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The Impact of Adding Silver Nanoparticles on the Physical Properties and Antimicrobial Activity of PVA Polymer

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ABSTRACT: Poly (vinyl alcohol) (PVA) thin films with different amounts of silver (Ag) nanoparticles (0.001, 0.002, 0.003, 0.004 and 0.005)g were created using the solution cast method. PVA/Ag nanocomposite structural and optical characteristics have been investigated. Using the X-ray Diffraction (XRD) technique, the produced nanocomposite's structural analysis was conducted.The crystalline structure of Ag nanoparticles was confirmed by the XRD pattern. The chemical bond and stretching vibrations attributed to different peaks were evaluated using FTIR spectra.UV-Vis spectroscopy has been used to investigate the impact of AgNPs on the optical properties of PVA, including absorbance, transmittance, absorption coefficient, refractive index, extinction coefficient, and real and imaginary components of the dielectric constant. The energy gap decreases the most plausible reason is the creation of a localized level in the forbidden energy gap as a result of increasing the fillers nanoparticle weight. This work demonstrates that the amount of AgNPs influences all of these features. The antibacterial activity of the nanocomposite must be measured.Hence, positive bacteria (Staphylococcus aureus, Streptococcus pneumonia), negative bacteria (Escherichia coli, Pseudomonas aeruginosa), and fungus (Candida albicans) were taken as models in this work. The maximum inhibitaion zones for PVA+(0.005g) Ag in Candida albicans and Staphylococcus aureus were about 18 mm, respectively.

Keywords: Nanocomposites, optical properties, PVA/Ag nanoparticles, Antimicrobial Activity.

1. INTRODUCTION

Recently, nanotechnology has emerged as a highly distinct field that includes everything from precisely new techniques to create new materials with dimensions on the nanoscale to revolutionary extensions of conventional device physics. Metal nanoparticles' extraordinary optical, electrical, and magnetic properties have been used to develop novel nanowires and nanorods as well as other nanocomposites. The synthesis and production of one-dimensional nanomaterials have come a long way in the last ten years. Metal nanoparticle integrated polymers have garnered a lot of interest because of the broader range of applications that these hybrid materials can be used for Silver (Ag) nanoparticles have drawn a lot of interest because of their unique physical and chemical characteristics. It is utilized in a variety of applications, including non-linear optics [1].

One of the most significant polymers that is available in powder, fiber, and film forms is PVA, commonly referred to as polyvinyl alcohol. This is a well-known water-soluble polymer that is often utilized in films, adhesives, paper coating agents, warp sizing agents, and other products. Due of its numerous industrial applications and low manufacturing costs, PVA has recently been the subject of intense research [2].

Typically, natural polymeric materials (such as: wool, silk, cellulose, and natural rubber) were employed textiles, papers, meals, pharmaceuticals, as well as in a number of technical processes for solar cells, energy storage devices, nano-dielectric structures, and optoelectronic devices [3]. Silver nanoparticles have a variety of interesting and distinctive abilities. It can be used in a wide range of applications, including those involving catalysis, electronics, non-linear optics, antimicrobials, and biomaterials. The ability of the secondary alcohol groups to reduce, as well as the polyvinyl alcohol's

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*Corresponding author: fatima_abd@uomustansiriyah.edu.iq outstanding film-forming capabilities and optical clarity, make it desirable to employ PVA as the host polymer for Ag nanoparticles [4].

The effects of incorporating silver nanoparticles into a mixture of polyvinyl alcohol (PVA) and polyethylene glycol (PEG-400) on the mixture's optical properties were investigated by researchers B. H. Rabee et al in 2015. PVA-PEG blends with Ag nanoparticle ratios of 0, 3, 6, and 9 by weight percent were used to make nanocomposite samples. Preparing the films through casting [5].

A new PVA/PEG/BaTiO3 nanocomposite that is utilized in a range of optoelectronic applications was produced in 2021 by researchers B. Mohammed et al. A PVA/PEG combination was used to generate PVA/PEG/BaTiO3 nanostructures with different BaTiO3 NP ratios. The results demonstrated this, PVA/PEG's optical qualities improved as the proportion of BaTiO3 NPs in the mixture rose. Its nature allows it to be used in many different photonics and electronics applications [6].

In 2023 Researchers G. Abdul, H. Abdul et al, prepared PVA/ZnO Nanocomposites to fuond optical characteristics and antibacterial activity by using solution casting method. to create nanocomposites with different amounts of Zinc Oxide nanoparticles (0.001-0.005)g. The structural and optical properties of PVA/ZnO nanocomposites were examined. The absorbance spectrum and absorption coefficient of PVA polymer are improved by adding more ZnONPs to nanocomposites, according to experiments; the energy band gap of PVA polymer shrank as ZnONP concentration increased[7].

Investigating how introducing silver nanoparticles affects the physical properties and antimicrobial activity of polymer polyvinyl alcohol is the goal of this investigation.

2. THEORETICAL PART

The relationship between the incident light intensity (I_0) and the absorbed light intensity (I_A) of a material is known as absorbance (A) [8]:

$$A = \frac{I_A}{I_0} \tag{1}$$

Using the optical absorption spectrum and the following equation, one can estimate the absorption coefficient (α) [6]:

$$\alpha = 2.303\frac{A}{d} \tag{2}$$

Where, d is the film thickness in cm

From the following equation can calculate extinction coefficient (k)[6]:

$$k = \frac{\lambda\alpha}{4\pi} \tag{3}$$

Where λ: is the wavelength to incident light

From the following equation can calculate the energy band gap[9]:

$$\alpha h\upsilon = B(h\upsilon - E_g)^x \tag{4}$$

Where, α is the absorption coefficient, υ is the frequency, h is Planck's constant, B is a constant, E_g is the optical energy band gap between the valence band and the conduction band and x is the power that characterizes the transition process. Specifically, x can take the values 1/2, 3/2 , 2 or 3 for transitions designated as direct allowed, direct forbidden, indirect allowed, and indirect forbidden respectively. The determination of the value of optical energy band gap E_g involves the plotting of $(\alpha h\upsilon)^{1/x}$ against hυ.

The reflectance(R) has been found from values of transmission (T), and Absorbance (A), using the relationship[10]:

$$A + R + T = 1 \tag{5}$$

From the following equation can calculate the refractive index(n)[11]:

$$n = \frac{1+R^{1/2}}{1-R^{1/2}} \tag{6}$$

Where R: is the reflectance

From the following equation can calculate the real and imaginary parts of dielectic constants[11].

$$\varepsilon_r = n^2 - k^2 \tag{7}$$

$$\varepsilon_{i=2nk} \tag{8}$$

Moreover, the average crystallite size (D) has been estimated by using the Scherer formula [2].

$$D = \frac{K\lambda}{\beta COS(\theta)} \tag{9}$$

K: shape factor = 0.94

β is FWHM (full width at half maximum) is equal to the width of the line profile (measured in radians) at half the maximum intensity(radian).

3. EXPERIMENTAL WORK

The very good properties of poly (vinyl alcohol) that has a molecular weight (M_W=14000g/mol) produced in India and comes from Thomas Baker. It was selected as the host material for silver nanoparticles with particle size less than 50 nm produced in Nanjing Nano Technology co, Ltd. in this study. In order to form films of pure PVA and PVA containing Ag nanoparticles, 0.9g of PVA was combined with (0.001, 0.002, 0.003, 0.004 and 0.005) g of silver nanoparticle in 10ml of hot distilled water. Stirred for 2 to 3 hours at room temperature with a magnetic stirrer (hot plate) in (25–30 °C). Pour the solution into glass plates with a diameter of 3 cm and let it slowly evaporate at room temperature for 3 to 4 days to obtain a homogeneous film. UV-Visible spectrophotometer was used to measure the absorption and transmission spectra type ((T70/T80) series UV/Vis spectrometer) in the wavelength range (200-900) nm. The thickness of the films was measured via a digital micrometer type (Tesha) with (0.001mm) measurement accuracy and measurment range (0-150) mm made in Japan. Fourier Transform Infrared spectroscopy (FTIR) was conducted for all films using (Bruker-Tensor 27 with ATR unit). In the wavenumber scope (400-4000) cm^{-1}, the instrument works. Acompletely computerized X-ray diffractometer (XRD; X'Pert PRO, PANalytical, the Netherlands) was used to study the structure of PVA polymer and PVA/Ag nanocomposite film. The FESEM (model: EM 10C) from the German company Zeiss was used to take topography of as-prepared films.

The antimicrobial activity was examined by diffusion method in agar wells in microbiology labrotary. The microbial included gram-positive bacteria (*Staphylococcus aureus*, *Staphylococcus pneumonia*) and gram-negative (*Escherichia coli and Pseudomonas aeruginosa)* bacterial cultures, as well as *Candida albicans*. This study used sterile petri dishes with a 90 mm diameter and sterile nutritional agar material, along with diffusion techniques in agar wells. When fungi and bacteria were incubated for 24 hours at 25°C, the diameter of the inhibitory zone (mm) was measured.

4. RESULTS AND DISCUSSION

4.1 X-Ray Diffraction

The X-Ray Diffraction pattern of pure PVA and PVA with different Ag amounts (0.003 and 0.005 g) are shown in Figs.1 and 2, respectively. Fig.1 illustrates the X-Ray Diffraction pattern of pure PVA polymer. It will be noticed one wide peak 2θ=19.4046° and two small peaks 2θ =22.6160° and 39.9739°, indicating the synthesis pure PVA film is semi-crystalline; these peaks are agreed with [2, 19].

Fig.2-A shows that the XRD pattern of PVA + 0.003g Ag nanoparticles films. It can be notice from the figure that the major peak is identified to 2θ = 19.4174° for pure PVA and another small peak at 2θ=38.06 ° attributed to (111) plan of the Ag crystalline structure. With increasing the amount of Ag nanoparticles (PVA+ 0.005g Ag), two peaks were observed related to the (111) and (200) of Ag structure at 2θ=38.06 ° and 2θ=44.24°, respectively as shown in Fig.2-B .

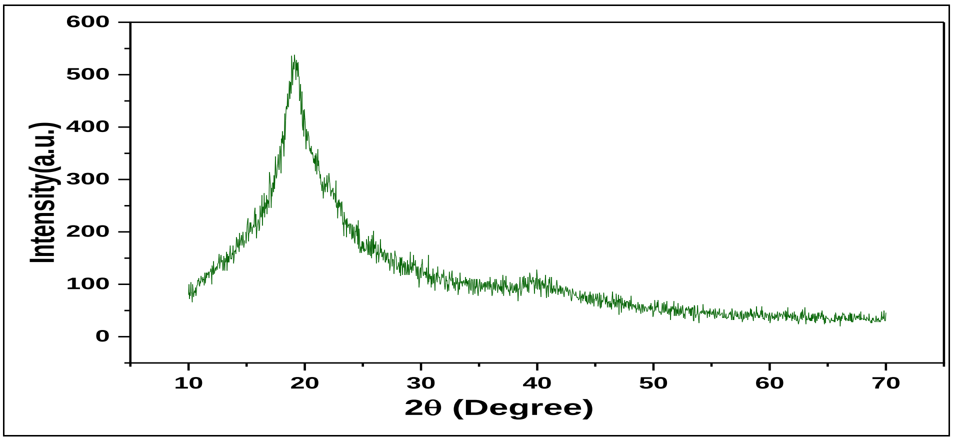

Figure (1): XRD for pure PVA polymer

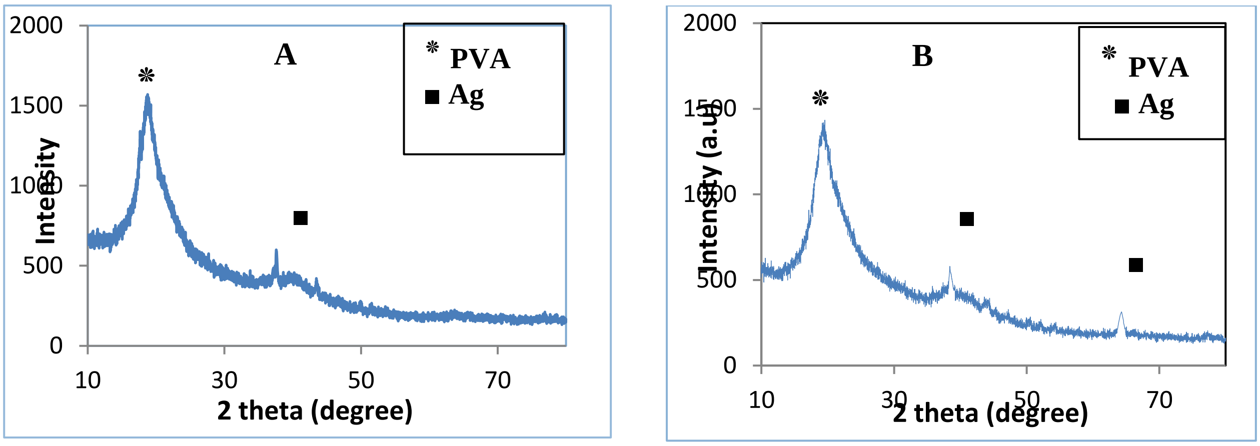

Figure (2): XRD for A- PVA +0.003g Ag, B- PVA + 0.005g Ag nanocomposits films

Fig.3 showed XRD pattern of Ag nano powder. The polycrystalline structure of Ag is indicated by 2θ ≈ 38˚, 44˚, 64˚, and 77˚ in the X-Ray diffraction of pure Ag nanopowder, which is consistent with the findings of F.H. Abd El-kader et al. [20]. The magnitude of (2θ) in Table (1) with full width half maximum (FWHM), intermolecular distance (d), and crystalline size (D) that calculated from eq. (9). The XRD pattern agrees with crystallography open database (COD) card no. (9013045) [21].

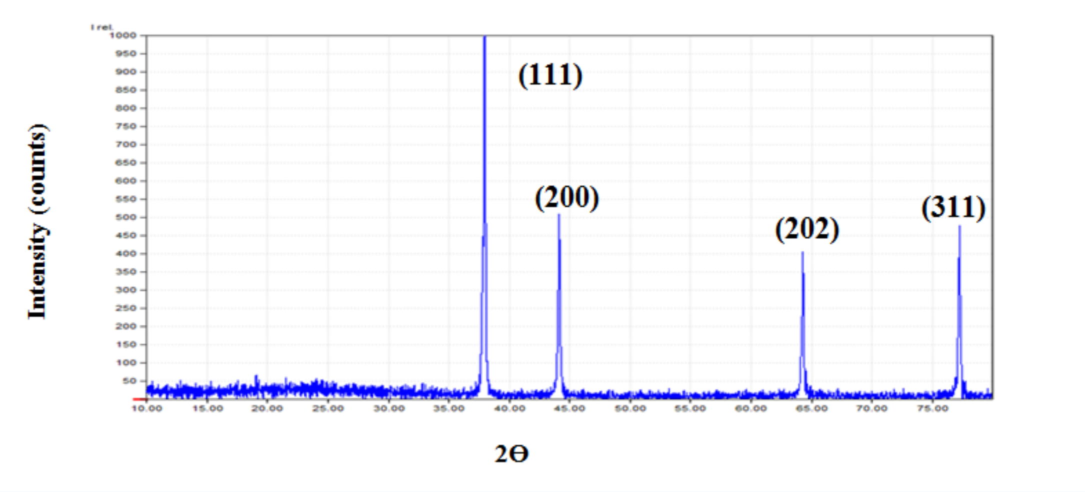

Figure (3): XRD pattern for Ag Nano powder

Table (1): X-Ray Diffraction Parameters of Ag Nano powder

2θ (deg)	d (˚A)	FWHM (deg)	D (nm)	(hkl)
38.0632	2.36223	0.20340	40.39684	111
44.2449	2.04548	0.20800	39.25314	200
64.3825	1.44590	0.20930	48.23876	202
77.3482	1.23270	0.20390	49.91782	311

4.2 FESEM Images

Fig.4 shows the PVA polymers FESEM picture. These images show a large cluster on a PVA surface.

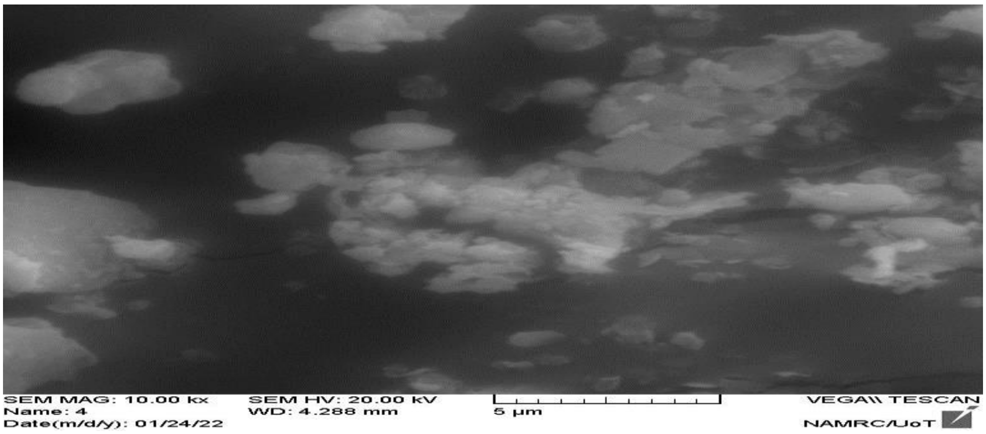

Figure (4): SEM image for pure PVA polymer

FESEM image for PVA and (0.003g) Ag films show in the Fig.5, the surface is rough [12].

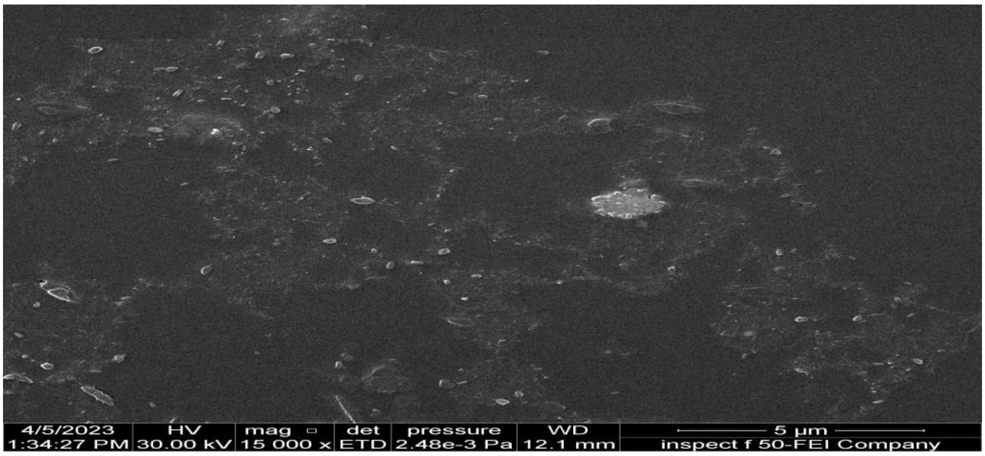

Figure (5): SEM image for PVA/Ag NPs 0.003g

FESEM image for PVA and (0.005g)Ag films show in the Fig.6 , the surface is rougher due to the increase in Ag NPs[12].

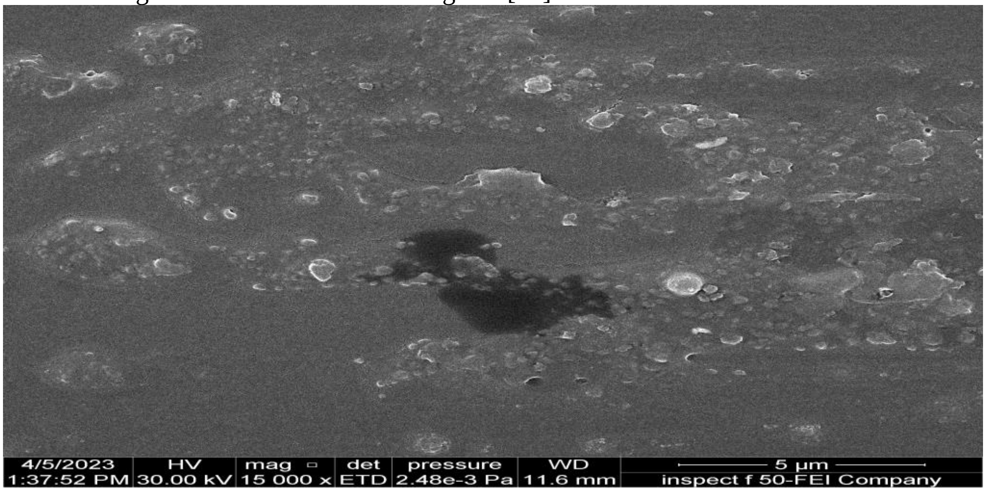

Figure (6): SEM image for PVA/Ag NPs 0.005g

4.3 FTIR Spectroscopy

The FTIR spectroscopy is significant because it specifies the primary features of polymer peaks and nanoparticles. All FTIR spectra were measured in transmittance mode.The FTIR spectrum of pure PVA film is shown in Fig.7 it is noted that the band C-H bending vibrations out of the plane appears at the peaks of (603.42, 835.45, 919.01 and 945.54) cm^{-1}. The IR peaks at (1023.47 cm^{-1} , 1087.79 cm^{-1} and 1242.59 cm^{-1}) refers to the C-O stretching mode. Furthermore, the IR transmittance peaks at (1329.20 cm^{-1} and 1373.90 cm^{-1}) represent CH_2 bending. The asymmetric bending vibrations of ($C–CH_3$) bond are noticeable at IR peak 1418.73 cm^{-1}. While, the C=C stretching peak appeared at 1567.69 cm^{-1} . The C=O stretching vibration of ester group appears around IR peaks (1716.33 cm^{-1} and 1732.76 cm^{-1}). Lastly, the C-H stretch aliphatic peaks appeared at (2855.80 cm^{-1} and 2921.56 cm^{-1}), and O–H stretching broad bands at 3263.32 cm^{-1}. Current FTIR results are matched with important findings obtained by H. Kamal, and N. Thayumanavan, et al. [2, 13, 14].

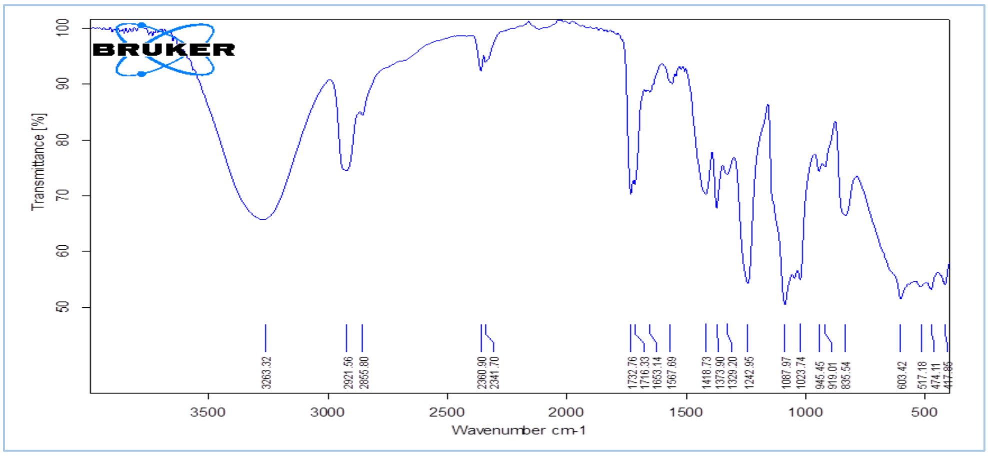

Figure (7): FTIR spectrum of pure PVA film

The FTIR spectra of powdered pure silver (Ag) nanoparticles was displayed in Fig.8 To find out the composition and purity of the metal nanoparticles, infrared research was done. In the fingerprint area, or below 1000 cm^{-1}, metals typically exhibit absorption bands due to interatomic vibrations. Ag-O stretching and deformation vibration are shown by the peaks at (1044.00 and 1251.93) cm^{-1}, respectively. The observed nanometer metal-oxygen frequencies for each distinct metal oxide are consistent with published values[12].

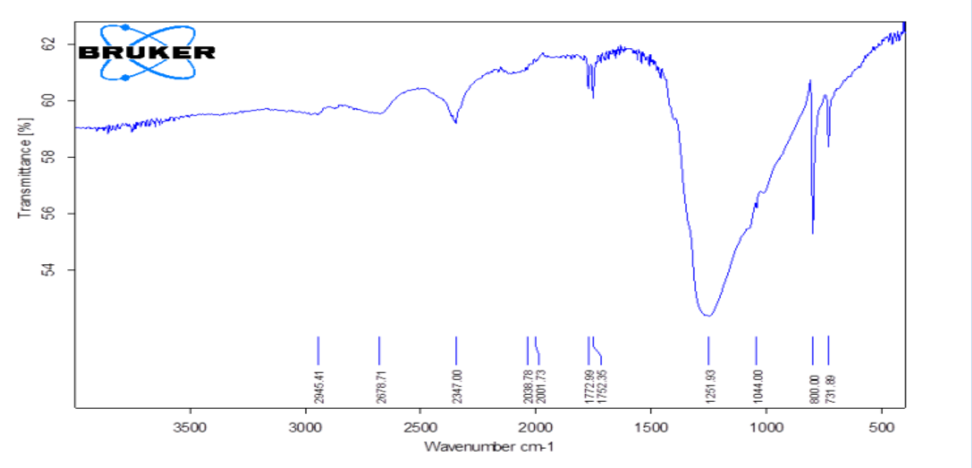

Figure (8): FTIR spectrum of powder pure Ag NPs

To prove probable intermolecular interaction between the components of nanocomposites, FTIR spectra were studied for (PVA/Ag) nanocomposite and for different amounts (0.001to 0.005g) step 0.001g as shown in Fig.9 and to clarify the positions of the peaks that appeared with types of bonds of polymer. It was observed that are no chemical interactions between the nanoparticle and the polymer. The transmittance decreases marginally with an increase in the amount AgNPs because of the increase in the density of the nanocomposite with an increase in the amount of nanoparticles[15].

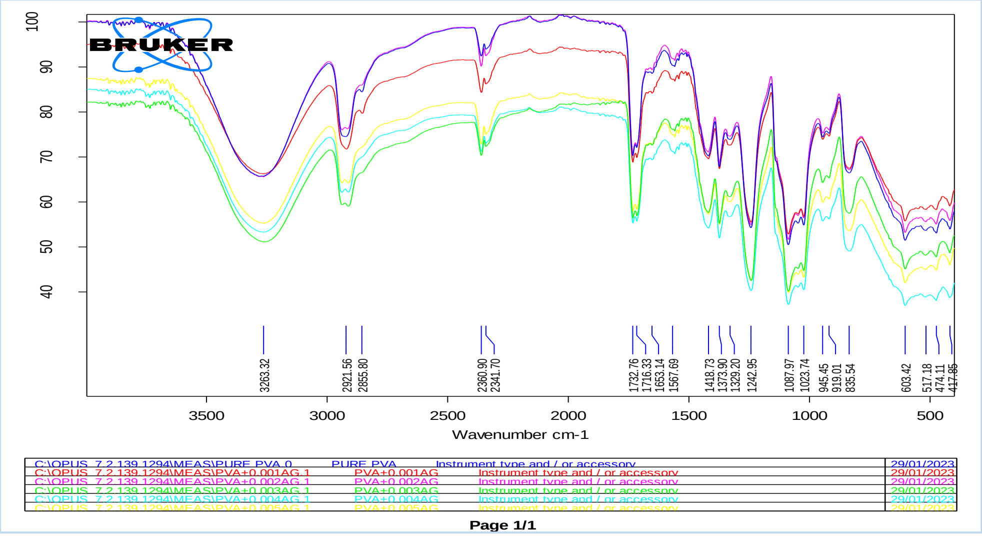

Figure (9): FTIR spectra of PVA/Ag nanocomposites

4.4 Optical Properties

The UV- Vis. spectra of pure PVA and PVA-Ag nanocomposite with different amounts of nanoparticles plotted in Fig.10 . It is clear that the maximum absorption wavelength is located at 280 nm with maximum absorbance value (0.128) for pure PVA polymer. The pure PVA film has a nearly zero absorbance in the visible region because it is colorless polymer and show no bands in the range of measurements. The absorption spectrum of PVA/Ag nanocomposite films shows two peaks at 280 nm and at 330 nm with a reduced intensity which corresponded to the Ag is present in the film. These peaks were due to as PVA absorption and Ag nanoparticle excitons. The UV absorption spectrum demonstrates this, the more the amount of Ag in the matrix of PVA, the larger the absorbance of PVA host in the ultraviolet–visible range. As show in Table(2),the maximum absorption wavelength doesn't chang with increasing the amounts of AgNPs .where the intensity of peaks inerwosed.this explained the role of adding nanomateral to enhance the absorbance polymer due to good benefit in using polymer nanocoposite films in many optical applications [16]**.**

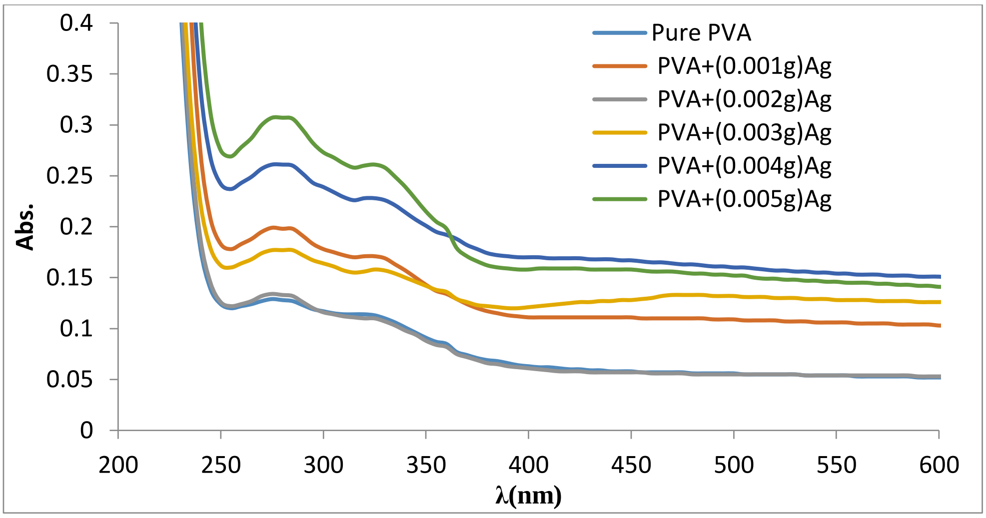

Figure (10): Absorption spectra of pure PVA and PVA/Ag nanocomposite films

Table (2): show the highest peak of the absorption spectra of PVA/Ag nanocomposite films

Samples	Wavelength(nm)	Absorbance
Pure PVA	**280**	**0.128**
PVA/Ag NPs (0.001g)	**280** **330**	**0.198** **0.169**
PVA/Ag NPs(0.002g)	**280** **330**	**0.133** **0.107**
PVA/Ag NPs(0.003g)	**280** **330**	**0.177** **0.157**
PVA/Ag NPs(0.004g)	**280** **330**	**0.261** **0.226**
PVA/Ag NPs(0.005g)	**280** **330**	**0.307** **0.258**

The transmittance (T), defined as the ratio the intensity of transmitted radiation and the intensity of incident radiation. Transmittance of pure PVA polymer and PVA/Ag nanocomposites is seen in Fig. 11. Pure PVA polymer transmittance is greater than that of PVA/Ag nanocomposites. The transmittance in the UV field of pure polymer and nanocomposites shows a high decrease than in the visible region. The effect of append Ag nanoparticles is obvious; in the UV region they have a very low transmittance that has decreased with increasing AgNPs content[5].

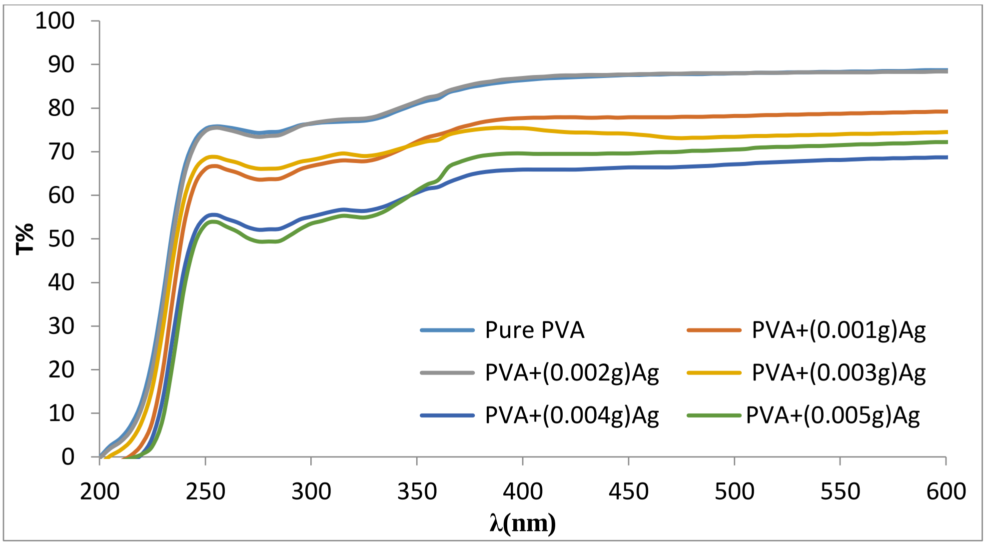

Figure (11): Transmission spectra of pure PVA and PVA/Ag nanocomposite films

The absorption coefficient (α) is defined as the ability of material to absorption light with a limited wavelength per unit length and it is a characteristic property for every absorber molecule or ion. The absorption coefficient can be calculated from eq. (2). The value of the absorption coefficient gives us information about the nature of electronic transition. When the high absorption coefficient value ($\alpha > 10^4 cm^{-1}$) at higher energies, the direct electronic transitions expected and the energy momentum preserve of the electron and photon. While the value of absorption coefficient low($\alpha < 10^4 cm^{-1}$) at low energies, indirect electronic transitions expected. Fig.12 showed the absorption coefficient as a function of the wavelength for PVA/Ag nanocomposite films. According to Fig.12, the value of absorption coefficient for all sample less than ($10^4 cm^{-1}$), so that the indirect electronic transitions have been deduced [16].

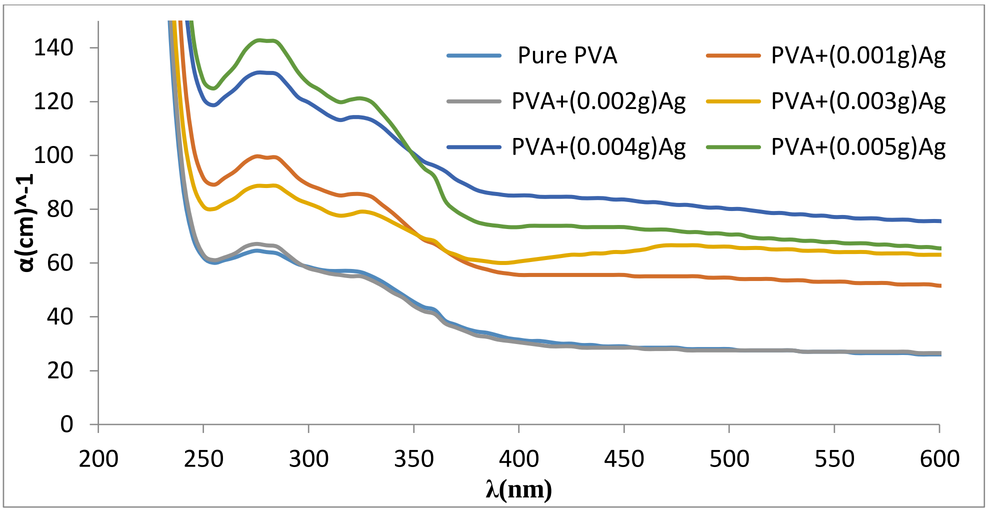

Figure (12): Absorption coefficient of pure PVA and PVA/Ag nanocomposite films

The refractive index is a significant optical characteristic that may be computed using equation (6), which is depend on reflectance. Fig.13 shows the variation of the refractive index of nanocomposites as the function of wavelength. The refractive index decrease with an increase amount of AgNPs[5].

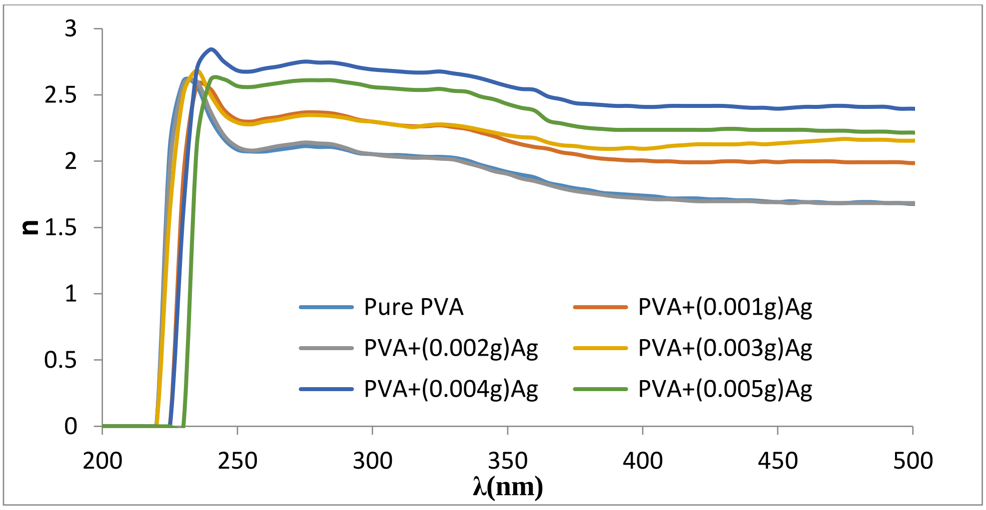

Figure (13): Refractive index of pure PVA and PVA/Ag nanocomposite film

Experimental calculations of a nanocomposites' energy band gap are shown in Fig.14. Energy band gap E_g for indirect transitions in (PVA-Ag) nanocomposite can be calculated. According to the results, when the amounts of (Ag) nanoparticles increases, the energy gap decreases from(5.19 to 4.95eV) as show in Table (3).The most plausible reason is the creation of a localized level in the forbidden energy gap as a result of increasing the fillers nanoparticle weight percentage[17].

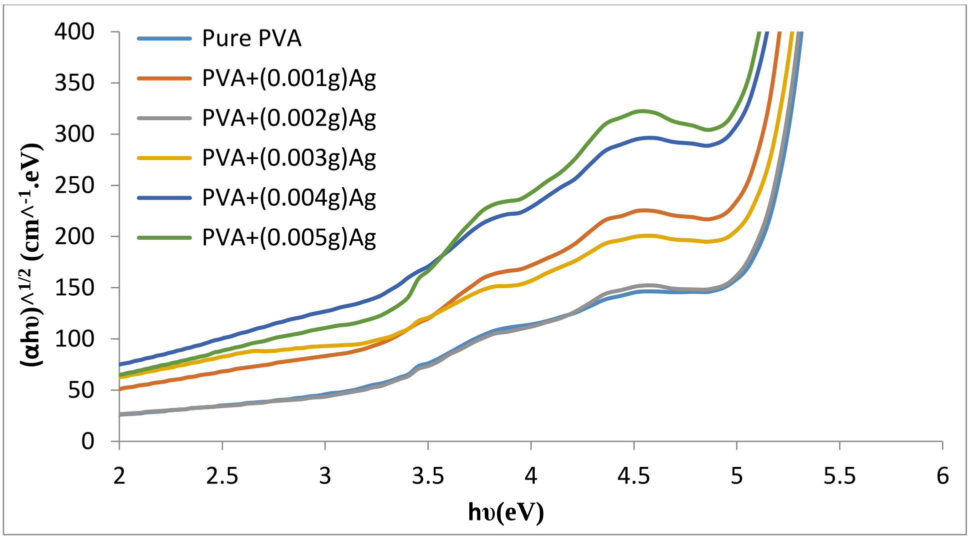

Figure (14): Optical energy gap of pure PVA and PVA/Ag nanocomposite films

Table (3): Energy gap (E_g) for PVA/Ag nanocomposite films

Samples	Energy gap(eV)
Pure PVA	**5.19**
PVA/Ag NPs(0.001g)	**5.16**
PVA/Ag NPs(0.002g)	**5.03**
PVA/Ag NPs(0.003g)	**5**
PVA/Ag NPs(0.004g)	**4.98**
PVA/Ag NPs(0.005g)	**4.95**

The coefficient of extinction can be calculated by Equation (3). The extinction coefficient depends on absorbance. The variation of extinction coefficient(k) with wavelength for (PVA-Ag) nanocomposites is as shown in Fig.15. The extinction coefficient increases with increasing of amount AgNPs. This behavior of extinction coefficient can be ascribed to high absorption coefficient. The extinction is high at the longest wavelengths and high amount[5].

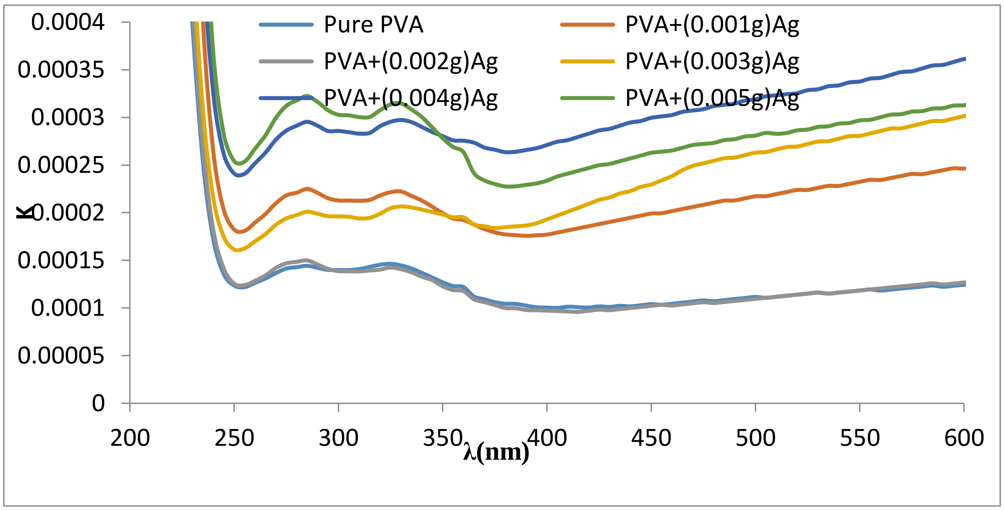

Figure (15): Extinction Coefficient of pure PVA and PVA/Ag nanocomposite films

For (PVA-Ag) nanocomposites, Fig.16 depicts the fluctuations in extinction coefficient as a function of wavelength. It demonstrates that adding more Ag nanoparticles to (PVA) increases the extinction coefficient because (PVA-Ag) nanocomposites have a high absorption coefficient. Whereas the host (PVA) will change as a result of the manganese oxide nanoparticles. An intriguing finding is that the absorbance in the visible area increases as AgNP concentration increases[7].

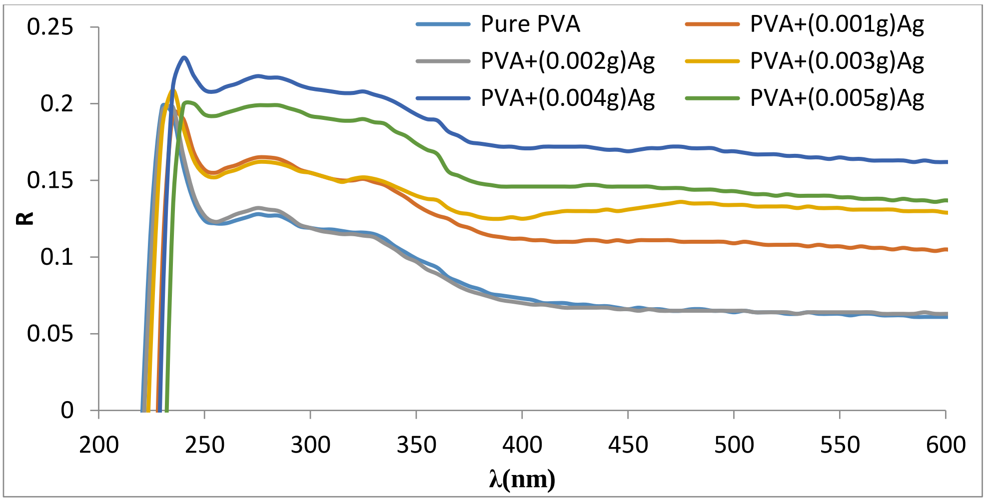

Figure (16): Reflection spectra of pure PVA and PVA/Ag nanocomposit films

The connection between the real and phantom dielectric constants of PVA/Ag nanoparticles with photon wavelength and various Ag nanoparticle grafting ratios is depicted in Figs. 17 and 18. The figures lead us to the conclusion that the imaginary(εi) and real(εr) parts of the dielectric constants rise as the percentage of grafted Ag nanoparticles increases, which is explained by an increase in incident light absorption and scattering[10].

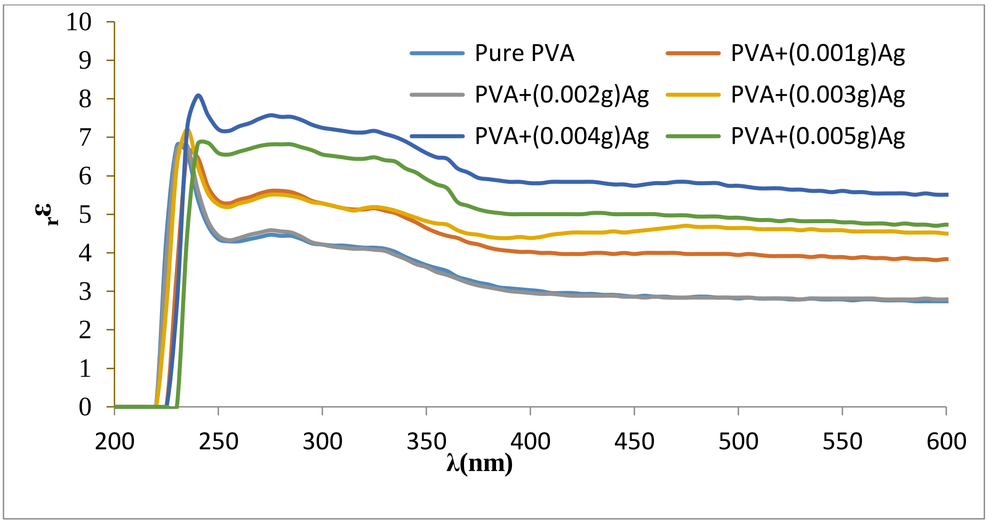

Figure (17): Real dielectric constant of pure PVA and PVA/Ag nanocomposite films

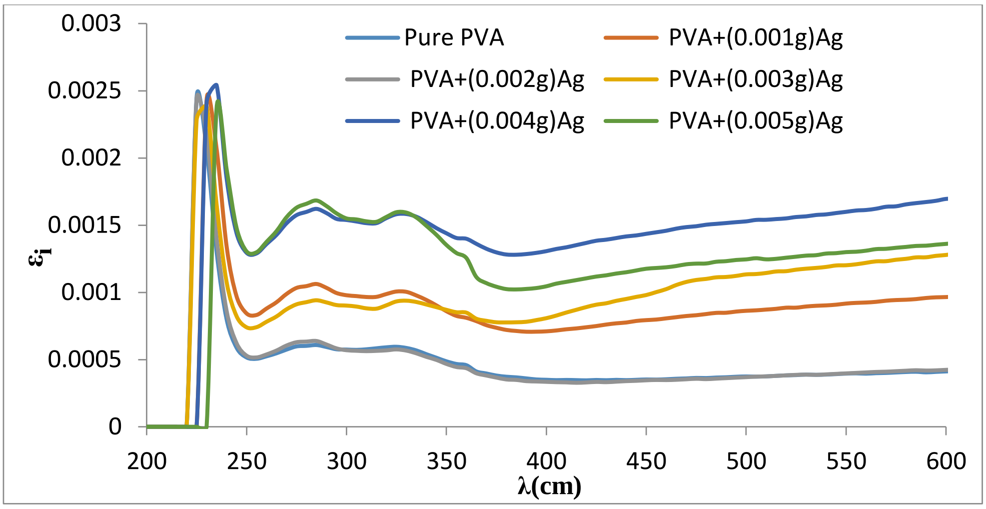

Figure (18): Imaginary dielectric constant of pure PVA and PVA/Ag nanocomposite films

5. ANTIMICROBIAL ACTIVITY

The pure PVA polymer and the PVA/Ag nanocomposite films have antimicrobial characteristics. During the agar plate method, researchers test against positive bacteria (*Staphylococcus aureus*, *Streptococcus pneumonia*), negative bacteria (*Escherichia coli*, *Pseudomonas aeruginosa*), and fungus (*Candida albicans*). The outcomes are given in Table (4). In most samples, positive bacteria like *Staphylococcus aureus* and *Streptococcus pneumonia* showed weaker antibacterial activity than negative bacteria like *Escherichia coli* and pseudomonas aeruginosa and fungi like (*Candida albicans*). The best results were noted for PVA+(0.005g) Ag in *Staphylococcus aureus* and *Candida albicans* about 18mm.

Many studies have found that Ag NPs suppress bacteria growth. The actual mechanism of action of Ag NPs is yet unclear, suggeing that bacteria are inhibited by AgNPs interacting directly with the cell surface. This interaction to impacts the permeability of the bacterial cell membrane, allowing nanoparticles to penetrate the cell system and producing oxidative stress in the bacterial cell. Bacterial growth will be inhibited and bacteria will be died as a resul[18].

Table 4:The PVA polymer and PVA/Ag nanocomposite antimicrobial efficacy against several harmful bacterial strains

Samples	**Pure zone of inhibition (mm)**				
	staphylococcus aureus	**streptococcus pneumonia**	**Escherichia coli**	**pseudomonas aeruginosa**	**Candida albicans**
pure PVA	**11**	**9**	**9**	**10**	**7**
Pure Ag	**12**	**14**	**16**	**15**	**16**
PVA+(0.003g)Ag	**10**	**13**	**11**	**12**	**13**
PVA+(0.005g)Ag	**18**	**16**	**17**	**16**	**18**

6. CONCLUSIONS

By using the solution cast approach, PVA nanocomposite thin films with varying amounts of AgNPs have been created. characterized using various characterization techniques, including UV-Visible spectroscopy, FTIR, and XRD. Clear evidence of the crystalline nature of the Ag nanoparticles was provided by XRD analyses. After making the composites, the crystal structure of Ag did not change, according to XRD. AgNPs had no detrimental effects on the polymer structure, according to FTIR, because there were no covalent connections established between PVA and AgNPs. With the exception of transmittance and energy gap, the absorbance, absorption coefficient, extinction coefficient, refraction index, and reflectance of Ag doped PVA films increase with increasing doping quantity. Increasing doping content also results in an increase in the dielectric constant (real and imaginary). The energy gap decreases the most plausible reason is the creation of a localized level in the forbidden energy gap as a result of increasing the fillers nanoparticle weight. This study shows that all of these characteristics are affected by the number of AgNPs.

The antimicrobial activity of nanocomposite was measjured. Hence, *positive bacteria (Staphylococcus aureus, Streptococcus pneumonia*), negative bacteria (*Escherichia coli, Pseudomonas aeruginosa*), and *fungus* (*Candida albicans*) were taken as models in this work. The maximum inhibitaion zones for PVA+(0.005g) Ag in (*Candida albicans and Staphylococcus aureus*) were about 18 mm, respectively.

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