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COMMUNICATION

Optimization of the Mechanical Properties and Water-Vapor Transmission Properties of Free Films of Hydroxypropylmethylcellulose

M. S. Nagarsenker* and D. D. Hegde

Bombay College of Pharmacy, Kalina, Mumbai 400098, India

ABSTRACT

Free films of hydroxypropylmethylcellulose were prepared by a spraying technique. Methocel E5 and Methocel E15 were used in varying proportions in the preparation of films. The free films were studied for their mechanical properties and moisture permeability characteristics. A 2^2 factorial design was used to quantitate the effect of each polymer on the tensile strength and permeability constant of the films.

INTRODUCTION

Film coating of tablets with hydroxypropylmethylcellulose (HPMC) has become popular because of its solubility in water; stability in presence of heat, light, air, and moisture; and ability to coat using a fully aqueous system (1). A transparent, tough, flexible, and nontacky film can be formed from an organic or aqueous solution of HPMC. It is reported that water-vapor permeability of HPMC films tended to decrease as the viscosity decreased. However, when HPMC of low viscosity was used, both tensile strength and elongation decreased (2). These observations suggested the possibility of cracking the coated film when an HPMC of lower viscosity is used. These properties of film coat can be optimized by blending of HPMC of low and high viscosity grades. In the present study, free films of combinations of HPMC 5 counts per second (cps) and HPMC 15 cps were evaluated for tensile strength and water-vapor permeability. A 2^2 factorial design was used to assess the effect of each type of HPMC used on these properties.

MATERIALS AND METHODS

Materials

The materials used were Methocel E5 and Methocel E15 (Dow Chemical Company) and propylene glycol (s·d Fine Chemicals, Mumbai, India).

* To whom correspondence should be addressed.

Table 1

Composition of Free Films: A Two-Factor Two-Level Design

Experiment	Methocel E5 (%w/w) X_1	Methocel E15 (%w/w) X_2	Propylene Glycol (% w/w)
1	3	0.5	3
a	6	0.5	3
b	3	3	3
ab	6	3	3

Methods

Preparation of Free Films

A total of 50 ml of the polymer solutions was sprayed onto clean dry glass plates with a PILOT spray gun (Manik Radiators Pvt. Ltd., Mumbai, India). The films were dried using a hot air blast (40–50°C), cut into squares of 3 × 3 cm, and were stored at room temperature protected from moisture. Propylene glycol (3% w/w) was used as a plasticizer in all solutions. The thickness of the free films that were cast in triplicates was measured at six places, avoiding the edges using a micrometer (Mitutoyo, Japan). Films demonstrating large variations in thickness and inhomogenous structure were rejected.

A simple 2^2 factorial design was used to quantitate the effect of each type of Methocel on the tensile strength and the permeability constant. Table 1 describes the concentration in which Methocel E5 and Methocel E15 were combined for the preparation of free films. In addition to the experiments mentioned in Table 1, films were spray cast using aqueous solutions of HPMC 5 cps in a concentration of 8% w/w (experiment F1) and HPMC 15 cps in a concentration of 4% w/w (experiment F2).

Mechanical Evaluation of Free Films

Mechanical properties of free films were evaluated using an Instron 1026 tester (Instron Ltd., UK) at a strain rate of 5 mm/min, and the distance between the two jaws of the tester was fixed to 10 mm. Films were cut into strips of (5 × 30 mm), and a minimum of five samples of each type was used for the study.

Water-Vapor Transmission Rate Studies

Water-vapor transmission rate (WVTR) studies were carried out using a modified ASTM E 96-53T method. The apparatus consisted of a shallow cup with a flange and a matching gasket, which was held in place by means of three screw clamps. The film of appropriate dimensions was placed on the cup and the gasket placed on the film followed by the metal ring. The film was secured in place with the help of three screw clamps. A humidity gradient was established on either side of the film and the WVTR measured. The test was conducted with 94% relative humidity outside the cup and negligible relative humidity inside the cup.

RESULTS AND DISCUSSION

Mechanical Evaluation of Free Films

The mechanical properties of a film should be such that they remain intact during handling and transport. Ideally, a film coat should be hard and tough without being brittle. These properties are reflected in a high value of tensile strength, a high modulus of elasticity, and substantial percent elongation at break when subjected to a tensile elongation test (1,3).

The mechanical properties of free films can be defined by stress–strain data (4). The stress–strain data for free films are given in Table 2. It was found that the value of Young's modulus was high when the total polymer (E5 and E15) concentration used in the solution was high. The film (experiment ab) cast with the solution containing 6% w/w of Methocel E5 and 3% w/w of Methocel E15 showed highest value of modulus of elasticity. As the results of this part of the study (Table 2) indicated interaction of independent variables X_1 and X_2, a term X_1X_2 was included in a polynomial equation $Y = B_0 + B_1X_1 + B_2X_2 + B_{12}X_1X_2$, which was proposed to describe the relationship between the independent variables X_1 and X_2 and response variable, tensile stress. Mathematical treatment of the tensile strength data gave the following relationship

$$Y = 5.156 + 1.4566X_1 - 1.672X_2 + 0.8053X_1X_2 \quad (1)$$

Table 2

Evaluation of Stress–Strain Parameters

Experiment	Stress × 10^8 (dynes/cm^2)	Strain	Elongation (%)	Youngs Modulus × 10^8 (dynes/cm^2)
F1	1.573	0.084	8.39	18.85
F2	1.828	0.14	14.00	13.34
1	0.990	0.04	4.0	24.76
a	1.548	0.071	7.14	22.09
b	1.176	0.060	6.00	19.97
ab	2.388	0.080	8.00	29.86

where tensile stress of films is given by $Y \times 10^7$ in dynes/cm^2.

WVTR Studies

WVTR studies determine the effectiveness of the polymer film as a moisture barrier. An extensive review of mathematical derivation and theory for calculating rates of moisture permeation was covered by several workers (5–7). In the present study, the water-vapor permeability constant was calculated as

$$\text{Permeability constant (P)} = \frac{W \times L}{A \times T \times \Delta P}$$

where W is the grams of water diffusing through the film having thickness L and area A, T is the time in hours during which water diffuses, and ΔP is the vapor-pressure gradient. P is expressed in terms of g/cm mm Hg per 24 hr. The results of WVTR studies have been tabulated in Table 3. The water-vapor permeability of the films decreased with decrease in viscosity. This conforms with the results reported by Nagai et al. (2). The weight of moisture transmitted (in grams) through all films studied was found to be directly proportional to time, over a period of 7 days, when the temperature, vapor pressure, and the area of film exposed were held constant. Considering interaction between independent variables, the polynomial equation derived (from data in Table 3) to describe the relationship was as follows:

$$Y = 2.776 - 0.0258X_1 - 0.1336X_2 + 0.0916X_1X_2 \qquad (2)$$

where the permeability constant of the film is given by $Y \times 10^{-6}$ in g/cm mm Hg per 24 hr. Equations (1) and (2) were made use of to construct contour plots, which are shown in Figs. 1 and 2, respectively. Each line in these contour plots represents a number of combinations of E5 and E15 that have the value of tensile stress and permeability constant indicated in Figs. 1 and 2, respectively. Using these plots, it is possible to select combinations of E5 and E15 that will have low WVTR and better tensile strength.

Table 3

Water Vapor Transmission Rate Studies

Experiment	Film Thickness (μm)	Weight Gain (g)	Permeability Constant ×10^{-6} (g/cm mm Hg/day)
F1	49	2.056	3.99
F2	38	2.303	3.47
1	34	2.07	2.77
a	42	1.699	2.83
b	43	1.815	3.12
ab	51	1.933	3.87

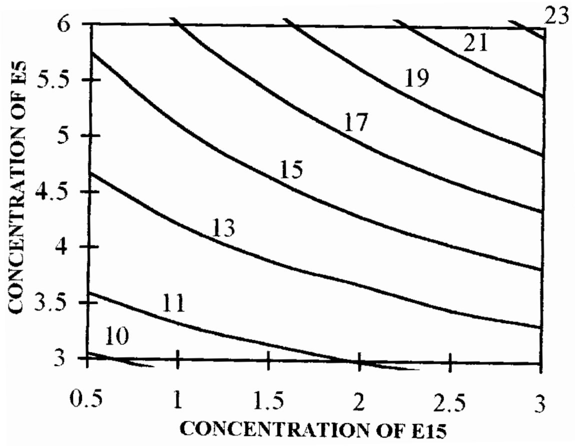

Figure 1. Contour plot for tensile strength of free HPMC films.

To test the validity of the design, films containing 3.5% w/w of Methocel E5 and 3.5% of Methocel E15 (experiment F3) were spray cast in triplicate and evaluated for tensile stress and WVTR studies. The observed value and the values calculated for the tensile stress were 1.84×10^8 and 1.43×10^8, respectively, and those for WVTR studies were found to be 4.65×10^{-6} and 3.34×10^{-6}, respectively.

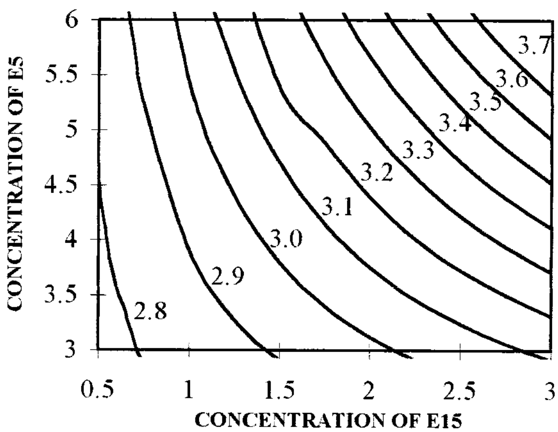

Figure 2. Contour plot for moisture permeability constant of free HPMC films.

CONCLUSIONS

Tensile strength and water-vapor transmission properties of HPMC films can be optimized by mixing HPMC of low and high viscosity. Various combinations of E5 and E15 can be selected from contour plots to give good film coat of desirable properties. These combinations can be further screened based on parameters like cost of polymer, ease of spraying, and time required for film coating to arrive at an optimized mix of polymers.

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