



**SYNTHESIS, CHARACTERIZATION AND BIOCIDAL EFFECT
OF COPPER NANOPARTICLES USING L-ASCORBIC ACID AS A
REDUCING AND CAPPING AGENT**

By
Younus Rashid Taha Al-Mashhadani

A Thesis Submitted to the
Faculty of Engineering at Cairo University
in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF SCIENCE
in
Chemical Engineering

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
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Title of Thesis:

Synthesis, characterization and biocidal effect of copper nanoparticles using L-ascorbic acid as a reducing and capping agent

Key Words:

Metallic nanoparticles, Copper Nanoparticles, Chemical reduction, Antibacterial, L-ascorbic acid

Summary:

Chemical reduction method is one of chemical techniques for synthesis nanoparticles. It has been used to reduce metals such as copper by using copper salts with reducing agents. In this work, copper nanoparticles were synthesized by reduction of copper salt (copper chloride dehydrate; $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ or copper sulfate pentahydrate; $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) by using L-ascorbic acid as reducing and capping agent at the same time. This thesis presents a study of factors affecting copper nanoparticles synthesis using L-ascorbic acid, where the factors selected are reaction time, molar ratio of L-ascorbic acid to copper salt. Dynamic Light Scattering (DLS) used for measuring the size of nanoparticles and size distribution of these nanoparticles while Transmission Electron Microscopy (TEM) was used to provide information about morphology and crystallographic structure of copper nanoparticles as well as the size of nanoparticles.

Antibacterial activity of the copper nanoparticles solutions was examined against pathogenic organisms such as Gram positive bacteria, Gram negative bacteria and fungi. The Minimal Inhibitory Concentration (MIC), Minimum Bactericidal Concentration (MBC) and inhibition zones were determined. The results obtained from this work in antimicrobial field confirm that copper nanoparticles have inhibitory effects and fatal on bacterial and fungal strains selected.

In the name of Allah, the Most Merciful, the Most Compassionate

(And not absent from your Lord is any [part] of an atom's weight within the earth or within the heaven or [anything] smaller than that or greater but that it is in a clear register)

Sorah Yunus (61)

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DEDICATION

This Thesis manuscript is dedicated to my parents and my grandmother (May Allah have mercy on them). Their love and unwavering support has always been the bedrock upon which every worthwhile achievement in my life has been built.

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ABSTRACT

A chemical reduction method is one of methods which used to produce copper nanoparticles. In this method, copper salt such as (Copper (II) chloride dihydrate; $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ and copper (II) sulfate pentahydrate; $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) were used as precursor. L-ascorbic acid was used as a reducing and capping agent with copper salt.

The parameters such as reaction time and molar ratio of L-ascorbic acid to copper salt were changed in this work to study their effect on size of particles. The size of particles and size distribution were measured by Dynamic Light Scattering (DLS) while the morphology of copper nanoparticles was determined by Transmission Electron Microscopy (TEM).

The results showed that increasing time of reaction and increasing the molar ratio of L-ascorbic acid to copper salt decrease the copper nanoparticles size. The copper nanoparticles with size less than 12 nm and spherical in shape were prepared using the chemical reduction method. The copper nanoparticles colloidal solution was stored in ambient conditions after preparation for more than four months but no any change was showed. The synthesis process using of L-ascorbic acid has the advantages such as non-toxic, inexpensive, use simple equipment under ambient conditions and environmental friendly.

Antimicrobial tests of copper nanoparticles were carried out on various types of bacteria (gram positive bacteria and gram negative bacteria) and fungi. The prepared aqueous solutions of copper nanoparticles were diluted to different concentrations and 10 μl of each dilution was spotted on the overlay of each bacterial culture by impregnating the as-synthesized copper nanoparticles using micropipette on paper discs.

The Minimum Inhibitory Concentration (MIC) is the lowest concentration for an antimicrobial agent which inhibits a microorganism growth. The zone of inhibition was measured after 24 h of incubation.

The Minimum Bactericidal Concentration (MBC) is defined as the lowest concentration for an antimicrobial agent that will kill 99.9 % of a microorganism.

The Minimum Bactericidal Concentration (MBC) was measured after completion of Minimum Inhibitory Concentration (MIC) test.

The results showed that the copper nanoparticles exhibited antimicrobial activity and that the lower the particles, the higher the biocidal effect on both bacteria and fungi.

Keywords: Metallic nanoparticles, Copper Nanoparticles, Chemical reduction, Antibacterial, L-ascorbic acid

Chapter one: Introduction

1-1 Background

The concept of nanotechnology is attributed to Richard Feynman (American physicist, 1959). He was the first scientist invited to discuss nanoscience in his famous lecture "There's Plenty of Room at the Bottom" Richard highlighted the importance of controlling and manipulating on a small scale and nanotechnology [1].

The nanotechnology idea started in (1974) when the word "nanotechnology" was used by Norio Taniguchi for the first once in a technology production paper to describe precision machining of materials to within atomic-scale dimensional tolerances [2]. In (1977) the concept of Molecular Nanotechnology was put forward by Kim Eric Drexler [3]. The scanning tunneling microscope used in (1981) to help researchers for seeing individual atoms which invented by Binnig and Rohrer. Also in (1981), the first research on engineering of molecular was issued.

In (1985), Fullerenes or called Bucky-balls were one of the first nanoparticles discovered by Richard Smalley, Harry Kroto and Robert Curl.

Bucky-balls are consisted of carbon atoms joined to three another carbon atoms by covalent bonds. Therefore, the atoms of carbon are connected in the same layout of hexagons and pentagons in the form of a hollow sphere, ellipsoid or tube.

The most common Bucky-ball consists of 60 atom of carbon and it is at times called C₆₀. There are other sizes of Bucky-balls such as a balls consisting of 20 atom of carbon to a balls consisting of over than 100 atom of carbon. Bucky-balls very strong because of the covalent bonds between their atoms and the carbon atoms easily form covalent bonds with a variety of other atoms. Bucky-balls are used in composites to enhance material. Bucky-balls have the interesting electrical property of being very good electron acceptors, which means they accept freed electrons from other materials. This property is important, for example, in increasing the efficiency of solar cells in transforming sunlight into electricity.

In (1989), scientists at the IBM Research Center, California, used 35individual xenon atoms to spell out the IBM logo. They further proved how nanoparticles can be manipulated and applied in nanotechnology [2].

In (1995), the term nanofluids were coined by Choi and other researchers, have shown that it is possible to break down the limits of conventional solid particle suspensions by conceiving the concept of nanoparticle fluid suspensions. These nano particle fluid suspensions are called nanofluids, obtained by dispersing nanometer sized particles in a conventional base fluid like water, oil, ethylene glycol, etc. The thermal properties of nanofluids are better than those of their base fluids [4].