

# **Preparation and Study Some Physical Properties of (CMC/PAA: MgO) Nano Composites**

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Pure polymeric films (CMC/PAA) prepared by solution casting method (solution casting) and reinforced with magnesium oxide particles ( $MgO_{NPs}$ ) by Precipitation method with different weight ratios (0, 3, 5, and 7) wt%, Calcined at different temperatures (800°C). The results of Electron microscopy (SEM) reveal that the polymeric film (CMC/PAA) appears to be homogeneous and interconnected and, when applied (CMC/PAA:MgO 7%), forms well-dispersed aggregates in the nanocomposites films. (FTIR) the spectrum revealed that  $(MgO_{NPs})$  does not have a destructive effect on the polymer structure as there are no covalent bonds between (CMC, PAA, and  $MgO_{NPs}$ ). The optical properties of the nanocomposites  $(CMC/PAA:MgO)$  were measured in the wavelength range  $(200-$ 1000) nm. The optical results showed that by increasing the nanomaterial concentration, the (reflectance, absorption coefficient, reflection index, extinction coefficient, and the real and imaginary dielectric constant) increased while the transmittance and energy gap decreased. The results show that the thermal conductivity coefficient (K) is significantly increased when magnesium oxide nanoparticles  $(MgO_{NPs})$  are added to the polymeric mixture, and this increase in the thermal conductivity coefficient is a result of filling the polymer with nanoparticles.

Keywords:  $CMC$ , PAA,  $MgO_{NPs}$ , Structural properties, Optical properties, Thermal properties.

## **1. Introduction**

**ABSTRACT**

Polymeric materials have piqued the interest of scientists and technologists due to their wide range of applications. This is owing to its high mechanical strength, light weight, and optical qualities, which enable it to be used in novel industrial, electronic, and medicinal applications [1-2]. Nanoparticles are the most essential factor in the nano structure business. specific magnetization, Mechanical strength, specific optical characteristics, larger core surface area, and lower melting temperatures are all physical and chemical qualities of metal nanoparticles that can be beneficial in a variety of industrial applications [3]. In the areas of microelectronic processing, drug distribution, vehicles, optical integrated circuits, injection molded components ,membranes, sensors, packaging materials, coatings, fire retardants, autos, adhesives, medical equipment, consumer goods, and other industries [4]. Because of its importance in producing and changing the physical properties of polymeric materials, polymer blending is attracting a lot of scientific attention [5-7].

Nano composites made from mixes are a relatively new and promising direction in nano composites research. Developing novel polymeric materials with superior qualities by combining two or more polymers is frequently less expensive and time demanding than developing new polymer chemistry. The inherent qualities of basic polymers, on the other hand, may not be sufficient to suit certain of the specific requirements of emerging sectors. Because of the tiny size, large specific area, quantum confinement effects, and strong interfacial contact of nano materials, a modest amount of nano additions can improve the overall performance of polymeric materials in the nano composites system [8]. Metal nanoparticles have recently gotten a lot of attention because of their unique features compared to bulk metals [9]. Metal oxides, such as (MgO), are of particular importance since they are not only stable under extreme process conditions, but they are also widely acknowledged as human and animal-safe materials. In the human body a specific metal oxide, such as (MgO) is a necessary mineral [10]. Due to its ability to form a continuous matrix, excellent viscosity, biocompatibility, and availability, carboxymethyl cellulose (CMC)  $((CH<sub>2</sub>COOH)<sub>n</sub>)$  is a chemically adapted water soluble derivative of cellulose, an alternative polymer used in blending with proteins to generate flexible and strong films [11-12]. It has been widely employed in the pharmaceutical, food, and packaging industries due to its safety and non-toxicity. CMC has also improved the mechanical, thermal, and barrier properties of the films [13-15]. Poly acrylic acid  $(PAA)$   $((C_3H_4O_2)_n)$  is a water soluble thermoplastic polymer that forms a stable combination with metals and has been safely utilized in a number of therapeutic applications such as medications, body lotions, and pharmaceuticals [16-18].

The aim of the research is to use a simple, non-toxic and low-cost preparation method for the preparation of high-purity and crystalline magnesium oxide nanoparticles (MgO<sub>NPs</sub>) by precipitation method. These semiconducting particles were used to prepare polymeric nanocomposite films .The effect of weight addition ratios  $(3, 5, 7)$  wt% of nanoparticles on the final performance of the overlapping secondary films was studied, in order to obtain flexible polymeric nano films with the desired specifications and the

possibility of using them in electrical applications.

# **2. . Experimental Part**

# **2.1. Preparation Of (MgONPs)**

The  $(MgO_{NPs})$  were prepared by precipitation method [19] by dissolving a weight of aqueous magnesium chloride  $(MgCl<sub>2</sub>.6H<sub>2</sub>O)$  amounting to  $(0.2M)$  in  $(100ml)$ of distilled water and the dissolution was done by using a magnetic mixer at a temperature of (50℃) and leaving the solution for half on the other hand, diluted ammonium hydroxide (NH4OH) solution was used. The distillation process was carried out using a burette with a capacity of (100ml), as the diluted ammonium hydroxide was placed in the burette and the distillation was at a rate of (3ml) per minute with the continuation of the mixing process and after the completion of the process Distillation, the resulting solution has a pH (12), This solution is filtered with filter paper and then washed several times with distilled water. Then the resulting material is taken and dried in an electric dryer at a temperature of (150℃) for two hours. After the drying process is completed, it is crushed until we get magnesium hydroxide powder. And we conduct a calcination process in a furnace at different temperatures (400, 600, and 800) ℃ to form magnesium oxide powder ( $MgO_{NPs}$ ).

#### **2.2. Preparation Of (CMC/PAA:MgO) Nano composites**

Pure (CMC/PAA) and its nanocomposite samples were prepared through the simple method "solution casting". First, the two polymers (CMC) and (PAA) were dissolved at (50°C) in the distilled water for (1h), (0.5g) of (CMC) in (10ml) of the distilled water and (0.5g) of (PAA) in (10ml). Second, the two polymers were added together for (3h) at the same previous temperature to obtain  $(CMC/PAA)$  blend. Third,  $(MgO_{NPs})$  were added to the solution of polymer with different weight percentages  $(0, 3, 5, 5, 7)$  wt%.

# **3. Results and discussion**

## **3.1. TGA Analysis**

Thermo gravimetric analysis (TGA) of magnesium oxide nano powder (MgO<sub>NPs</sub>)

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prepared within the thermal range (0-1100) °C was studied to characterize the physical and chemical properties as a function of temperature in the presence of oxygen. Figure (1) shows the TGA curve of magnesium oxide nanoparticles (MgO<sub>NPs</sub>) before heat treatment. The weight loss curve showed that the material lost about (2%) of its initial mass at a temperature of (44-323) °C due to the loss of water molecules (moisture) absorbed by the sample from the external environment [20], on the other hand, another weight loss was observed of About (29%) of its initial mass at a temperature ranging from (430-323) °C is due to the decomposition of magnesium hydroxide [21], in addition to that, another weight loss of about (32%) of its initial mass was observed at Temperature ranges between (430-686) °C. This is due to the possibility of its association with the continuous removal of condensed types of hydroxyl group on the surface of nano powdered magnesium oxide (MgO<sub>NPs</sub>) [22], from the curve we notice the loss of a small part of the weight at temperatures higher than (686°C) This indicates the end of the reaction which indicates weight loss, pure nano crystalline magnesium oxide (MgO<sub>NPs</sub>) powder was obtained. The results show that after a temperature (686°C), most of the reactants involved in the preparation of nano magnesium oxide  $(MgO_{NPs})$  will volatilize upon heat treatment [23].



*Fig. 1. TGA curve of (MgO).*

# **3.2. SEM Analysis**

Micrographs of magnesium oxide Nano powder (MgONPs) show before and after

(Calcination Process) at (800 °C) with granular size distribution chart. Magnesium oxide nanoparticles (MgONPs) were represented before the calcination process and as shown in Figure (2) as dense aggregates of nanoparticles. in the form of scales, the Nano powder is in a state of increase in Crystallinity and granular size and the change of nanostructure, and this is due to the high temperature of calcination leading to the continuation of (Crystalline growth) of the granules and their merging with each other and then increasing the granular size [25,24].



Figure (2): (a, b) FE-SEM images of the prepared (MgONPs) powder prior to the (Calcination process) with the granular volume chart of the prepared powder, (d, e) FE-SEM images of (MgONPs) powder prepared after (Calcination Process) at (800 °C).

## **3.3. FTIR Spectra**

Infrared spectroscopy (FTIR) of the prepared films was carried out in order to know the effective aggregates by measuring the transmittance spectrometry as a function of wave number. According to Figure (3), we note that there is no interaction between the nanoparticles and the polymer mixture due to the fact that the density of the nano composites increases with the increase in the nanoparticles content, and the permeability decreases slightly with the increase in the nanoparticles content [26]. Broad bands are observed for all samples at about  $(3286.59 \text{ cm}^{-1})$  due to  $(0-H)$  groups in the polymer matrix chain [28], The peaks at (2938.67 cm-1) are related to the stretching of the existing (C-H) bond [29], At (2388.85, 2162.22, and 1639.53) cm-1 the stretching mode occurs in the bonds (C=O=C, C≡C, C=C) respectively .This is caused by the absorption of FTIR rays by the bond [27], At (1412.1 cm-1) the asymmetric bending vibrations of the (-CH2) group occur [30, 31, 32], while at  $(1317.14 \text{ cm}^{-1})$  it refers to the bending vibrations of the (-OH) group [32], At  $(1063.38, and 889.42)$  cm<sup>-1</sup> as a result of the absorption of the (FTIR) rays, the stretching mode occurs in the (C-O, C=C) bonds respectively [27].



*Fig. 3. FTIR spectra for (CMC/PAA*:*MgO).*

# **3.4. Optical properties**

The study of the transmittance and reflectivity spectrum is considered an effective way to understand and develop the energy gap and synthesis of polymers. Figure (4) shows the transmittance and reflectivity spectrum of pure (CMC/PAA) films supported by different weight ratios of calcined magnesium oxide nanoparticles  $(MgO_{NPs})$  at a temperature of (800°C). The obtained results showed that the addition of magnesium oxide nanoparticles  $(MgO_{NPs})$  led to a decrease in the transmittance ratio of the prepared film (CMC/PAA), and this decrease increases with the increase in the content of nanoparticles (MgO<sub>NPs</sub>), also The increase in the weight of nanoparticles increases the density of nanoparticles, and this increases the occurrence of the scattering process, which is the main process that affects the transmittance of light at the boundaries of defects and grains, and this leads to a decrease in transmittance [33]. While the reflectivity of the prepared film (CMC/PAA) increases when adding magnesium oxide nanoparticles (MgONPs), and this is due to the fact that the magnesium oxide nanoparticles (MgO<sub>NPs</sub>) are within the composition of the film (CMC/PAA) The prepared absorbs part of the light falling on the membrane and scatter the other part [34].



*Fig. 4. Spectrum of transmittance and reflectivity of (CMC/PAA) films.*

The absorption coefficient  $(\alpha)$  and energy gap (Eg) were calculated for pure (CMC/PAA) films supported by different weight ratios of calcined magnesium oxide nanoparticles (MgO<sub>NPs</sub>) at a temperature of  $(800^{\circ}C)$ , as shown in Figure  $(5)$ . We can see that with the increase in the concentration of nanoparticles  $(MgO_{NPs})$  the absorption coefficient increases, the explanation of this is that with the increase in the number of charge carriers the absorbance and the absorption coefficient of nano membranes increase [35,36]. On the other hand, the absorption coefficient is at its lowest at high wavelengths (low energy), this indicates that the electron transfer potential is low because the energy of the incident photon is not enough to pass the electron from the valence beam to the conduction beam, but at higher energies it is possible, On the other hand, we note that the energy gap value of the pure (CMC/PAA) film recorded (4.724 eV), while the increase in the concentration of nanoparticles  $(MgO_{NPs})$  leads to a decrease in the energy gap values of the reinforced films to become (4.508, 4.345 and 4.215) eV for the nanoparticle reinforcement ratios. (3%, 5% and 7%), respectively, the decrease in the energy gap value is due to defects in the films when the concentration of nanoparticles  $(MgO_{NPs})$  increases, as these defects produce localized states in the energy gap, and thus lead to lower energy gap values, that is, the decrease in the energy gap reflects the increase in the degree of turbulence in the prepared films [37].



*Fig. 5. The absorption coefficient and energy gap of the (CMC/PAA) films.*

The refractive index and Extinction Coefficient were calculated for pure (CMC/PAA) films reinforced with different weight ratios of calcined magnesium oxide nanoparticles ( $MgO_{NPs}$ ) at (800°C), as shown in Figure (6). It was observed that with the increase in the concentration of Magnesium Oxide nanoparticles  $(MgO_{NPs})$  both the refractive index and the Extinction coefficient increased. The behavior of the refractive index is due to the increase in the density of the nanoparticles, which leads to the scattering of the incident photon. While the increase in the Extinction coefficient is due to the increase in the absorption coefficient, as the Extinction coefficient depends largely on the absorption coefficient [38]. This agrees with many researchers [39,40,41].



*Fig. 6. Refractive index and Extinction Coefficient of (CMC/PAA) films.*

The real part  $(E_r)$  and the imaginary part (Ei) of the dielectric constant were calculated, and as shown in Figure (7) which shows the change of the real part and the imaginary part of the dielectric constant of (CMC/PAA) films. The results show that the values of the real and imaginary part of the dielectric constant of (CMC/PAA) films increase when adding magnesium oxide nanoparticles (MgO<sub>NPs</sub>) calcined at a temperature of (800°C), which is arranged by an increase in the absorption and scattering of incident light due to the increase in the content of nanoparticles [42,43].



*Fig. 7. The real and imaginary part of the (CMC/PAA) films dielectric constant.*

## **3.5. Thermal properties**

The study of thermal conductivity of polymeric materials and their compounds is one of the physical properties through which the suitability of these materials for the required thermal applications is determined. Figure (8) shows the thermal conductivity coefficients (K) of the as-prepared polymeric films (CMC/PAA) before and after cementation with calcined magnesium oxide nanoparticles (MgO<sub>NPs</sub>) at (800 $^{\circ}$ C), The results obtained show that the thermal conductivity coefficient increases significantly when adding magnesium oxide nanoparticles (MgO<sub>NPs</sub>) to the polymeric mixture, also by increasing the weight ratio of magnesium oxide nanoparticles  $(MgO_{NPs})$  the thermal conductivity coefficient increases, Where we note that the thermal conductivity coefficient of the pure film  $(CMC/PAA)$  is equal to  $(0.00127 \text{ W/m}$ .K) and when reinforced with nanoparticles, an increase in the thermal conductivity coefficient was observed by increasing the percentage of reinforcement down to the ratio (CMC-PAA: 7%  $MgO_{NPs}$  at 800 °C), where the thermal conductivity coefficient (0.00514 W/m.K), as a reason for the increase in the coefficient of thermal conductivity values (K) is caused by the addition of nanoparticles to the polymeric materials and this increase in the thermal conductivity coefficient is due to the filling of the polymer with nanoparticles accordingly, The molecules fill the structural gaps (vacuoles) formed within the polymer during the formation process, On the other hand, the added nanoparticles (MgO<sub>NPs</sub>) compared to the polymeric mixture (CMC/PAA) are considered to be high thermal conductivity materials due to the difference in the structural structure between them, as the nanoparticles have a thermal conductivity coefficient (K) of a high value compared to the base material and this leads to increase the thermal conductivity coefficient (K) by adding nanoparticles [44-47].



*Fig. 8. Thermal conductivity coefficient for (CMC/PAA*:*MgO).*

# **4. Conclusions**

The solution casting technique was used to prepare films (CMC/PAA) and magnesium oxide nanoparticles (MgO<sub>NPs</sub>). TGA study of magnesium oxide nano powder show that upon heat treatment most of the reactants involved in preparing the nanomaterial will volatilize, as we notice a weight loss that starts at a temperature of (44°C) and stabilizes at a temperature of (686°C). SEM electron microscopy reveals that nanoparticles form aggregates and diffuse into (CMC/PAA:MgO) films. The optical properties of the nano composites (CMC/PAA:MgO) were measured in the wavelength range (200-1000) nm. The optical results showed that by increasing the concentration of the nanomaterial, the (reflectance, absorption coefficient, reflection index, extinction coefficient, and the real and imaginary dielectric constant) increased, while the transmittance and energy gap decreased. When adding nanoparticles, we notice that the thermal conductivity coefficient (K) increases

as a result of adding nanoparticles to the polymeric material.

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