International Journal of Pharmaceutics, 46 (1988) 179–180
Elsevier

IJP 01601

Use of energy dispersive X-ray analysis to determine solute concentration profiles

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(Received 10 March 1988)
(Accepted 15 April 1988)

Key words: Energy dispersive X-ray analysis; Solute concentration profile; Poly(2-hydroxyethyl methacrylate) (PHEMA)

The bombardment of a material by electrons causes characteristic X-rays to be emitted as the energized electrons return to their original energy states after excitation (Murr, 1982). The energy of these emitted X-rays is characteristic of the atomic species in the sample. The frequency of the emitted X-rays increases with increasing atomic number of the atomic species. Thus, the electron beam can be focused on a particular spot of a sample; the emitted X-rays can be collected and their frequencies and intensities measured. By comparing the relative intensities of emitted X-rays the relative concentrations of each atomic species can be determined. This technique is generally ineffective in detecting nitrogen, carbon or oxygen. It is, however, capable of measuring levels of heavier elements such as chlorine. Here we show how the method can be used to determine drug concentration profiles in releasing polymeric samples.

Cylinders of poly(2-hydroxyethyl methacrylate) (PHEMA) were prepared by bulk polymerization of HEMA in the presence of 0.05 wt % benzoyl peroxide as an initiator and 0.15 wt % ethylene glycol dimethacrylate as a cross-linking agent. The polymerization was conducted in Teflon cylinders, 0.625 cm in diameter and 10 cm long for 72 h. The temperature was gradually increased during the reaction from 40 to 80 °C.

Some samples were loaded with a 3 wt % solution of sodium chloride and others with iodine, using as a swelling medium for loading a 50/50 mixture of water and ethanol. These samples were then air-dried for 24 h and placed in a vacuum oven at 50 °C for 72 h. Subsequently, the dry glassy samples were placed in deionized-distilled water for 72 h and water diffusion was allowed to take place. At periodic intervals the samples were removed and 0.25-cm-thick slabs were cut from the centers of each cylinder. They were then placed in a vacuum oven at 50 °C for 48 h and allowed to dry. Subsequently they were spot-analyzed with a scanning electron microscope (JEOL JSM 35CF scanning electron microscope) in a spot mode with an EDAX 911 analyzer using 25 kV excitation voltage to determine chlorine- and iodine-containing concentration profiles.

Using the scanning electron microscope we were able to examine the iodine concentration distribution in the gel studied here. Some results for two different cylinders are presented in Fig. 1. Here, the iodine diffuses very slowly out of cylindrical samples of swollen PHEMA. Thus, the solute con-

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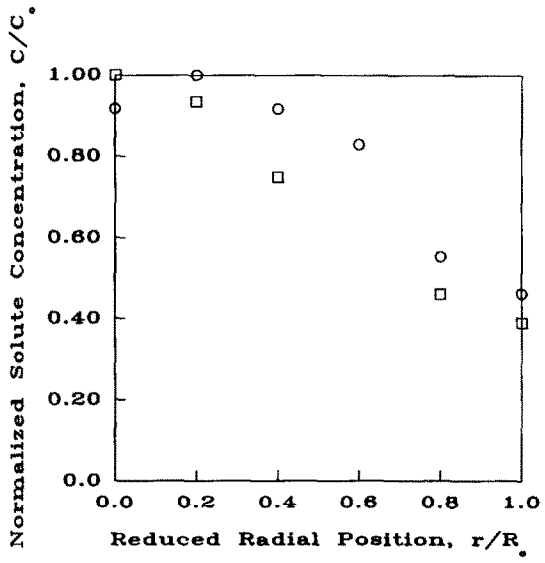

Fig. 1. Normalized iodine concentration, c/c_0, as a function of radial position, r/R_0, in two cylindrical, water-swollen poly(2-hydroxyethyl methacrylate) network samples. Here, c_0 is the external solution concentration at the maximum radius R_0 of the samples.

centration profiles can be recorded even at long times. The data presented here are for samples that have swollen so much that the glassy core has disappeared (Klier and Peppas, 1987).

The solute concentration is at a maximum in the center of the polymer samples and decreases toward the edges. Iodine concentration was detected throughout the samples including the region near the polymer/penetrant interface. Indeed, this Figure indicates a normalized concentration of 0.40 at the polymer/solvent interface, indicative of an iodine partitioning.

For small solute concentrations this technique gives a noticeable solute peak. However, the background noise is large (about half of the peak amplitude) and consequently the resolution may be poor. Better resolution is expected with higher solute concentration.

The technique of X-ray emission spectroscopy is effective in determining the concentration of heavy atoms in polymeric media. Relatively high solute concentrations are desirable; organic constituents may be undetectable. Thus, this technique may be useful in examination of drug release predominantly from polymer matrix systems where the drug contains a heavy inorganic molecule (such as chlorine salts) and in which the drug is present in large quantities.

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