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Gamma Radiation-Induced Crosslinked PVA/Chitosan Blends for Wound Dressing

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Crosslinked polyvinyl alcohol (PVA) and chitosan polymer blends have been prepared by using gamma irradiation. Chitosan was used in the blends to prevent microbiological growth, such as bacteria and fungi on the polymer. The physical properties of the blend, such as gelation, water absorption, and mechanical properties were examined to evaluate the possibility of its application for wound dressing. A mixture of PVA/chitosan, with different ratios, were exposed to gamma irradiation doses of 20, 30, 50 KGy, to evaluate the effect of irradiation dose on the physical properties of the blend. It was found that the gel fraction increases with increasing irradiation dose and PVA concentration in the blend. Swelling percent increased as the composition of chitosan increased in the blend. The PVA/chitosan blend has a water content in the range between 40% and 60% and water absorption between 60% and 100%. The water vapor transmission rate value (WVRT) of the PVA/chitosan blend varies between 50% and 70%. The examination of the microbe penetration shows that the prepared blend can be considered as a good barrier against microbes. Thus, the PVA/chitosan blend showed satisfactory properties for use as a wound dressing.

Keywords: PVA/chitosan; blend; absorption; water vapor transmission; wound dressing

1 Introduction

Irradiation is recognized as a very suitable tool for the formation of hydrogels. Radiation processes have various advantages, such as easy process control, possibility of joining hydrogel formation and sterilization in one step and finally, it is not necessary to add any chemical initiators (1). Hydrogels, that form a three–dimensional network, consist of a hydrophilic polymer which contains a large quantity of water. They are one of the most promising materials for biomedical applications, having several advantages for wound dressing, contact lenses, drug delivery systems, etc. These wide applications of hydrogels are due to their biocompatibility with blood, body fluids, and tissue (2–4).

An ideal substrate used as a wound dressing must have basic features such as absorption of the exudates, prevention of excessive loss of body fluids, good adhesion of the wound, non-toxicity and prevention of infection (5).

PVA has wide commercial applications due to its unique chemical and physical properties. It is a nontoxic, highly crystalline, water-soluble polymer, having good film-forming and high hydrophilic properties. However, PVA as a soluble polymer cannot be used in wide applications. Thus, it has to be converted to completely insoluble materials with high mechanical properties (6).

Chitosan, the partially deacetylated form of chitin, is a material known in the wound management field for its hemostatic properties. In addition to its hemostatic properties, chitosan possesses many other biological properties including bacteriostatic and fungistatic properties that are particularly useful for wound treatment. Chitosan has, therefore, been employed in various physical forms for wound treatment, for example, as a solution, gel, film, membrane, sponge powder or fiber. Moreover, hydrogels consisting of chitosan and PVA/poly-N-vinylpyrrolidone (PVP) were prepared for using as wound dressings (7).

Poly(N-vinyl-pyrolidone)-k carrageenan hydrogels (PVP/KC) were prepared by irradiation. The mixture of aqueous solutions of PVP, KC by gamma rays at different doses. Some preliminary experiments were evaluated to identify their usability in the wound dressing application (8). Also, novel chitosan-polysaccarides composite membrane was prepared for wound dressing and was evaluated *in vitro* (9).

In this work, attempts were made to prepare a blend for a wound dressing that consists of PVA and chitosan. Blends from a mixture of PVA/chitosan were made using a cobalt

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60 gamma-ray irradiation technique. The physical properties such as gellation, swelling, mechanical properties and water uptake were examined to evaluate the usefulness of the blend for wound–dressing.

2 Experimental

2.1 Materials

Laboratory grade PVA used in this study, in the form of a powder, having an average molecular weight of 125,000, was obtained from the Laboratory Rasayan. Chitosan was supplied by Pronova Biopolymer, Inc. (USA). Its degree of deacetylation and molecular weight were determined as 85% and 50,000, respectively. Both polymers were used without further purification. Double-distilled water was used as solvent.

2.2 Preparation of PVA/Chitosan Polymer Blend

Films of a PVA/chitosan blend with a different composition were prepared by the casting solution technique. PVA powder was dissolved in distilled water at 95°C, while chitosan was dissolved in a 5% acetic acid solution at room temperature. The polymer solutions were then mixed with continuous stirring, to complete miscibility and subsequently cast onto glass dishes for transparent films. The cast films were dried in a vacuum oven at 50°C for 24 h. Then, they were exposed to Co-60 gamma irradiation doses to evaluate the effect of irradiation on the physical properties of the blend.

2.3 Determination of Gel Fraction (GF %)

A known weight (W_1) of the irradiated samples were extracted by water in a Soxhlet apparatus for 24 h. then dried to a constant weight in vacuum (W_2). The soluble fraction was determined according to the following equation:

$$SF = W_1 - W_2/W_1 \times 100$$

Thus, GF (%) was calculated as follows:

$$GF\ (\%) = (1 - SF) \times 100$$

2.4 Determination of Swelling Percent (SW %) and Equilibrium Water Content Percent (EWC %)

A known weight of the samples (W_1) are soaked in water and left for 24 h at room temperature. The swollen part of the samples was weighed (W_2). The swelling percent is determined by:

$$SW(\%) = W_2 - W_1/W_1 \times 100$$

EWC % was calculated by immersing the irradiated sample in water with the proportional mass of samples to the mass of water about 1:500 at room temperature. Swelling is continued to reach a constant weight. Before weighing the sample, any surface water was removed with filter paper.

The EWC % was calculated as follows:

$$EWC\ \% = Ws - Wd/Ws \times 100$$

where Ws and Wd are the weights of swollen state and dried state, respectively.

2.5 Determination of Water Absorption Percent

The irradiated samples were immersed in water at room temperature for different time intervals. The water absorption percent was calculated as follows:

$$Aw\ (\%) = W_2 - W_1/W_1 \times 100$$

where Aw is water absorption, W_1 is the weight of initial blend sample (before being immersed in water) and W_2 is the weight after being immersed in water.

2.6 Measurement of Water Vapor Transmission

The water vapor transmission rate (WVRT) was measured according to a monograph of the European Pharmacopiae. It consists of measuring the weight loss of a bottle which contains 25 ml of water. The bottle has a mouth with a diameter of 35 mm. The sample with a diameter of 40 mm was then put at the bottle mouth as a cap, and placed in an oven at 35°C for 24 h. The water vapor transmission rate (WVRT) was calculated by using the following formula:

$$WVRT = W_i - W_t/A \times 24 \times 106\ g\,m^2/h$$

where WVRT is expressed in, A is the area of bottle mouth (mm^2), W_i and W_t are the weight of bottle before and after placed in oven, respectively.

2.7 Determination of Mechanical Properties

The mechanical properties were measured in accordance with ASTM 638 specifications. The unirradiated and irradiated polymer blend films were tested for tensile strength and elongation at break. Dumbell shape specimens were cut from sheets using a steel die of standard width (4 mm) and length (40 mm). An Instron Universal Tester (Model 1195) was used throughout this work.

2.8 Microbe Penetration Test

A sample with a thickness of 2–3 mm was cut in a Laminar air flow cabinet (under aseptic conditions) into a small pieces ($2 \times 2\ cm^2$). The pieces were put on the surface of a sterile solidified TSA (Tryptose soy Agar) plates and/or PDA (Potato Dextrose Agar) plates. Next, the plates were

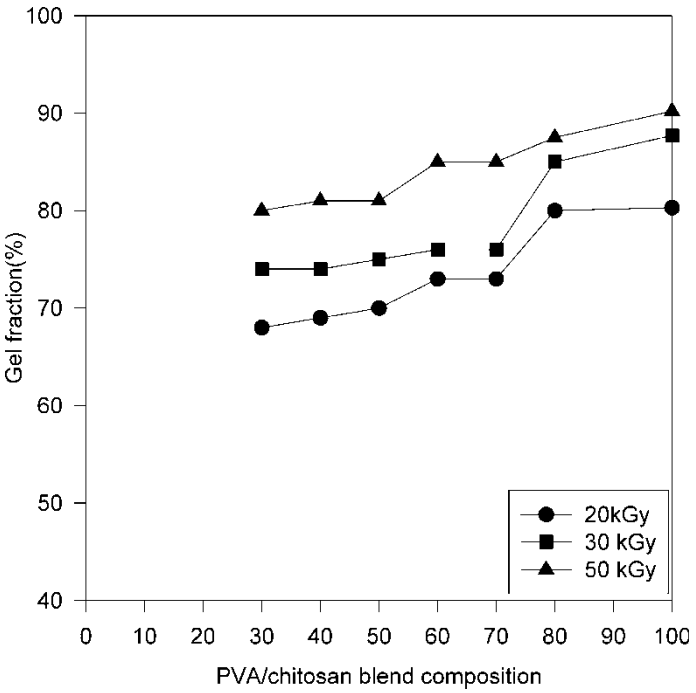

Fig. 1. Effect of various compositions of PVA/Chitosan polymer blends on the gel fraction (%) at different irradiation doses.

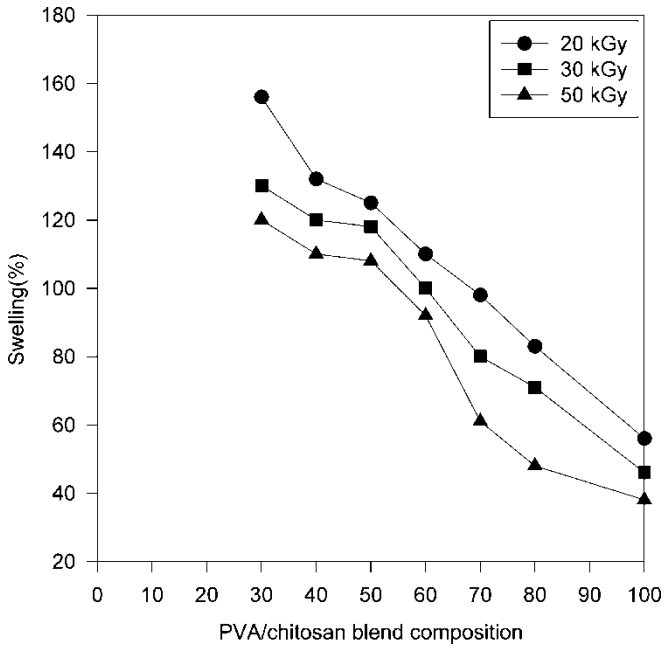

Fig. 2. Effect of various compositions of PVA/Chitosan polymer blends on the swelling (%) at different irradiation doses.

incubated at 30°C for 24 h. On the upper surface of each piece, 0.1 ml of pathogenic bacteria (Bacillus Cereus) or Pathogenic yeast (Canadida Albicans) with a concentration of 107 cells/ml was dropped and flated by sprayer, then the plates were incubated at 30°C for 14 days. The observation of microbial passing through the blend was recorded day by day for 14 days.

3 Results and Discussion

3.1 Gel Fraction and Swelling Percent

The dependence of GF % and SW % on the blend compositions, at different irradiation doses, was studied. The results obtained are shown in Figures 1 and 2. It can be seen from Figure 1 that GF % increases with increasing irradiation dose for all blend compositions. This may be explained by the occurrence of a crosslinking process due to irradiation of PVA/Chitosan polymer blends. This crosslinking process results from the coupling of the polymer radicals that were directly and indirectly produced from PVA or chitosan by γ-rays (10).

On the other hand, it was found that increasing PVA content in the blend is accompanied by an increasing in gel fraction for each irradiation dose under investigation. It is well known that this increase is due to the crosslinking nature of PVA (11, 12).

On the other hand, the SW % increased as chitosan concentration in the blend increased as observed in Figure 2. For example, at 70% PVA, the SW % is equal to 58%, 78%, 98% at 20, 30, 50 kGy, respectively, whereas at 70% chitosan, the SW % is equal to 160, 135%, 115% at the same irradiation dose. At higher doses, the decrease in SW % can be attributed to an increase in the extent of crosslinking. The increase in swelling at high chitosan is due to the fact that chitosan is considered as a degradable polymer (13).

3.2 Equilibrium Water Content Percent (EWC %)

EWC % of the blend with its different compositions was measured at two irradiation doses, namely 20 kGy and 50 kGy. The results obtained are given in Table 1. From this Table, it was observed that the EWC % of PVA/chitosan blends tends to increase with increasing PVA content in the blend at all irradiation doses under investigation. The PVA/chitosan blend shows the EWC % to be in the range between 36 and 56%.

3.3 Water Absorption Percent

Water absorption % was measured as a function of blend compositions at different irradiation dose at a fixed immersion time

Table 1. Effect of blend compositions on the EWC % at different irradiation dose

Blend composition PVA/Chitosan	Equilibrium water content (EWC %)	
	20 kGy	50 kGy
30/70	36	36
50/50	44	40
70/30	49	50
80/20	56	53

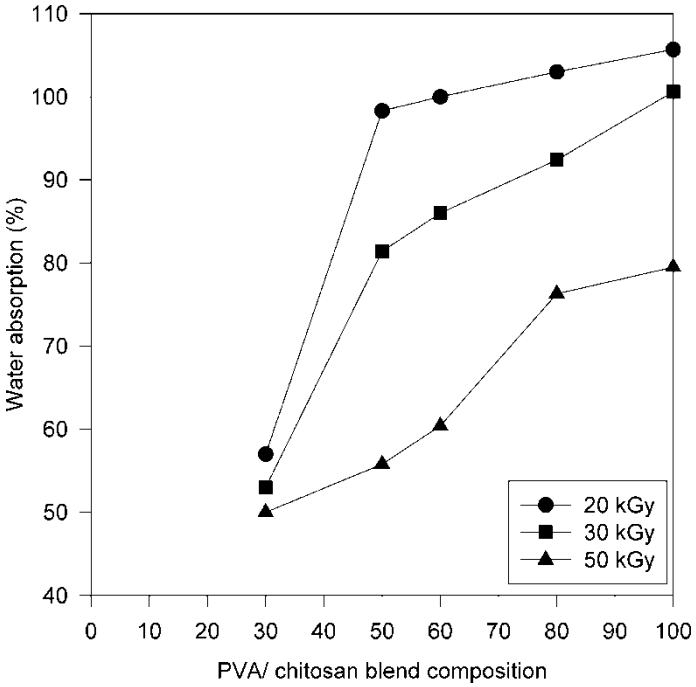

Fig. 3. Effect of various compositions of PVA/Chitosan polymer blends on the water absorption (%) at different irradiation doses.

equal to 24 h. The results obtained are shown in Figure 3. From these results, it was found that water absorption % decreases on increasing the irradiation dose. This decrease may be attributed to an increase in the degree of crosslinking between the polymer chains during irradiation (14). On the other hand, on increasing PVA content in the blend, there is an increase in water absorption. At an irradiation dose equal to 20 kGy, water absorption is equal to 100%, and 58% at 70% PVA and 70% chitosan, respectively. This is due to the fact that PVA is a water soluble polymer, while chitosan is not soluble in pure water, but it is soluble in acidified water.

3.4 Water Vapor Rate Transmission (WVRT)

According to Peppas et al., 1987 (15), the essential problem on using hydrogel in wound dressing is to control the loss of body fluid which occurred due to evaporation and exudation. Thus, the hydrogel prepared as wound dressing must decelerate the loss of body liquid from the wound and in the same time, maintain a suitable humidity in the wound area.

The water vapor transmission rate (WVRT) of non-irradiated and irradiated blends with different compositions was evaluated and the results obtained are shown in Table 2. From this Table, it was found that the values of WVRT are lying between 40–75 g m^2/h for irradiated samples.

From comparison of the obtained experimental values of WVRT with the commercial one, it was found that, according to Bruin et al. 1990, (16) an occlusive wound covering which has a value of WVRT $\leq$ 33 gm^2/h has a weakness point for its use as wound dressing, while the value of WVRT equal 53 gm^2/h is suitable. Hence, the values given in Table 2 corresponding to 30% chitosan or higher seem to fulfill the application of the prepared hydrogel for wound dressing.

Table 2. Effect of radiation dose on the water vapor transmission (WVRT)

Blend composition PVA/Chitosan	WVRT (g m^2/h)	
	Unirradiated	5 Mrad
100:0	25.4	40
80:20	29.14	50.9
70:30	38.8	55
60:40	49.68	60
50:50	23.6	73.35
40:60	23.6	73.11
30:70	20	57.5

The higher value of WVRT causes a faster drying of the wound, which causes a loss of the body liquid due to evaporation and exudation, which will decrease the body temperature and accelerate the rate of metabolism.

3.5 Mechanical Properties

The tensile strength and elongation at break of PVA/chitosan blend were measured as a function of blend compositions at different irradiation dose. The results are shown in Figures 4 and 5, respectively. It was found that the tensile strength increases with increasing both PVA concentration in the

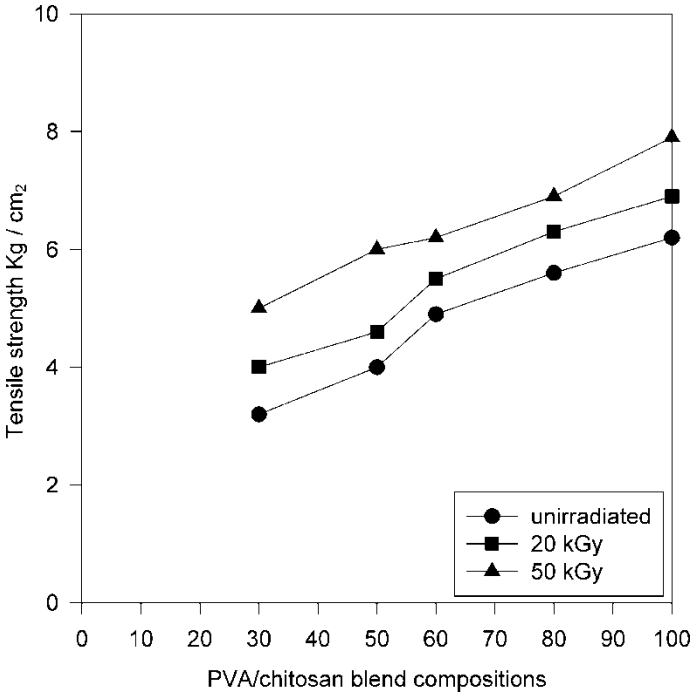

Fig. 4. Effect of various compositions of PVA/Chitosan polymer blends on tensile strength at different irradiation doses.

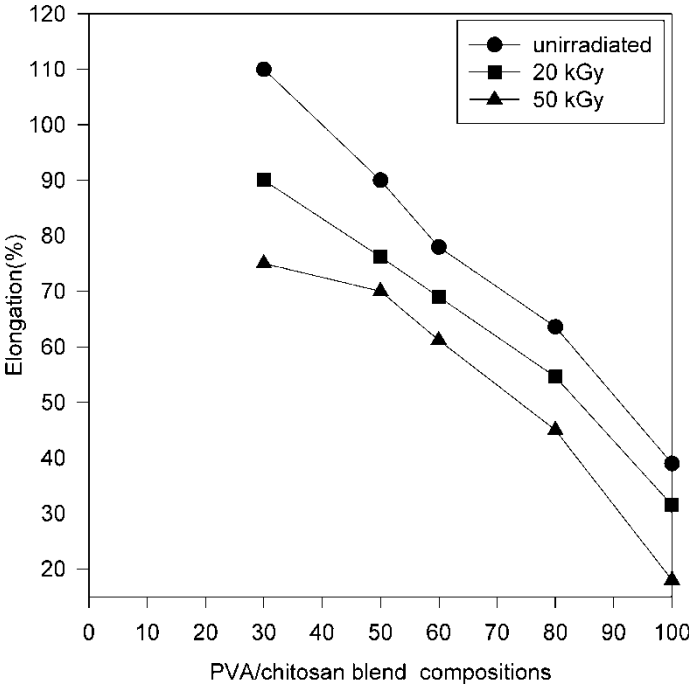

Fig. 5. Effect of different blend compositions on elongation (%) at different irradiation doses.

blend and irradiation dose. The increase of the tensile strength is affiliated with the radiation induced crosslinking of PVA. On the other hand, and as expected, the elongation at break values have encountered a systematic decrease with increasing the PVA content in the blend, as well as with increasing the irradiation dose for the same blend composition. Hence, the mechanical values attained in the present investigation seem to fullfil the requirements for wound dressings as reported by NHO and Park (16).

3.6 Microbe Penetration Test

Based on the microbe peneteration test, there were no bacterial and/or yeast passing through the blend during day-by-day observation for 14 days. Since no bacteria and yeast were found on the TSA and PDA medium, respectively, the PVA/chitosan blend could be considered a good barrier against microbes. This characteristic is very important for hydrogel wound dressing, especially in protecting the wound from further infection so that it may accelerate its healing.

4 Conclusions

In this work, a blend for wound dressing was prepared, which consisted of PVA and chitosan. The blend was crosslinked by a gamma ray irradiation technique. PVA-chitosan composition and irradiation dose had a greater influence on swelling than gel content. The prepared blend showed some properties which can meet the requirements of suitable wound dressing. For example, the blend gives a wet environment to a wound which causes faster healing. Mechanical properties of the blend are satisfactory as a dressing material. This blend could be considered as good barrier against microbe.

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