



STUDY OF WOUND DRESSING STRUCTURE AND HYDRATION/DEHYDRATION PROPERTIES

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ABSTRACT

Hydrogels manufactured by radio-induced crosslinking and simultaneous sterilisation of hydrogels of PVP, PEG and agar, according to the Rosiak method, have many desirable properties for using as wound dressings. However, some properties need to be improved or better controlled. The membranes need to be strong enough to be freely used. Another important property to be controlled is the capacity of absorption of exudate and the kinetics of drying. Therefore, it was necessary to understand the role of main parameters (agar, PVP, PEG concentration and dose) in the structure of the net and in the hydration and dehydration properties. The structure of the membranes was studied by sol analysis and the hydrating/dehydrating properties were studied by isothermal thermogravimetric analysis. The gel content for all samples were always in agreement with expected values considering that only PVP undergoes crosslinking. The hydrating and dehydration results did not show variation with the tested parameters. It was concluded that the network was solely composed of crosslinked PVP plasticized by the other compounds. The properties of hydration/dehydration is related rather to diffusion than to capillarity or osmose and to the chemical retention of water in the polymeric matrix.

KEYWORDS

Hydrogel, radiation, sterilisation, poly(vinylpyrrolidone), poly(ethylene glycol), agar, hydration, structure

INTRODUCTION

The wound dressings manufactured by the simultaneous radio-induced crosslinking and sterilisation of hydrophilic polymers were invented by Rosiak et al (Rosiak et al, 1989). They show usually good biocompatibility and are widely applied, not only as wound dressings, but also as drug delivery systems and many others. The wound dressing manufacture process technology was successfully transferred to Brazil as part of a IAEA program. As a consequence the dressings produced in Brazil have approximately the same characteristics of the original one sold as HDR™ or Aqua-Gel™ in the Polish market. However, the climate in Brazil is of course completely different from Europe. The climate is usually warm, being wet or dry depending upon the place. So, the necessity of replacement of dressings can change from place to place. The comprehension of the dehydrating properties is also important when those hydrogels are used as drug delivery systems as the concentration of chemicals is function of the water content of the hydrogel. Moreover, the medical culture and their conditions of work are also different. Therefore, it is important to understand the chemical structure of the membrane in order to improve its handability and general mechanical properties.

EXPERIMENTAL

The wound dressings tested were hydrogels composed of poly(vinylpyrrolidone), poly(ethylene glycol), agar and water. In the final form they were transparent sheets of few millimetres thickness, containing over 90% of water. The first step of manufacture consisted in the preparation of aqueous solution of dressing components. After dissolving and mixing them at elevated temperature a homogeneous solution was formed. Then the moulds, which can also be used as final packages, were filled with liquid solution of the dressing. After cooling, the solid physical gel was packed in the proper final boxes. Finally, the package of solid gel was subjected to ionising radiation to become crosslinked. Usually the dose applied was 25 kGy for assurance of the sterility. The irradiation was performed by a electron-beam "Dynamitron type" from Radiation Dynamics. The maximum energy was 1.5 MeV and current 15mA.

Sol fraction and Mechanical tests.

The gel content was determined by boiling in water 5 pieces of each sample individually wrapped in a polyester sock. A vacuum oven was used to dry the samples and the testimony. The mechanical properties (elongation and stress) were performed in a Instron, model 5567, according ABNT NBR 6241/80 similar to ASTM standards.

Hydrating/dehydrating behaviour.

Swelling experiments were used to estimate the hydrating behaviour of the membranes. Degree of hydration was determined by the percentage difference in weight after and before hydration. The dehydrating behaviour was followed by measuring the difference of mass when the sample was isothermally (310K) dried in a Shimadzu TGA-50 thermobalance, within a synthetic air atmosphere. The samples had 3mm diameter and weighted approximately 22 to 26mg. Further experimental details about the dehydration procedure can be found in Machado *et al* (1996).

RESULTS AND DISCUSSION

Figure 1 compares the sol percentage measured as the concentration of PEG was increased from 0 to 10% with the estimated values. The expected values were determined based on the hypotheses that only PVP underwent through crosslinking, and agar and PEG did not take part in the structural network by crosslinking or grafting. The results were surprisingly coincident, i.e., the measured values matched almost exactly the estimated ones. This very simple test showed that the network was composed by pure PVP molecules almost completely crosslinked and the PEG is acting mainly as plasticizer. It is know that PEG under radiation has a tendency to crosslink and to degrade at the same time. However, PEG in such low concentration at low dose is probably only degraded. Therefore, the non crosslinked components are acting as radical sink and this trapping effect can be used to fit the relation dose/properties.

In our previous work the mechanical properties of membranes were already analysed (Lugao *et al*). The results showed that the mechanical properties of the membranes produced in Brazil by the so-called Rosiak method are at the same level of the commercial products manufactured by the same process. It is also shown that the decrease in the tensile strength and increase in elongation was typical for plasticizers. This result is also in agreement with the sol analysis, showing that PEG and agar were not grafted or crosslinked to the network.

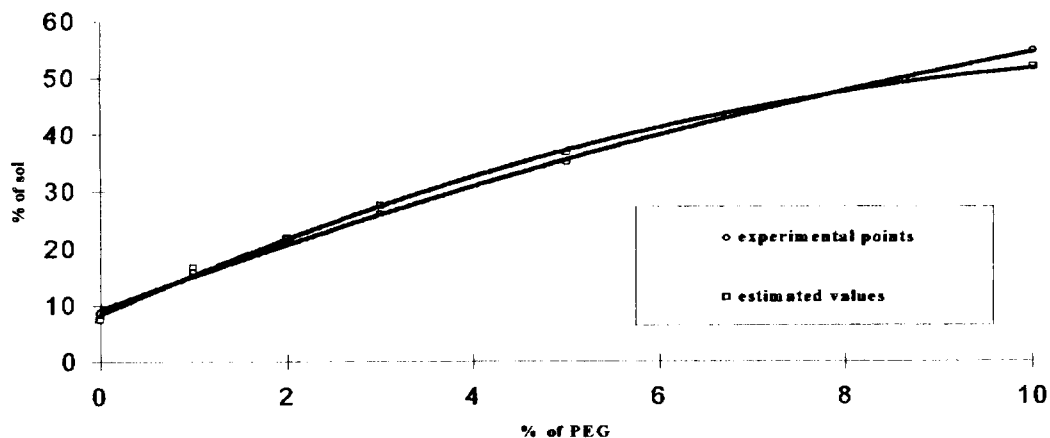


Figure 1. Percentage of sol as a function of PEG concentration

Nevertheless, the increase in PEG means a decrease in water content, so the decrease in tensile strength and increase in elongation could also be related to the decrease in the degree of crosslinking as a result of the smaller amount of OH radicals and increase amount of PEG acting as radical trap.

Dehydration Properties

The dehydration experiments were conducted at 310K in order to simulate the use of the membrane on wounded skin. The relative weight loss observed in all thermogravimetric curves from figure 2a and 2b shows that the dehydration behaviour does not depend on the composition of the sample. The residual mass is directly dependent on the concentration of solid material in the sample. There is no observable difference in dehydrating behaviour of the membranes for all tested concentrations and for all tested dose. The same experiments were repeated with membranes prepared by gamma irradiation to study also the dose rate effect and again there is no difference between all series. Therefore, one possible conclusion is that the effect of chemical retention of the water in the polymeric structure is neglectable for such high concentration.

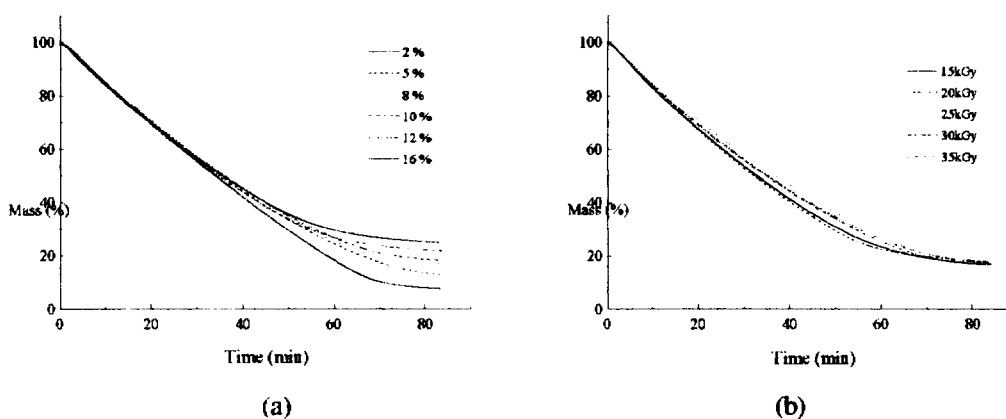


Figure 2. Dehydration for (a) various PVP concentrations at the same dose and (b) various total doses for the same composition

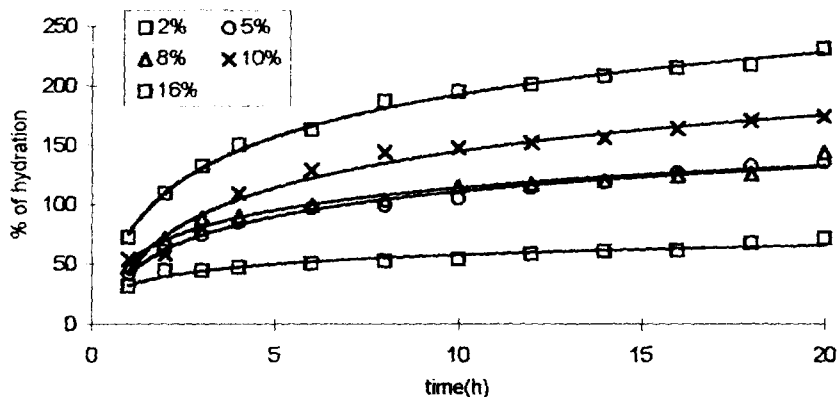


Figure 3. Percentage of hydration as a function of time for various PVP concentration.

It is interesting to observe that they all practically reach the equilibrium of hydration in a couple of hours. There are three steps:

1. the rate of hydration is very high, the water is finding very little resistance to penetrate in the net; and the rate is approximately the same for all PVP concentrations, showing that there is no effect of the concentration of hydrophilic groups on the water uptake;
2. there is a continuous slow down, showing an increase in the resistance of the net to swell;
3. it was reached almost the equilibrium as the net is completely stretched.

These results are in agreement with the dehydration experiments showing that the main phenomena governing the water release and uptake is the diffusion.

FINAL REMARKS

The sol analysis and mechanical properties showed clearly that PEG and agar did not participate in the net work and the dehydration/hydration properties showed that the water is loosely bound to the PVP network or PEG plasticizer. Therefore, the handability of the membranes is connected mainly with the amount of water and plasticizer present in the PVP structure.

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REFERENCES

- Rosiak J.M., Rucinska-Reybas A., Pekala W. (1989) U.S. Patent N.º 4,81,490, Method of Manufacturing of Hydrogel Dressings.
- Rosiak J.M., Ulanski, Pajensky L.A., Yoshi F, Makuuchi K. (1995) Radiation formation of hydrogels for Biomedical purpose. Some remarks and comments. *Radiat. Phys. Chem.*, 46(2); 161-168.
- Machado, L.D.B., Miranda, L.F., Penteadó L.C., Lugao, A.B., Andrade e Silva, L.G., Forti, M.L., Huzler B.W. (1997) Estudo da influência da composição e da dose de radiação na desidratação de hidrogéis à base de polivinilpirrolidona. IV ENAN, Poços de Caldas, Brasil. (accepted).
- Lugão A.B., Miranda L.F., Miranda A, Nakahira H.U., Andrade e Silva L.G. (1996) Desenvolvimento de Hidrogéis. Estudo do Comportamento em função do poli(óxido de etileno). III Encontro Nacional de Biomateriais, Sao Paulo, Maio de 1996.