochemical, electrochemical, sonoelectrochemical, thermal decomposition, laser ablation, mechanical / ball mill, wire discharge, the electric explosion of wire, and the biological method that used bacteria, fungi, and plants. Table 1 listed the copper nanopowder synthesis methods into the chemical, physical, and biological categories.

Table 1. Copper nanopowder synthesis methods.

Chemical methods	Physical methods	Biological methods
Chemical reduction [38, 39]	Laser ablation [24]	Bacteria [34]
Microemulsion reduction [40, 41]	Mechanical mill [25]	Fungi [35]
Sonochemical [42] &	Pulsed Wire Discharge [27]	Plants [37]
Sonoelectrochemical [43]	Electric Explosion of Wire [29]	
Microwave [15]	Gas evaporation [31]	
Photochemical [17]		
Electrochemical [44]		
Thermal decomposition [22]		

The characteristics of copper nanopowder such as size, morphology, and particle size distribution depending on the applied synthesis method. Table 2 listed the characteristics of copper nanopowder in each method.

2.1. Chemical reduction

The chemical reduction method for the synthesis of copper nanopowder is the easiest, simplest and most commonly used [1]. It is generally carried out in aqueous media using copper salts as a source of copper and reducing agents such as sodium borohydride [4], hydrazine [5], ascorbic acid [6], polyol [7], isopropyl alcohol with Cetyl Trimethyl Ammonium Bromide (CTAB) [8] & glucose [9]. Reverberi et al. in his study used a vanadium reducing agent (+2) salt [10]. In addition to being in aqueous media, other media in the form of the organic solvent was once used in the synthesis of copper nanopowder [11]. The morphology and size of the resulting nanopowder particles can be controlled by selecting the reducing agent and regulating the reaction conditions. The disadvantage of this method is its toxic and flammable chemicals. In addition, the surface of the nanoparticles is easily oxidized, so it is necessary to use inert environmental conditions (nitrogen or argon atmospheres) or surface-active agents to protect the surface of nanoparticles such as surfactants, dissolved polymers, weak acids, etc. known as capping agent [1].

2.2. Microemulsion reduction (colloidal)

The synthesis of copper nanopowder applies a chemical reduction method in an organic compound solvent in the form of microemulsion water in oil (W / O) such as Water / Isooctane / Cyclohexane [12] or water oil (O / W) or water in supercritical carbon dioxide (W / Sc. CO_2). Compared with the chemical reduction method above, the application of microemulsion method can produce copper nanopowder with morphology and size distribution more evenly, but its operation is more expensive and needs to separate the solvent from product [1].

2.3. Sonochemical & Sonoelectrochemical

Synthesis of copper nanopowder can be done by applying sonochemical & sonoelectrochemical method. The working principle is to use ultrasound radiation (frequency 20 kHz to 10 MHz) in the copper salt electrolyte solution. The chemical and physical effects of ultrasound arising from the species, growth, and implosive collapse of bubbles [13]. The sonoelectrochemical is the use of ultrasound radiation on the electrochemical method. Some different arrangements of equipment have been used for the introduction of the ultrasound irradiation into the electrochemical systems [14].

2.4. Microwave
A microwave method is a form of electromagnetic energy in the frequency range between 300 MHz to 300 GHz, in the synthesis of copper nanopowder, can accelerate volumetric heating and kinetics, rapid volumetric heating and kinetics, short reaction periods and increasing yields of products compared to conventional heating methods [1]. The size of the resulting nanoparticles can reach 10 nm [15].

2.5. Photochemical
The photochemical method for synthesis of copper nanopowder utilizes light intensity. Photochemical techniques provide several advantages over conventional chemical means, such as (a) the reduction of metal ions can be carried out without using reducing agents and so avoiding undesired by-products of the reductant, (b) through the choice of suitable wavelength and concentration the rate of reaction can be controlled, (c) the light, which works as a reducing agent, is uniformly distributed in the solution, (d) irradiation can be performed at room temperature [16]. Kapoor and Mukherjee [17] using photochemical (UV light) method with poly (N-vinylpyrrolidone) (PVP) as a stabilizer can produce copper nanopowder with an average size of 15 to 20 nm.

2.6. Electrochemical
Copper nanopowder is produced by an electrochemical method by applying an electric current between the anode and cathode electrodes in the electrolyte solution. The reduction process occurs on the surface of the cathode. The deposition of copper powder on the cathode surface can be obtained at higher overpotentials corresponding to the limiting diffusion current density. The reaction of hydrogen evolution also occurs on the surface of the cathode [18]. The electrochemical method is a simple method. The process is fast and more economical to synthesize copper nanopowder. This method offers a relatively clean, non-toxic, process flow process that can be done at room temperature and environmentally friendly [19]. Kadam [19] used copper sulfate as an electrolytic solution and supplied 2 V of voltage and 1.5 A of current for 30 minutes and successfully synthesized copper nanoparticles with the size around of 24 nm and spherical. The problem that still exists in synthesis with electrochemistry is the presence of ionic interactions and inhomogeneous divisions in particle size grains.

2.7. Thermal decomposition
The synthesis of copper nanopowder is carried out in pressurized containers and controlled temperatures such as autoclaves, where the solvent reaches a temperature above its boiling point. Following the type of solvent, called hydrothermal [20, 21] and solvothermal. Karthik et al. [22] used this method for the synthesis of 50 to 100 nm copper nanoparticles. They use copper (II) succinate precursor and oleylamine as a capping agent.

2.8. Laser ablation
The method consists of ablation of a target by intense laser radiation in a liquid, yielding to an ejection of its constituents and the formation of nanoclusters and nanostructures. Variety of liquids can be used in which the particles remain as a suspension [23]. Moniri et al. [24] produce nanoparticles of average 5 nm copper in a 1.2 J/cm^2 laser fluence.

2.9. Mechanical mill
Milling is a solid-state processing technique for nanoparticle synthesis. The micron-sized material is fed into the milling machine. The types of milling machines commonly used for the synthesis of copper nanoparticles are planetary, vibratory, uniball, and the attritor. This method has ease of operation and low production cost but has a long time for nanoparticle production. The important factors affecting the quality of the final product are the type of mill, milling speed, container, time, temperature, atmosphere, size, and size distribution of the grinding medium, process control agent, the

weight ratio of the ball to powder and extent of filling the vial [1]. Yadav et al. [25] produce a 21 nm copper nanopowder with a planetary ball mill.

2.10. Pulsed Wire discharge (PWD)

When a high-pulsed current is driven through a thin metallic wire, the Joule heating causes electrical energy deposition that can turn the whole wire into plasma. By cooling this plasma with ambient gas, we can obtain a large number of very fine solid particles. This method is not usually used in industry due to the high cost and possible inefficiency for some metals. This method is suitable for metals with high electrical conductivity, which can easily be formed into thin wires [26]. Murai et al. [27] using the PWD method with the energy of 68 J successfully synthesized 0.25 mm diameter copper wire into a copper nanopowder with an average size of 25 nm. He also examined the effect of giving coating oleic acid, and the result was 10 nm smaller than uncoiled and unoxidized for several months (over two months) after the preparation.

2.11. The Electric explosion of wire (EEW)

It is a process of explosive destruction of a metal wire under the action of high current density (more than 10^6 A/cm^2) [28]. The following peculiarities characterize EEW: time of explosion is 10^{-5} to 10^{-8} s; the temperature at the moment of explosion can reach the value more than 104 K; pressure up to 109 Pa; velocity of product recession is from 1 to 5 km/s. The material of the wire transmutes into particles of nano-sized range (10 to 100 nm) following specific conditions. Extremely non-equilibrium conditions of EEW cause some unusual properties of nanopowder [28]. Electro-explosive nanopowder has, as a rule, the spherical form of particles, they are steady against oxidation and sintering at room temperature and characterized by high diffusion activity at the heating [28]. EEW in an inert gas or hydrogen is applied to produce metal powders, alloys, and intermetallic compounds. Whereas EEW in an active chemical environment is applied to produce nanopowder of metal chemical compounds: oxides, nitrides, carbides, etc. [28]. Dash et al. [29] using the EEW method successfully synthesized 0.5 mm copper wire into a copper nanopowder with an average size of 36.34 nm and a spherical shape. The experimental conditions are 3 μF of capacitance, 22 kV of charging voltage, 0.1 MPa of Argon gas ambiance pressure.

2.12. Gas evaporation

Synthesis of metal nanopowder by the gas evaporation method is evaporation with the subsequent condensation of metal using high-intensity electron beams in an atmosphere of inert gas. The simple scalability to the industrial conditions, the possibility of evaporating any material and the high-purity of the product are advantages of particles synthesized from the gas phase [30]. Zavjalov et al. [31] obtain copper nanopowder by substance evaporation with the help of a powerful electron beam with the sizes of particles are limited in range from about 15 to 700 nm.

2.13. Biological method

It has been found that living organisms such as bacteria, fungi, and plants have great potential for the synthesis of metal nanoparticles. This method is easier to control the size of the distribution of nanoparticles synthesized than others and no toxic impact on the environment [32, 33]. Metal compounds usually reduce into their respective nanoparticles because of microbial enzymes or the plant phytochemicals with antioxidant or reducing properties [1]. Varshney et al. [34] used *Pseudomonas stutzeri* to synthesize copper nanoparticles from wastewater generated from electroplating. They have a cubic shape, and the size of nanoparticles produced by this method is 50-150 nm [3, 34]. Pavani et al. [35] used the *Aspergillus fungus* species to synthesize copper nanoparticles. Plants can be used for the synthesis of copper nanopowder through inactivated plant tissue, plant extracts, exudates, gums, and other parts of plants [36]. Lee et al. [37] have been biologically researching for the synthesis of copper nanopowder with a size of 40-100 nm using *Mangolia* plant leaf extract as a reducing agent.

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Table 2. Resume research of each copper nanopowder synthesis method.

Method	Material	Conditions	Morphology	Size	Ref.
Chemical reduction	Solvent: DI Water Precursor: $CuCl_2.2H_2O$ Reducing agent: Ascorbic acid	80°C, 14 h	Spherical	< 2 nm	[38]
Chemical reduction	Solvent: DI Water Precursor: $CuSO_4.5H_2O$ Reducing agent: Ascorbic acid Capping agent: Starch	80°C, 2 h	Cubic	25.19 nm	[39]
Microemulsion reduction	Solvent: Water in TX-100/n-hexanol/cyclohexane Precursor: $CuCl_2$ Reducing agent: $NaBH_4$	RT, 10 min	Spherical	5-15 nm	[40]
Microemulsion reduction	Solvent: Water in n-heptane, alcohol Precursor: $CuCl_2$ Reducing agent: $NaBH_4$ Stabilizer: Bis(ethylhexyl)hydrogen phosphate	25°C, 12 h	Spherical	60 nm	[41]
Sonochemical	Solvent: Water Precursor: $CuSO_4$ Reducing agent: Hydrazine monohydrate Stabilizer: Ethylene glycol		Spherical	108 nm	[42]
Sonoelectrochemical	Solvent: Water Precursor: $CuSO_4.5H_2O$ Stabilizer: poly(N-vinylpyrrolidone)	480 mA/cm^2, 30 min	Spherical	25-60 nm	[43]
Microwave	Solvent: Ethylene glycol Precursor: $CuSO_4.5H_2O$ Reducing agent: $NaH_2PO_2.H_2O$ Stabilizer: poly(N-vinylpyrrolidone)	5 min	Spherical	10 nm	[15]
Photochemical	Solvent: Water Precursor: $CuSO_4$ Stabilizer: poly(N-vinylpyrrolidone)	20°C, 20 min	Spherical	15-20 nm	[17]
Electrochemical	Solvent: Water Precursor: $CuSO_4$	2 V, 1.5 A	Spherical	24 nm	[44]
Thermal decomposition	Solvent: Oleylamine Precursor: Copper (II) succinate Stabilizer: Oleylamine	245°C, 45 min		50-100 nm	[22]
Laser abrasion	Copper metal plate	1.2 J/cm^2 RT, 10 min	Spherical	5 and 16 nm	[24]
Mechanical ball	Copper powder Wet milling medium: Toluene Rectified	40 h		21 nm	[25]
Pulsed wire discharge	Copper wire Coating: Oleic acid	473 K, 10 μF, 5.2 kV, 68 J	Spherical	25 nm	[27]
Electric explosion of wire	Copper wire	0.1 MPa, 3 μF, 22 kV	Spherical	36.34 nm	[29]
Gas evaporation	Cylindrical copper ingot Flux: Argon	5 MW/cm^2	Spherical	15-700 nm	[31]
Biological	Solvent: Water Precursor: $CuSO_4.5H_2O$ Biological entity: Bacteria *pseudomonas stutzeri* from electroplating waste			150 nm	[34]
Biological	Solvent: Water Precursor: $CuSO_4.5H_2O$ Biological entity: Fungi *aspergillus fungus*			50-150 nm	[35]
Biological	Solvent: Water Precursor: $CuSO_4.5H_2O$ Biological entity: *Mangolia* plant leaf extract			40-100 nm	[37]

3. Copper nanopowder characteristics

Copper nanopowder synthesis methods that have been applied in the industry are electrochemical, electrical explosion and laser ablation. Table 3 shows the copper nanopowder specifications that produced in the industry as well as the methods used.

Table 3. The copper nanopowder product specification and methods in industry[a].

Method	Purity Cu (% Metal basis)	Color	APS (nm)	SSA (m^2/g)	Morphology	Density (g/cm^3)		Other	Ref.
						Bulk	True		
EEM	99.9	Saddle brown	40	10-14	Spherical	0.21	10-14		[45]
ECM	99	Black brown	30	35		0.15-0.35	35	PP	[46]
LSM	99.8	Black	25	30-50	Spherical	0.15-0.35	8.94	CC	[47]

[a] US Research Nano Materials, Inc.
EEM=Electrical Explosion Method; ECM= Electrochemical Method; LSM= Laser Synthesized Method;
APS= Average Particle Size; SSA=Specific Surface Area; PP= Partially Passivated Cu_2O; CC= Carbon Coated.

A common characteristic of copper is a ductile metal that has high thermal and electrical conductivity. Pure copper has a soft and orange-reddish color. The characteristics of the above copper nanopowder product specifications can be determined using a test apparatus to determine the purity level of copper nanopowder, particle size, particle size distribution, morphology and specific gravity.

4. Commercial applications using copper nanopowder

Copper is an alternative metal substitute of precious metals that have high electrical conductivity and heat properties making it ideal for reducing production costs and used in industrial applications for high strength metals and alloys, high thermally conductive materials and capacitor materials. As a nanopowder, the main characteristic is to have a high SSA (Specific Surface Area) so that copper nanopowder can be used in a variety of industrial applications, including anti-biotic, antimicrobial, anti-fungal agent, conductive ink, & conductive paste for printed electronics, sintering additives, lubricant additives, and catalysts. [1, 3]

5. Conclusions

Several methods of synthesis of copper nanopowder have been delivered. Chemical methods are the most commonly used method for the preparation of nanoparticles but still involves toxic materials. Physical methods can produce faster results and do not involve toxic materials but are still costly. While biological methods known as nanoparticle synthesis are environmentally friendly but require a very long time synthesis.

Copper nanopowder has very high electrical and heat conduction, excellent physicochemical properties; good biocompatibility and high surface activity, and therefore is very promising for magnetic nano-devices and some electronic and medical applications as well as the incorporation of materials and medicines.

Research is still ongoing in search of the latest methods that offer the lowest and least expensive environmental damage to continue. Similarly, research to discover new properties of copper nanoparticles and new products.

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