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EFFECTS OF ADDITIVE AND GAMMA IRRADIATION ON THE STRUCTURAL AND OPTICAL PROPERTIES OF POLYVINYL ALCOHOL DOPED WITH SILVER NITRATE

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KeyWords

Composite material, XRF, XRD, Gamma irradiation, inorganic additives, polymers cross-linking, polyvinyl alcohol (PVA), extinction coefficient, dielectric constant, UV-Vis spectroscopy, degree of crystallinity (DOC).

ABSTRACT

Structural and optical properties of polyvinyl alcohol (PVA) doped with silver nitrate (AgNO₃) composite Films synthesized by casting method has been investigated by XRD and UV-Vis spectroscopy. Samples were irradiated with gamma rays in the dose range 0-10 Gy and the effects of γ -irradiation and the addition of AgNO₃ to PVA matrix were studied. The results indicated that the irradiation of the PVA/AgNO₃ composite films with γ -ray doses induced obvious color changes from white (non-irradiated film) to light yellow, golden then to brown and dark brown and the color intensity increased with increasing γ -ray doses. The films showed absorption band peaking at the wavelengths around 230 and 450 nm and the intensity of peaks were found to be in direct proportion with radiation dose; the surface Plasmon band was noted around 430–466 nm. Hence, the appearance of this very weak band may be related to the formation of small quantity of reduced silver nanoparticles. A clear linear correlation between the applied dose and the specific absorbance and the absorption coefficient of the prepared films was noted and the energy gap (Eg) was found to be smaller than that of the bulk silver. The extinction coefficient (K), refractive index (n) and optical conductivity of the prepared samples were measured. XRD results revealed that irradiation and the addition of AgNO₃ led to a more compact structure compared to the pure PVA which resulted in an improvement in its structural and optical properties. Thus, this work suggests the possibility of using the gamma irradiated PVA/AgNO₃ films in different electronic and semiconductors applications.

1 Introduction

Polymeric materials are of great interest in scientific and technological research. Solid polymer blends have been intensively studied due to its potential applications for novel systems and devices [1-3]. By addition of dopants, blends, or copolymers to the main polymer matrix, the optical, electrical, thermal, mechanical and electrochemical properties of these materials could be selectively

modified obtaining particular characteristics for various applications in different fields of science [3, 4]. This field of polymer additives has attracted strong interest in today's materials research as it is possible to achieve impressive enhancements of material properties as compared with the pure polymers [5, 6]. Furthermore, irradiation of polymers has established itself as one of the most acceptable, easy and non-toxic approach to alter polymer properties significantly [1-3]. Irradiation of polymers destroys the initial structure by introducing defects inside the material which leads to changes in the properties of these polymers. Ionizing radiation effects on polymers include cross-linking of the molecular chains, degradation of macromolecules (chains scission) and changes in the number and nature of the double bonds [2, 3]. This entire process can occur either in one or all together, depending on the chemical nature of the polymer, radiation dose applied and other external factors. Polyvinyl alcohol (PVA) is the most studied polymer throughout the last decades in scientific research and continues to attract considerable attention due to its fascinating and desirable characteristics specifically for various pharmaceutical and biomedical applications and also due to its relatively simple chemical structure with a pendant hydroxyl group [4, 7]. PVA is highly hydrophilic, non-toxic and biocompatible polymer with excellent film formation by casting [8-11]. PVA has been of particular interest also due to its abundance, high absorption coefficients in the visible light, chemical stability, easy polymerization, and low cost of monomer [12-15].

Inorganic additives, such as transition metal salts, have considerable effects on the optical, thermal and electrical properties of PVA [5, 8]. PVA doped with inorganic metal such as silver (Ag) [16], gold (Au) [17], Copper (Cu) [18], TiO₂ [19] and CeO₂ to form PVA/metal composite films and hydrogels have been used to improve physical, optical, mechanical, and electrical properties of these composite materials. Since silver has a very good electrical conductivity among all the metals; the combination of PVA/Ag with uniform dispersion can yield functional composite material with enhanced electrical properties. The obtained composites have characteristic advantages compared to their single component counterparts and can have potential applications in new devices [20-22]. In particular, transition metal particles dispersed/encapsulated within a polymer matrix can offer attractive and practicable routes for combining properties arising from metal particles and from that of polymers [9, 10]. This could greatly improve the optical and dielectric properties of the PVA composites. These particles could act as conductive junctions between the PVA chains that resulted in an increase of the electrical conductivity of the composites [2, 11]. Several methods have been employed to prepare polymer/metal composites such as blending of both components and irradiation [1-3]. All these metallic particles (Gold, Silver, Platinum...etc) exhibit unusual electrical, magnetic, optical and electrochemical properties due the near-free conduction electrons which are dependent on their size, surface Plasmon, surface free energy and surface area, as well as on the surrounding dielectrics [23]. Among these important metal particles used, silver (Ag) particles are of great interest in today's materials world due to its special electronic, optical, and magnetic properties [15, 24] and its wide variety of applications in catalysis [18], conducting inks, microelectronics, thick-film electrode material, and solar cells [1, 3]. These mentioned properties are dependent on the particle size, shapes and the method of composites synthesis. Varieties of synthesis method have been developed, including reduction from metallic salts, ultrasonic irradiation technique, ion implantation, and thermal process and microwave technique [5, 13 and 25]. However, fabrication of PVA/Ag composite films without using of toxic chemical agents still remains highly challenging. This challenge may be surpassed by using irradiation technique which has a capability of producing polymer and metal particle components without using any chemical agents. The synthesis of silver composites by using y- irradiation has been mentioned by Ali et al., (2007) and M.M. Abutalib [26]. The combination of PVA as polymer with silver as a noble metal may produce hybrid composed material that possesses very interesting behaviors [25].

Various modifications in PVA have been observed due to additive and/or irradiation [9-13]. Nouh S A and R A Bahareth [27], studied the effect of electron beam irradiation on the structural, thermal and optical properties of polyvinyl alcohol thin film, they concluded that the electron beam irradiation in the dose range 95–210 kGy enhances the scope of the polymer in high temperatures. In the present study, $AgNO_3$ is used as additive stabilizer to PVA. The aim is to investigate the feasibility of modifying its structural and optical properties to study the possibility of further enhancement of these properties using gamma irradiation.

2 Experimental

2.1 Samples

The PVA stock was supplied by TECHNO PHARMCHEM (Mol. wt = 85000 to 124000 g/mol Degree of hydrolysis 86-89 %). The PVA/AgNO₃ films were made using solvent casting technique with different concentrations of AgNO₃/PVA that is, $[C_1 = 5, C_2 = 10, C_3 = 15$ and $C_4 = 20]$ wt% AgNO₃ by dissolving each concentration of PVA powder in 100 ml distilled water at room temperature on a beaker. The solutions were magnetically stirred at room temperature for 3 hours then poured in a Petri-dish to form films by casting method in a dark room. Films were left to dry at ambient temperature at least 2 days, the ambient condition was measured using data logger [Temperature = 24.1 0 C, Humidity = 28.5%, atmospheric pressure = 961 mBar]. Films were peeled off the Petri-dish, cut into small films 2x2 cm (suitable for measurements), loaded in sealed dark dental film envelopes. The thickness of the films was measured using a micrometer and the average thickness was found to be 110 µm.

2.2 Irradiation facility

TERABALT Cobalt-60 unit was used for samples irradiation. Different radiation doses which were specify with treatment planning system (TPS) PlanW2000 (0 Gy, 5 Gy and 10 Gy) at Surface Source Distance (SSD) 95cm, field Size (FS) 10x10 cm², Gantry angle 0⁰, Collimator angle 0⁰. The films were placed at the center of the field size. ⁶⁰Co γ -rays irradiation facility (TERABALT - radiotherapy cobalt unit type 80–model SCS, UJP PRAHA, CZECH REPUBLIC) from National Cancer Institute, University of Gazira, Wad-Madani, Sudan, was used in this study.

2.3 Analysis of the samples

X-ray fluorescence analysis (XRF), UV-Visible spectroscopy and X-ray diffraction analysis (XRD) were performed on γ-irradiated and non-irradiated PVA/AgNO₃ films using X-ray Fluorescence Model CM/HM/ XRF-06 with serial number C13206, UV-visible spectrophotometer Model V-650 UV/VIS/NIR and GNR analytical instruments group XRD apparatus model EXPLORER.

3 Results and Discussion

3.1 XRF Analysis

XRF was used to validate the intensity of AgL α characteristic X-ray line dependence on the AgNO₃ concentration in the PVA/AgNO₃ composite films. **Fig. 1**, graphically represent the dependence of the intensity in part per thousand (PPT) of AgL α characteristic X-ray line on AgNO₃ concentration for non-irradiated and gamma-irradiated PVA/AgNO₃ films, strong linear correlation is observed. Moreover, **Fig. 2**, reveals the strong dependence of intensity of AgL α characteristic X-ray line on Ag concentration for 10 Gy γ -irradiated PVA/AgNO₃ films with correlation coefficient R² = 0.99881 and the correlation equation y =1.392x+951.2 as an example.

3.2 UV-Vis Analysis

UV absorption is mainly due to electron (or anion) transitions from the top of the valence band to the bottom of the conduction band. The absorption spectra produced two absorption bands in the visible region. These visible bands correspond to the excitation of outer electrons, which provide information on the electronic transitions of the molecules in the samples, they are attributed to the π - π^* transitions and to the presence of ions in the polymer. In this study, the electronic absorption spectra of the investigated samples have been recorded in the region from 200 to 1100 nm for non-irradiated and y-irradiated PVA samples. UV-Vis spectra of PVA\AgNO₃ recorded revealed an absorption band peaking at the wavelengths around 230 and 450 nm which are related to criteria of the pure PVA and PVA/AgNO₃ respectively. According to the literature, the band at 450 nm could be attributed to chelate formation of Ag+ coordinated with the hydroxyl group of PVA or to silver particle formation [28]. The absorption bands increased following the radiation dose increment, such phenomena has been reported by (Mohammed et al., 2014) [29] and (Ramnani et al., 2007) [30]. In one hand, it is well known that pure PVA films are transparent and contains only single bonds; therefore it would be expected to absorb radiation only in the far UV region (120-200) nm. On the other hand, the physical properties of PVA are dependent on its preparation technique whether from the fully hydrolysis, or partial hydrolysis, of poly vinyl acetate. Thus the absorption band at 230 nm may be assigned to π - π * transition. This transition is related to the carbonyl groups (C=O) associated with ethylene unsaturation (C=C) of the type –(CH=CH)2CO-. The existence of carbonyl functionalities is probably due to residual acetate groups remaining after the manufacture of PVA from hydrolysis of polyvinyl acetate or oxidation during manufacturing and processing [8].



Fig 1: Dependence of the intensity in part per thousand (PPT) of AgLα characteristic X-ray line on AgNO₃ concentration for nonirradiated and Gamma- irradiated PVA/AgNO₃ films.



Fig 2: Correlation between the intensity in part per thousand (PPT) of AgL α characteristic X-ray line and AgNO₃ concentration for PVA/AgNO₃ films irradiated with 10 Gy as an example.

Figs. 3, **4**, **5** and **6** show UV-Visible absorption spectra of gamma-irradiated PVA/AgNO₃ samples with (5, 10, 15 and 20) wt % Ag respectively. We observe that with increasing AgNO₃ content; a strong increase in the absorbance in the ultraviolet and visible regions; i.e., in the spectral range (200-600) nm, the absorption coefficient of the samples also increased with increasing gamma-irradiation dose. The dramatic enhancement in absorption is found to be in agreement with the color change of PVA samples. Furthermore, the peaks get narrower and sharper and also increase in intensity with increasing irradiation dose. **Tables 1 & 2** show the specific absorbance and the absorption coefficient respectively, measured from the absorption spectrum of all PVA/AgNO₃ films. We can deduce that both specific absorbance and absorption coefficient linearly increase with increasing radiation dose and AgNO₃ concentration. **Fig. 7**, shows the correlation between specific absorbance and radiation dose for gamma-irradiated PVA/AgNO₃ sample with 10 wt % Ag as an example, it shows strong linear correlation with R² = 0.99378 and the correlation equation y = 0.031x+3.435. **Fig. 8**, shows the correlation between absorption coefficient and radiation dose for gamma-irradiated PVA/AgNO₃ sample with 10 wt % Ag as an example, it shows strong linear correlation with R² = 0.9923 and the correlation equation y = 0.00065x+0.072.



Fig 3: UV-Visible absorption spectra of the non-irradiated and gamma-irradiated PVA/AgNO₃ samples with 5.0 wt% Ag.



Fig 4: UV-Visible absorption spectra of the non-irradiated and gamma-irradiated PVA/AgNO₃ samples with 10 wt% Ag.



Fig 5: UV-Visible absorption spectra of the non-irradiated and gamma-irradiated PVA/AgNO₃ samples with 15 wt% Ag.



Fig 6: UV-Visible absorption spectra of the non-irradiated and gamma-irradiated PVA/AgNO₃ samples with 20 wt% Ag.

Table 1: The specific absorbance values for PVA/AgNO₃ films with different concentrations of AgNO₃ irradiated with γ -radiation doses of (0, 5 and 10) Gy.

AgNO ₃	Specific Absorbance	Specific Absorbance	Specific Absorbance
Concentration	for PVA/AgNO₃ films non- irradiated (0 Gy).	for PVA/AgNO₃ films irradiated with 5 Gy.	for PVA/AgNO₃ films irradiated with 10 Gy.
5.0 wt %	2.88	3.5	3.25
10 wt %	3.43	3.6	3.74
15 wt %	3.3	3.44	3.73
20 wt %	3.12	3.45	3.26

Table 2: The absorption coefficient values of the PVA/Ag films with different concentration of AgNO3 irradiated with radiation dosesof (0, 5 and 10) Gy.

AgNO ₃	Absorption Coefficients	Absorption Coefficients	Absorption Coefficients
Concentration	for PVA/AgNO₃ films non- irradiated (0 Gy).	for PVA/AgNO₃ films irradiated with 5 Gy.	for PVA/AgNO₃ films irradiated with 10 Gy.
5.0 wt %	0.060	0.0733	0.068
10 wt %	0.0718	0.0754	0.0783
15 wt %	0.069	0.072	0.078
20 wt %	0.0653	0.0722	0.0683



Fig 7: Correlation between specific absorbance and radiation dose of γ-irradiated films with 10 wt % Ag as an example.





Table 3 lists the optical band gaps of PVA/Ag nanocomposites prepared with different $AgNO_3$ concentrations and gamma-irradiated with various doses. From this table it can be observed that the values of the band gap in case of PVA/Ag nanocomposite are lower than that of bulk Ag; this is may be ascribed to the strong quantum confinement in the nanocomposites; it may also be attributed to the creation of electronic disorder which becomes prominent with increasing gamma-ray doses; in other terms, due to the photo-degradation of dyed polymer and the formation of defects and clusters in the material [31]. **Fig. 9**, shows the relation between $(\alpha hv)^2$ and photon energy (hv) for PVA/Ag of 10 wt% Ag irradiated with 10 Gy as an example on energy gap (E_g) estimation.

Table 3: The optical band gaps of PVA/Ag nanocomposites prepared with different concentrations and gamma-irradiation doses.

Energy gap (eV)			
Dose (Gy)	Non-irradiated films (0 Gy).	Films irradiated with 5 Gy.	Films irradiated with 10 Gy.
5.0 wt %	3.72	3.36	3.4
10 wt %	3.658	3.17	3.26
15 wt %	3.57	3.16	3.37
20 wt %	3.52	3.19	3.56



Fig 9: Relation between $(\alpha hv)^2$ and photon energy (hv) for PVA/Ag of 10 wt% Ag irradiated with 10 Gy as an example on (E_g) estimation process.

The extinction coefficient (K) of the prepared PVA/Ag composite films linearly increases with increasing AgNO₃ concentration; same thing happens with the increment of radiation dose. Moreover, the peaks get narrower and shift continuously to longer wavelengths. **Fig. 10**, shows the extinction coefficient for PVA/Ag films of 10 wt% Ag irradiated with 10 Gy as an example. Refractive index of PVA/Ag composite films linearly decreases with increasing AgNO₃ concentration but tends to increase again at higher concentrations. It is also increases with the increment of radiation dose but again tends to decrease at higher doses. Moreover, the peaks get narrower and shift continuously to longer wavelengths. The change in the refractive index values of the irradiated samples can be attributed to varying electronic structure and crystallinity induced by gamma-irradiation depending upon whether degradation or cross-linking process is predominant. Also the increase in the refractive index of the prepared films with irradiation may be due to ionization or atomic displacements that resulted from gamma ray collision with the samples which may change the internal structure in the polymer films [2, 28 and 31]. **Fig. 11**, shows the refractive index for PVA/Ag films of 10 wt% Ag irradiated with 10 Gy as an example. Optical conductivity of PVA/Ag composite films linearly increases with increasing AgNO₃ concentration, similar thing happens with the increment of radiation dose. Moreover, the peaks get narrower and shift continuously to longer wavelengths. In both cases, the value of optical conductivity which is highly linked to the dielectric constant degrades at higher dopants concentration and higher doses. **Fig. 12**, shows the optical conductivity for PVA/Ag films of 10 wt% Ag irradiated with 10 Gy as an example.



Fig 10: The Extinction Coefficient of PVA/Ag composite films doped with 10 wt % Ag concentration γ-irradiated at 10 Gy.



Fig 11: The Refractive Index of PVA/Ag composite films doped with 10 wt % Ag concentration γ-irradiated at 10 Gy.



Fig 12: The Optical Conductivity of PVA/Ag composite films doped with 10 wt % Ag concentration y-irradiated at 10 Gy.

XRD is the most common bulk analysis technique used to identify the crystalline phase present and crystal particle sizes. XRD analysis has been routinely used for crystalline phase identification based on diffraction peak position and pattern and the measurement of the average crystalline sizes and lattice parameters. It can also supply information about deformation of a crystalline sample in other terms the strain of a crystal lattice which will result in a change in the inter-atomic distances [32, 33]. In this study, X-ray diffraction technique has been used to investigate and characterize the structure of the prepared samples. It revealed that PVA/AgNO₃ samples have in large an amorphous feature. It is important to note that there are two halos cited at 19.66° and 40.5° characteristic of PVA. The first one has a clear crystalline peak at a diffraction angle $2\Theta = 20.04^{\circ}$ which corresponds to a (101) spacing, this main peak centered at 19.66° corresponds to the PVA crystalline phase with the accepted monoclinic unit cell with a = 7.81, b = 2.52, c = 5.51 Å and $\beta = 91.42^{\circ}$; one unit cell comprises two monomer units of vinyl alcohol (CH2CHOH) [32]. The second halo has a low intensity and broad shape which corresponds to noncrystalline zones within the crystalline polymeric matrix. Moreover, there are several very low intense peaks in these figures which are assigned to the Ag nanoparticles; their low intensities are due to the very low concentration of Ag NPs compared with amount of PVA as shown in **Fig 13**.

It is obvious from the XRD pattern that the diffraction peak at $2\Theta = 19.66^{\circ}$ revealed a clear crystalline peak of maximum intensity implying the more organized distribution of the AgNO₃ in the polymeric matrix. It was observed that the addition of AgNO₃ has no effect on the crystal type of the film; which means that it has been uniformly distributed inside the host polymer. Also, it is clear that no peaks were observed corresponding to the AgNO₃ or other impurities indicating the purity extent of the reduced Ag NPs. On the other hand, no significant effect of gamma irradiation noticed on either the intensity of the diffraction peak or on its full width at half maximum (FWHM); but with the increase in the applied dose, peaks intensity tends to slightly decrease, broaden and shift to lower angles reflecting the fact that the polymer matrix suffers from some kind of structural rearrangement due to irradiation treatments [34]. The average crystallite size (D) of the non-irradiated and gamma irradiated films is calculated from the Scherer's formula and listed in **Table 4**. From the table, we observe that particle size slightly increased with increasing AgNO₃ concentration and the combined effects of γ -irradiation dose resulted in increasing particle size as well. These results are in good agreement with those obtained from UV-visible absorption measurements. The semicrystalline nature of PVA is attributed to the strong intermolecular interaction among PVA chains through hydrogen bonding. The addition of AgNO₃ to PVA matrix leads to a decrease in the intermolecular interaction between chains and thus to a decrease in the degree of crystallinity (DOC) as shown in **Table 5**; this in turns means more amorphous crystal and increasing in intera-atomic distances (d) as shown in **Table 6**.



Fig 13: Diffraction patterns of PVA/Ag composite films doped with different AgNO₃ concentrations γ-irradiated at 10 Gy as an example on XRD measurements.

Table 4: Particle size in PVA matrix after γ-irradiation for PVA/AgNO₃ samples with different concentrations.

AgNO ₃	Particle Size of PVA/AgNO ₃	Particle Size of PVA/AgNO ₃	Particle Size of PVA/AgNO ₃
Concentration	films non-irradiated (0 Gy).	films irradiated with 5 Gy.	films irradiated with 10 Gy.
10 wt %	12.117	11.041	13.174
15 wt %	12.65	11.239	13.480
20 wt %	12.977	11.776	13.947

Table 5: Degree of crystallinity (DOC) in PVA matrix after γ-irradiation for PVA/AgNO₃ samples with different concentrations.

AgNO ₃ Concentration	DOC for PVA/AgNO₃ films non- irradiated (0 Gy).	DOC for PVA/AgNO₃ films irradiated with 5 Gy.	DOC for PVA/AgNO ₃ films irradiated with 10 Gy.
10 wt %	21.75 %	15.03 %	13.43 %
15 wt %	15.30 %	12.10 %	11.77 %
20 wt %	13.70 %	10.69 %	10.35 %

Table 6: Distances in PVA matrix after γ -irradiation for PVA/AgNO₃ samples with different concentrations.

AgNO ₃ Concentration	For PVA/AgNO₃ films non- irradiated (0 Gy).	For PVA/AgNO₃ films irradiated with 5 Gy.	For PVA/AgNO₃ films irradiated with 10 Gy.
10 wt %	4.5132	4.563	4.507
15 wt %	4.521	4.565	4.512
20 wt %	4.605	4.569	4.542

Conclusion

Functional composite material of PVA\AgNO₃ films with enhanced optical and thermal properties was successfully synthesized using controlled temperature and pressure by casting method alongside y-ray irradiation. The irradiation of the films with y-ray doses induced obvious color changes from white (non-irradiated film) to light yellow, golden then to brown and dark brown and the color intensity increased with increasing y-ray doses. X-ray Fluorescence (XRF) analysis validated that the intensity of AgLa characteristic X-ray line depends on the Ag concentration in the prepared films with a correlation value near to 1. The irradiated films showed absorption band peaking at the wavelengths around 230 and 450 nm and the intensity of peaks is in direct proportion with radiation dose. There exist a clear linear correlation between the applied dose and the specific absorbance and the absorption coefficient of the prepared films. There exist a clear linear correlation between AgNO₃ concentration and the specific absorbance and the absorption coefficient of the prepared films. The surface Plasmon band was noted around 430–466 nm. Hence, the appearance of this very weak band may be related to the formation of small quantity of reduced silver nanoparticles. Values of the band gap (E_e) in case of PVA/Ag nanocomposites are found to be lower than that of bulk Ag; this is may be ascribed to the strong quantum confinement in the nanocomposites; it may also be attributed to the creation of electronic disorder which becomes prominent with increasing gamma-ray doses. Values of the extinction coefficient (K) linearly increase with increasing AgNO₃ concentrations and radiation dose. The overall effect of the radiation dose is increasing the value of the refractive index (n). Values of the optical conductivity linearly increase with increasing AgNO₃ concentration and radiation dose. Values of the dielectric constant linearly increase with increasing AgNO₃ concentration and radiation dose and was found to be very big. These high values of dielectric constant are very crucial in electric applications and for capacitance applications of small sizes as it gives possibilities of new devices with minimized dimensions. XRD patterns validated UV-Vis observations regarding the fact that the polymer matrix suffers from some kind of structural rearrangement due to gamma-irradiation treatments. XRD diffraction peak at 20 = 19.66⁰ revealed a clear crystalline peak of maximum intensity implying the more organized distribution of the AgNO₃ in the polymeric matrix. These results

indicate that the non-irradiated copolymer films or those irradiated with low dose up to 10 Gy may be suitable for applications as a semiconductor polymeric material in the electrical and electronic industries.

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