An Investigation on the Influence of a Vinyl Pyrrolidone/Vinyl Acetate Copolymer on the Moisture Permeation, Mechanical and Adhesive Properties of Aqueous-Based Hydroxypropyl Methylcellulose Film Coatings

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Received December 1, 2003; accepted April 3, 2004

Polymers for aqueous film coating, such as hydroxypropyl methylcellulose (HPMC), often require the inclusion of a plasticizer to reduce brittleness and increase flexibility and ductility. A vinyl pyrrolidone/vinyl acetate copolymer (S630) was investigated for its influence on HPMC film coating parameters, comparing the results with a commonly used plasticizer, polyethylene glycol and another copolymer, polyvinyl alcohol. The viscous properties of the solutions and the glass transition temperatures of the equivalent polymer films were evaluated. Its effect on the film properties, such as appearance, surface roughness, moisture permeation and mechanical properties, as well as its ability to promote better adhesion of the film coat to the core surface, was compared. S630 was able to reduce the viscosity of the polymer solution and glass transition temperature of HPMC, as well as, enhance the mechanical properties of the cast film. The moisture permeation was slightly reduced but not to the same extent as polyethylene glycol. A 10% concentration of S630 increased the adhesive strength and toughness of the HPMC film coat. In conclusion, S630 was effective as a film-former, substrate adhesive and plasticizer. It has the potential to be used to replace the more volatile plasticizers which have problems of loss or migration.

Key words hydroxypropyl methylcellulose; plasticizer; moisture permeation; surface roughness; mechanical property; adhesion

Film coating of tablets is commonly used to protect the integrity of the core material against environmental factors, for taste masking or providing a tough and better quality finish to minimize possible damage from mechanical handling during high speed packing.[1] In view of environmental health and safety issues, as well as the higher costs associated with organic solvents, aqueous film coating is currently the method of choice in the pharmaceutical industry. One of the most widely used polymers for aqueous film coating of tablets is hydroxypropyl methylcellulose (HPMC).[2] Generally, to minimize coating time and maximize production efficiency, the lowest possible viscosity grade of the polymer is the preferred choice as a high solid content in the coating solution is possible and, thus less water needed to be removed. The sprayable solution viscosity for aqueous film coating should be in the range of 150 to 400 mPa s. The apparent viscosity of aqueous HPMC solution is related to the molecular weight of the HPMC polymer and lowering of the molecular weight reduces the physical properties of the film coat.[3] The advantage of using low viscosity grade polymer is limited if film quality, such as vapour permeabililty and tensile strength, is compromised.

Due to the brittleness of plain HPMC films, plasticizers are often included in HPMC coating formulations. The functions of a plasticizer include softening films, reducing brittleness, increasing flexibility and ductility. Plasticization can increase the segmental mobility of HPMC, resulting in depression of the glass transition temperature.[4] As plasticizers lower both the mechanical properties and glass transition temperature, their compatibility and concentration are very important in coating formulations. Over-plasticization can lead to increased diffusivity,[5] making the film coat a poor protector of its core. In high humidity environments, adsorbed water itself has a plasticizing effect. Another additive, polyvinyl alcohol (PVA), had been reported to interact with HPMC resulting in decreased diffusivity.[5]

Mechanical properties, such as tensile strength or maximum tensile stress, elastic modulus indicating stiffness and rigidity,[6] tensile strain at breakage or elongation (%) and work of failure (energy) are often used to gauge the performance of the film. An ideal film coat should be hard and tough with a high tensile strength and elastic modulus,[7] as well as a large work of failure and moderate elongation before break.

Film coating on a tablet should be in intimate and continuous contact with the substrate surface.[1] Strong film-tablet adhesion is also essential for good quality coated tablets as poor adhesion not only indicates visible film-coating defects but also affects the stability of moisture sensitive drugs and compromises the level of mechanical protection the film coat confers to the core. Evaluation of the adhesion characteristics provides useful information on the internal stress in films and film coating defects.[8] The adhesiveness of film coating onto tablet substrate is difficult to determine. Nonetheless, it is important to evaluate adhesiveness of coatings to determine the effectiveness of additives such as plasticizers in not only affecting the film tensile properties but also confer superior adhesion properties. Variations of the butt adhesion technique for detaching the entire film from the surface of the tablet under constant rate of deformation had been employed to evaluate the adhesive properties of the tablet film coat.[9—11] This study investigates the use of vinyl pyrrolidone/vinyl acetate copolymer, a 60 : 40 copolymer of vinyl pyrrolidone and vinyl acetate in modifying the properties of HPMC films.

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Experimental

Materials Polymer employed for the film coat was a low viscosity hydroxypropyl methylcellulose (HPMC; Methocel E3 LV®, Dow Chemical, U.S.A). Molecular weight for Methocel E3 is about 20000 daltons.[12] The additives used included vinyl pyrrolidone/vinyl acetate copolymer (S630; Plasdone S-630®, ISP Technologies, U.S.A.), 98—99% hydrolyzed polyvinyl alcohol (PVA; Aldrich Chemical, U.S.A.) and polyethylene glycol 1500 (PEG; Merck-Schuchardt, Germany). The molecular weights for the additives, PEG, S630 and PVA are 1400 to 1600, 58000, and 85000 to 146000 daltons, respectively. Desiccant used was anhydrous calcium oxide (Merck, Germany).

Capsule-shaped placebo tablets, used as cores for coating, were prepared from lactose (86%) with povidone K-25 (2%), tapioca starch (9.3%), sodium starch glycolate (1%), talcum (1%) and magnesium stearate (0.7%). These caplets weighed 650 mg each, length 15.7 mm, width 6.1 mm, thickness 6.6 mm, approximate surface area 300 mm^2, hardness at 12.1—17.8 kP, disintegration time of approximately 5 min and friability at 0.35%.

Preparation of HPMC Solutions HPMC solutions were prepared according to the formulations in Table 1. HPMC was dispersed in water heated to above 80 °C. The additive, previously dissolved in water, was incorporated gradually with vigorous stirring. The dispersion was then transferred to an ice bath for rapid hydration. Water was added to make up to weight, followed by further agitation (i) by stirring with a magnetic stirrer at 500 rpm for 30 min for solutions to be used for casting films or (ii) by using a turbine homogenizer (L4R, Silverson Machines, U.K.) at high speed for 20 min for the film coating solutions. Solutions were then allowed to hydrate overnight at 5 °C and equilibrated to room temperature before use.

Determination of Apparent Viscosity Viscosities of HPMC solutions with 3% w/w solid content were measured using a rotational viscometer (LV1, Brookfield Engineering Lab, U.S.A.) at shear rates of 12 and 30 rpm at 25 °C. For the film coating solutions with 8% solid content, viscosity was obtained using a rheometer (RheoStress 1 with RheoWin QC, HAAKE Instruments, U.S.A.) with the Z10 DIN sensor at shear rates of 50 to 500 rad s^{-1} and temperature condition was controlled by a thermocontroller (DC30 ThermoHaake, HAAKE Instruments, U.S.A.) for various temperatures between 30 °C and 60 °C. Triplicates from each formulation were determined and viscosity values obtained at 450 rad s^{-1} were used for comparison.

Preparation of Cast Films Films were cast with 20 g of film casting solution using flat glass dishes of diameter 97 mm. These dishes were placed on glass slabs, previously leveled with a spirit level, in a hot air oven set at 45 °C for 24 h. The oven was validated for uniformity of drying. This was determined by the rate of water evaporation in different parts of the oven and spots with similar drying rates were determined. Casting dishes containing HPMC solutions were placed in positions where the rates of evaporation were between 0.7 to 0.9 g h^{-1}.

Dried films were collected, sealed in polyethylene bags and kept in an environment-controlled room set at 25±1 °C and relative humidity of 55±5% for no less than 5 d before evaluation.

Surface Roughness Analysis Quantification of the surface roughness of HPMC films was determined using a scanning probe microscope (SPM-9500J, Shimadzu, Japan). The scanning probe microscope provides direct quantitative values of the surface texture by parameters, such as Ra (arithmetic mean roughness), Ry (maximum height) and Rz (10-point mean roughness). Surface roughness was best represented by Ra values because Ry and Rz were calculated based on selected points only and would be less representative of the whole surface roughness.[13] Samples were scanned over 3 sites, each with an area of 25 μm by 25 μm with Z ranges (scanning depth) set at 1 and 2 μm and at a frequency of 1 Hz. Each sample was measured on both surfaces of the film. Measurements were in triplicates and results averaged.

Moisture Permeation Studies Disks of diameter 20 mm were cut from cast films and only films with thickness of 0.08±0.01 mm were selected for use. The test disk was clamped between two sets of polytetrafluorethylene (PTFE) and stainless steel washers. The diameter of the opening of the washers was 12 mm. The washers with the film disk were secured using an aluminium screw cap with a central opening over a 28 ml glass bottle. Each bottle contained 20 g calcium oxide to produce a gradient for moisture permeation. The bottles were placed in a controlled environment chamber (KBF 115, WTC Binder, Germany) set at 25 °C and 75% relative humidity. Controls without barrier film and with 0.08 mm thick polyethylene film were also included. The samples were weighed periodically over a period of 30 d and evaluations were carried out with five replicates. The moisture permeation rate was the gradient of the slope of weight gained against time.

Table 1. Formulae of Coating Solutions for Film Casting and Caplet Coating

Formula	Solution for casting film	Solution for coating film
Additive (g)	0, 0.15, 0.225, 0.3, 0.45, 0.6, 0.9	0.8
HPMC (g)	qs[a]	7.2
Total solid content (g)	3.0	8.0

a) Quantity sufficient to make up to 3 g of solid.

Determination of Glass Transition Temperature The films cast from solutions with 8% solids were left to equilibrate in a desiccator for not less than 2 d before determining the glass transition temperature (T_g) using a differential scanning calorimeter (DSC-50, Shimadzu, Japan). The sample was first heated at 20 °C min^{-1} to 160 °C to remove moderately bound moisture.[14] It was then rapidly cooled to 25 °C and reheated at 20 °C min^{-1} to 200 °C. Accurately weighed samples of about 6 mg were placed in crimped aluminium sample pans and the T_g values were determined from the thermograms of reheated samples. Triplicate measurements were carried out to determine the T_g and the mean calculated.

Film Coating Film coating was carried out using 4 kg caplets in a vertical fluidized tablet coater (Ultra-coater MP1, Aeromatic-Fielder, U.K.) rotated at 22 to 25 rpm, inlet temperature at 80 °C and fluidizing air flow rate at 70 m^3 h^{-1}. Coating solution was sprayed tangentially using a pair of diagonally arranged two-fluid nozzles set at 40° from the normal in the direction of tablet flow, at a combined spray rate of 13 to 16 ml min^{-1} and atomizing pressure at 3 bars.

Determination of Film Thickness Thickness of the cast films was measured using a thickness gauge (7305, Mitutoyo, Japan) at random spots over the entire film. Only samples within the desired thickness and uniformity were used.

Thin and uniform cross-sections of coated caplets were sliced using a sharp cutting blade. A cut sample was placed under a stereomicroscope (SZH, Olympus, Japan) equipped with a video camera (CCD-IRIS, Sony, Japan) and the image was displayed directly on a monitor (PVM145E, Sony, Japan). Thickness was determined using digital vernier callipers (500-216, Mitutoyo, Japan), calibrated using a 10 mm stage micrometer in place of a sliced cross section. Ten cut samples were used to determine film thickness for each batch of coated caplets. For every sample, thickness was measured at 10 points along a section of film coat and the mean of all thickness values of each batch was calculated.

Evaluation of Mechanical Properties of Cast Films The mechanical properties of both the free cast film strips and coated film were determined using a tensile tester (E-Z Tester, Shimadzu, Japan). Film strips were tested using a 100 N load cell and crosshead speed of 10 mm min^{-1}. Stress–strain curves giving maximum stress, elasticity (elastic modulus), % strain (% elongation at breakage) and energy for work done (area under the curve) were obtained. Measurements were taken under environmental conditions of 25±1 °C and 55±5% relative humidity. For each formulation, 10 samples were tested using 50 mm×10 mm film strips of 85±10 μm thickness.

Evaluation of Adhesive Properties of Film Coats The butt adhesion technique was performed using the tensile tester at initial gauge length of 24 mm and crosshead speed of 200 mm min^{-1}. A laboratory-designed tablet holder was fitted to the tester in place of the lower grip (Fig. 1). A 6.25 mm wide adhesive tape was attached centrally over the caplet film coating, around the circumference of the caplet, leaving a short length of folded tape to be clamped to the upper grip of the tester. The ends of the caplet were truncated, leaving a test width of approximately 8.5 mm. The film on the coated caplet surface nearest to the folded part of the tape was carefully detached using a scalpel and lifted up together with the attached folded tape to facilitate the peel test. The caplet was held in place by a pin one each side of the adjustable holder. The pins secured the sample yet allowed free rotation for peeling the film. Twenty samples from each batch of coated caplets were evaluated. For control, a piece of non-stick tape wrapped tightly twice around the circumference of an uncoated caplet was unrolled. Five samples were carried out for the control and results averaged. Maximum adhesive force of the film coat was corrected by deducting the measured value with the value obtained for the control.

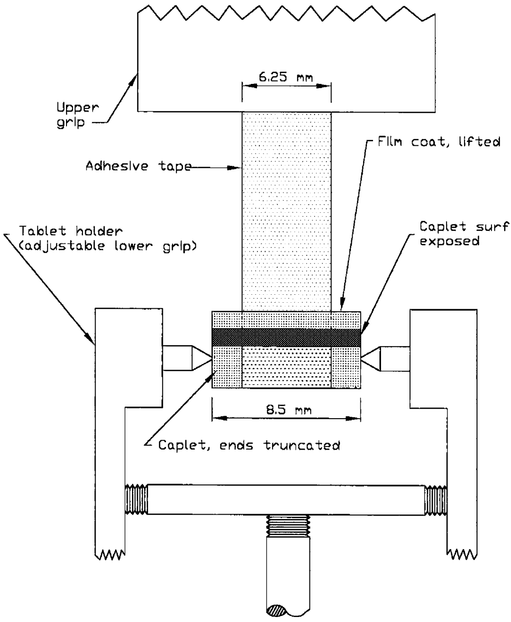


Fig. 1. Custom-Designed Tablet Holder Fitted to the Tensile Tester for Evaluating Film Coat Adhesion

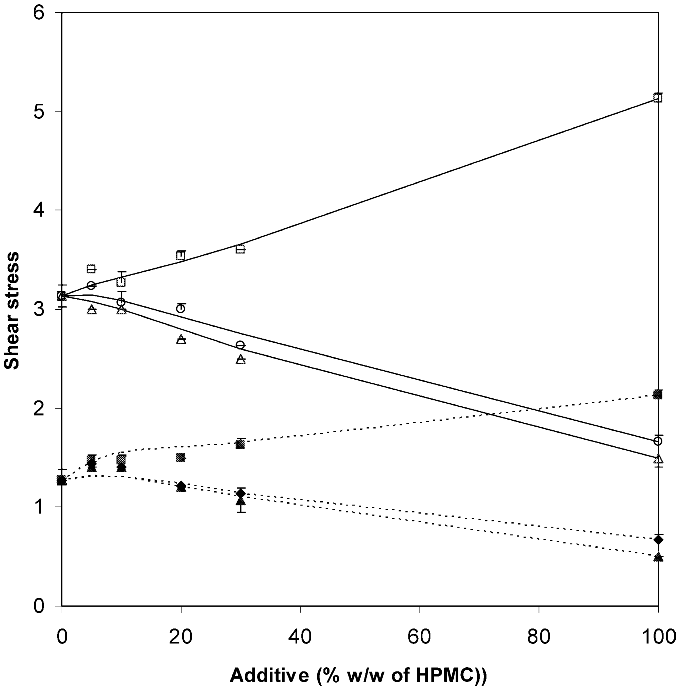


Fig. 2. Shear Stress of 3% HPMC Solutions Containing Various Concentrations of Additives, PEG (▲, △), PVA (■, □) and S630 (●, ○), Measured at Shear Rates of 12 (Closed Symbols) and 30 (Open Symbols) rpm

n=9; bars are ±1 standard deviation.

Results and Discussion

HPMC had been known to be able to produce an excellent film coat but there were instances where there were difficulties relating to the ability of the film coat to adhere satisfactorily to the tablet surface.[2] One of the objectives of this study was to evaluate the adhesiveness of film coats onto tablet cores and the influence of various additives on coat-substrate adhesion. The additives studied were PEG, PVA and S630. PEG and PVA are commonly used as additives for HPMC, as a plasticizer and copolymer respectively. The purpose of this study was to compare S630 with PEG and PVA and to evaluate its influence on HPMC films. Unplasticized HPMC films were clear, transparent and colourless. When cast, they were not strongly adhering and could be easily detached from the surface of the casting dish. Some portions, particularly at the edges were self-detached from the glass surfaces. HPMC-S630 films were similar in appearance but considerably more adhesive. Films with more than 10% S630 had portions adhered very strongly to the glass surfaces. The HPMC films plasticized with less than 20% PEG appeared clear and transparent immediately after harvest but hazy patches developed during storage. Similar films with concentrations of 20% or higher were not clear but hazy in appearance with signs of blooming. The reason for this observation was a problem with co-additive's solubility and compatibility causing the excess plasticizer to be separated and reassembled as tiny spots on the surface of the film as was also reported by other investigators.[15] Upon exposure to more humid environment of a relative humidity of 60% or higher, HPMC-PEG films also turned hazy and had an oily feel. Moisture enhanced this effect shown by PEG as it facilitated the uptake of water vapour, making the films soft, flexible and moist. HPMC films, in which PVA was incorporated were not transparent but translucent in appearance with patches and raised spots, more evident with increased PVA concentration. This effect was attributed to PVA being separated from the system after it had exceeded the level at which it was compatible or soluble in HPMC.[5] Nonetheless, such films were also evaluated for their moisture permeability and mechanical properties.

Viscosity of HPMC Film Formulations Viscosity measurement for solutions with 3% solids showed that the HPMC solutions had non-Newtonian flow properties as shown by the increase in shear stress between measurements taken at shear rates of 12 and 30 rpm (Fig. 2). Although S630 is a copolymer, its effect on the viscosity appeared to be very similar to that of PEG, a plasticizer, especially at the shear rate of 12 rpm. At concentration above 10%, both S630 and PEG reduced the viscosity of HPMC solution. The trend was reversed with PVA, when increased PVA concentration increased the viscosity of HPMC proportionally.

Solutions with 8% solids were also prepared for viscosity measurements at 450 rad s^{-1} from 30 °C to 60 °C. This was performed as coating was carried out with 8% solids. Similar trends to those with 3% solid were found. PVA increased the viscosity of HPMC solutions whereas S630 behaved like PEG in reducing the viscosity. It was also noted that all HPMC solutions, with or without an additive, had their minima in viscosity at temperatures around 55 °C (Fig. 3). Generally, HPMC solutions exhibit pseudoplastic flow behaviour and pseudoplasticity increased with increasing molecular weight or concentration.[3] Overall, the changes in viscosity with the inclusion of additives were small as the major factor determining viscosity in the coating solution was the HPMC polymer grade used and its concentration in the solution.

Effect of Additives on Film Surface Roughness HPMC produced very smooth films. From the results on the surface roughness evaluated with the scanning probe microscope (Fig. 4), it appeared that S630 was most compatible with HPMC as there was hardly any change in the surface

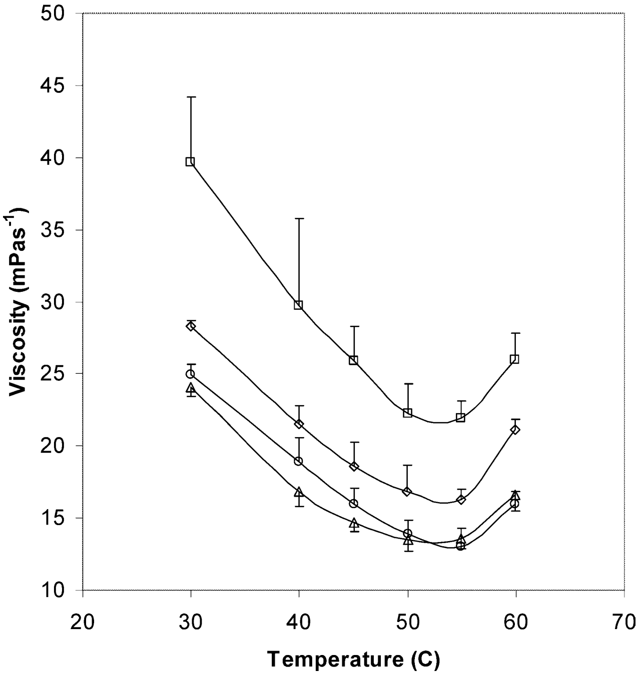


Fig. 3. Apparent Viscosity of 8% HPMC Solutions at Varying Temperatures Measured at Shear Rate of 450 rad s^{-1}: without Additive (◇) and with PEG (△); PVA (□); and S630 (○)

n=9; bars are ±1 standard deviation.

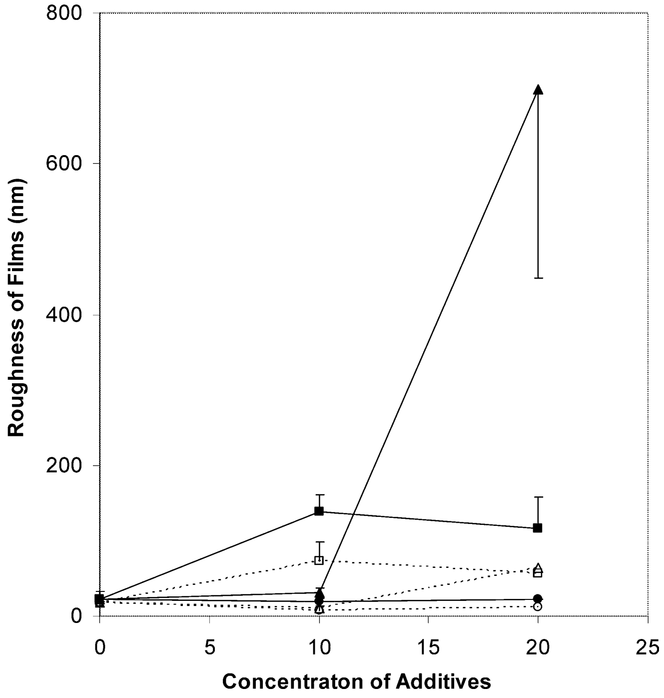
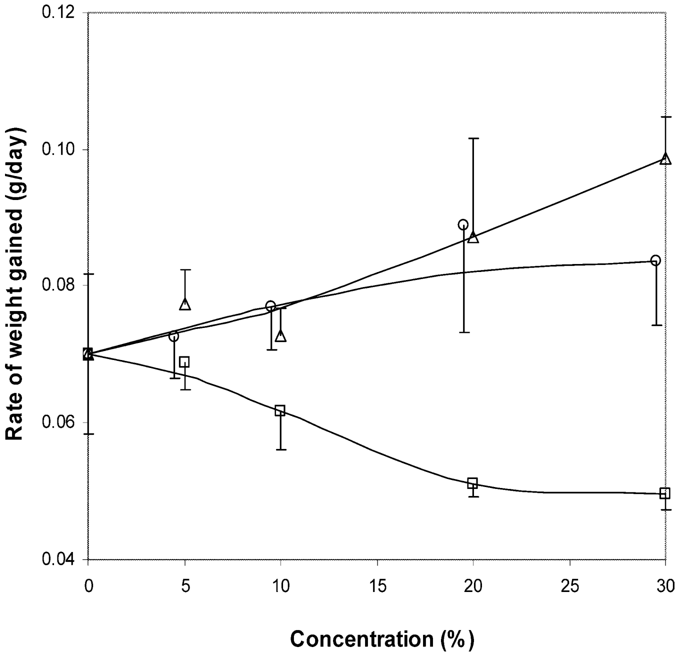


Fig. 4. External (———) and Internal (– – – –) Surface Roughness of HPMC Films with Additives of 10% PEG (△), PVA (□) and S630 (○)

n=9; bars are ±1 standard deviation.



Fig, 5. Moisture Permeation of HPMC Films in Weight Gained over Days with Varying Concentrations of PVA, S630 or PEG: PVA (□); S630 (○); PEG (△)

n=5; bars are ±1 standard deviation.

roughness for such films. Both the external surface exposed to the environment and the inner surface against the glass dish, were equally smooth. Surface roughness increased for films containing PEG and PVA. In the case of PEG, there was a very pronounced increase in surface roughness above the 10% concentration. The increase could be due to its hygroscopic nature. The films containing 20% PEG appeared moist and sticky rapidly when exposed to the environment. The hygroscopic PEG could dry poorly and in the process, became separated from the HPMC film when the moisture level in the partially dried film was low.

Moisture Permeation of HPMC Films Results from the moisture permeation test (Fig. 5) showed that both PEG and S630 had little influence in the moisture permeability of HPMC films when used below a 5% plasticizer level. However, after this concentration the moisture permeability of HPMC films increased with increasing concentrations. The reason for the initial influence, as attributed by other investigators,[16)] could be due to the densification of the HPMC polymer films when smaller plasticizer molecules were added. In high PEG concentration range, PEG increases the diffusion coefficient of HPMC when the diffusion pathways become enlarged and passage of water vapour molecules is facilitated.[5)] In order to maintain the integrity against moisture permeability, the concentration of PEG in HPMC films cannot exceed 10% of the polymer weight. S630 showed a gradual increase in moisture permeability to concentrations up to 20% after which the influence was stabilized. Overall, the increase in moisture permeability in HPMC-S630 films was quite close to those with PEG, except for those with 30% additive. Like PEG, the use of S630 beyond 10% did not function well as a moisture barrier. PVA was found to reduce the moisture permeability of HPMC films, the reduction increasing with the concentration of PVA. The decrease in moisture permeability in HPMC-PVA films was due to decreased moisture diffusivity. This could be attributed to the crystallinity of PVA enhancing the crystalline phase in the HPMC-PVA films, thereby reducing the diffusion pathways.[4,5)]

Effect of Additives on the Glass Transition Tempera-

ture The glass transition temperature (T_g) of H PMC varies from 170 °C to 180 °C.[17] The T_g of HPMC determined by differential scanning calorimetry in the study was 167.15 °C. The T_g of HPMC incorporated with PVA could not be detected. This could be attributed to the presence of a crystalline phase in the blend. Both PEG (T_g, 161.03 °C) and S630 (T_g, 163.23 °C) reduced the T_g of HPMC films by approximately 6 °C and 4 °C respectively. Plasticization lowered the T_g values and the incorporation of PEG or S630 in the HPMC formulation was proposed to increase the segmental mobility of the HPMC polymer. The glass transition temperature has been described as the characteristic temperature at which a polymer changes from a state of relative molecular or segmental rigidity (glassy phase) to one of considerable chain mobility (rubbery phase).[4] Incorporation of a plasticizer affects the glass transition temperature of the coating polymer and the more efficient the plasticizer, the greater the lowering of the T_g.[18] Glass transition temperature influences the physical properties, such as viscosity of the coating solution and the mechanical properties and moisture permeability of the film coat.[6]

Effect of Additives on the Mechanical Properties of HPMC Cast Films Mechanical tests on free films dried from coating solutions with 3% w/w solid content, either HPMC alone or HPMC incorporated with varying concentrations of an additive, were performed. An ideal film should be hard, tough and extendible, characterized by high tensile strength, high elastic modulus and moderate elongation.[5,19] The maximum stress at break, a measure of the tensile strength, is an indicator of the abrasion resistance of the film.[20] HPMC-PVA films showed a downward trend in tensile strength at higher concentrations of PVA (Fig. 6a). It had been reported in other studies that PVA decreased film hardness and elastic modulus but not to the same extent as HPMC-PEG films.[5] PEG affected the tensile strength of HPMC films adversely, reducing the tensile strength of HPMC films by more than 60% when 30% PEG was incorporated. Elastic or Young's modulus is a measure of the stiffness or rigidity of the film, an indicator of its resistance to mechanical deformation.[21] Figure 6b shows that the additives used in this study reduced the elastic modulus of HPMC films, with PEG showing the most pronounced effect. PVA showed little change above the 10% concentration. Figure 6c shows the work of failure or energy used in straining the sample to failure and is related to film toughness. This property is important in film coating as it relates to the ability of the coating to withstand shock loads without affecting adversely the integrity of the film.[6] Work of failure was found to increase initially at concentrations of additives of less than 10%. At concentrations of 10% and higher, there was a general decline in work of failure for HPMC films containing PEG or PVA. The work of failure for HPMC-S630 films was highest of the three additives used. For concentrations between 5 to 10%, the decrease in work of failure from 5 to 10% was slight compared to the other additives. Mechanical properties of HPMC-S630 films above the 10% S630 concentration were not evaluated because they adhered very strongly to the surface of the glass dish, making it almost impossible to remove the test samples. This strong adhesion was not observed with the films containing PEG or PVA. From these results (Fig. 6), it could be seen that S630 had the least effect in reducing the elastic modulus of HPMC and was most influential in improving film toughness. Hence, S630 effectively promoted the formation of hard and tough HPMC films. Reduction in film properties may give rise to problems such as cracking, resulting in lost of integrity of the film coat.[16] The decrease in mechanical properties with the incorporation of PVA beyond 10% concentration showed that PVA had a positive influence only at 10% or less, the level at which the components of the blend are compatible. PEG exerted an adverse influence in mechanical properties, such as tensile strength, elastic modulus and work of failure. It had been reported that plasticizers, such as PEG, often used in HPMC formulations to enhance film flexibility, adversely reduce hardness and tensile strength, thus lowering the resistance to high-speed packaging and handling.[7]

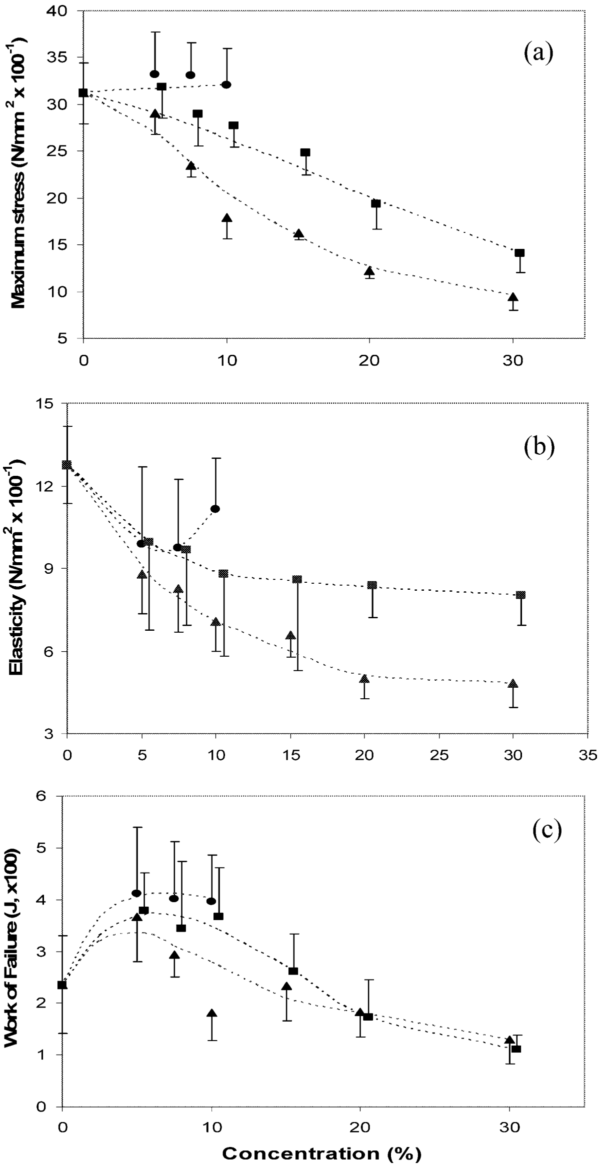


Fig. 6. Effect of PEG, PVA and S630 on (a) Tensile Strength, (b) Elasticity and (c) Work of Failure of HPMC Films

PEG (▲) and PVA (■) and S630 (●). $n=10$; bars are ±1 standard deviation.

Difference in Film Coat Thickness of HPMC Films with and without 10% Additive Aqueous film coating was carried out on caplets in a vertical coater to produce film

Table 2. Film Coat Thickness of Different HPMC E3 Formulations

Film type	HPMC	HPMC+S630	HPMC+PVA	HPMC+PEG
	Thickness (μm)			
Mean	84	82	86	84
S.D.	7	5	5	6

coat thickness of 82 to 86 μm (Table 2). The quantity of solid content in the coating solution was a major factor in determining the thickness of film coat. Different additives in the HPMC solution did not alter the coat thickness significantly. PEG had minimal effect on film coat thickness while PVA increased and S630 decreased the coat thickness marginally by 2 μm.

Effect of Additives on Film Coat Adhesion The film coating process should produce a film coat in intimate contact with the core substrate.[1)] The force of adhesion and area under the stress–strain curve can provide an accurate quantitative measure of the film-tablet adhesion. The force of adhesion is the force required to remove the film coating from the tablet.[10)] Strong adhesion is evidenced by the increase in adhesive force. Increased adhesive toughness is reflected by the increase in area under the curve or the work done to detach the film from the surface of the solid core.[9,10)] Thus, adhesive toughness of the polymer in conjunction with the force of adhesion, provides a more complete understanding of the mechanisms involved in the adhesion process.

It is imperative that a good film coat has strong substrate adhesion in addition to good film properties. The measurement of adhesion is extremely difficult due to the limited surface area and geometry of tablets. A method to determine film coat adhesion was devised by the use of elongated caplets that, after truncating the two semi-circular ends, allowed a length of film coats for detachment and measurement of adhesion force. The film coat was cut transversely prior to detachment with an adhesive tape over the film coat. Both S630 and PVA enhanced the force of adhesion in HPMC film coats as indicated by the maximum stress in the stress–strain data obtained from the modified butt adhesion technique (Fig. 7a). The increase was approximately 37±1% higher than the film coat comprising of HPMC without any additive. Adhesive toughness, indicated by energy for the work of failure (area under the stress–strain curve) or work done to detach the film from the caplet core, was highest for HPMC-S630, followed by HPMC-PVA films (Fig. 7b). However, only S630 enhanced the toughness HPMC film significantly. Both adhesive force and toughness were reduced in HPMC films containing PEG. From the results obtained on the tensile test of cast films and adhesion test of film coats, films plasticized with PEG were adversely affected, with reduction in both the mechanical and adhesive properties of HPMC film, whereas PVA and S630 enhanced these properties. Clearly, S630 showed an overall advantage in the force of adhesion as well as adhesive toughness of HPMC E3 films when compared to the other two additives evaluated.

Conclusion

From this study, S630 was shown to function both as a plasticizer and copolymer in HPMC film coating formulations. Its influences in reducing the glass transition temperature and viscosity of HPMC solutions were similar to PEG, a well-recognized plasticizer for HPMC. These effects of plasticization on coating solutions are beneficial as the drying temperature is lowered and less energy is required to spray the coating solution.

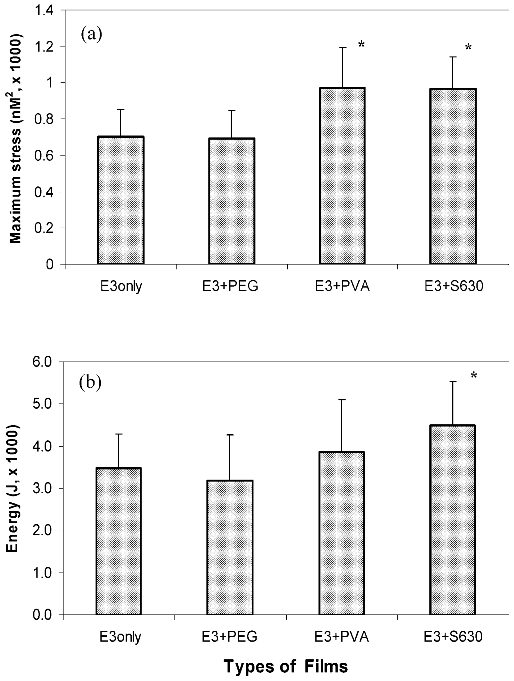


Fig. 7. Adhesive Strength (a) and Adhesive Toughness (b) of HPMC Film Coat without and with PEG, PVA and S630

* Significant difference with HPMC film (ANOVA, $p<0.05$) $n=20$; bars are ±1 standard deviation.

The tensile strength and elastic modulus of HPMC films were decreased with the inclusion of PEG and PVA although the effect by the latter was comparatively less. The effect of S630 in reducing the mechanical properties of free films was minimal, as compared to PEG or PVA. At 10% concentration, S630 reduced the film elasticity slightly but increased both the tensile strength and work of failure of HPMC films. The most prominent influence of S630 on HPMC films was the increase in substrate adhesion as evidenced by the cast films adhering tightly to the glass surfaces. When S630 was added at 10% by weight of the total solid content, the resultant film coat had enhanced adhesive property as shown by the butt adhesion technique. In conclusion, S630 showed promise in improving HPMC film coats, providing clear and smooth appearance as well as good mechanical and adhesive properties without adversely affecting the water vapour permeability.

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