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Preparation of Al_2O_3/MgO Nano-Composite Particles for Bio-Applications

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K E Y W O R D S

Nano composite particles, sol-gel method, bio Applications.

A B S T R A C T

This study describes the preparation and study of the properties of Nano composite particles prepared in a sol-gel method which consists of two materials (Al_2O_3-MgO). The powder was evaluated by x-ray diffraction analysis, scanning electron microscopy analysis (SEM), particle size analysis, and energy dispersive x-ray analysis (EDX) and antibacterial test. The evaluation results of the nanocomposite particles shows a good distribution of the chemical composition between aluminum oxide and magnesium oxide, smoothness in particles size where it reached to (54.9, 59.8) nm at calcination in (550 ^{0}C and 850 ^{0}C) respectively, formation of different shapes of nanoparticles and different phases of the Al_2O_3 particles (kappa and gamma) and nanopowder have well antibacterial action, Therefore, this reflects the efficiency of the proposed method to manufacture the nanocomposite powder and the possibility of using this powder as a strengthening material for the composite materials and using these composite materials in bio applications, especially in the fabrication of artificial limbs.

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1. Introduction

Nanoscience is a broad and multidisciplinary area of research that has increased dramatically worldwide in recent years and nanomaterials represent the backbone of Nano science and nanotechnology [1,2]. Nanomaterials are advance-ceramic materials of growing scientific interest because they have stunning properties compared with alternate sorts, of other materials [3]. Nano composite particles consist of a ccombination of two different materials into a single hybrid particle and give a multifunctional material which can be used in different fields including biomaterials, electronic and engineering applications or be used to enhance existing properties [3, 4]. So, the enthusiasm for this sort of materials has expanded and the strategies used to create it have been enhanced [5, 6]. Nano materials can be prepared from natural materials such as particles and ccellulose fibers from sugar palm fiber, and thin films from sago trees can be used in many applications like packaging and reinforcement materials. Nanomaterials can also be prepared from industrial materials and here the structure and properties of the nanoparticles can be controlled [7, 8]. There are different ways to fabricate nanoparticles including mechanical, physical and cchemical methods [9, 10]. The chemical methods are the most important amongst the techniques Used due to fast production and short time. The cchemical methods incorporates a few strategies include sedimentation, dispersions, and sol-gel [11]. The sol-gel techniques is an essential strategy compared with other technique's since it gives the powder homogeneity, high purity, and smoothness [12]. This method includes basicc steps starting with breaking, up the crude material (nitrate, alkoxides or chloride) in an appropriate dissolved, encourages particle precipitations. To form the gel and finally uses heat treatment (drying and calcination) to create the powder. Alkoxides are the most widely recognized materials used to create nanomaterials [13]. The advantages of the sol-gel method are: improving adhesions between the substrate and the top coat, materials can be melded into complex geometries, high-purity products are obtained due to the ceramic oxides precursors which are dissolved in the appropriate solvent for the sol-gel transformation, lower processing temperatures, simple, economics, no need for special or expensive equipment and an effective method to proud high-quality coatings [1,3].

MgO is a paramount inorganic material, it utilized in numerous application, for example, catalysis, impetus underpin, harmful waste remediation, obstinate materials, and adsorbents, added substance in substantial fuel oils, reflecting and hostile to reflecting coatings, superconducting and ferroelectric thin movies as the substrate, superconductor and lithium particle batteries, and so forth In medication [14,15]. MgO is utilized for the help of acid reflux, sores to Mach, and for bone recovery. As of late, MgO nanoparticles have appeared for applications in tumor treatment. MgO nanoparticles additionally have extensive potential as an antibacterial operator [15]. Alumina (Al_2O_3) is generally utilized as an impetus support and wear-safe material, because of its fascinating concoction, mechanical, and thermal properties [16,17]. Alumina is one of the idle biomaterials utilized in orthopedic [16]. It is in this way, a biodegradables material by the natural conditions [18]. Ultrafine Al_2O_3 powder has the extensive, potential for and a wide scope of uses including high-quality materials, electronics production, and medical applications. The wide uses make it a typical, material and there has been an expanding enthusiasm for the amalgamation of ultrafine Al_2O_3 [16]. Al_2O_3 is a basically unpredictable, oxide being a few distinctive metastable stages conceivable, which inevitably converts to stable Al_2O_3 [16, 18]. The reason for these examinations is to utilize nitrates for both magnesium and aluminum to produce a Nano powder of two oxides. Its physical and chemical properties were studied for use in subsequent medical applications and to demonstrate the success of this method.

2. Experimental

The following tools and equipment are utilized in this work:

- Sensitive four digits balance type (Sertorius, Germany). As shown in Table 1 Figure a.
- Magnetic stirrer with hot plate type (India). as shown in Table 1 Figure b
- Filter papers.
- PH meter indicator type (Champ, Germany).
- Drying oven. As shown in Table 1 Figure c.
- Furnace up to 1200°C for calcination type (nabertherm, Germany). As shown in Table 1 Figure d.
- X-Ray diffraction. as shown in Table 1 Figure e.
- Scanning electron microscope and EDX. as shown in Table 1 Figure f.
- Particle size distribution PSD (Brook even Nano-Brook090 Plus)

In this, work used the "aluminum" nitrate (Al $(NO_3)_3$. $9H_2O$ (purity 99% by Thomas Baker, India)) and calcium: nitrate (Mg $(NO_3)_2.6H_2O$ (purity 98% by Thomas Baker, India)), and surfactant, material (sucrose extra pure) ($C_{12}H_{22}O_{11}$) supplied by alpha chemicals India with purity 99% were used to prepare the Nano composite particles. Initially, to prepare the Nano composite particles, one molar of aluminum nitrate no hydrate was dissolved in 100 ml of deionized water and one molar of calcium nitrate was dissolved in another 100 ml of deionized water with continuous mixing on hot plate stirrer at 60 °C until the salt dissolved, then the dissolved salts for both types are placed in a flask and mixing for two hours at

60 °C. The next step is the addition of surfactant material (sucrose extra pure) (2%), then drops of ammonium hydroxide (NH_4OH) were added with/continuous mixing to increase the viscosity until the gel was formed. Then, the solution was filtered with filter papers to get out the gel and dried at 80°C for 6 hours. The next step is heat treatment at 550°C and 850°C for 2 hours. The technological path of the preparation process is shown in Figure 1. The resulted Nano composite powder was evaluated by X-ray diffraction (lap x-automated powders diffract meters and x-rays were generated using copper (Cu-Kα) radiation at 35kV 40mA, Germany), scanning electron microscope (SEM) and energy dispersive x-ray (EDX) (inspect S 50, FEI, Netherlands) and particle size analysis (PSA) (Brook, nanoBrook-90 plus). One sample for each test. Table 2: the starting materials for the preparation Nano composite powders of Al_2O_3-MgO.

Table 1: The Tools and Equipment. a) Four digits balance, b) Magnetic stirrer, c) Drying oven, d) Furnace up to 1200° C, E) X-Ray Diffraction. F) Scanning electron microscope and EDX.

no.	the tools	no.	the tools
a		b	
b		e	
c		f	

Table 2: The starting materials for the preparation nano composite powders of Al_2O_3-MgO .

Raw materials	Formulation	Purity %	Physical state
Aluminum nitrate	$Al(NO_3)_3.9H_2O$	>98	Solid
Magnesium nitrate	$Mg(NO_3)_2.6H_2O$	98%	Solid
Ammonium hydroxides	NH_4OH	28-30 %	Liquid
sucrose extra pure	$C_{12}H_{22}O_{11}$	99 %	Solid
Ethanol absolute	C_2H_5OH	≥99.8	Liquid

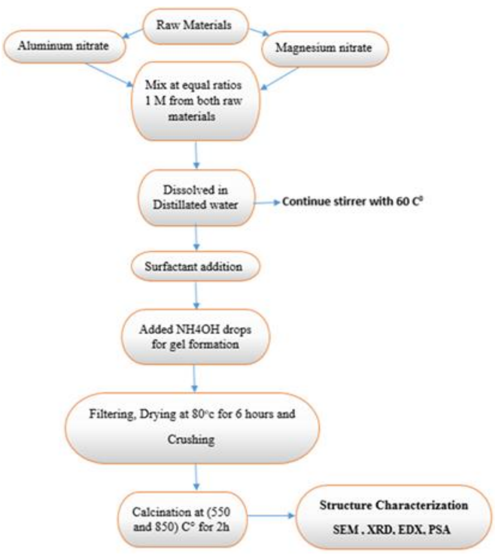


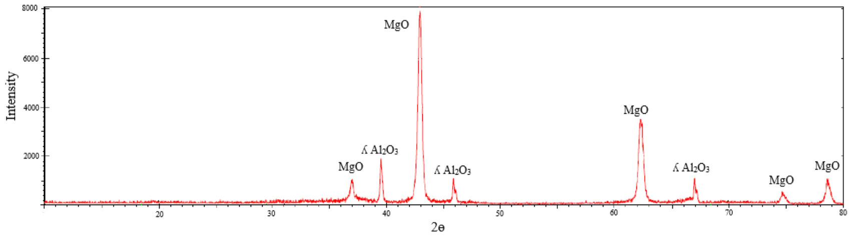
Figure 1: The technological path of the preparation process

3. Results and discussion

I. X-Ray diffraction result

XRD was used to determine the type of material or element and the special arrangement of atom's within crystalline phases and materials. Figure 2 (a) shows the results of the X-ray diffraction analysis of the composite Nano powders prepared from (aluminum and magnesium) nitrate by a sol-gel method dried at 80 °C for 6 hours and treated at 850 °C for two hours. The resulting alumina is (kappa χAl_2O_3) and magnesium oxide (MgO) according to the analysis card of this test (013-0373 and 045-0946) JCPDS respectively. Figure 2 (b) shows the results of the X-ray diffraction test of the same powders treated at 550 °C for two hours. The resulting alumina is ((ʎ - χ) Al_2O_3) and magnesium oxide (MgO) obtained by the analysis card (013-0373, 010-0425 and 045-0946) JCPDS respectively.

The results of this analysis indicate the effect of heat on the formation of the phases since two different transformer phases are produced (kappa and gamma). In addition, the heat affects the “particle size” produced for both phases as will be demonstrated in the particles size analysis results. The samples resulted from the treatment ccontain a small number of phases because ccrystallization heats to form phases of Al_2O_3 and MgO need higher temperatures. Table 3 shows the components of the ccomposite Nano powder from the phases and the existed ccrystalline systems as well as cell parameters for each phase.



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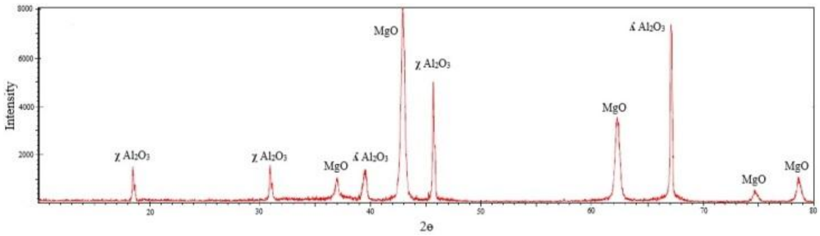


b

Figure 2: X-ray diffraction patterns for composite nanoparticles (a) The composite nanoparticles (Al_2O_3-MgO) with heat treatment at 850 °C. (b) The composite nanoparticles (Al_2O_3- MgO) with heat treatment at 550 °C.

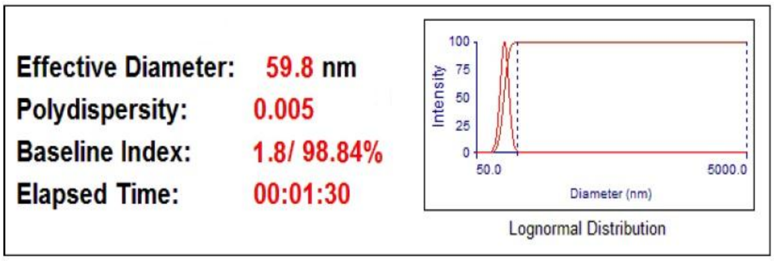
Table 3: Shows the phase components of the composite nanopowder of Al_2O_3-MgO.

Nano particle	Phase content	Crystal system	Cell parameters
(Al2O3-MgO) at 850 °C	Gamma(ʎAl2O3) MgO (Periclase, syn)	Cubic Cubic	a=3.9500 Å a=4.7990 Å
(Al2O3-MgO) at 550 °C	Kappa (χAl2O3) Gamma(ʎAl2O3) MgO (Pericles, syn)	Orthorhombic Cubic Cubic	χ=(a=4.8437 , b=8.3300, c=8.9547) Å ʎ=(a=3.9500 Å) lime=(a=4.7990 Å)

II. Particle size distribution (PSD) result

PSD uses a single-lens system that receives all the scattered signals emitted from particles in the nanometer to the millimeter size. The use of high-quality lenses results in high-resolution images; of low and diffuse light, ensuring that the device will receive all signals - even weak signals at high angles resulting from the dispersion of smaller particles.

Figure 3 (a and b) explains the particle size of the composite nan powders prepared from nitrate (aluminum and magnesium) nitrate and treated at 550 °C and 850 °C for 2 hours. As noticed, the low temperature (550 °C) gives more smoothness, about 54.9 nm, with a multiplicity of phases where two phases of alumina appeared compared with the high temperature (850 °C), which gives less smoothness, about 59.8 nm, and fewer phases.



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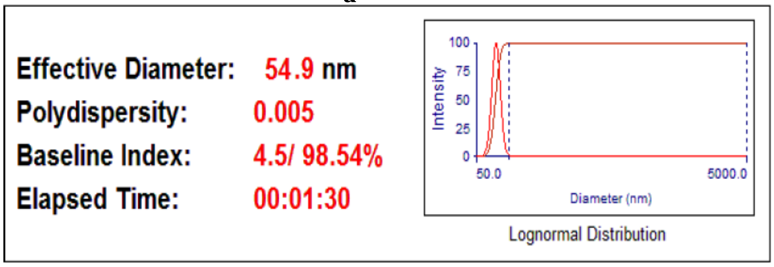

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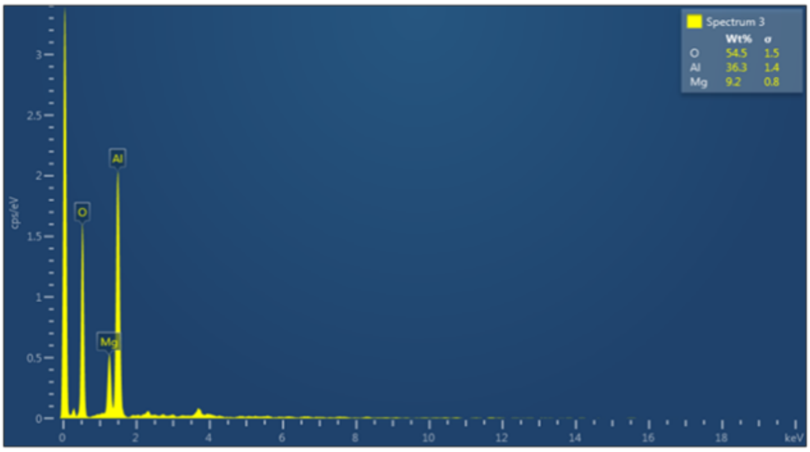
Figure 3: The particle size analysis of the composite nanoparticles. (a) The composite nanoparticles (Al_2O_3- MgO) with heat treatment at 850 °C. (b) The composite nanoparticles (Al_2O_3- MgO) with heat treatment at 550 °C.

III. Energy dispersive x-ray analysis (EDX) result

EDX analysis was used to identify the chemical elements and their concentration within the material. The EDX results in Figure 4 (a and b) shows the homogenous distribution of elements in the structure of Nano composite powder prepared from aluminum nitrate and magnesium nitrate and treated at 550 °C and 850 °C for two hours.

The initial composition of the powder was assessed by the presented elements in the analysis which match with the resulted Nano composite powder. Also, the results showed the absence of impurities and this indicates the purity of the resulted material. The chemical analysis of the powder, after calcination at (550 and 850) °C for two hours, confirmed the possibility of Nano composite powder synthesis by the sol-gel method.



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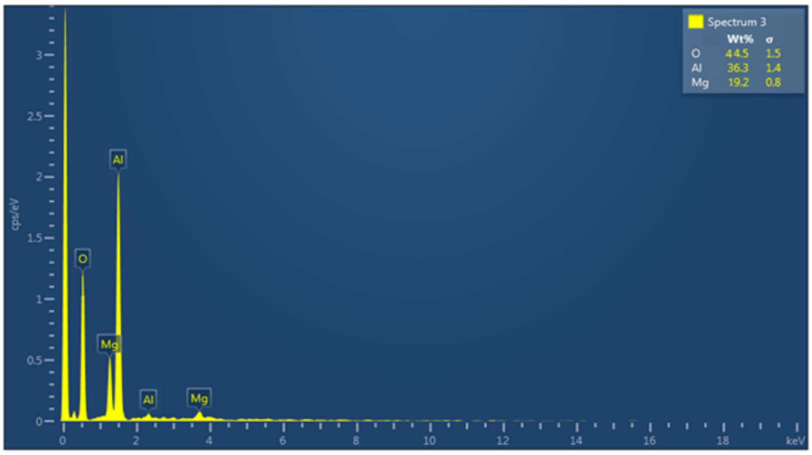

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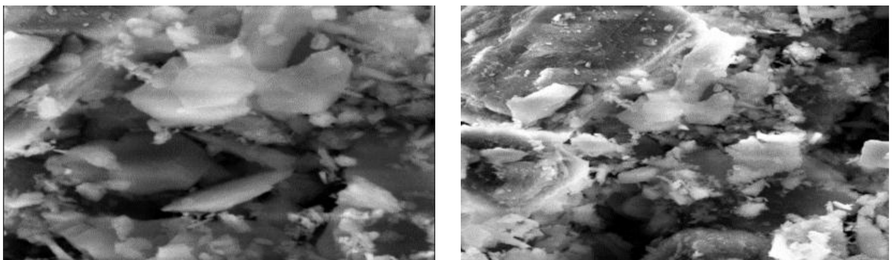
Figure 4: Energy dispersive x-ray analysis (EDX) of the composite nanoparticles. (a) The composite nanoparticles (Al_2O_3- MgO) with heat treatment at 850 °C. (b) The composite nanoparticles (Al_2O_3-MgO) with heat treatment at 550 °C.

IV. Scanning electron microscope (SEM) result

Scanning electron microcopy SEM was used to examine sample topographies at very high magnification using a piece of equipment called electronic microscopy. During the SEM test, the electron beam. Concentrated on the sample point, leading to the transfer of energy to the spot and then translated into the signal. In this analysis, a scanning electron microscopy was used to study the morphological and histological characteristics of the composite Nano powder produced by the sol-gel method. Figure 5 (a) shows an image with high enlargement intensity for the ccomposite Nano powder (aluminum and magnesium) oxide dried at 80 °C for 6 hours and calcination at 850 °C for 2 hours. The random shape of the particles helped in the emergence of some clusters between particles. Also, it was noticed that the shape of the resulted particles is irregular due to the different geometricc formation of the particles. While Figure 5 (b) shows an image with high magnification intensity for the ccomposite nanoparticles of the oxides (aluminum and magnesium) prepared under the same cconditions and ccalcination at 550 °C. As shown, the distribution, of particles and concentration is in some areas of particles and this helps in the emergence of some clusters between the large particles. Also noticed that the resulted particles were irregulars in shape because of the difference in geometric shapes, of the particles and it showed more smoothness due to the low heat of the treatment compared to the previous heat.

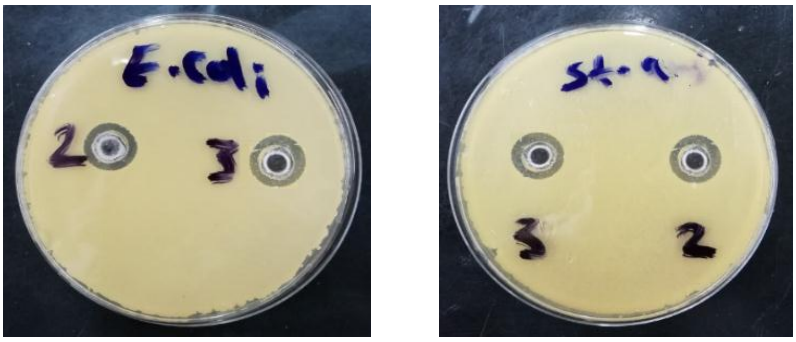


a **b**

Figure 5: SEM images for the composite nanoparticles. (a) The composite nanoparticles (Al_2O_3- MgO) with heat treatment at 850 °C. (b) The composite nanoparticles (Al_2O_3- MgO) with heat treatment at 550 °C.

V. Antibacterial activity of composite nanoparticles.

Observe from the examination pictures Figure 6 that there is a clear effect of the powder on (E. coli and st.aur) bacteria type. This indicates the effectiveness of the powder as an antimicrobial agent. This is in line with what the researchers presented (Elans et .al in 2019) where they found that the alkaline effect of (MgO) Nano powder on bacteria as the antibacterial mechanism, ions in the surface of nan powder cause bacterial death due to interconnection with the cell membrane [19]. And the researchers (Hewit et al. in 2001) they found that the increase of the surface area of (magnesium Nano powder) will cause an increase of the O_2 concentration in the environment and cause the death of the bacteria [20].



a **b**

Figure 6: Shows antibacterial activity for Nano composite powders. a) E. coli bacteria activity for powders at (850-550) °C. b) st.aur bacteria activity for powders at (850-550) °C.

4. Conclusions

The evaluation of the ccomposite nanoparticles using XRD, particle size analysis, SEM and EDX tests showed that “nanoparticles" have an irregular shape and a homogeneous chemical structure composed of (aluminum and magnesium) oxides and free of impurities. In addition, the results showed that the temperature effects on the smoothness and the formation of phases of the Nano composite particles. The heat treatment at 550 °C gave more smoothness and phases than heat treatment at 850 °C. Antibacterial activity test of ccomposite nano powder from the examination pictures that there is a clear effect of the powder on (E. coli and st.aur) bacteria types. This indicates the effectiveness of the powder as an antimicrobial agent.

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