

SYNTHESIS, CHARACTERIZATION AND ANTIFUNGAL ACTIVITY OF METAL DOPPED CuO-MCM-41 NANOCOMPOSITES

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Abstract

Nanomaterials made from metal oxides such as CuO, Fe₂O₃, and ZnO demonstrate a wide range of pharmacological characteristics, including antibacterial, anti-inflammatory, anti-cancer, wound healing, and anti-diabetic properties. In particular, Copper Oxide nanomaterials have become increasingly important due to their growing applications in diverse areas such as heterogeneous catalysis, antimicrobial agents, antioxidants, imaging agents, cancer therapy, and drug delivery systems. The CuO-MCM-41 nanomaterials were synthesized through a hydrothermal method. The combination of the produced nanomaterials was achieved using a solution dispersion technique. Specifically, 0.5 g of the synthesized nanomaterials was dissolved in 50 ml of water and sonicated for 10 minutes. EDX analysis confirmed the presence of oxygen, copper, and silicon in the created nanocomposites. SEM images of the produced copper oxide nanocomposites showed irregular shapes with some aggregation, particularly at higher concentrations of MCM-41. The antifungal effectiveness was evaluated using the disc diffusion approach, revealing inhibition zones of 21 mm against *Aspergillus niger* and 22 mm against *Candida albicans*, thus confirming the strong antifungal properties of the synthesized nanocomposites.

INTRODUCTION

The initial idea behind nanomedicine stemmed from the notion that tiny, autonomous nanodevices and similar technologies could be conceptualized, developed, and incorporated into biological systems to mend cells at the molecular level. Today, nanomedicine has progressed into various specific applications, each reflecting the core understanding that the ability to engineer particles and devices at the molecular scale can lead to considerable progress in both scientific research and healthcare practices (Saikia, Borah, & Goswamee, 2017; Saikia, Borah, Goswamee, et al., 2017). It is described as the manipulation,

repair, production, and management of human biological systems at the molecular scale using engineered nanostructures and nanomachines. These nanoscale therapies are set to transform drug delivery from the foundation upwards (Khan et al., 2020).

Nanotechnology offers the potential for atomically precise manufacturing, which is beneficial for producing particles, various devices, and systems (Roco, 2002; Saiduzzaman, 2019). A recent advancement in the growing field of nanotechnology allows for the creation of highly organized nanoparticles of diverse sizes and

shapes, contributing to the development of

modern technology (Baig et al., 2021).

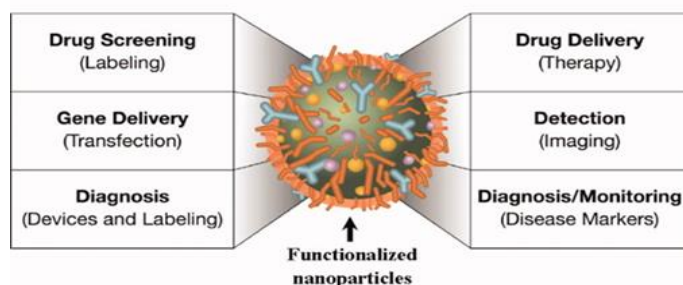


Fig.1.1: Features of Nano-Medicines

In copper oxide, the copper atom has an oxidation state of Cu^{2+} . The unit cell displays monoclinic symmetry. Within each cell, there are four copper oxide dimers and two copper oxide

units present in the primitive cell. Each CuO atom is located at the center of an oxygen parallelogram (Gattinoni & Michaelides, 2015).

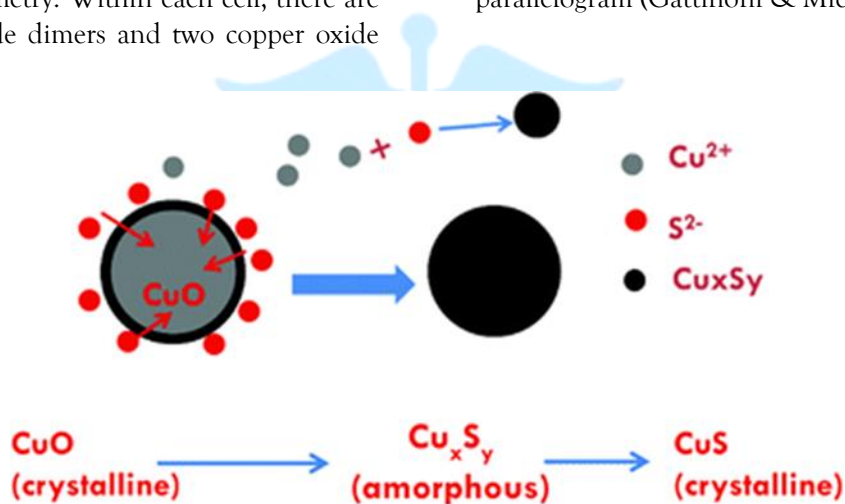


Fig: 1.2: CuO nanoparticles

Nano-composites are perceived as a contemporary solution to address the requirements of our society and the present era. Their enhanced functional capabilities and outstanding technological options and features lead to an approximate annual growth rate of 25% (Bhateria & Singh, 2019).

These nanocomposite materials are utilized in various applications, incorporating amorphous components across one, two, and three dimensions, made from notably diverse materials, and integrated at the nanoscale. The significant properties of both metal-based nanomaterials and

ceramic-based nanocomposites are particularly applicable in dental applications, where non-polymer-based nanoparticles or inorganic materials such as metals or ceramics, like hydroxyapatite, calcium phosphate, and bioactive glass nanomaterials, are essential for enamel repair and alveolar bone regeneration (Hafeez, 2022). To synthesize MCM-41, a solvent (which can be aqueous or alcoholic), a silica precursor (such as tetraethyl orthosilicate (TEOS), tetrabutyl orthosilicate (TBOS), or tetramethyl orthosilicate (TMOS)), either anionic or cationic, or non-ionic surfactant, and a catalyst are

required. Depending on the methods used, the reaction can occur in either type of ionic medium with various ratios of SiO₂ to surfactant.

The mixture is combined and allowed to age for a few hours at room temperature or at 100 degrees Celsius before being placed in a static autoclave. Copper (II) sulfate, often called copper sulfate, is an inorganic compound with the formula (CuSO₄ · H₂O)_n, where n varies from zero to five. The most prevalent form of this compound is the pentahydrate, with n equal to five. In the past, this substance was known by names like copper vitriol and Roman vitriol (Duraismy et al., 2021).

METHODOLOGY

The CuO-MCM-41 nanomaterials were synthesized using a hydrothermal method (Vadia et al., 2013). A specific quantity of 2.4 grams of cetyl trimethyl ammonium bromide was combined with 10 ml of distilled water and stirred gently for 30 minutes while gradually heating until the solution became clear. After that, 10 ml of NH₄OH solution was added, and the mixture was stirred again for 10 minutes while maintaining the pH between 9 and 12. Next, 10 ml of tetraethyl orthosilicate was added dropwise, and the mixture was mixed for 3 hours while keeping the pH at 10 through shaking or stirring. A solution of 0.5 grams of CuSO₄ was prepared in 10 ml of water and added dropwise to the previous solution. Subsequently, the mixture was transferred to a Teflon autoclave and heated at 110 °C for 36 hours in an oven.

The resultant sample was then cleaned using ethanol and purified water, filtered, and dried in an oven at 80 °C for 12 hours. Once dried, the sample was ground into a fine powder with a mortar and pestle. Finally, the fine powder was heated at 400 °C for 4 hours.

Silver Coating:

Silver Coating: The solution dispersion method was used to combine the synthetic nanomaterials (Liang et al., 2018). After dissolving 0.5 g of synthesized nanomaterials in 50 ml of aqueous medium, the mixture was

sonicated for ten minutes. After that, it was left on a magnetic stirrer for half an hour. Following a 5-minute sonication step, a silver nitrate solution was created by precisely measuring 0.1 g of silver nitrate and adding it to 15 ml of aqueous medium. After mixing the two solutions dropwise, they were shaken for three hours. Next, 0.5 g of NaBH₄ was dissolved in 15 ml of pure aqueous medium to create the NaBH₄ solution. The previously created solution was then mixed with the prepared borohydride solution and shaken for 60 minutes. Following the clarification of the mixture, the filtrate was dried for 60 minutes at 700°C in an oven. Lastly, the dried sample was heated for four hours at 400°C in a muffle furnace.

Antifungal activity assessment:

3.6.1: Disc diffusion method for evaluating antifungal activity

The disc diffusion method was used to examine the susceptibility (Bibi et al., 2011). The University of Sindh, Jamshoro, Pakistan's Department of Pharmacy provided the chosen fungal strains, which were cultivated in a 0.02% Tween 20 solution with turbidity adjusted to the McFarland standard. For eighteen hours, the synthesised samples were incubated at 37°C. Sabouraud dextrose agar, a medium for fungal growth, was added to Petri plates along with 100 µL of the fungal culture. The test samples (5 µl of 10 mg/ml DMSO) were then injected onto filter paper discs and placed on the agar's surface. Likewise, discs containing 5 µl of reference standards (4 mg/ml DMSO) served as positive controls. The common antifungal medication is miconazole. DMSO-treated discs were used as negative controls to verify that DMSO is non-toxic. After that, the plates were incubated at 28°C for a full day. Following this time, callipers were used to measure and record the diameter of the inhibition zones from the drug-treated discs and the standard discs in millimetres.

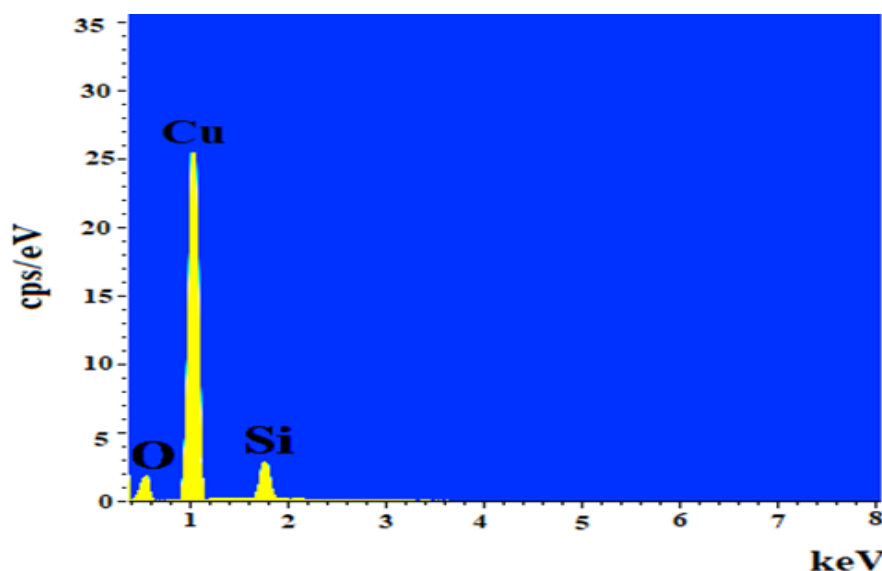


Figure 3.1: Energy Dispersive X-ray spectroscopy (EDX) of CuO-MCM-41 nanocomposites

RESULTS

An energy dispersive X-ray spectrophotometer was used to analyse the elemental content of the generated nanocomposites. The Energy Dispersive X-ray Spectrophotometer (EDAX) was

running at a 129 KV increasing voltage. EDX confirms that silicon, copper, and oxygen are present in the generated nanocomposites, as shown in figure 2.

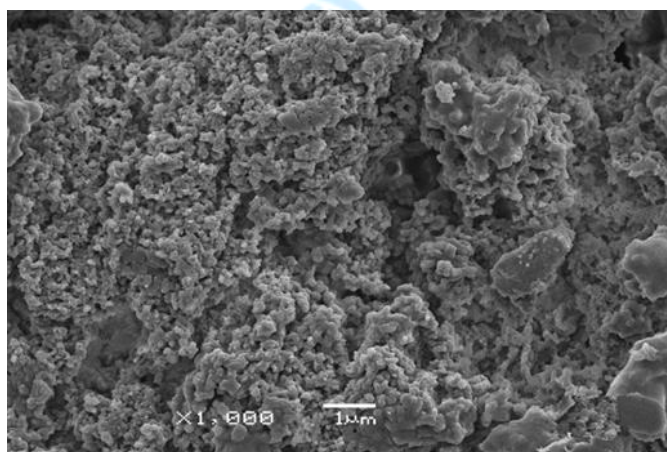


Figure 3.2: SEM of CuO-MCM-41 nanocomposites

Figure 3.2 shows the surface morphology of the produced CuO-MCM-41 nanocomposites as determined by SEM examination. The figure displays a SEM image of the generated CuO nanocomposites. Because the concentration of

MCM-41, the stabilising agent, was lower than the quantity of salt precursor used, the nanocomposites exhibited an irregular shape and some aggregation. As the illustration shows, the generated nanocomposites have a rough surface.

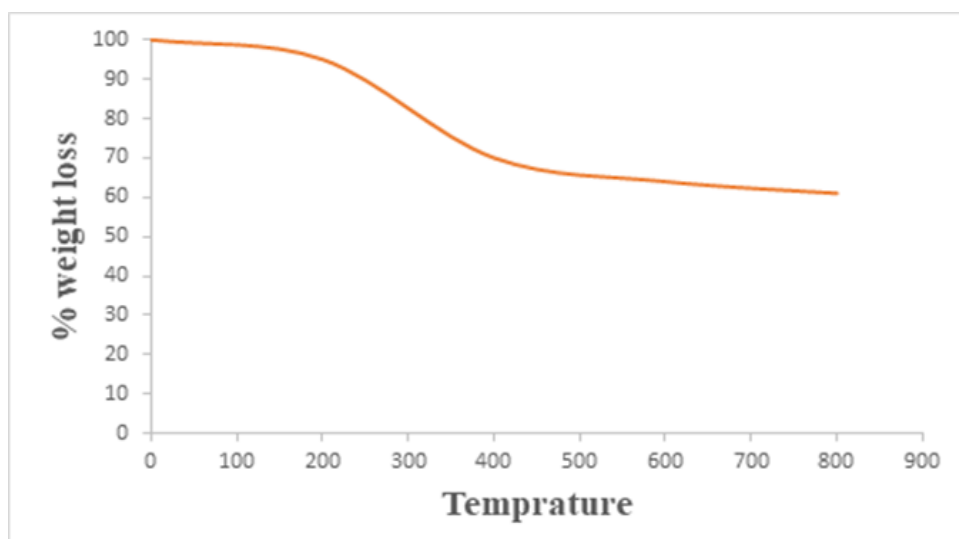


Figure 3.3: TGA of CuO/MCM-41-Ag nanocomposites

shown in Figure 3.2. The TGA curve showed three distinct phases of weight decrease. Up to 800 °C, around 30% of the weight of the synthesised substance is lost. It was demonstrated that the initial weight loss decreased the quantity of water that was readily absorbed and the volatility of several solvents up to 425°C. Between 300 and 500 °C, an additional amount

of weight loss was observed; this loss is only attributable to the removal of bound water and any impurities that could have experienced heat degradation. Ultimately, the weight loss process was significantly slowed down, and CuO/MCM-41 nanocomposite production started above 600 °C.

Table 01: Using the disc diffusion technique, copper oxide-MCM-41 nanocomposites (20 mg/ml) inhibit the formation of several fungal spores.

S. No	Organism	Diameter of growth inhibition zone (mm) 20 mg/ml	Positive control	Negative control
1	<i>Aspergillus niger</i>	21	7	-
2	<i>Aspergillus oryzae</i>	20	8	-
3	<i>Candida albicans</i>	22	16	-
4	<i>Candida parasilopsis</i>	27	21	-

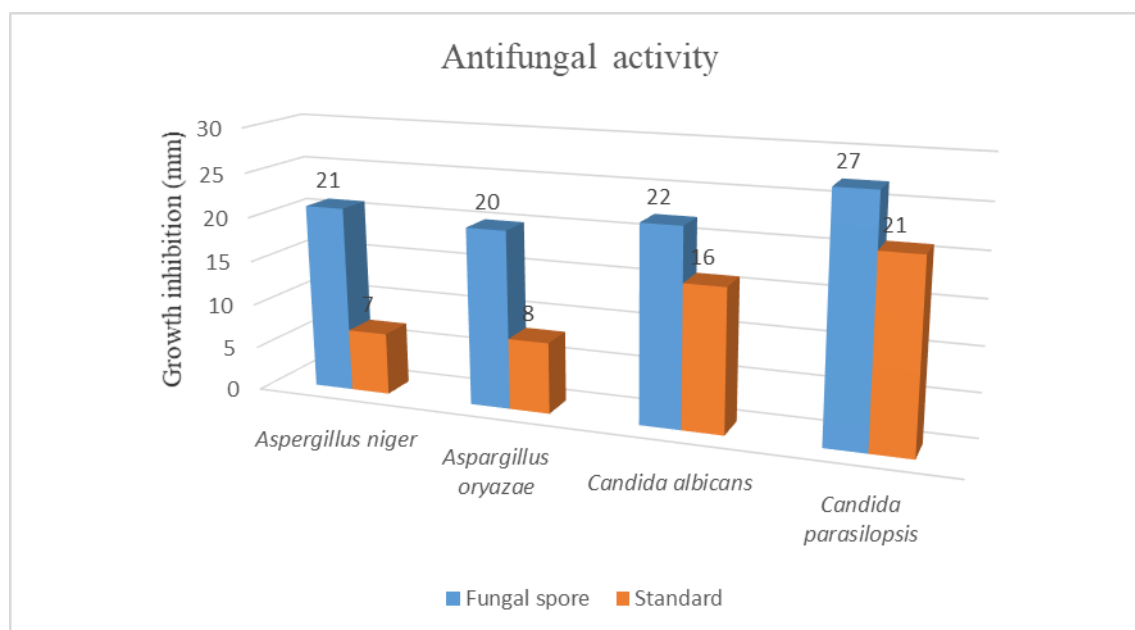


Fig. 3.4: Growth inhibition of fungal spores by disc diffusion method (20 mg/ml)

Figure 3.4 shows the zone of inhibition of several fungal spores against 20 mg/ml using the Disc Diffusion Method.

DISCUSSION

According to Yang & Zhong (2013) and Behera et al. (2019), nanomaterials are solid materials with a range of phases, one of which has a single, double, or triple dimensional structure with a size of no more than 100 nm or an arrangement with nanoscale reoccurring length between the various phases that make up the composite. The current study has only focused on the chemical synthesis of CuO-MCM nanocomposites, in contrast to Krishnan & Pravitha (2020), where the researchers performed an analogous analysis on the antioxidant effect of the copper oxide-MgO nanomaterials synthesised by chemical and biological methods.

The adverse effects of pharmaceuticals with co-occurring illnesses in terms of treatment costs and consumption are lessened when drugs are distributed to a designated target. Increasing bioavailability for a set period of time and at specific sites within the biological system was the aim of medication delivery. Molecular targeting using nanotechnology is the only method to do

this. (Patra and others, 2018). One advantage of employing nanoscale medical technology is that it may be easily incorporated into biological systems, reduce the spread, and accelerate biochemical reactions. Nanotechnology instruments work faster than conventional medication delivery.

successful drug delivery by nanotechnology depends on three key elements: i) systemic encapsulation of the active agents; ii) efficient drug release; and iii) successful drug transport to the targeted location of the biological system. Numerous nano-delivery drugs were available by 2019. The basic idea behind nanomaterials is to use functional units with widths in the nm range to organise and produce novel materials with remarkable flexibility and physical feature improvement. The current work is at odds with that of Krishnan and Pravitha (2020), who investigated the antioxidant activity of copper oxide-MgO in a similar manner. The current work has exclusively concentrated on the chemical synthesis of CuO-MCM nanocomposites among nanomaterials produced by biological and chemical methods. The present investigation aligns with the findings of Phiwang et al. (2013), who investigated the

antifungal characteristics of copper oxide/zinc nanoparticles produced by the co-precipitation technique. CuO/MCM nanocomposites were produced in this work by annealing at different temperatures for a few years. The XRD results of the reference research showed the extremely crystalline nature of the nanocomposites, which is similar to the current experiment. Similar to the current work, the authors of the reference study found that the generated nanocomposites exhibited very great antifungal activity against many fungal species. In all studies, the antifungal activity was determined using the disc diffusion technique.

CONCLUSION

The hydrothermal synthesis of CuO nanocomposites using MCM-41 and silver in an aqueous medium, as well as the assay of the generated nanocomposites and their bio-analysis against the chosen drug-resistant fungal strains, were all successfully finished in this work. The biosynthesised copper oxide nanocomposites were comprehensively tested using a variety of characterisation methods. The configuration characteristic features of the biologically generated copper oxide nanomaterials were assessed using a scanning electron microscope, EDX, and TGA. *Aspergillus niger* ATCC 16404, *Candida albicans* ATCC 10231, *Aspergillus oryzae* ATCC 10124, and *Candida parapsilosis* ATCCB78 were also employed as fungal pores. The nanocomposites showed exceptional efficacy against various fungal species.

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