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Conflict of Interest

All the authors declare no conflicts of interest otherwise disclose in the manuscript.

Data Availability

The data required in this research can be accessed by contacting the corresponding author.

Author Contributions

Dr. Sabah H. Juma prepare the samples according to the best conditions. While Dr. Reem S. Khalil carried out the measurements on the samples and contributed to writing the manuscript, Dr. Mustafa Shaker Hashim provided general supervision of the work, writing and correspondence.

ORIGINAL STUDY

Bioactivity of the Synthesized MgO Nanomaterial by Hydrothermal Technique

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Abstract

MgO was synthesized using the hydrothermal technique. The autoclave containing material based on magnesium $(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was filled and heated to 100 °C for 3 h. Electrophoretic deposition technique was utilized to deposit MgO powder on a Ti plate. To examine its bioactivity (formation of hydroxyapatite (HAp)), coated Ti with MgO was then submerged in concentrated SBF for a month. The formation of pure HAp without additional phases including calcium and phosphorus was confirmed by the XRD test. Scanning electron microscope (SEM) images refer to the formation of semispherical HAp nanoparticles with a diameter ≈ 20 nm. The inhibition zones of MgO were evaluated in order to assess their antibacterial effectiveness against *S. aureus*, *S. epidermidis*, *Escherichia coli*, *Klebsiella pneumoniae* and *Candida albicans*. MgO was not shown to have any antibacterial properties against the dangerous bacteria that were identified.

Keywords: Hydroxyapatite, Bioactivity, MgO, Hydrothermal, Electrophoretic

1. Introduction

Numerous factors, such as the surface chemistry and physics, the implant material's biocompatibility, the implantation bed itself, and the surgical implant process, are crucial for long-term implants. Bioactive biomaterials are able to bind to the tissues of their live hosts [1]. These materials behave like natural bone and have characteristics that are so close to those of normal bone that they are broken down and replaced by natural bone by osteoclasts, which are cells that dissolve bone [2].

The bioactive materials' mechanical strength limits their application in scenarios involving mechanical stress. Many approaches to solving this issue have been put out and researched. One of these methods involves creating a bioactive layer of calcium phosphate, bio composite, and glass-ceramic, and then covering the implants with these substances [3].

Bone tissue is a naturally occurring composite substance made up of both inorganic and organic components, such as collagen [4]. Approximately

65 % of the total mass of bones is made up of HAp, which has the chemical formula $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ [5]. The remaining portion is made up of water and organic matter, primarily collagen.

The high surface to volume ratio of nanomaterials gives birth to their unique features. The majority of recent technological advancements in a range of biological, physical, pharmacological, and medicinal applications may be directly attributed to these materials' characteristics [6].

Because modern implant materials are incompatible with osteoblasts (cells that make bone), implant failure can happen for a variety of reasons. Comparing nanoparticles to corresponding conventional micron-scale materials, improved cytocompatibility has been shown thus far. When compared to traditional materials, the nanostructural characteristics, bioactive surfaces, and advantageous surface chemistry of nanomaterials which substantially resemble bone—promote the production of new bone. As a result, these materials may represent the orthopaedic implant material of the future [7].

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Several methods have been used to form MgO. Mustafa et al. transformed magnesium element into MgO powder nanoparticles using the rapid break down anodization technique [8]. Also, Molecular beam epitaxy sputtering [9], sol-gel [10], solvothermal [11], thermal decomposition [12] were used to synthesized MgO.

Many oxides materials were studied for bioactivity applications, like TiO₂ and ZnO. Also, based on BaTiO₃, MgTiO₃, CaTiO₃, and SrTiO₃ perovskite oxides and their composites, these materials were investigated due to their extremely significant functions in tissue engineering (especially in bone tissues) [13].

But the using of hydrothermal technique for producing MgO is a conventional method due to its advantages. This chemical reaction takes place in a solvent at a temperature higher than its boiling point in sealed containers. The temperature can be increased by heating simultaneously as the self-pressure rises to a pressure greater than 1 bar. Its homogenous deposition, affordability, environmental friendliness, ease of expansion, excellent crystallization, pure final product, exact control over size, and ability to regulate morphology based on crystal growth conditions are what made it significant [14].

MgO has garnered significant interest in the biomedical field due to its unique properties and bioactivity towards harmful microorganisms. It has long been utilized as a dietary supplement in clinical settings to increase bone density [15]. One of the most commonly used metallic oxide nanoparticles in labs for therapeutic applications is MgO. MgO nanoparticles can be used as an additive or reinforcing material in metals, ceramics, and polymers [16]. For a coating to be more resistant to corrosion and to allow tissue to develop between the HAP coating and substrate, a magnesium oxide layer must be produced [17].

Since metal oxide nanoparticles have more stability, durability, and less toxicity than other materials, they have emerged as promising candidates for use in the production of antibacterial compounds [18].

Researchers in the field of biomaterials are inspired to discover novel materials with effective antimicrobial characteristics by the need to develop active antibacterial materials. The traditional antibacterial materials stop working and need to be replaced with new ones. The creation of novel antibacterial materials, such as inorganic compounds with special qualities including durability, physical and chemical stability, and ease of formulation and extraction from readily available and

affordable raw materials, serves as a rational replacement [19].

The aims of current contribution are: i) production of MgO nanoparticles and investigate its bioactivity using bio mimic procedure. ii) Antibacterial efficiency of the produced MgO powder.

2. Experimental part

2.1. Producing MgO nanoparticles using a hydrothermal process

To achieve full dissolve, 1.02 g of Mg (NO₃)₂·6H₂O and 0.56 g of HMT were combined with D.W. at a volume of 80 ml and swirled for 30 min. The autoclave was filled with the solution and firmly closed. It was then heated for 3 h at 100 °C. Centrifugation was used to separate the white powder that was produced after the heating procedure. After being cleaned with ethanol and distilled water, the finished powder was baked for 3 h at 400 °C. After baking it is still the while powder.

2.2. EPD of MgO on Ti

The EFD details are given elsewhere [20]. A glass container was utilized to deposit the MgO powder for utilizing EPD. After adding 0.5 g of MgO powder to 50 mL of methanol, the mixture was stirred for 10 min using a magnetic stirrer. The cathode and anode were two pieces of Ti foil. Both electrodes were linked to a 30-V power source. A 30-min deposition was held.

2.3. Bioactivity tests (BACT)

The details of BACT is reported else ware [21]. Coated plate (with produced nanoparticles by RBA process) was immersed separately in SBF; concentrated five times (SBF × 5) for one month to investigate the creation of BHAp. Table 1 shows the composition of SBF [22].

The formation of pure HAP without additional phases including calcium and phosphorus was confirmed by the XRD test.

Table 1. The composition of SBF and the concentration of (SBF × 5).

ITEM	ITEM SBF*5 (g/l)
NaCl	40.18
NaHCO ₃	1.76
KCl	1.125
K ₂ HPO ₄	1.15
MgCl ₂ ·6H ₂ O	1.555
CaCl ₂	1.465
NaSO ₄	0.36

2.4. Antibacterial test

A comprehensive description of the samples' antibacterial activity evaluation was provided by Ref. [23]. The diameter of the inhibition zones was measured in millimeters.

3. Results and discussion

XRD pattern of prepared MgO powder is illustrated in Fig. 1.

The MgO nanoparticles have a crystalline form, as seen by many peaks in the XRD data. The diffraction peaks are in close agreement with the standard cubic phase of Magnesium Oxide (JCPDS card No. 01-087-0653). At an angle of 42.7° and a relative intensity of 100 %, the (200) plane of MgO displays the greatest peak. At angles of 36.6° , 62° , 74.3° , and 78.3° , the (111), (220), (311), and (222) planes each have their own distinct peaks.

Fig. 2 illustrates SEM image of MgO nanoparticles. In general, the shape of nanoparticles is

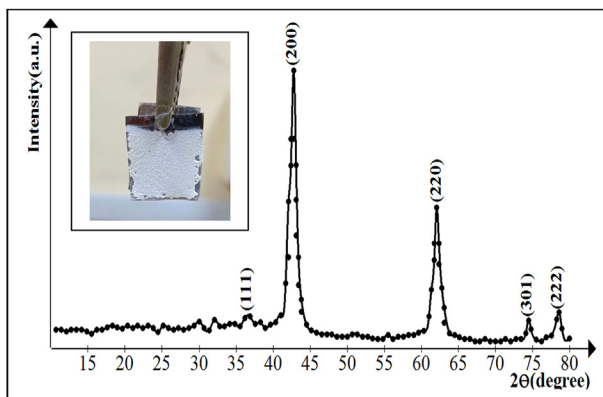


Fig. 1. The produced MgO powder's XRD pattern. Picture of MgO powder deposited on Ti substrate is shown in the inset.

semi spherical and the diameter of each particle is around 25 nm. These particles tend to form clusters due to the high energy of the surfaces of these nanoparticles. The surface energy may be defined as the as excess energy, i.e., the difference in the energy between a particle and the same number of atoms in an infinitely extended solid [24].

A white layer was formed on MgO nanoparticles upon immersion in SBF; Fig. 3 displays the XRD pattern of this layer.

The XRD consists of three phases: the first is the Ti phase (the base) with eight peaks (002), (101), 203), (102), (220), (110), (103), and (112). Only one peak (110) appeared for TiO_2 phase. The dominant peak (211) is corresponding to HAp phase with three another peaks (210), (311), and (222). What is striking about the pattern shown in Fig. 3 is the absence of peaks attributed to MgO phase. It appears that the MgO phase acted as a catalyst for the deposition of HAp on titanium.

Fig. 4 depicts the shape of the HAp particles that developed on the surface of Ti. SEM images demonstrated that HAp took the shape of interconnected nanoballs. HAp nanoparticles have nano sizes (≈ 20 nm) and have tendency to aggregate to form micro balls. Also the figure illustrates MgO particles and the deposition of HAp on these oxides particles.

The ratio of $\text{Ca/P} = 1.67$ for HAp marks it aside from the other members of the calcium phosphate family; in the current work, this value is 1.04. This decrease can result from the non-stoichiometric HAp that forms from SBF. The current results are in agreement with report else ware [25]. A change in phase composition may be the cause of the Ca/P reduction [26]. In the diagram of Fig. 5, the peaks shown are due to the elements used in the bio-mimetic process, specifically those forming the SBF.

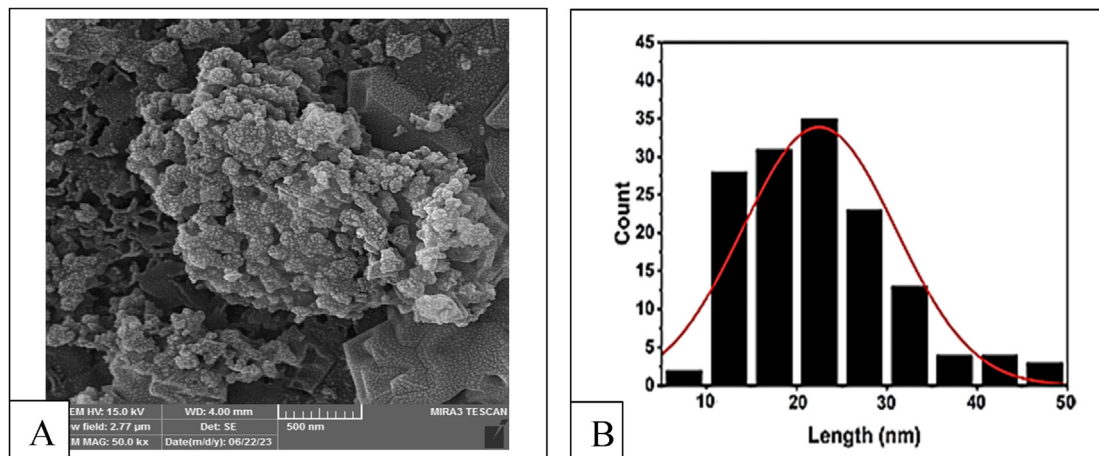


Fig. 2. A: An MgO nanoparticle SEM image, B: The distribution of MgO nanoparticle.

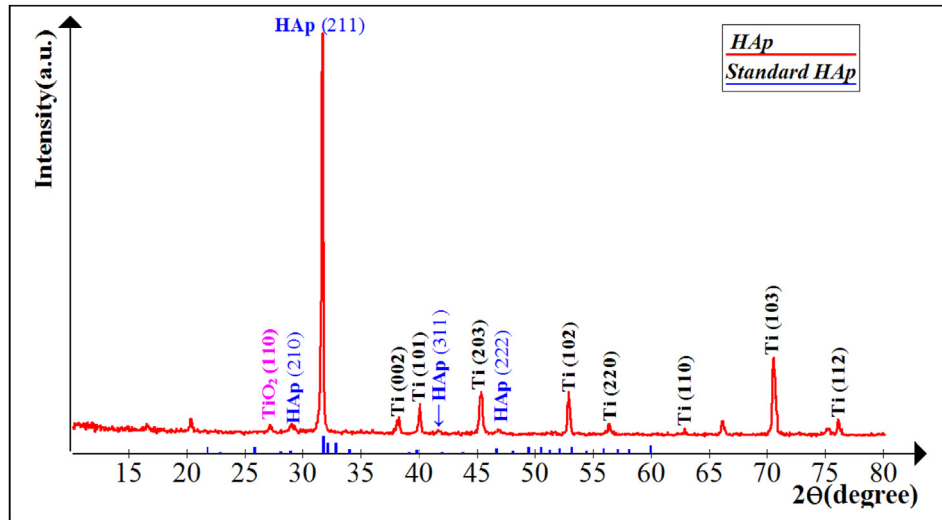


Fig. 3. After soaking in SBF, the deposited HAp layer on MgO nanoparticles XRD pattern.

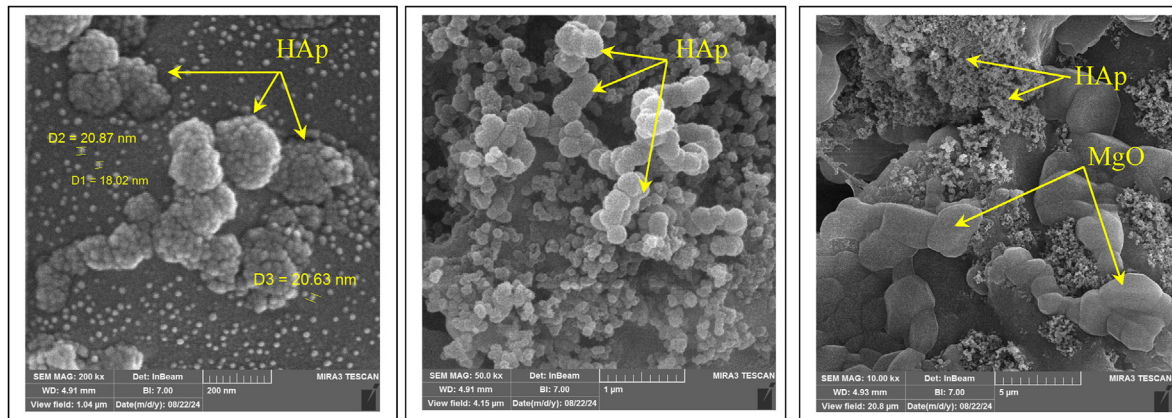


Fig. 4. SEM images of HAp that was deposited using a biomimetic method.

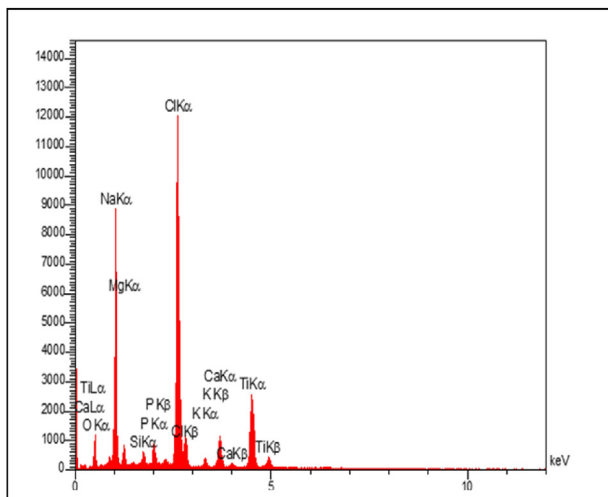


Fig. 5. The elements of the layer that was deposited using the biomimetic procedure.

MgO did not exhibit any antibacterial properties, see Fig. 6. These explanations, any or all of them, might be responsible for this:

- 1- The nano size of the materials used is one of the parameters that affect the killing of bacteria. The micro-sized aggregations of this oxide might be the cause of its antibacterial characteristics. This indicates that the examined bacteria were not killed by taking use of the benefits of nano sizes [8].
- 2- The binding with the outer shell (membrane) of microorganisms is likely the mechanism responsible for many antibacterial actions; however, this process may not function with MgO powder [27]. Current finding contradicts that of Keren et al., who verified that the antibacterial activity of Mg^{2+} ions promotes the formation of safer and healthier food.

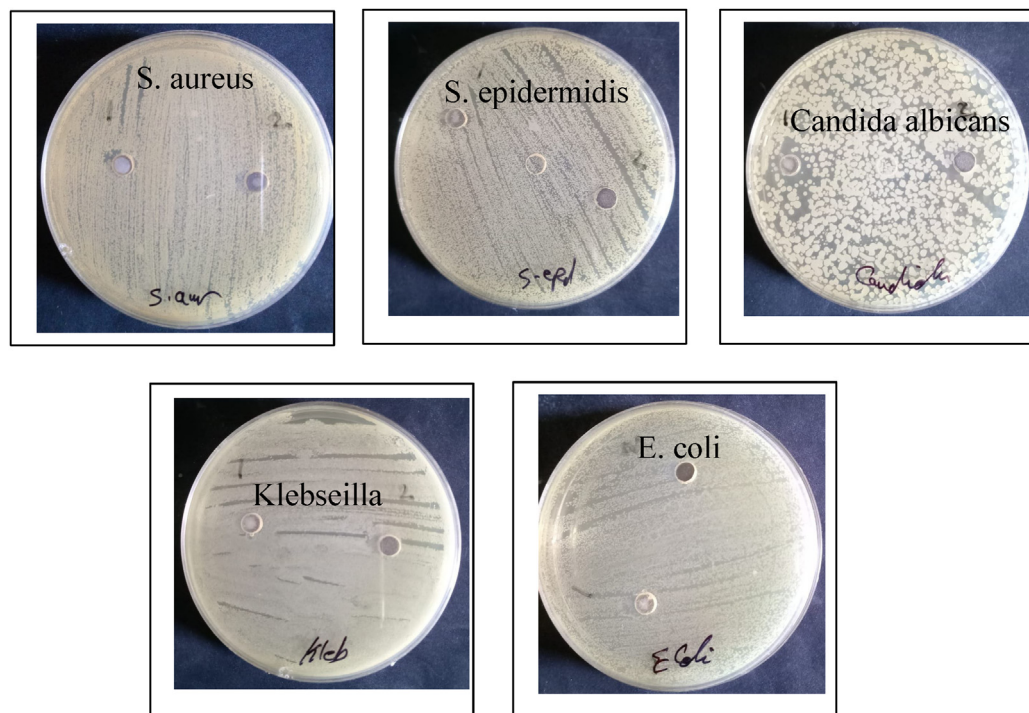


Fig. 6. Inhibition zones of MgO nanoparticles.

4. Conclusions

Hydrothermal technique is an active method to produce bioactive MgO powder but due the aggregation it has no antimicrobial resistance against five harmful bacteria.

Source of Funding

The researchers did not receive financial support from any party and relied entirely on their own personal efforts.

Conflict of Interest

All the authors declare no conflicts of interest otherwise disclose in the manuscript.

Ethical Approval

Not applicable.

Data Availability

The data required in this research can be accessed by contacting the corresponding author.

Author Contributions

Dr. Sabah H. Juma prepare the samples according to the best conditions. While Dr. Reem S. Khalil carried out the measurements on the samples and

contributed to writing the manuscript, Dr. Mustafa Shaker Hashim provided general supervision of the work, writing and correspondence.

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