Preparation and Study of Structural and Electrical Properties of (CMC/PAA:MgO) Nano Composites

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Abstract

Magnesium oxide particles (MgO_{NPs}) were prepared by using the precipitation method at different calcination temperatures (400, 600, and 800) °C, and the polymeric (CMC/PAA) films filled-MgO_{NPs} with films were prepared by weight ratios (0, 3, 5, and 7) wt% by casting method. The results of X-ray diffraction proved that the magnesium oxide phase is obtained at (400 °C), and with the increase in temperature, the phase increases crystallization, and the particle size increases with the increase in the calcination temperature at (600 and 800) °C. The FE-SEM results indicate that the nanoparticles (MgO_{NPs}) tend to form aggregates dispersed in the films of the nanocomposites. The electrical properties reveal that the dielectric constant and the dissipation factor decreases with increasing frequency, and they increase with increasing concentration of nanoparticles, while the electrical conductivity increases with increasing both frequency and nanoparticle concentration.

Keywords: CMC, PAA, MgO_{NPs}, Structural properties, Electrical properties.
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1. Introduction

The process of mixing two polymers has attracted researchers interest [1-3].Because of their dielectric characteristics and high resistivity, polymers were used as insulators in early studies. Electrical gadgets use polymer-based insulators to separate conductors without transferring current through them. Polymers are used as insulators in corrosion-resistant electronic equipment, printed circuit boards, and cable sheathing materials. Low cost, easy processing, flexibility, outstanding mechanical qualities, and great strength are just a few of the benefits of polymers. It is utilized in the photolithography process in the microelectronic fabrication sector.[4-17].

Nanotechnology is an interdisciplinary technology that has gained traction in a wide range of industries during the previous decade. It has been suggested that its huge societal influence was the catalyst for the beginnings of a second industrial revolution [18]. Nanotechnology is a discipline of science concerned with the atomic and molecular research and design of materials and technologies. Nanotechnology is concerned with structures with a diameter of (100) nanometers or fewer, and it encompasses the

creation of materials and technologies on this size [19,20]. Nanotechnology, as defined by Norio Taniguchi (Nanotechnology), was first coined in Japan in 1974 and is defined as the separation, consolidation, and deformation of materials by one atom or one molecule [21].This the description refers to a wide range of rapidly emerging technologies that are based on scaling down existing technologies to the next level of precision and miniaturization, i.e., technologies that provide nanometer-level precision in material industrialization [22].Nanomaterial's have gained increasing popularity in recent years due to their characteristic surface effect, quantum size effect, and size effect in ceramics, biotechnology, microelectronics, pharmaceuticals, chemical and other molecular assembly disciplines. Nanoparticles (MgO) are a new type of fine functional inorganic nanomaterial's that arose with the advancement of nanomaterial's technology. It has a surface base that separates it from acidic, neutral, and volatile adsorbents, making it a potential material for catalyst [23], support for catalyst [24,25], superconductivity, electrical membranes, and thin films as a substrate [26]. The main production methods for nano(MgO) currently documented are liquid precipitation, alkoxide hydrolysis, sol-gel solution method, electrolysis, gas phase method, solid phase method, etc. [27].

The goal of this research is to use a simple, non-toxic, and low-cost precipitation process for producing high-purity and crystalline magnesium oxide nanoparticles (MgO_{NPs}). Polymeric nanocomposite films were made with these semiconducting particles. In order to generate flexible polymeric nano films with the appropriate specifications and the ability to use them in electrical applications.

2. Experimental Part

2.1. Preparation of (MgO_{NPs})

Magnesium oxide particles (MgO_{NPs}) were prepared using the precipitation method by following the following steps: (0.2M) of aqueous magnesium dichloride ($MgCL_2.6H_2O$) was dissolved in (100 ml) distilled water under constant stirring using a magnetic mixer at (50°C) and leave the solution for half an hour to obtain a homogeneous solution. Then, a solution of ammonium hydroxide (NH_4OH) was added slowly (drop by drop) using (100 ml) burette. 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. After the distillation process was completed, the resulting solution would be pH ranges between (12) with continuous heating at (50°C) for the water to evaporate, then this solution is filtered with filter paper and then washed several times with distilled water. Finally, the resulting material was dried for Three hours at (150°C) and then followed by calcination at (400, 600, and 800 °C) for Four hours in order to obtain magnesium oxide particles (MgO_{NPs}).

2.2Preparation of (CMC/PAA:MgO) Nanocomposites

The pure (CMC/PAA) and nanocomposite samples were prepared using the solution casting process, and the two polymers (CMC) and (PAA) were dissolved in distilled water for one hour at (50°C), (0.5g) for both polymers in (10ml) of distilled water, Then the two polymers were mixed together for three hours at the same temperature as before to produce (CMC/PAA) blend, and varied weight percentages of (MgO_{NPs}) (0, 3, 5, and 7) wt% were added to the polymer solution.

3. Results and Discussion

3.1. Structural Analysis

XRD Analysis.X-ray diffraction assay was performed in order to determine the crystal structure and crystal size of magnesium oxide nanoparticles (MgO_{NPs}) before and after the calcination process prepared by precipitation method. Figure (1) shows the X-ray diffraction patterns of magnesium oxide (MgO_{NPs}) before and after the calcination process at (400, 600, 800) °C, the results revealed the detection of diagnostic peaks for the powder prepared before the calcination process at (2Θ = 32.86°, 38.01°, 50.88°, 58.71°, 62.12°, 68.31°, 72.12°) for the crystal levels (100), (011), (012), (110), (111), (103) and (201) respectively, Which refers

to the formation of pure magnesium hydroxide ($Mg(OH)_2$) with a hexagonal crystal structure with crystal structure(P-3m1 no.164), crystalline dimensions (a=b = 3.142 ˚A and c = 4.766 ˚A) and crystal angles (α=β=90° and γ= 120°), which corresponds to the standard X-ray diffraction spectrum (JCPDS 01-076-0667)[28,29].

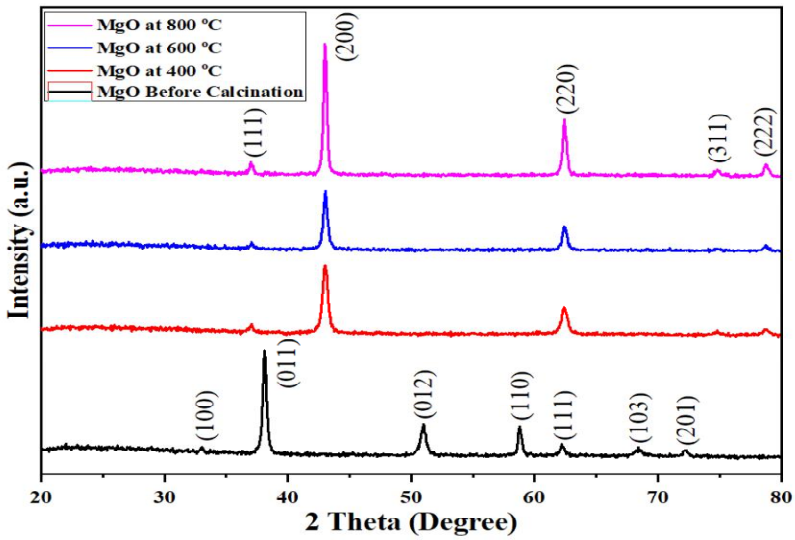


Fig.1. X-ray diffraction patterns of magnesium oxide nanoparticles before and after calcination at (400, 600, 800) °C.

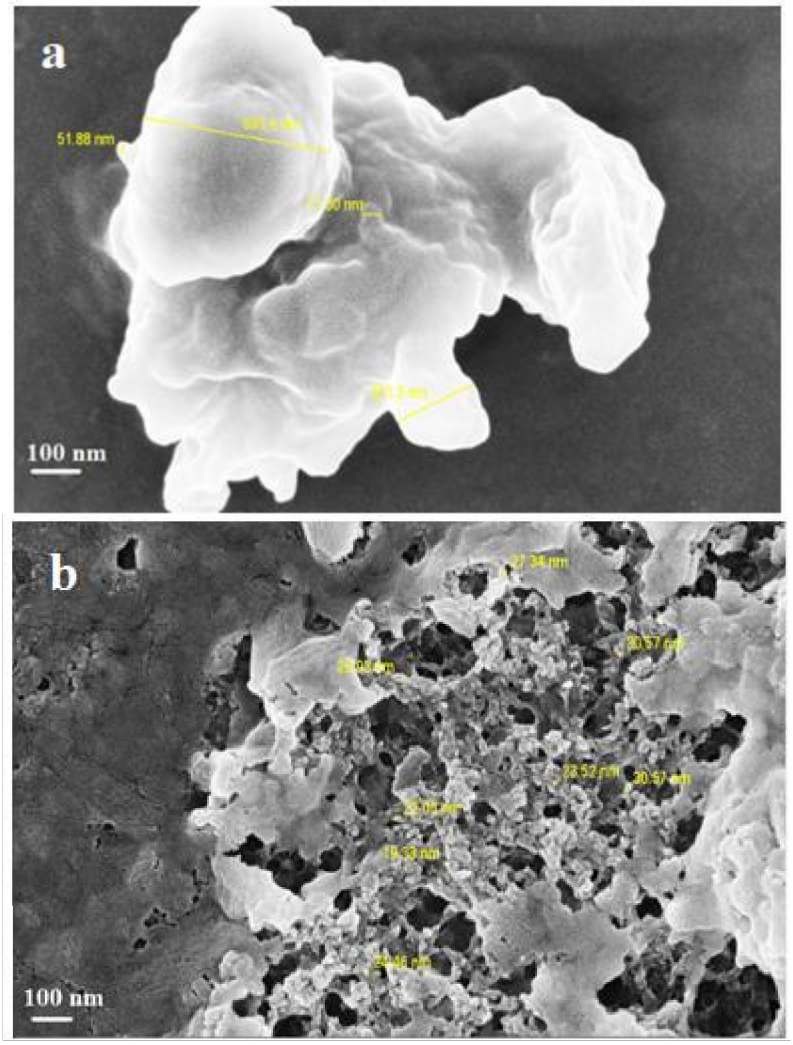
From Figure (1) we notice that after the calcination process at (400, 600 and 800) °C a phase shift occurred where new diagnostic peaks were detected at (2Θ = 36.98°, 42.98°, 62.38°, 74.81°, 78.76°) for crystalline levels (111), (200), (220), (311) and (222), which indicate the formation of pure magnesium oxides (MgO_{NPs}) at (400 °C) with a cubic crystal structure with crystal structure (Fm-3m no.225), crystalline dimensions (a= b = c = 4.209 ˚A) and crystalline angles (α=β=γ= 90°) Which matches the Standard Spectrum for X-ray diffraction (JCPDS 01-077-2364). It was observed that with the increase in the calcination temperature at (600 and 800) °C, the diagnostic peaks become sharper and higher in intensity, and this is due to the high crystallization of the magnesium oxide nano powder [30], and the crystal growth is in the same cubic system at all temperatures. The results of X-ray diffraction proved that a pure magnesium oxide phase is obtained at the temperature (400 °C), and with the increase in temperature, the phase crystallizes further. No other peaks were detected indicating the presence of impurities within the crystal structure of the prepared magnesium oxide.

Through the use of Debye-Scherrer equation, the crystalline size of magnesium oxide particles (MgO_{NPs}) before and after calcination was calculated to be (19.33 nm) before calcination and (13.74 nm), (17.01 nm), (21.24 nm) after calcination at (400, 600 and 800) °C respectively, where the results showed a decrease in the granular size after the calcination process at (400 °C) as a result of the drying process of ($Mg(OH)_2$) to obtain magnesium oxide (MgO_{NPs}), and that the granular size For the particles, it increases with the temperature of calcination at (600 and 800) °C, and this is due to the decrease in the width of the diagnostic peaks (the decrease in FWHM values) with the increase in temperature, and this agrees with many researchers [31-35]. Table (1) presents the values of crystalline size and some crystalline parameters of the prepared powder before and after the calcination process that were calculated by X-ray diffraction.

Table.1. Some Crystalline Parameters of Magnesium Oxide Particles Before and After the Calcination Process at (400, 600 And 800) °C.

	MgO Powder		2Θ (deg) Practical	2Θ (deg) Standard	FWHM (deg)	Crystalline size (nm)	dhkl (°A) Practical	d_{hkl} (°A) Standard	(hkl)
	Before calcination		38.12	38.05	0.3884	19.33	2.3588	2.36304	(101)
400 °C	43.02	42.941	0.5377	13.74	2.1008	2.1045	(200)		
600 °C	43.04	42.941	0.4343	17.01	2.0999	2.1045	(200)		
800 °C	43.02	42.941	0.3479	21.24	2.1008	2.1045	(200)		

SEM Analysis. Examination of the prepared films (CMC/PAA) by FE-SEM was carried out before and after reinforcement with magnesium oxide nano powder (MgO_{NPs}) with a percentage of (7%) at (800 °C). Figure (2a) shows that The pure (CMC/PAA) film is homogeneous and regular [36,37], and when magnesium oxide nanoparticles (MgO_{NPs}) are added with a percentage of (7%) at (800 °C) as shown in Figure (2b), the picture show that polymeric films with(CMC/PAA:MgO_{NPs}) nanoparticles contains many clusters distributed on the surface, where the results indicate that nanoparticles (MgO_{NPs}) tend to form aggregates dispersed in the films of nanocomposites [38-40].



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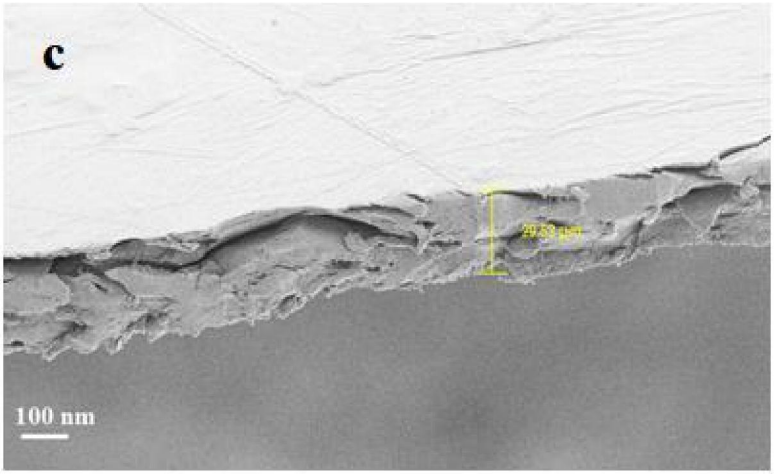

Fig.2. (a) FE-SEM Images of Pure Film,(b)Nano Composites Film(CMC/PAA:MgO_{NPs}) and (c) Cross-Section Image.

3.2. Electrical Properties

Dielectric Constant. The values of the dielectric constant ('ε) were calculated for the prepared polymeric single (CMC/PAA) films and (CMC/PAA) films reinforced with magnesium oxide nanoparticles (MgO_{NPs}) with different weight ratios (3, 5, 7) wt% and calcined at (800 °C), within the frequency range from (1-5MHz) at room temperature (25 °C). Figure (3) shows the behavior of the dielectric constant ('ε) for single (CMC/PAA) films supported by magnesium oxide particles (MgO_{NPs}) nanoparticles with different weight ratios were calcined at (800 °C) as a function of frequency.

The results showed a decrease in the values of the dielectric constant ('ε) for all the prepared single and reinforced films with increasing frequency values as shown in Table (2), which is because the electric dipoles inside the material particles arrange themselves in the direction of the electric field applied to the frequencies values at Low frequencies, while at high frequencies the electric field periodically reverses very quickly, as there is no further diffusion of ions towards the applied electric field, which leads to a decrease in the dielectric constant values with increasing frequency [41]. Most polymeric materials share a decrease in dielectric constant values with increasing frequency [42].

On the other hand, the results showed that the addition of magnesium oxide nanoparticles (MgO_{NPs}) increased the dielectric constant values for all films (CMC/PAA) prepared at each frequency, the higher the ratio of the material to nanoparticles. The higher the dielectric constant values, the higher the dielectric constant (CMC:PAA: 7wt% at 800°C) the films reached the higher the dielectric constant as shown in Table (2) The reason for this is due to the addition of magnesium oxide nanoparticles (MgO_{NPs}) To fill the gaps or defects that separate the adjacent levels and reduce the voltage barrier between them, which facilitates the transport of ions between the levels within the structure of the reinforced polymeric membranes and the addition of magnesium oxide nanoparticles, and thus leads to an increase in the polarization ratio[43,44].

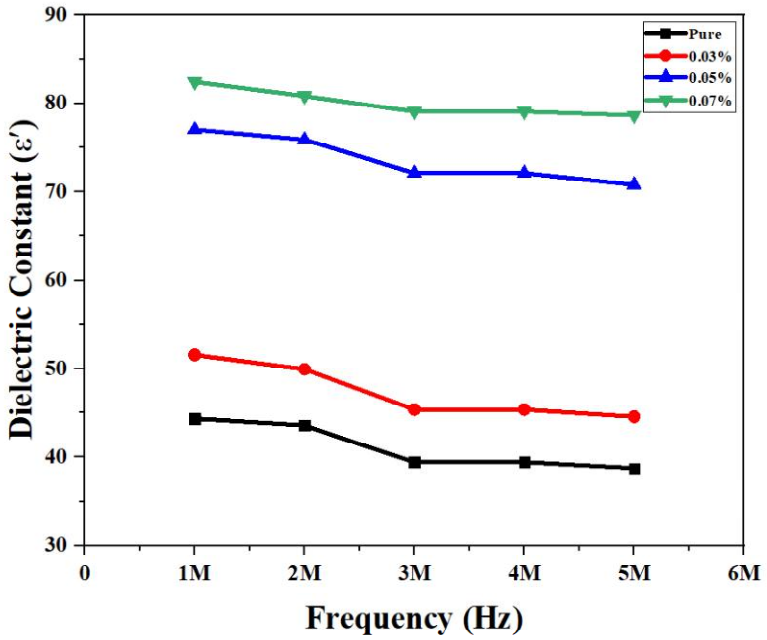

Fig.3. Variation of the dielectric constant as a function of frequency for single (CMC/PAA) films with (MgO_{NPs}) .

Table.2. values of the dielectric constant for films prepared at different frequencies.

			Dielectric Constant ('ε)	
(MHZ)	Frequency	CMC:PAA 3 wt% MgO_{NPs} at 800°C	CMC:PAA 5 wt% MgO_{NPs} at 800°C	CMC:PAA 7 wt% MgO_{NPs} at 800°C
1	44.33	51.54	77.01	82.39
2	43.6	49.93	75.86	80.82
3	39.4	45.33	72.12	79.09
4	39.4	45.33	72.12	79.09
5	38.72	44.54	70.82	78.64

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Dissipation Factor. The study of the dissipation factor (tanδ) is one of the most important factors that are directly related to the applications of polymeric composite materials, and the loss factor is defined as a measure of the percentage of energy loss in electrically insulating materials to the total energy passing through the insulator, where the loss factor is directly proportional to the energy dissipated within the range From (1-5MHz) at room temperature (25 °C). The dissipation factor values were calculated for the prepared films. Figure (4) shows the behavior of the dissipation factor (tanδ) for the single (CMC/PAA) films with (MgO_{NPs}) nanoparticles with different weight ratios were calcined at (800°C) as a function of frequency.

The results showed a decrease in the values of the dissipation factor (tanδ) for all the prepared single and reinforced films with an increase in the frequency values as shown in Table (3). It limits the mobility of these diodes, which leads to a decrease in the number of charge carriers with an increase in frequency values. Therefore, the dipoles need higher energy for relaxation to occur, and consequently, the values of the dissipation factor (tanδ) decrease [45].

On the other hand, the results show that the addition of magnesium oxide nanoparticles (MgO_{NPs}) led to an increase in the values of the dissipation factor (tanδ) for all the prepared

films (CMC/PAA) at each frequency. (CMC:PAA: 7wt% at 800 °C) the highest value of the dissipation factor and as shown in Table (3), the reason for this increase is due to the increase in the number of electrons in the nano composites [46,36].

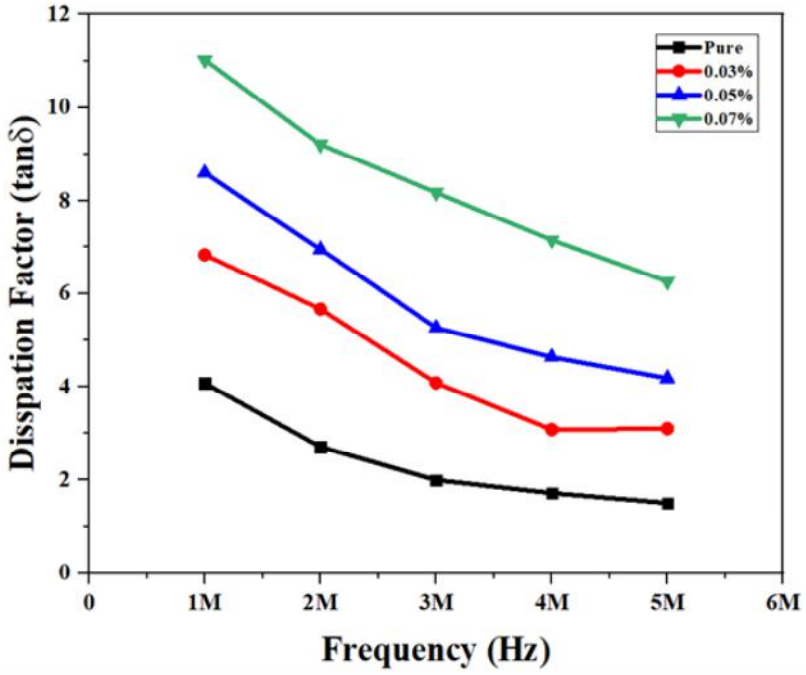

Fig.4. Variation of the dissipation factor as a function of frequency for single (CMC:PAA) films with(MgO_{NPs}).

Table.3. values of the dissipation factor for the films prepared at different frequencies.

Frequency (MHZ)	Dissipation Factor (tanδ) CMC:PAA	CMC:PAACMC:PAACMC:PAA 3 wt% MgO_{NPs} at 800°C	5 wt% MgO_{NPs} at 800°C	7 wt% MgO_{NPs} at 800°C
1	4.07	6.83	8.6	11.014
2	2.71	5.66	6.95	9.21
3	2	4.08	5.26	8.18
4	1.72	3.08	4.63	7.156
5	1.51	3.1	4.18	6.25

A.C Electrical Conductivity. Alternating electrical conductivity values ($\sigma_{a.c}$) were calculated for all the as-prepared films as a function of the frequency of the applied electric field within the range (1-5 MHz) and at room temperature (25 °C) to understand the mechanism of electrical conductivity and the nature of the polarization that occurs within Prepared films, Figure (5) shows the AC conduction ($\sigma_{a.c}$) behavior of single (CMC/PAA) films with(MgO_{NPs})nanoparticles (MgO_{NPs}) with different weight ratios calcined at (800 °C) as a function of frequency.

The results showed an increase in the electrical conductivity values of alternating current ($\sigma_{a.c}$) for all the prepared single and reinforced films with increasing frequency values as shown in Table (4). This is due to the increase in the frequency values and thus the increase in the ratio of electric polarization within the prepared membranes, which results in a rapid jump in the charge carriers between adjacent levels, In general, polymeric materials and semiconductors share an increase in electrical conductivity values with increasing frequency [47].

On the other hand, the results show that the addition of magnesium oxide nanoparticles (MgO_{NPs}) led to an increase in the A.C electrical conductivity values ($\sigma_{a.c}$) for all (CMC/PAA) films prepared at each frequency. The films (CMC:PAA: 7 wt% at 800 °C) achieves the highest A.C electrical conductivity value and as shown in Table (4), the reason for this increase is due to the addition of conductive particles to the magnesium oxide nanoparticles (MgO_{NPs}) creating conductive pathways inside The structure of the prepared films thus increases the values of A.C electrical conductivity and decreases the voltage barrier between the conduction levels [48,49].

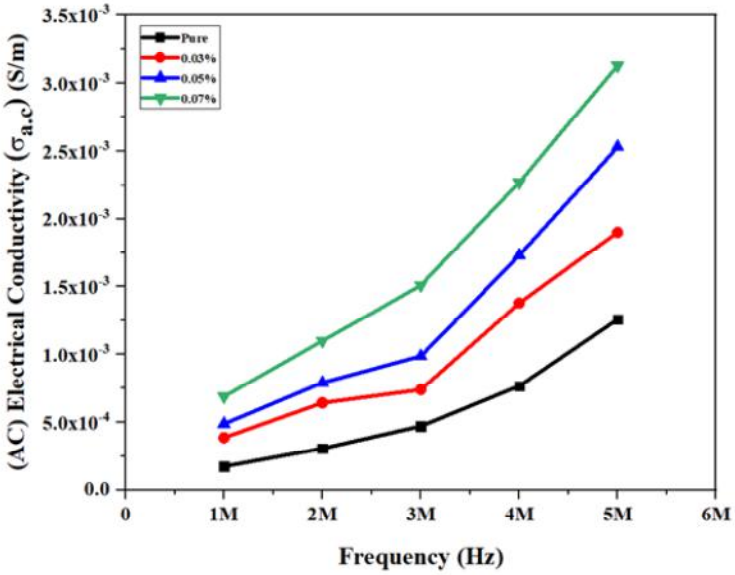

Fig.5. Variation of the electrical conductivity of alternating current as a function of frequency for individual films (CMC/PAA) with (MgO_{NPs}) nanoparticles.

Table.4. values of the alternating electrical conductivity coefficient for films prepared at different frequencies.

A.C Electrical Conductivity ($\sigma_{a.c}$)*10^{-5} (S/m)					
	Frequency (MHZ)	CMC:PAA 3 wt% MgO_{NPs} at 800°C	CMC:PAACMC:PAA 5 wt% MgO_{NPs} at 800°C	CMC:PAACMC:PAACMC:PAA 7 wt% MgO_{NPs} at 800°C	
1	1.74*10^{-4}	3.88*10^{-4}	4.88 *10^{-4}	6.88*10^{-4}	
2	3.08*10^{-4}	6.42*10^{-4}	7.89*10^{-4}	0.0011	
3	4.72*10^{-4}	7.39*10^{-4}	9.87*10^{-4}	0.00151	
4	7.66*10^{-4}	0.00138	0.00173	0.00227	
5	0.00126	0.0019	0.00253	0.00313	

4. Conclusions

The solution casting method was used to prepare pure films and with (MgO_{NPs})nanoparticles. The results of X-ray diffraction proved that with increasing temperature, the phase crystallization increases. FE-SEM results show that polymeric films withnanoparticles (CMC/PAA:MgO_{NPs}) contain many agglomerates distributed on the surface.The electrical results showed that with increasing frequency, the dielectric constant and the dissipation factor increased, and the electrical conductivity decreased, but when nanoparticles were added, the (dielectric constant, dissipation factor, and electrical conductivity) increased.

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[18] Scanning electron microscopy (SEM) shows the surface morphology of the (PAA-CMC-MgO)nanocomposites films many aggregates or chunks randomly distributed on the top surface, homogeneous and coherent.
[19] D.C electrical conductivity for (PAA-CMC-MgO) nanocomposites increased with increasing of temperature and magnesium oxid nanoparticles wt.% concentration.
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