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THE MECHANICAL AND ADHESION PROPERTIES OF AQUEOUS-BASED HYDROXYPROPYL METHYLCELLULOSE COATING SYSTEMS CONTAINING POLYDEXTROSE AND TITANIUM DIOXIDE

V-M. Lehtola[1], J.T. Heinämäki[2], P. Nikupaavo[2], J.K. Yliruusi[2]

1 Leiras Oy, P.O.Box 33, FIN-33721 Tampere, Finland
2 Department of Pharmacy, Pharmaceutical Technology Division, P.O. Box 15, FIN-00014 University of Helsinki, Finland

SUMMARY

The effect of titanium dioxide and polydextrose on film adhesion to microcrystalline cellulose tablet surface and mechanical properties of aqueous-based hydroxypropyl methylcellulose free films were evaluated using a Lloyd LRX materials testing machine. The free films and the films applied to tablets were prepared by using a pneumatic spraying technique similar to that used in fluidized-bed coaters.

The film adhesion was found to increase with increasing concentrations of titanium dioxide and polydextrose in the film. The addition of polydextrose to the film increased only slightly the moisture permeation of coated

tablets. This may be due to the hygroscopic nature of polydextrose. Over the range studied, the addition of polydextrose reduced the elongation and the tensile strength of the film, indicating decreased deformation capacity of the film and a risk of cracking.

INTRODUCTION

Hydroxypropyl methylcellulose has been widely used for aqueous film coating of pharmaceutical tablets. It is normally possible to produce an excellent film coating with HPMC, but difficulties may sometimes arise with respect to the ability of the film coating to adhere satisfactorily to the tablet surface. This is especially true when considering multivitamin tablets.

In confectionery and food industry, polydextrose has been used alone or in combination with other polymers to produce film coatings with excellent adhesive properties (1). There is only little information available about the effect of polydextrose on the adhesion to tablet surfaces and mechanical properties of polymer films in pharmaceutical industry.

Titanium dioxide is used in film coating formulations as a pigment. There are conflicting studies concerning the effect of titanium dioxide on the adhesion between polymer film and tablet surface (2 - 4).

The objective of this work was to investigate the effects of some solid additives (polydextrose, titanium dioxide) on the adhesion and mechanical film properties of hydroxypropyl methylcellulose (HPMC).

MATERIALS AND METHODS

Tablet preparation

The tablets were compressed from microcrystalline cellulose (Emcocel 90 M, Edward Mendel, Finland). Magnesium stearate (Ph. Eur.) was used as an external lubricant at the concentration of 1.0%. The tablet cores were compressed in an instrumented single-punch Korsch EK-O tablet machine (Korsch GMBH, Germany) using flat-faced 11-mm punches and a compression pressure of 150 MPa. The weight of the tablets was 300 mg.

Tablet coating

The aqueous film coating solution consisted of hydroxypropyl methylcellulose 10% w/w (Methocel E5, Dow Chemical, U.S.A.) and polyethylene glycol (Macrogol 400, Hoechst, Germany) 20% w/w of the polymer weight as a plasticizer. Titanium dioxide (Jahrens Fabrikker, Norway) was used 10%, 20% and 30% of polymer weight and polydextrose (Pfizer Inc., U.S.A.) 0%, 50%, 75% and 100% of polymer weight.

The aqueous film coating of tablets was made using a fluidized-bed coater (Aeromatic Strea-1, Aeromatic AG, Swizerland). The conditions have been described elsewhere (5).

Free film preparation

Free films were prepared by using a spraying equipment which consisted of elements of a fluidized-bed coater (Aeromatic Strea-1, Aeromatic AG, Swizerland). The system has been described in detail elsewhere (5). The conditions was same as used in tablet coating.

Evaluation of free films and coated tablets

A Lloyd LRX materials testing machine (Lloyd instruments Ltd, England) was used to determine the mechanical and stress-strain properties of free films. The environmental conditions were 22 ± 1°C and 40 ± 5% R.H. The thickness of each film was measured at five different points and the films of a thickness of 75 ± 5 μm were cut into 10 x 1.5 cm strips. The measurements were performed using a 50 N load cell, initial gauge length of 4.0 cm and a cross speed of 5 mm/min. Tensile strength, elongation (strain) at break, modulus of elasticity (Young's modulus) and work done were calculated from stress-strain curve. Measurements were repeated three times.

The adhesion of film coatings to the tablet surface was also measured with a Lloyd LRX materials testing machine. Before testing, the film from around the edges of the tablets was removed with a sharp blade. The tablet was mounted into the lower grip of a materials testing apparatus with a piece of double-sided adhesive tape. The upper grip was then driven onto the tablet surface with a piece of adhesive tape, and a fixed force of 3.0 N was then used to get a firm adhesion of the tape to the film. Adhesion strength measurements were performed using a 50 N load cell and a cross speed of 7.5 cm/min. The measurements were repeated six times.

The coated tablets were stored at 10%, 20%, 40%, 70% and 90% relative humidities in 21°C for one month. Each sample consisted of three tablets, which were weighed together. The samples were weighed before and after storage.

Film morphology

The morphology of the film was studied by scanning electron microscopy by taking micrographs (JEOL JSM-820, Japanese Electron Optical Ltd., Tokyo, Japan).

Statistical analysis

Statistical evaluation (t-test) was made using the Windows version of Systat 5.0 (Systat Inc., U.S.A.).

RESULTS AND DISCUSSION

Film morphology

Cross-section of HPMC-coated tablet is presented in Figure 1. As seen on the S.E.M. photo, the tablet surface is covered by homogenous and continuous film.

Mechanical properties of free films

The mechanical properties of free films can be defined by stress-strain data (6). Young's modulus is the constant of proportionality of stress to strain, and is equal to the slope of the straight-line portion of the stress-strain curve. In the straight-line portion of the curve deformation of the film is elastic. The rest of stress-strain curve indicate the plastic deformation of the film before breaking. The stress-strain results of HPMC free films containing different amounts of polydextrose are shown in Figures 2 and 3. Addition of polydextrose reduced the values of elongation at break. According to Banker (6), elongation is defined as a measure of the capacity of a film to deform prior to failure. Thus the lowered elongation indicates a low deformation capacity of the film and a brittle film structure (6).

The addition of polydextrose to film resulted in a decrease in the tensile strength (Fig. 3). Tensile strength is the maximum stress applied to a point at

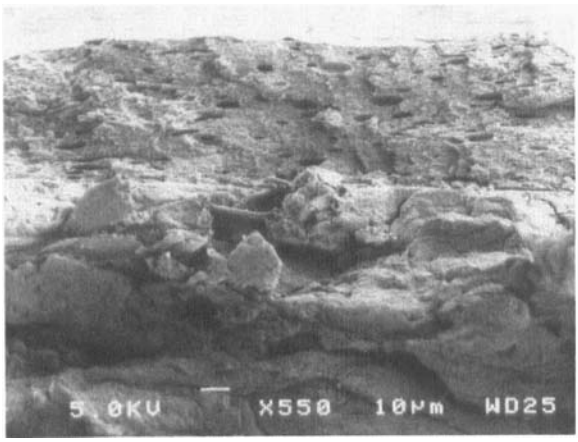

FIGURE 1

SEM micrographs of the cross section of a coated tablet. Bar in figure 10 μm.

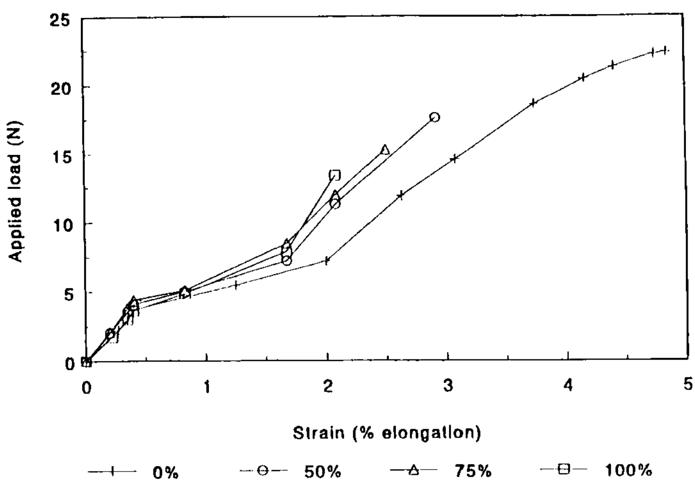

FIGURE 2

The stress-strain profiles of HPMC free films containing polydextrose 0 - 100% of the polymer weight.

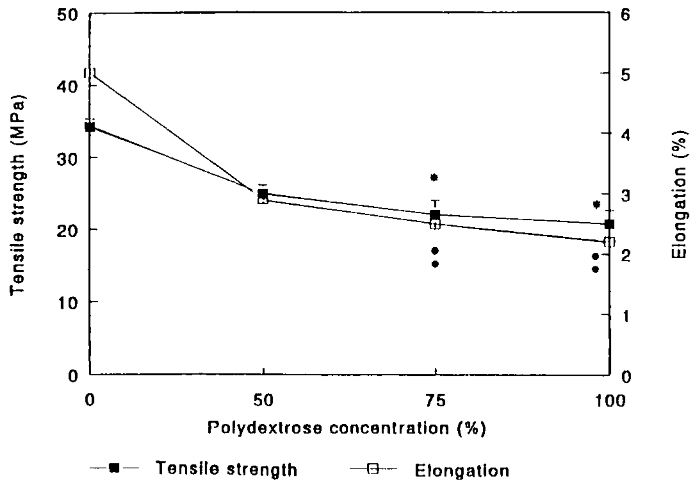

FIGURE 3

The tensile strength and elongation profiles of HPMC free films containing polydextrose 0 - 100% of the polymer weight (n =3). Closed asterisks (tensile strenght) and closed circles (elongation) show the statistically significant differences compared to the films containing 50% of polydextrose: * $p < 0.05$, ** $p < 0.01$. *** $p < 0.001$.

which the film breaks. According to the literature, the risk of cracking of a film increases with decreasing tensile strength (6).

The results obtained with free films indicate that the mechanical properties of HPMC films impairs with increasing concentration of polydextrose in the film. Despite this no signs of film defects in the coated tablets was observed.

<u>Adhesion and Young's modulus</u>

Adhesion between tablet cores and film coating increased with increasing concentration of titanium dioxide and polydextrose (Fig. 4).

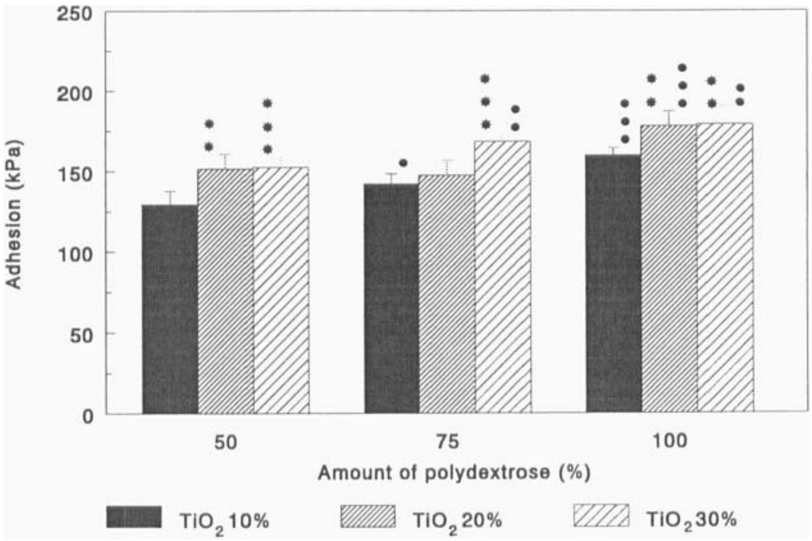

FIGURE 4

Effect of titanium dioxide and polydextrose concentrations on the adhesion between MCC tablets and HPMC film (n = 6). Closed asterisks show the statistically significant differences compared to the films containing 10% of titanium dioxide and closed circles to the films containing 50% of polydextrose: * p < 0.05, ** p < 0.01, *** p < 0.001

Okamafe and York (3) suggested that the pigment-polymer interaction stiffens and strengthens the polymer segments at the polymer-pigment interface as well as at the film-tablet interface. Thus, the adhesion between film and tablet surface increases with increasing concentration of titanium dioxide in the film. Our results are in accordance with those presented by Okamafe and York (3).

A poor adhesion between film and tablet surface is due to a high internal stress in the film coating. According to Sato (7), Young's modulus is proportional to internal stress; the value of Young's modulus increases with increasing internal stress.

TABLE 1

Young's modulus values for hydroxypropyl methylcellulose free films (n = 6).

Amount of polydextrose of polymer in film (%)	Young's modulus (N/mm^2) mean ± S.D.
0	557 ± 39
50	404 ± 23
75	394 ± 24
100	365 ± 30

In our studies, addition of polydextrose to the film slightly decreased the values of Young's modulus (Table 1) and increased the adhesion between film coating and tablet surface (Fig. 4). Obviously, polydextrose decreased the internal stress in the film and thus improved the adhesion.

It has been presented earlier that polydextrose can produce a film also without polymer (1). It can be assumed that a polydextrose film is brittle, but it has good adhesion properties. This may be due to hydrogen bonds between tablet structure and polydextrose.

Elongation and tensile strength results obtained with free films indicate the risk of film cracking. However no sign of cracking in the coated tablets were obtained. This may be due to lower values for Young's modulus and increased adhesion between film and tablet surface, when the amount of polydextrose was increased.

TABLE 2

Mechanical strength of coated tablets (n = 10).

Amount of titanium dioxide of polymer in coating solution (%)	Amount of polydextrose of polymer in coating solution (%)	Mechanical strength (N) mean ± S.D.
10	50	302 ± 25
20	50	293 ± 16
30	50	284 ± 18
10	75	278 ± 18
20	75	289 ± 20
30	75	275 ± 20
10	100	285 ± 17
20	100	287 ± 24
30	100	284 ± 19

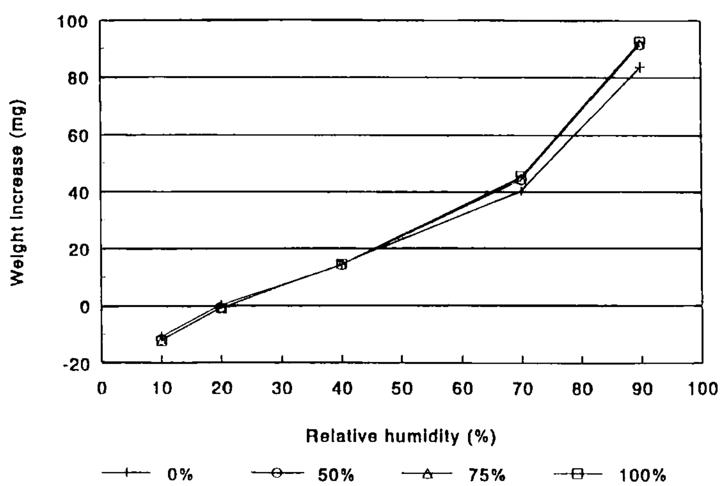

FIGURE 5

The moisture permeation of tablets coated with HPMC film containing polydextrose 0 - 100% of the polymer weight. The amount of titanium dioxide was constant (20%).

Mechanical strength and moisture permeation of coated tablets

The mechanical strength of uncoated tablets was 218 ± 9 N. The film coating increased the mechanical strength of tablets (Table 2), but the amount of polydextrose and titanium dioxide did not affect the mechanical strength.

The moisture permeation on uncoated tablets in 40% R.H. and 90% R.H. were 41 mg and 332 mg, respectively. The polymer film applied to tablet surface decreased the moisture permeation on tablets (Fig. 5). The addition of polydextrose to the film increased only slightly the moisture permeation on coated tablet. This may be due to the hygroscopic nature of polydextrose.

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