Journal of Applied Physical Science International
8(1): 36-46, 2017
ISSN: 2395-5260 (P), ISSN: 2395-5279 (O)

International Knowledge Press
www.ikpress.org

PHYSICAL PROPERTIES AND ANTIBACTERIAL ACTIVITY OF TiO_2 NANOPARTICLES WITH POLYVINYL ALCOHOL/POLYVINYL PYRROLIDONE POLYMER

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AUTHORS' CONTRIBUTIONS

This work was carried out in collaboration between all authors. All authors read and approved the final manuscript.

Received: 8th December 2016
Accepted: 27th January 2017
Published: 25th March 2017

Original Research Article

ABSTRACT

The structural properties which include X-ray diffraction (XRD) and scanning electron microscopy (SEM) of the polymer PVA/PVP and a mixture of polymer PVA/PVP with titanium dioxide with two particle sizes (45.7 and 15.7) nm have been studied. The absorption and transmission spectra of these films have been measured. The casting method was used to prepare films of PVA/PVP and this composite polymer with titanium dioxide with two particle sizes in plastic and glass plates in order to study the activity of these films to reduce bacterial growth. The antibacterial activity against pathogenic bacteria *Staphylococcus aureus* as gram positive bacteria and *Escherichia coli* as gram negative bacteria was tested. The results showed that the best reduction of *S. aureus* growth for TiO_2 (15.7 nm)/PVA/PVP film on plastic plate reached to (62%) and on glass plates reached to (79.7%), while the best reduction of *E. coli* growth for TiO_2 (15.7 nm)/PVA/PVP film on plastic plates reached to (84.7%) and on glass plates reached to (76%).

Keywords: TiO_2 nanoparticle; PVA/PVP polymer; physical properties; antibacterial effect; pathogenic bacteria.

1. INTRODUCTION

Polyvinyl Alcohol (PVA) is a white and granular, powder, tasteless [1]. It has been applied in the industrial, medical, commercial and food sectors and it has been used to produce many end products, such as surgical threads, lacquers, resins and food packaging materials that are often in contact with food [2]. Polyvinyl Pyrrolidone (PVP) is a white, hygroscopic powder with a weak characteristic odor. In contrast to most polymers, it is readily soluble in water and a large number of organic solvents, such as alcohols, amines, acids, chlorinated hydrocarbons, amides and lactase. On the other hand, the polymer is insoluble in the common esters, ethers, hydrocarbons and ketones [3]. Titanium dioxide (TiO_2) is a photo catalyst and widely utilized as a self-cleaning and self-disinfecting material for surface coating in many applications, titanium dioxide has a more helpful role in our environmental purification due to its nontoxicity, photo induced super-hydrophobicity and antifogging effect [4]. These properties have been applied in removing bacteria and harmful organic materials from water and air, as well as in self-cleaning or self-sterilizing surfaces for places such as medical centers [5-8]. This study was aimed to

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evaluate the physical properties, antibacterial activity of PVA/PVP, TiO_2 nanoparticles/PVA/PVP films and used these films to cover the plastic and glass surfaces coating inhibits the growth of bacteria on them.

2. EXPERIMENTAL WORK

2.1 Synthesis TiO_2 Nanoparticles/PVA/PVP Films

Pure PVA/PVP and TiO_2 nanoparticles doped PVA/PVP films have been prepared by casting method [9]. Hot distilled water (~55°C) (10 ml) was used to dissolve (0.3 g) from PVA as a granular powder with molecular weight (M_w=14000 g/mole) obtained from (BHD Chemicals Ltd) and (0.2 g) from PVP as a granular powder with molecular weight (M_w=40000 g/mole) obtained from (Ourchem for Laboratory Use Only). This mixture solution was magnetically stirred continuously for (3 hrs.) until became homogeneous viscous solution. Then it poured into plastic petri dish with diameter (10 cm) and keeps under room temperature (~30°C) for (7 days) to evaporate all solvent slowly and obtained PVA/PVP thin film with thickness about (0.00015 μm).

In order to prepare TiO_2 nanoparticles/PVA/PVP composite films with two particle sizes for TiO_2 nanoparticles; (45.7 and 15.7 nm); the amount of powder for each particle size as used (0.01 g) with (10 ml) distilled water. (6 ml) of this TiO_2 nanoparticles solution was added to PVA/PVP solution and the same method mentioned above used to get 45.7 nm TiO_2/PVA/PVP and 15.7 nm TiO_2/PVA/PVP films with thickness about (0.00675 and 0.00683) μm, respectively.

The transmittance and absorbance spectra for all samples were measured by using UV-Visible spectrophotometer type (T80- series UV-Visible spectrometer). Scanning Electron Microscopy (SEM) type (INSPECTS50) made in Holland and. X-Ray Diffraction (XRD) instrument is from type (SHIMADZU XRD – 6000) made in Japan, with following specifications are Target CuK_α, Wavelength is (1.5406 A˚), Current is (30 mA) and Voltage is (40 KV).

2.2 Antibacterial Effect of PVA/PVP, TiO_2 Nanoparticles/ PVA/PVP Films against Pathogenic Bacteria

Antibacterial effect of PVA/PVP films doped with TiO_2 nanoparticles was determined against pathogenic bacteria *Staphylococcus aureus* and *Escherichia coli* (obtained from Department of Biology/College of Science / Al-Mustansiriyah University/ Baghdad /Iraq). Glass and plastic plates were coated with PVA/PVP and TiO_2 nanoparticles/PVA/PVP, and then dried for 7 days. After drying the bacterial suspension (10^8 cell/ ml) was poured onto the film of glass and plastic plates and allowed to settle on the top of the film, the control plates contained bacterial suspension only without PVA/PVP and TiO_2 nanoparticles/ PVA/PVP films. All coated plates and control were incubated at 37°C for 24 h. After the incubation (1ml) of each culture were serially diluted. Then 0.1 ml of each dilution was taken, spread on nutrient agar (Hi Media) and then incubated at 37°C for 24 h [10]. The colonies were counted and inhibitory effect was evaluated and calculated percent reduction of bacterial growth using the following equation described as [11]:

$$R\ (\%) = \frac{(A-B)}{A} \times 100 \quad (1)$$

R = the reduction rate, A = the number of bacterial colonies from control plates and B= the number of bacterial colonies from plates coated with PVA/PVP or TiO_2 nanoparticles /PVA/PVP films.

3. RESULTS AND DISCUSSION

3.1 X-Ray Diffraction

3.1.1 Pure PVA/PVP polymer

The X-ray diffraction for PVA/PVP film was showed in Fig. 1. These three peaks are observed for PVA/PVP at 2θ = 16.2296º,13.4161º and 43.819º corresponding d-spacing 5.45.7705 Aº, 6.59445.7 Aº, and 2.06436 Aº with intensities (392), (42) and (83) respectively, which confirms the polymer blend formation. These results indicate that the blends become more semi-crystalline with the increase in the concentration of PVA in PVP [12]. The particle size is nearly (14.178 nm). Table 1 illustrated some structural properties for pure PVA/PVP film.

Table 1. XRD parameters for pure PVA/PVP film

2θ (deg)	FWHM (deg)	Intensity (counts)	d (Å)	D (nm)
13.4161	0.69830	92	6.59445	11.460
16.2296	0.67	392	5.45705	11.982
43.8190	0.448707	83	2.06436	19.092

3.1.2 Pure TiO_2 nanoparticles powder

The X-ray diffraction of pure TiO_2 nanoparticles powder with two particles sizes are shown in Figs. 2A an B, respectively. Strong diffraction peaks at 25º, 48º and 37° indicating TiO_2 in the anatase phase, the intensities of XRD peaks of the sample reflects that the formed nanoparticles are crystalline and broad diffraction peaks indicate very small size crystallite [13]. From this study, considering the peak at degrees, average particle size has been estimated by using Debye-Scherer formula in eq. (2) and Specific Surface Area (SSA) can be calculated using eqs. (3 & 4) and the dislocation density (δ) in the sample has been determined using eqs. (5 and 6).

$$D = (0.9\,\lambda)/\,(\beta \mathrm{Cos}\theta) \quad (2)$$

Where, λ is wavelength of X-Ray, β is FWHM (full width at half maximum), θ is diffraction angle, d is d-spacing and D is particle diameter size.

$$SSA = \mathrm{SApart}/(V_{part} * \mathrm{density}) \quad (3)$$

$$S = (6x10^3) / (D.\ \rho) \quad (4)$$

Where SSA & S are the specific surface area, V_{part} is particle volume and SApart is Surface Area, D is the size (spherical shaped), and ρ is the density of TiO_2 (4.23 g/cm^3).

$$\delta = \frac{15\,\beta\,cos\theta}{4\,a\,D} \quad (5)$$

$$\delta = 1/D^2 \quad (6)$$

Where, β- diffraction broadening - measured at half of its maximum intensity (radian), θ- diffraction angle (degree), and α-lattice constant (nm) [14]. The intensity is increase with decrease the Particle size, the particle size is nearly (15.7 nm) for Fig. 2A and (45.7 nm) for Fig. 2B. Table 2A and B illustrated some structural properties for pure TiO_2 nanoparticles, respectively. It can be concluded from Table 2A and B the specific surface area (S) and dislocation density (**δ**) are decreased with increasing the particle sizes of TiO_2 nanoparticles.

3.1.3 TiO_2/PVA/PVP films

The X-ray diffraction pattern for TiO_2 (for two particles sizes (15.7 nm) and (45.7 nm))/PVA/PVP films are shown in Fig. 3A and B), respectively. The effect of TiO_2 nanoparticles on PVA/PVP films become more semi-crystalline with decreasing the particle size of TiO_2 nanoparticles and the intensity is increasing with decrease particle size [15]. The particle size of TiO_2 (15.7, 45.7) nm/PVA/PVP films are nearly (15.094, 11.9613) nm, respectively. The Table 3A and B illustrated the magnitude of (2θ) for three high peaks with their intensities, FWHM, d-spacing and particle size.

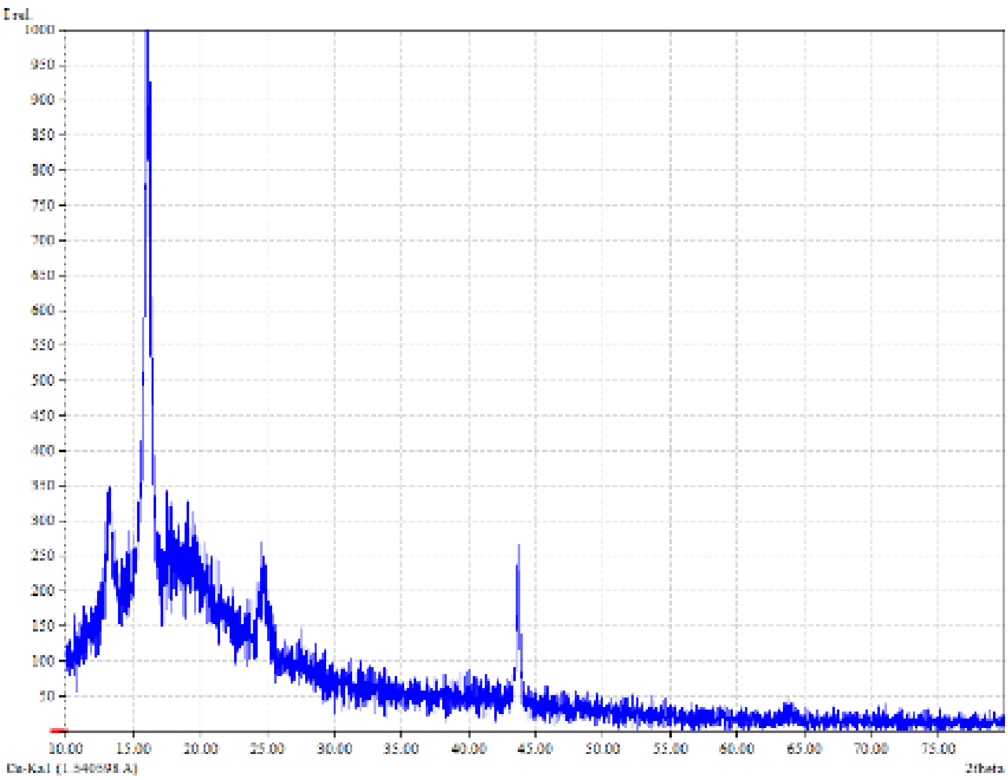


Fig. 1. XRD pattern for pure PVA/PVP film

Table 2A. XRD parameters for pure TiO_2 (15.7 nm) powder

2θ (deg)	FWHM (deg)	Intensity (counts)	d (Å)	hkl	D (nm)	$Sx10^6$ ($m^2.g^{-1}$)	$\delta x10^6$ (m^{-2})
25.3424	0.54100	285	3.51165	011	15.1	0.1018	4.385
36.9362	0.28000	16	2.43168	013	30.4	0.0505	1.082
37.8804	0.67000	50	2.37321	004	12.6	0.1220	6.298
38.7148	0.50000	12	2.32396	112	16.9	0.0910	3.501
48.0716	0.59500	77	1.89120	020	14.7	0.1046	4.627
53.9817	0.75000	39	1.69727	015	11.9	0.1292	7.061
55.0311	0.73000	39	1.66735	121	12.2	0.1260	6.718
62.0836	0.40000	12	1.49380	123	23.4	0.0657	1.826
62.7034	0.76000	28	1.48052	024	12.3	0.1250	6.609
68.8616	0.76000	10	1.36237	116	12.7	0.1211	6.200
70.3212	0.64000	12	1.33763	220	15.2	0.1011	4.328
75.0799	0.84000	16	1.26421	125	11.9	0.1292	7.061

Table 2B. XRD parameters for pure TiO_2 (45.7 nm) powder

2θ (deg)	FWHM (deg)	Intensity (counts)	d (Å)	hkl	D (nm)	$Sx10^6$ ($m^2.g^{-1}$)	$\delta x10^5$ (m^{-2})
25.3712	0.21100	759	3.50773	011	39.5	0.0358	6.409
37.0077	0.19670	46	2.42715	013	43.0	0.0329	5.408
37.8515	0.20100	172	2.37496	004	41.9	0.0338	5.696
38.6286	0.18750	48	2.32895	112	45.9	0.0308	4.746
48.0967	0.19930	253	1.89027	020	44.6	0.0317	5.0272
53.9434	0.20180	159	1.69838	015	44.4	0.0319	5.0726
55.1201	0.215.70	147	1.66487	121	42.2	0.0336	5.6153
62.1691	0.17900	25	1.49196	123	52.2	0.0271	3.6699
62.7443	0.20810	119	1.47965	024	45.1	0.0314	4.9163
68.8009	0.21860	45	1.36343	116	44.2	0.0320	5.1186
70.3422	0.19800	55	1.33728	220	49.9	0.0284	4.0160
75.0910	0.214200	85	1.26405	125	47.3	0.0299	4.4696
76.0766	0.186000	25	1.25011	031	55.0	0.0257	3.3057

Table 3A. XRD parameters for TiO_2 (15.7 nm)/PVA/PVP film

2θ (deg)	FWHM (deg)	Intensity (counts)	d (Å)	D (nm)
13.3526	0.64430	142	6.62567	12.720
16.2048	0.64120	527	5.46534	12.520
43.7891	0.42740	83	2.06570	20.042

Table 3B. XRD parameters for TiO_2 (45.7 nm)/PVA/PVP film

2θ (deg)	FWHM (deg)	Intensity (counts)	D (Å)	D (nm)
14.1576	0.64000	132	6.25070	12.514
15.5633	0.72670	82	3.48180	11.214
16.9928	0.66110	490	5.21363	12.155

3.2 Scanning Electron Microscopy (SEM)

3.2.1 Pure TiO_2Nanoparticles powder

The SEM images of TiO_2 at different magnification are shown in Fig. 4 for TiO_2 (15.7 nm) and Fig. 5 For TiO_2 (45.7 nm) which confirms that the TiO_2 nanoparticles are pseudo spherical in shape. It has been observed TiO_2 nanoparticles were agglomerated to from clusters. The effect of increasing particle size of TiO_2 led to form big agglomerated [16].

3.2.2 TiO_2/PVA/PVP films

Figs. 6 and 7 show SEM micrographs of (6 ml) TiO_2 nanoparticles with two particles sizes (15.7, 45.7) nm doped with PVA/PVP films, respectively. The SEM micrograph for TiO_2 (15.7 nm)/PVA/PVP film show that some areas of the surface is smooth and some TiO_2 nanoparticles white granule scattered randomly and the SEM image of TiO_2 (45.7 nm)/PVA/PVP films shows that rough surface with some gathering of TiO_2 nanoparticles in PVA/PVP [17].

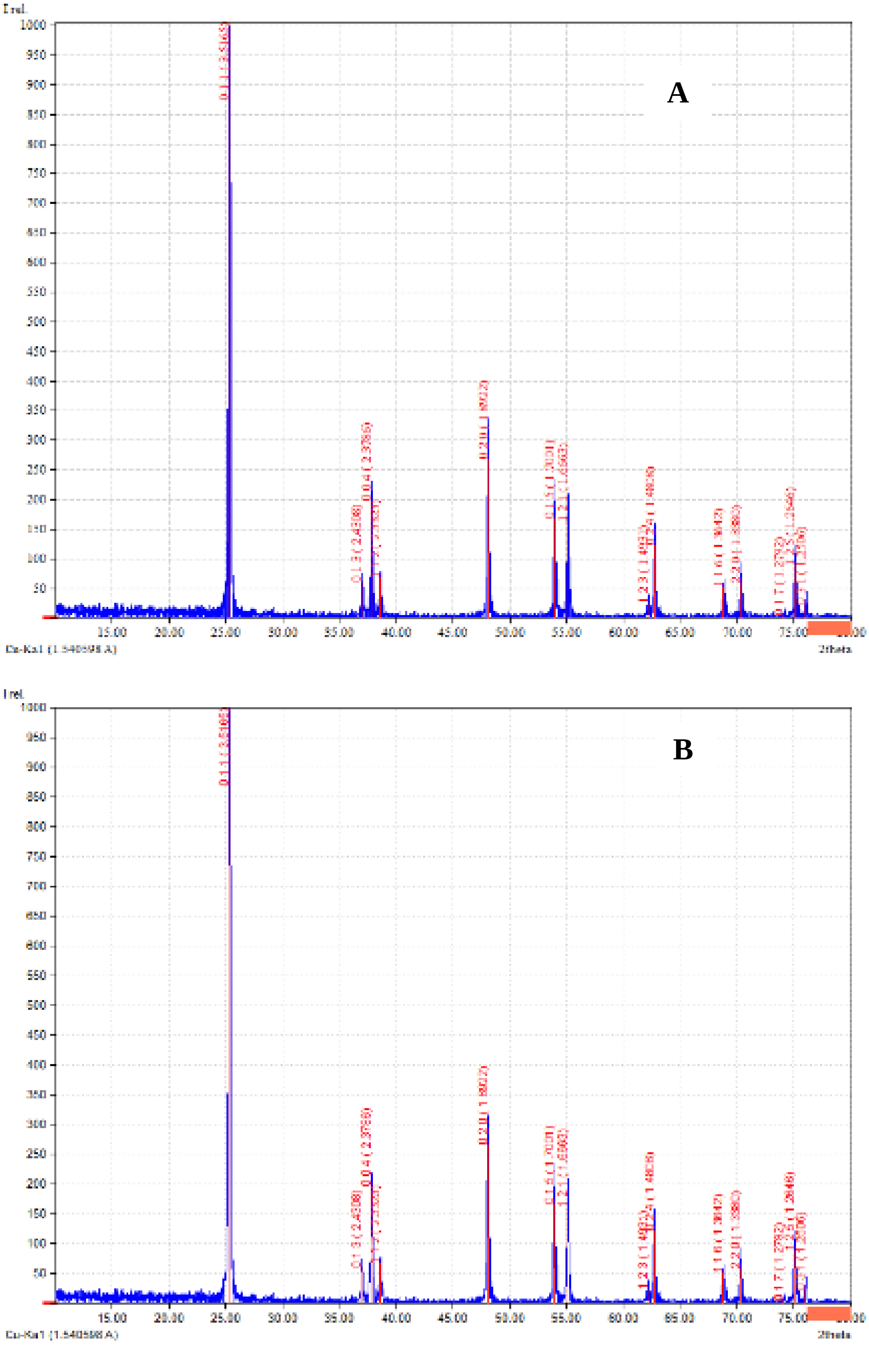
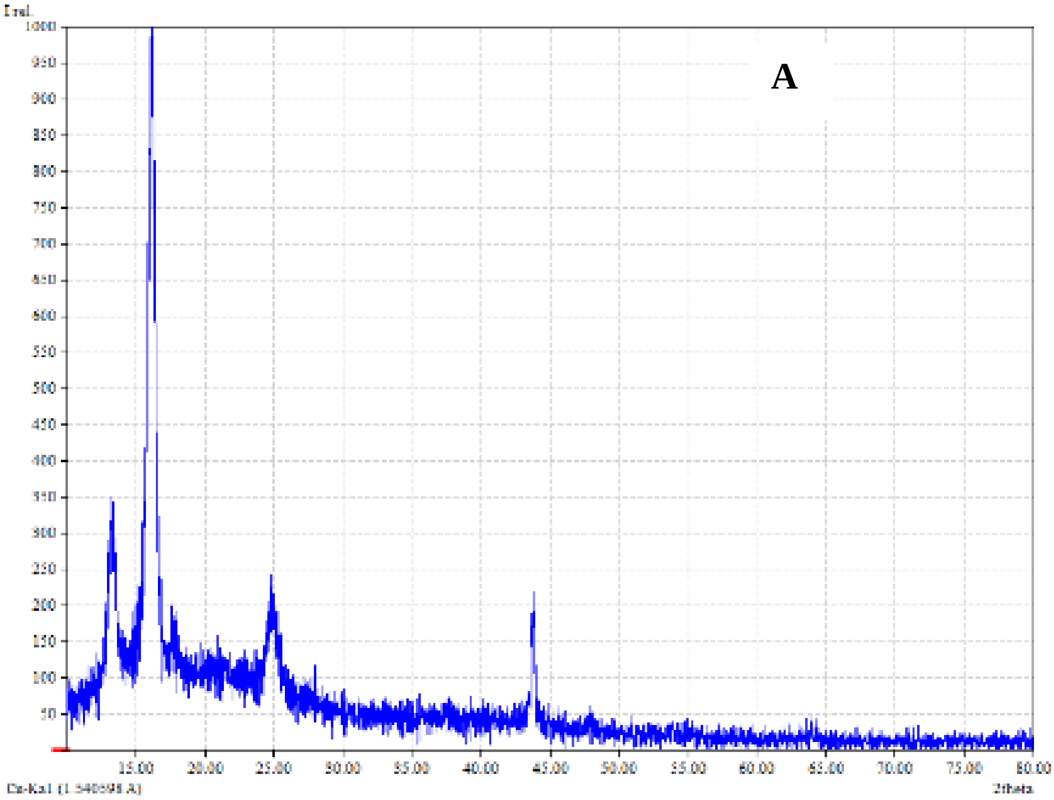


Fig. 2. XRD pattern for pure TiO_2 nanoparticles powder with two particles sizes A-(15.7 nm) B-(45.7 nm)



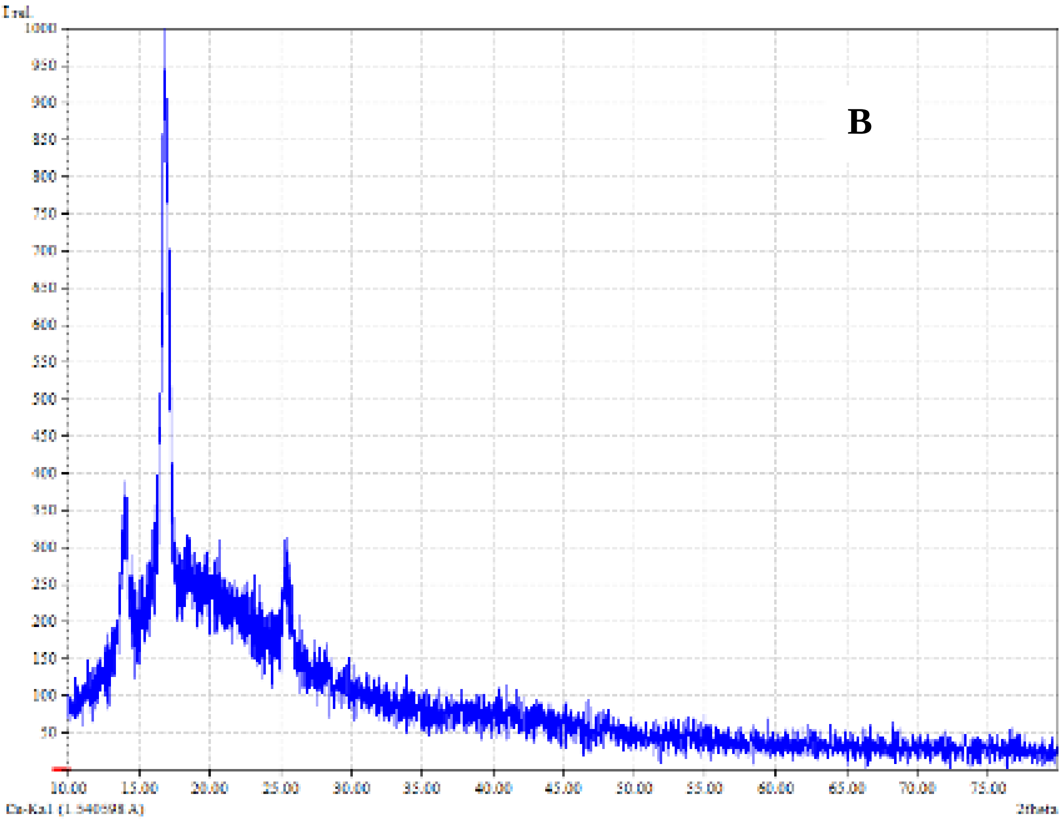


Fig. 3. XRD pattern for A- TiO_2 (15.7 nm)/PVA/PVP and B- TiO_2 (45.7 nm)/PVA/PVP films

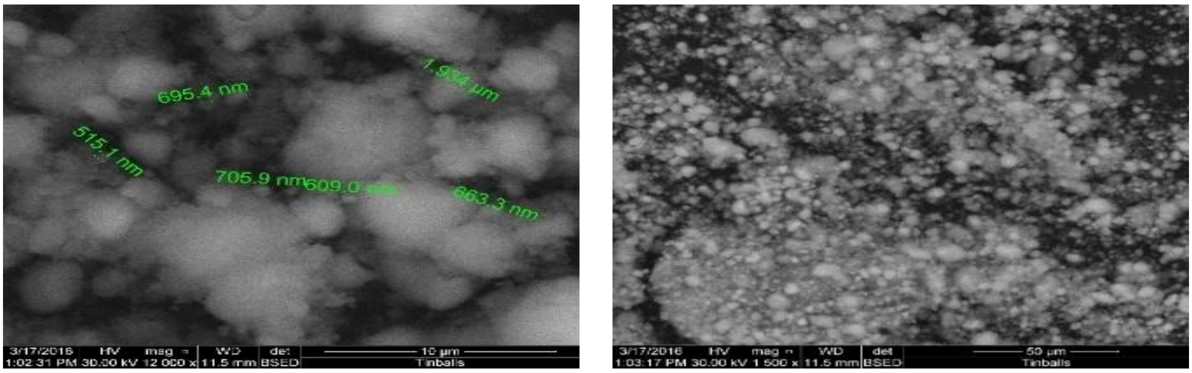


Fig. 4. SEM micrographs for TiO_2 powder with particle size (15.7 nm)

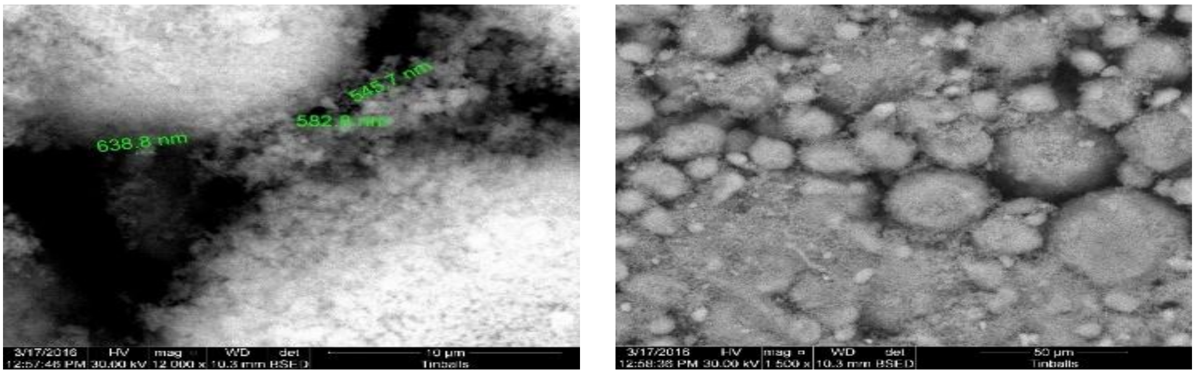


Fig. 5. SEM micrographs for TiO_2 powder with particle size (45.7 nm)

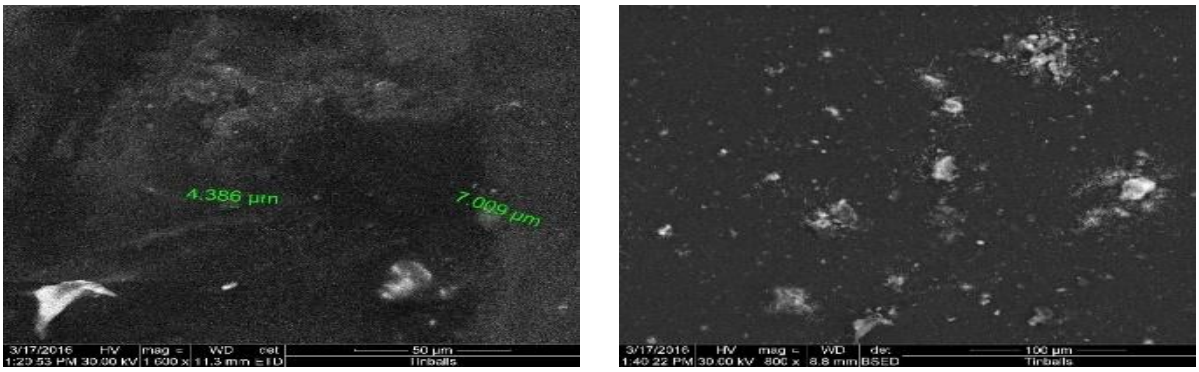


Fig. 6. SEM micrographs for TiO_2 (15.7 nm)/PVA/PVP films

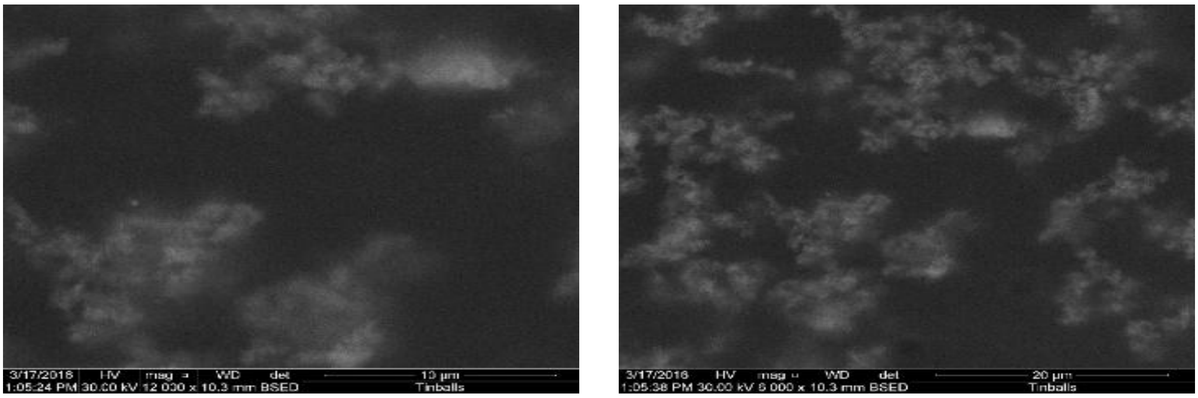


Fig. 7. SEM micrographs for TiO_2 (45.7 nm)/PVA/PVP films

3.3 UV-Visible Spectroscopy

The absorbance spectra for pure PVA/PVP and Nano TiO_2/PVA/PVP with two particle sizes were shown in Fig. 8. There is a peak of absorption spectrum for pure PVA/PVP at (285 nm) with intensity (0.435). The role of TiO_2 nanoparticle clearly appeared when mixing with PVA/PVP polymer. The peak of absorption spectrum is at 360 nm for TiO_2 with intensities (0.164 and 0.195) for (15.7 and 45.7) nm, respectively. The effect of increasing the particle size of TiO_2 nanoparticle led to increase intensity of absorption spectrum. This behavior attributed to titanium oxide nanoparticles absorbs more the incident light [18].

From the value of absorption spectrum (A), the absorption coefficient (α) of thin films prepared was calculated in the fundamental absorption region from the relation [19]:

$$\alpha = 2.303\frac{A}{t} \tag{7}$$

Where (t) is thickness of thin film. The relation between the absorption coefficient and wavelength demonstrated in Fig. 9 for all films. The behavior of these spectra was the same as absorption spectra. The values (α) were less than 10^4 (cm^{-1}), indirect transition suggested. $(\alpha h\upsilon)^{1/2}$ plotted against (hυ) and from line intercept x-axis, the value of E_g founded as in Fig. 10 and Table 3. To calculate optical energy gap from eq. (8) [20]:

$$(\alpha h\upsilon) = B(h\upsilon - E_g)^r \tag{8}$$

Where:

hυ :photon energy,E_g: Optical energy gap,B: constant depends on the type of transition.

r: expontional constant, it is value depended on type of transition, r :2 for the allowed direct transition, r :3 for the forbidden direct transition.

The value of E_g for pure PVA/PVP was (3.99 eV). The effect of adding TiO_2 nanoparticles on E_g of PVA/PVP was to decrease it with increasing particle size. Because the TiO_2 nanoparticles is act an occupies local states between valence band and conduction band. Reflectance(R) and Refractive index (n) of all films were calculated from eqs. (9 and 10) [21], respectively and there tablet in Table 4.

$$R + T + A = 1 \tag{9}$$

$$n = \frac{1+\sqrt{R}}{1-\sqrt{R}} \tag{10}$$

3.4 Antibacterial Effect of TiO_2/PVA/PVP

Reduction of *S. aureus* and *E. coli* growth was tested by using PVA/PVP (Pure) and TiO_2 nanoparticle (45.7 nm)/PVA/PVP, TiO_2 nanoparticle (15.7 nm)/PVA/PVP films on plastic plates. The results illustrated in Table 5 showed that the best reduction of *S. aureus* growth for TiO_2 (15.7 nm)/PVA/PVP film on plastic plates reached to (62%), while the best reduction of *E. coli* growth for TiO_2 (15.7nm)/PVA/PVP film on plastic plates reached to (84.7%). The antibacterial activity is

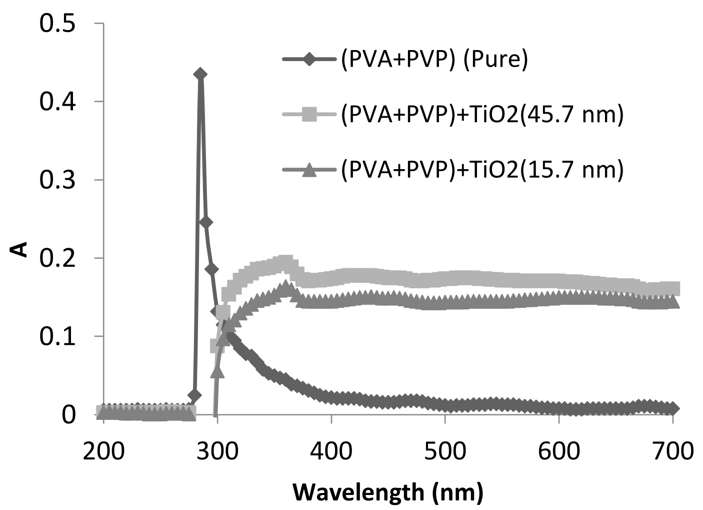


Fig. 8. Absorbance spectra for pure PVA/PVP and TiO_2 nanoparticles/PVA/PVP

increased with decreasing the particle size of TiO2 nanoparticles with increasing the specific surface area (S) and dislocation density (δ). For another study, the use of an antimicrobial coating consisting of chitosan PVA is a viable alternative in shelf-life extension of minimally processed tomato. Chitosan-PVA antimicrobial film may be a promising material as a packaging film [22]. The antibacterial activity of PVA against *E. coli* and the PVA membrane reduces bacteria reproduction [23]. The metal oxides carry the positive charge while the microorganisms carry negative charges; this causes to electromagnetic attraction between microorganisms and the metal oxides which leads to oxidization and finally death of microorganisms [24]. They causes pits in bacterial cell walls, leading to increase permeability and cell death [25]. A strong binding of nanoparticles to the outer membrane of microorganisms causes the inhibition of active transport, dehydrogenase and periplasmic enzyme activity and inhibit the DNA, RNA and protein synthesis, due to which cell lysis occurs [26].

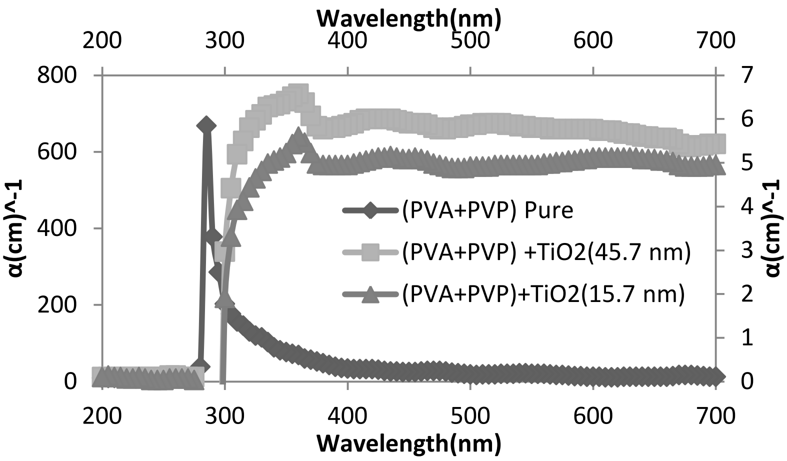


Fig. 9. Absorption coefficient as a function of incident photon energy for PVA/PVP and TiO_2 nanoparticles/PVA/PVP

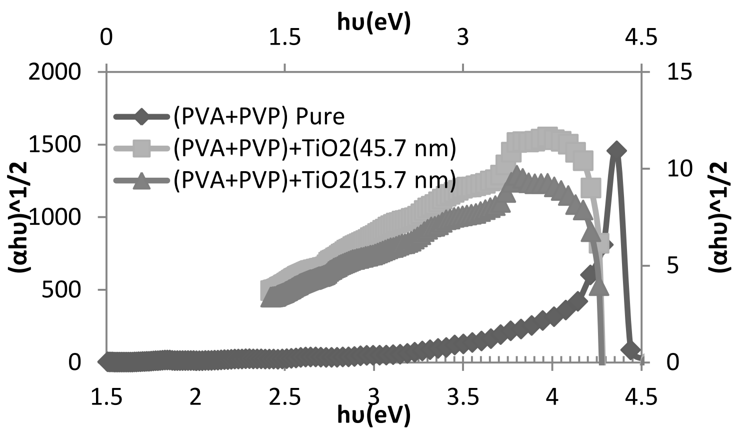


Fig. 10. The allowed indirect transition for PVA/PVP and TiO_2 nanoparticles/PVA/PVP

Table 4. Optical properties of pure PVA/PVP and TiO_2 /PVA/PVP

Substrate	λ (nm)	intensity	α $(Cm)^{-1}$	R	n	E_g (eV)
PVA/PVP(Pure)	285	0.435	677.87	0.201	2.625	3.99
PVA/PVP+TiO_2(15.7 nm)	360	0.164	5.595	0.151	2.27	2.80
PVA/PVP+TiO_2(45.7 nm)	360	0.195	6.575	0.149	2.25	2.30

Table 5. Reduction of bacterial growth for PVA/PVP and TiO_2 nanoparticles/PVA/PVP on plastic plate

Bacterial isolate	Treatment	Reduction of growth (%)
Staphylococcus aureus	(PVA+PVP) (Pure)	53.7
	(PVA+PVP)+TiO_2(45.7 nm)	34.1
	(PVA+PVP)+TiO_2(15.7 nm)	62
Escherichia coli	(PVA+PVP)(Pure)	54
	(PVA+PVP)+TiO_2(45.7nm)	64
	(PVA+PVP)+TiO_2(15.7 nm)	84.7

4. CONCLUSIONS

From all results it can be concluded that TiO_2 nanoparticles/PVA/PVP films had antibacterial activity against pathogenic bacteria on plastic and glass plates. Also the physical properties of these films studied and showed the semicrystalline behavior and the effect of increasing particle size of TiO_2 nanoparticle on decreasing energy gap. The TiO_2 NPs addition enhances the antibacterial activity and the enhancement is mainly against gram negative bacteria to (84.7%). The increase for gram positive is limited (10%) and also a decrease for TiO_2 NPs of 47.5 nm is clearly visible.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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