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Effect of CoO Nanoparticles Percentage on Dielectric Properties and Antibacterial Activity of CoO/Chitosan Nanocomposite films

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ABSTRACT

Cobalt Oxide (NP) were synthesized by two different methods, simple chemical routine and sol-gel. Energy Dispersive X-ray Analysis (EDAX) result showed that the weight percentage of Coblet in smple C1 larger than sample C2. Transmission electron microscopy (TEM) images indicated that the particles shape of CoO (NP) are Cubic, and the partials size of sample C1 smaller than sample C2. The partials size of sample C1 was between 20.79 to 14.5 nm and for sample C2 was between 62.37 to 16.5 nm.

Frequency dependence dielectric was observed in nanocomposite films of CoO/Chiosan were prepared using Chitosan with two types of CoO (NP). Dielectric constant ε' and dielectric loss ε" for all samples were found to be decrease with increasing frequency and semiconducting behavior. Also, antibacterial activity of the nanocomposite films showed increase ln the inhibition area towards the bacteria with increasing of CoO nanoparticles weight percentage in the C11 sample.

Keywords: Cobalt Oxide, Chitosan, nanocomposites, Dielectric properties, Antimicrobial activity.

1. INTRODUCTION

Metals, can be toxic to microorganisms at very low concentrations, metals have been widely used as antimicrobial agents in a many applications in the industry. Cobalt Oxide (CoO) nanoparticles is interesting because of its multiple oxidation states, e.g. CoO and Co_3O_4. Nanocomposite have received increasing attention in recent years. These materials have the capability to combine a many properties in a one compound as mechanical, electric and antibacterial properties[1].Nanoparticles show considerable changes in optical, electrical and magnetic behavior due to the change in shape and size[2]. Chitosan is usually used for the synthesis of biopolymer composites,

which are active carriers of antimicrobial[3].The poly cationic nature of Chitosan intervene with the negatively charged cell membrane of microbe causes membrane seepage, which stop the microbial growth and metabolic activity[4].

In this study we have studied the effect of synthesis method on weight percentage of Co in CoO nanoparticles and there effect on dielectric and antibacterial behavior of CoO/Chitosan nanocomposite films, and we used simple chemical routine and sol-gel method because they are simple methods with less energy consumption and no need for special or expensive equipment.

2. MATERIALS AND METHODS

Coblt(II) Chloride purum p.a., anhydrous, ≥98.%, Ammonium hydroxide Solution, Chitosan with 85 percent degree of deacetylation, and Ethanol absolute were purchased from Sigma Aldrich Co. Ltd (USA). Acetic acid, Glacial biochemical grade 99.86% was purchased from ACROS. Two bacterial strains Pure cultures of Gram negative human bacteria viz., *Pseudomonas aeruginosa (PA)* and gram positive bacteria viz., *Staphylococcus aureus (SA)* were obtained from "Culture Collection of Antibiotic Resistant Microbes (CCARM)" Military Hospital Tabuk. All the chemicals were analytical grade and used without further purification.

i. Synthesis of Chitosan

We were dissolving 1 g of Chitosan in 100 ml of 2% (v/v) aqueous glacial acetic acid with stirring at 100ºC for 4 hours for prepare Chitosan solution.

ii. Synthesis of CoO (NP) powder

For prepared CoO (NP) sample C1 (simple chemical routine), we were dissolving 1.29 g of $CoCl_2$ in (80 mL) of water and (20mL) Ethanol then added 5mL of NH_4OH drop by drop for 60 mint at 60° C with stirring for three hours. The powder was dried in the oven at 200 °C for 24 hours to obtain the nanoparticle powder of Cobalt oxide(C1).

But for prepared CoO (NP) sample C2 (sol-gel)**,** we were dissolving 1.29 g of ($CoCl_2$) in (20 mL) of water and (5mL) NH_4OH then added 80mL of Ethanol drop by drop for 60 mint at 60° C with stirring for three hours. The powder was dried in the oven at 200 °C for 24 hours to obtain the nanoparticle powder of Cobalt Oxide(C2).

iii. Synthesis of CoO/Chitosan nanocomposite film

For preparing CoO/Chitosan nanocomposite films we mixed Chitosan solution (10 mL) with 0.05 gm of CoO nanoparticles (C1 and C2) and stirring for 3 hours a room temperature. Then it was casted in petri dish until it dried and formed film (C11 and C21) at room temperature.

iv. Characterization and measurement

From EDAX the samples were prepared by attaching the particles to 12.5 mm diameter Al. Accelerating Voltage=20.0 kV, Working Distance=10 mm, Spot Size=4.5 (EDX). The images were digitally recorded in secondary electron imaging mode at different magnifications at resolution setting of 1024x884 pixels. EDX analysis was performed on multiple particles in area mode (~25-50 m^2) using EDAX Genesis XM4 system. The elemental contents were determined using standardless ZAF option.

The Transmission Electron Microscopy (TEM) studies were performed (TEM, JEOL JEM-3010, 300 kv). The samples for TEM were prepared by making suspension from the powder in deionized water. A drop of the suspension was put into the carbon gride and left to dry.

The electrical properties was measured at room temperature by (Hioki,LCR Hitester 3532-50).The frequency dependence of electrical properties for prepared samples were measured from 50Hz to 5Mhz.

In vitro antibacterial activity was examined for nanocomposite films of CoO/Chiosan (C11, C21). Pure cultures of Gram negative human bacteria viz., *Pseudomonas aeruginosa (PA)* and gram positive bacteria viz., *Staphylococcus aureus(SA)* were obtained from "Culture Collection of Antibiotic Resistant Microbes (CCARM)" Military Hospital Tabuk. The bacterial strains were maintained and grown in a nutrient agar medium.

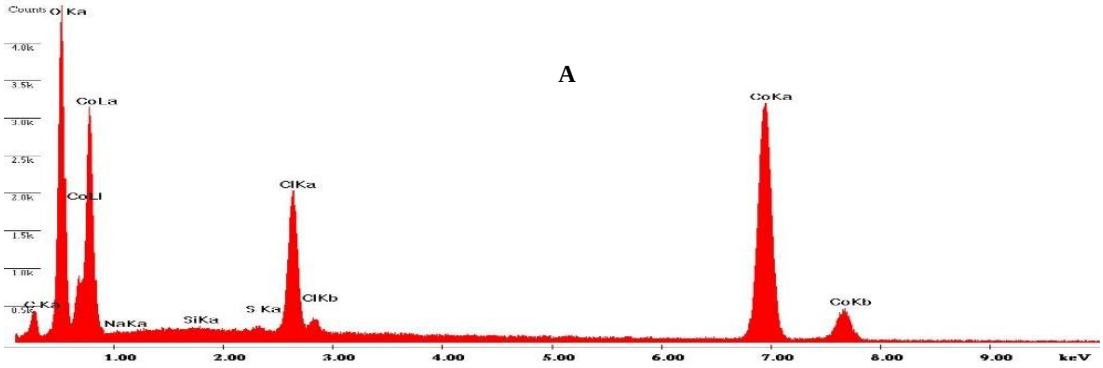
3. RESULTS AND DISCUSSION

3.1 EDAX and TEM study

EDAX analysis was performed on multiple particles in area mode (~25-50 m^2) using EDAX Genesis XM4 system. The elemental contents for nanoparticles (C1,C2) were determined using standardless ZAF option shows in Fig.1(a, b). Also, EDAX result showed that the weight percentage of Cobalt in sample C1 higher than sample C2.

TEM images are used to study the microstructures of the prepared samples. Fig (2 a, b, c and d) shows the representative TEM images of the prepared samples. The morphology of the all samples were found to be nearly Cubic. TEM results indicated dark sites may be due to the accumulation of nanoparticles[5-8].

The result of TEM shows that, the partials size of sample C1 smaller than sample C2. The partials size of C1 was between 20.79 to 14.5 nm and for sample C2 was between 62.37 to 16.5 nm.



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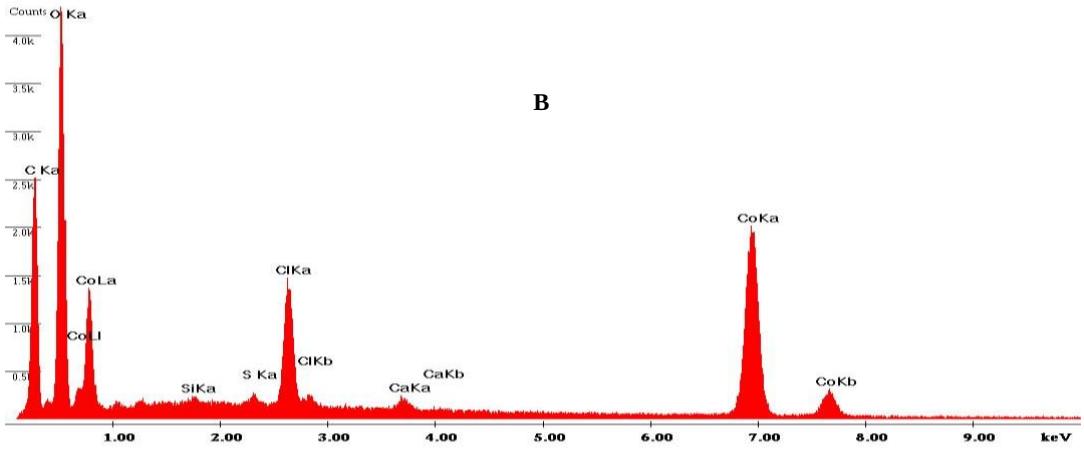

Fig.1: The EDAX images for (A) C1 and (B) for C2.

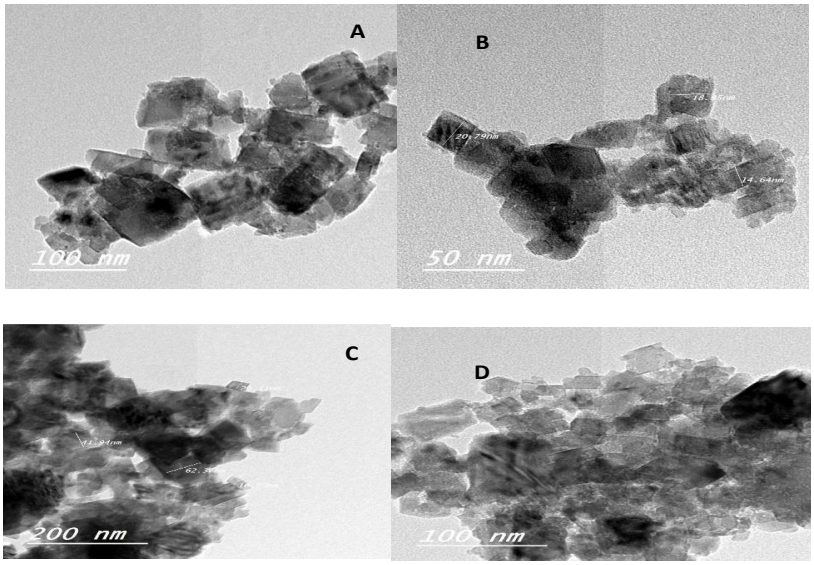

Fig.2: (2a,b) The TEM images for sample(C1) and .(2c,d) The TEM images for sample(C2)

3.2 Dielectric Properties

Dielectric constant (ε') and dielectric loss (ε") frequency of pure Chitosan and nanocomposite films of Chitosan/CoO with different weight percentage of Co (C11, C21), all samples collected in the frequency range (50 Hz to 5 MHz) at room temperature. Fig.3a Show the relation between the frequency and Dielectric constant (ε') for all samples (pure Chitosan, C11 and C21). The spectrum for all samples show that Dielectric constant (ε') increased with frequency

decreased. Also, the Dielectric constant (ε') proximate a limiting constant value at high frequencies, The analysis results of Dielectric constant mentions that the behavior accountable in this case are due to the dipolar orientation and space due to molecular movements linked with the side chains and charge effects resulting from trapping of filler charge carrier in dole out traps. Charge carriers trapped at the polymer– nanoparticles interfaces produce a large assistance to the dielectric constant at lower frequencies[7]. In Fig.3a it obviously seen that, Dielectric constant of pure chitosan film is higher than that of all nanocomposite films of Chitosan/CoO samples (C11,C21), This is a result to nano effect, the nanoparticles loading in polymer led to higher free charge carriers from the nanoparticles to accumulate in the polymer. In This case may reduce distance between NP and decrease the charge trapping sites. Hence, lower (ε') value and these situation effect in the Dielectric loss tangent (*D*) value to be smaller than that of pure polymer (fig3.C)[8]. So, if nanoparticles were added to pure polymer, the Dielectric constant of a polymer nanocomposite films would decrease. Also, the value of the Dielectric constant for sample C11 was less than the value of C21, it is may be due to difference in weight percentage of Co between sample C11 and C21 nanocomposite films (Co weight percentage in sample C11 larger than sample C21). Fig.3b showing Dielectric loss (ε") at room temperatures as a function of frequency for pure chitosan, C11 and C21 nanocomposite films. In Fig.3b Relaxation peaks appeared for (C11, C21) due to the efforts done by ionic charge transport in the CoO nanoparticles in C11 and C21 to follow the change in the Direction of applied field[9].

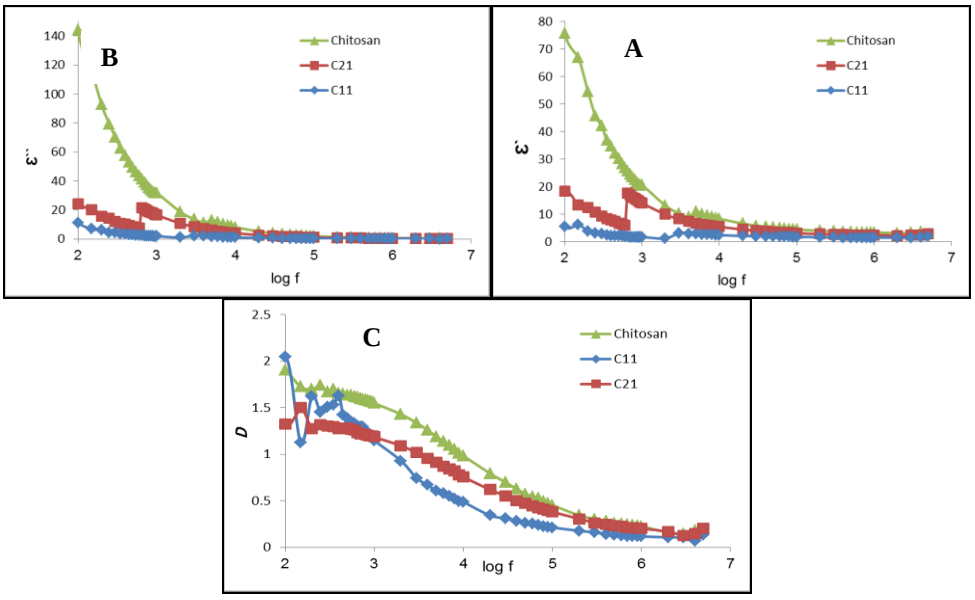

Fig.3: (3a) Dielectric constant as a function of frequency and (3b) Dielectric loss ε'' (3C) Dielectric loss tangent as a function of frequency for pure chitosan, C11 and C21 films at room temperatures

3.3 Antibacterial behavior

The antibacterial activity of nanocomposite films C11 and C21 have tested against two different types of microorganisms by using disc diffusion method. For the determination of antibacterial activity of nanocomposite films C11 and C21, the Gram-negative *Pseudomonas aeruginosa(PA)*, and Gram-positive *Staphylococcus aureus (SA)* were used. Mueller Hinton Agar (MHA) media was prepared and poured into sterilized Petri plates and then plates were spreader with of the bacteria separately. The loaded disks were applied carefully to the surface of the agar plates using sterile forceps. The experiment was carried out and the diameters of the zones of inhibition were measured after 24 hours of incubation at 37°C. Standard antimicrobial agents including Gentamicin (100 mg/mL) was used as control[10]. Fig. 4 show antibacterial test results of the nanocomposite films C11 and C2. The inhibitory activities were evaluated from the area measurement of clear inhibition zones a rounding the film discs. It can be seen from the Fig. 4 that there was a negligibly small inhibition zones surrounding all samples against Gram - negative Pseudomonasa (PA) but in the state of Gram - positive Staphylococcus aureus (SA) all nanocomposite films obvious large inhibitory zones a rounding the films. Also, the behavior of antibacterial nanocomposite films C11 larger than C21. Antibacterial behavior can be explained in all films due to two reasons: the first reason is the effect of amino groups NH^{3+} of chitosan on antibacterial activity. These positive ions can interact with the negatively charged cell walls of the bacteria and stop there growth[11]. The second reason is the release of the metal ions that result to the weakening of the bacteria wall and then its death. Therefore, the reason for increasing the inhibition area towards the bacteria as a result to the increase of weight percentage of Co in the C11 sample, resulting in an increase in the number of released cobalt ions[12].

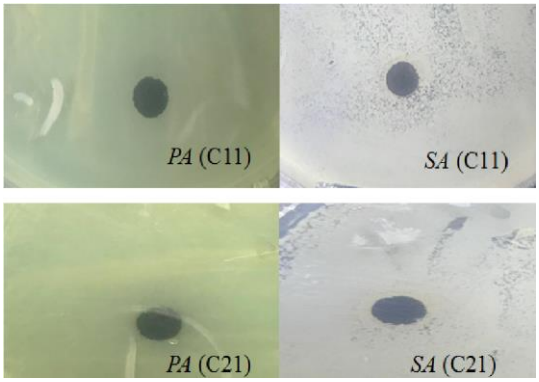

Fig.4: The antibacterial activity of nanocomposite film of (C11, C21) against Gram negative *Pseudomonas aeruginosa (PA)*and Gram-positive *Staphylococcus aureus (SA)*

CONCLUSION

- CoO nanoparticles with cubic structure have been synthesized successfully by two different methods.

- The partials size of sample C2 larger than sample C1. C1 was between 20.79 to 14.5 nm and for sample C2 was between 62.37 to 16.5 nm.
- The dielectric measurements indicate that the dielectric constant of pure Chitosan film and nanocomposite films (C1,C2) decrease, in the high-frequency range.
- The value of the Dielectric constant for sample C11 was less than the value of C21, it is may be due to difference in weight percentage of Co between sample C11 and C21 nanocomposite films (Co weight percentage in sample C11 larger than sample C21).
- The reason for increasing the inhibition area towards the bacteria was increasing of weight percentage of Co in the C11 sample, resulting in an increase in the number of released Cobalt ions.

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