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Quantitative estimation of internal stress development in aqueous HPMC tablet film coats

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Abstract

Data have been produced on the parameters which influence the development of internal stresses within aqueous HPMC film coats plasticized with glycerol in an attempt to calculate the magnitude of internal stress generation. Films have been prepared under conditions which equate to those experienced during film coating processes. The changes in mechanical and thermal properties of fresh films formed at different tablet bed conditions have been studied during their ambient storage. The contribution of internal stresses from three sources are calculated individually and a single equation is used to estimate the total internal stress within a film. This equation considers the contribution made by the volumetric changes of the tablet core during ambient conditions in addition to those caused by other well-established mechanisms. The results of these calculations showed that the total internal stress is influenced, not only by these volumetric changes, but also by the conditions in which the film coating process was performed. The possibility of the film coating undergoing failure is also discussed.

Keywords: Aqueous film coating; Internal stress; Coating defects; Mechanical properties; Thermal analysis

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1. Introduction

During pharmaceutical tablet film coat formation from polymeric solutions, some residual internal stresses are generated within the plane of the film. The origins, development and distribution of stresses within thin films and the interrelationship with film mechanical properties have been discussed by Hoffman (1981). Internal stresses have been shown to be due to (i) shrinkage of the film after its solidification point as further solvent evaporates from the system (Croll, 1979), (ii) the differences between the thermal expansion coefficient of the film coat and the tablet substrate, and the difference between the glass transition temperature of the film coat and the actual temperature of the film at any time (Sato, 1980) and (iii) any volumetric changes in the tablet core occurring during coating and subsequent storage (Rowe, 1983; Okutgen et al., 1991b). Many studies have shown that adhesion and cohesion related film coating defects are the manifestations of these internal stresses building

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up within the film coat to the point where the total internal stress exceeds the tensile strength of the film causing defects, such as cracking, edge splitting and peeling to occur (Rowe, 1981a, 1992). Additionally, as the internal stress exceeds the adhesive forces acting at the film coat/tablet substrate interface, local detachments and bridging of any intagliations may result (Rowe, 1981b). Equations are given in the literature to calculate the magnitude of the internal stresses resulting from individual sources (Croll, 1979; Sato, 1980; Rowe, 1983). These equations have been utilised to predict the incidence of internal stress related film coating defects (Rowe, 1981a, 1983; Okhamafe and York, 1985). However, the quantification of internal stresses in these studies is based on assumed film coatings applied to assumed tablet cores, and possible dimensional changes of the tablet cores have received little attention as an origin of internal stress.

The present work was designed to measure the mechanical, physical and thermal properties of freshly formed films and those which have been exposed subsequently to ambient storage conditions and to use these data in calculations of the total internal stress (P) expected to develop within the film coats during the coating process itself and during ambient storage of the coated tablets. Eq. 1, which considers the contribution of volumetric changes of the tablet core (Okutgen et al., 1991b) in addition to the other well-established mechanisms, is used in this present work to calculate total internal stress using laboratory-determined data.

$$P = \frac{E}{3(1-\nu)} \cdot \left[\left(\frac{\phi_S - \phi_R}{(1-\phi_R)} \right) + \Delta\alpha_{(\text{cubic})}\Delta T + \Delta V/V \right] \quad (1)$$

In Eq. 1, E is the elastic modulus of the film, ν denotes its Poisson's ratio, ϕ_S is the volume fraction of solvent at the solidification point of the film, ϕ_R represents the volume fraction of solvent remaining in an 'air dry' film, $\Delta\alpha$ is the difference between the cubical coefficient of thermal expansion of the film coat (α_c) and the substrate (α_S), ΔT denotes the difference between the glass transition temperature of the film (T_g) and the temperature of the film during manufacture or storage (T), ΔV is the volumetric change of the tablet core and V represents the original volume of the tablet core.

2. Materials and methods

Free films were cast from aqueous solutions containing 10% w/w hydroxypropyl methylcellulose (HPMC) and 2% w/w glycerol. This resulted in approx. 15% w/w plasticizer in the 'air dried' film. The films were cast on glass plates using a film applicator (similar in design to those used for thin-layer chromatography) whose clearance was selected to produce dry films with a thickness of approx. 65 μm. Film casting and drying were carried out within a Controlled Environment Cabinet (Fisons Scientific Apparatus, UK) which was capable of simulating the conditions of a tablet bed during actual film coating process. Two conditions were selected, 30°C/45% RH and 45°C/20% RH, based on the results of a previous study (Okutgen et al., 1991a) which evaluated the temperature and humidity conditions within a perforated-drum tablet coater during actual film coating processes. Actual mean conditions over the simulation periods were 30.1°C/45.1% RH and 44.9°C/20.5% RH.

After the films were formed, but whilst still under simulated coating/drying conditions, strips were cut using a standard stainless-steel template (ASTM D882-75b). Each strip was marked, peeled from the glass, its thickness measured in five places. Only films with a uniform thickness between 65 and 67 μm were used in subsequent mechanical testing. These were placed within individual paper envelopes in order to prevent curling during storage. Three randomly selected strips were left in the simulated coating conditions to be used for immediate (zero-time) tests and the remainder were transferred into a controlled temperature and humidity cabinet for 'ambient' storage at 25°C and 50% RH. The humidity of the ambient conditions was controlled using a saturated salt solution of magnesium nitrate (Nyqvist, 1983). The films were exposed to these conditions for up to 48 h, at which time earlier experimentation (Okutgen et al., 1991b) had shown dimensional stability of the tablet cores. Various tests were performed at 6, 24 and 48 h after their formation. At each of these time points, three randomly selected strips obtained from the same film sheet were assessed for elastic modulus, tensile strength, strain at break and T_g. Additionally, the water content of the film at solidification and at 'air dryness' and linear coefficients of expansion were determined.

The tensile testing of films was performed using a Monsanto Tensometer 10 (a tensile/compression testing machine, Monsanto, UK) during which each test sample was surrounded by a controlled temperature and humidity cabinet. For the immediate tensile testing of freshly formed films, the testing environment was equilibrated to the simulated coating conditions, i.e., either 30°C/45% RH or 45°C/20% RH using saturated solutions of chromium trioxide or potassium acetate, respectively (Nyqvist, 1983). Following these 'zero-time' tests, the environment for tensile testing was changed to ambient in order to perform the tests on films which had been stored under such conditions. Running the tests at the same environmental conditions at which the films had been equilibrated is necessary since HPMC films rapidly interchange water with their environment and this, in turn, markedly affects their mechanical properties (Aulton, 1994).

The moisture content of the films was measured using an Infrared Moisture Determination Apparatus (Mettler, Switzerland). These data were used in the determination of the weight fraction of residual solvent (water) at the solidification point and when air dried.

The thermal properties of films were investigated using a Thermomechanical Analyser (TMS-2, Perkin-Elmer, USA). The T_g determinations were made using the instrument in its penetration mode with a 7 g load, and the coefficient of thermal expansion of the film (α_c) and the tablet cores (α_S) were determined in expansion mode with 3 g load application. For determination of α_c, 980 μm thick film samples were prepared in order to obtain a satisfactory linear expansion with increasing temperature. Measurements of α_S were performed on tablet cores which had been stored at ambient laboratory conditions for 1 year. It was assumed that these tablets were in a completely relaxed state and thus dimensionally stable.

Previous work (Okutgen et al., 1991b) has quantified the time-dependent volumetric changes of maize starch tablets. These compacts were initially 3.1 mm thick, 10 mm diameter, flat-faced, round tablets compressed from maize starch BP at an upper punch speed of 25 mm min^{-1} up to a maximum compaction pressure of 100 MPa. The subsequent dimensional changes of these compacts during exposure to actual film coating conditions and during subsequent ambient storage for 48 h have been used here for calculation of P_V (the stress created within the film as a result of volumetric changes of tablet core).

3. Results and discussion

3.1. Mechanical properties

Measured values for the tensile strength and modulus of elasticity of the films under test at various time, temperature and humidity conditions are listed in Table 1. Data for two time periods are detailed. The zero-time data, i.e., zero post-coating storage time, are for films prepared, dried and tested at either 30°C/45% RH or 45°C/20% RH to simulate realistic tablet coating conditions. Tensile testing was also performed on replicate films after their subsequent ambient storage at 25°C and 50% RH for 6, 24 and 48 h. There was little difference in the measured properties after the first 6 h of storage under these conditions and thus the mean of the measurements between 6 and 48 h is presented in the '6–48 h' rows of Table 1. This was generally true of other measured mechanical and physicochemical data generated on films and thus the mean of the 6–48 h data is presented where appropriate in subsequent tables.

When first formed, the films produced at 45°C/20% RH are slightly stronger and have a greater modulus of elasticity than those prepared at 30°C/45% RH. This effect results from the

Table 1
Mechanical properties of aqueous HPMC films containing approx. 15% w/w glycerol

Coating temperature and humidity	Storage time at 25°C and 50% RH (h)	Tensile strength of film (σ) (MPa)	Modulus of elasticity (E) (MPa)	$E/3(1-\nu)$ (MPa)
30°C and 45% RH	0 [a]	27.45	817	419
	6–48	27.39	864	443
45°C and 20% RH	0 [a]	30.35	929	476
	6–48	26.53	823	422

[a] 'Zero-time' tensile evaluations were performed under the simulated coating conditions.

differences in the moisture content of the films at this point. Comparison of the weight fractions (w_R) recorded in Table 2 shows that the freshly formed 45°C/20% RH films have a lower moisture content than films prepared at 30°C/45% RH. The moisture sensitivity of HPMC film mechanical properties due to the plasticizing effect of water has been recorded previously (reviewed by Aulton, 1994). During ambient storage and subsequent water uptake, both films equilibrate to similar mechanical properties.

3.2. Estimation of internal stress

From data obtained in this study, the internal stresses expected to develop in the plane of an HPMC film containing glycerol and applied on commercial-sized, flat-faced maize starch tablets under different tablet bed conditions could be calculated. In the calculation of the first element of the internal stress equations (i.e., $E/3(1-\nu)$), the Poisson's ratio of the film was taken as 0.35 since this value has been reported to approximate the actual value for many polymeric materials (Croll, 1979; Rowe, 1981a; Stanley et al., 1981) and the elastic modulus E was determined from tensile evaluations. Values for the quotient $E/3(1-\nu)$ for the films studied are shown in Table 1.

The following sections discuss the calculated internal stress values arising from various sources in fresh films formed under conditions which simulate tablet bed conditions during a coating process and the changes in these values during subsequent ambient storage.

3.3. Estimation of internal stress resulting from solvent evaporation (P_S)

The formula for the calculation of internal stress resulting from the volumetric strain (shrinkage) of the polymer film due to the evaporation of solvent (P_S) is given by Eq. 2 (Croll, 1979).

$$P_S = \frac{E}{3(1-\nu)} \cdot \left(\frac{\phi_S - \phi_R}{1-\phi_R} \right) \tag{2}$$

In order to calculate P_S, one needs to know the values of ϕ_S (the volume fraction of solvent at the solidification point of the film) and ϕ_R (the volume fraction of solvent remaining in an air dry film). Measurements performed on film samples

Table 2
Calculation of internal stress (P_S) resulting from shrinkage of the film around the tablet

Coating temperature and humidity	Storage time at 25°C and 50% RH (h)	Volume fraction of water at solidification (ϕ_S)	Weight fraction of water when 'air dry' (w_R)	$(\phi_S - w_R)/(1-w_R)$	P_S (Eq. 2) (MPa)
30°C and 45% RH	0 [a]	0.162	0.129	0.0374	15.69
	6–48	0.162	0.148	0.0160	7.06
45°C and 20% RH	0 [a]	0.162	0.074	0.0955	45.44
	6–48	0.162	0.129	0.0376	15.94

[a] 'Zero-time' evaluations were performed on films immediately after removal from the simulated coating conditions.

which had been dried under infrared heating conditions to a constant weight showed that the true density of these absolutely dry samples was 1.055 g cm^{-3}. Since the density of the films which contain residual water (i.e., air dry samples) would be even closer to unity, it was thought reasonable to use weight fraction (w_S and w_R) of the residual water in films instead of the corresponding volume fractions (ϕ_S and ϕ_R) which are difficult to assess easily in practice.

Due to practical difficulties in determining the exact solidification point, values of w_R were obtained which resulted in unrealistically high values of P_S, indicating that the internal stress would greatly exceed the measured strength of the films reported in Table 1. This would indicate that such films would be unserviceable in practice, yet this is clearly not the case since HPMC/glycerol films are used frequently and successfully.

Croll (1979) suggested that ϕ_S can be determined as the solvent concentration at which the T_g of the polymer-solvent mixture coincides with ambient temperature. Using this approach, he determined the ϕ_S of polyisobutyl methacrylate films as 0.162. Later, Rowe (1981a) used this value for estimation of internal stress development in a HPMC film. Due to the practical difficulties encountered in determining ϕ_S and w_S in this study, this value of 0.162 was also taken as the volumetric fraction of water in the films at solidification point (ϕ_S). The corresponding data and calculations are presented in Table 2.

Table 2 lists the weight fraction of water in the films formed at 30°C/45% RH and 45°C/20% RH immediately after manufacture and then after subsequent ambient storage. The moisture content of fresh films increased on exposure to ambient conditions until they reached a plateau after 6 h.

The values of P_S will be reduced as the difference between ϕ_S and w_R decreases. This can be seen from Table 2 when the P_S values under different film forming conditions and at ambient storage are compared. The films formed at 45°C/20% RH had a larger initial difference between ϕ_S and w_R and the resulting initial internal stress was much higher than for films formed at 30°C/45% RH.

In both cases, the highest internal stress was developed in the fresh films and this stress decreased during ambient storage. This was a consequence of less residual water in the films when they were freshly formed. As the films were exposed to ambient storage, the $(\phi_S - w_R)$ value was reduced as increasing amounts of moisture were absorbed, thus P_S is also reduced. Croll (1979) suggested that coating systems should be chosen such that ϕ_S and ϕ_R are close together, in order that the polymer chains are free to move for a longer time before the solidification point is attained during solvent evaporation. This would also give more time for cohesive and adhesive bonds to be established within the polymer matrix, and between the film and the substrate, respectively. Since the value of ϕ_S is not influenced by the drying conditions, the development of P_S in a freshly formed film can only be controlled by the parameter w_R (or more correctly ϕ_R). This parameter was higher, and thus closer to the ϕ_S value, when the films were formed at higher relative humidities. Although the magnitude of P_S developed in fresh films which were formed at low relative humidity conditions (45°C/20% RH) was reduced significantly as the film equilibrated to ambient conditions, the high initial P_S value increased the risk of a possible failure occurring in these films immediately after the coating process.

3.4. Estimation of internal stress resulting from thermal strain (P_T)

The internal stress created as a result of thermal strain (P_T) is given by Eq. 3 (Sato, 1980):

$$P_T = \frac{E}{3(1-\nu)} \cdot \Delta\alpha_{(\text{cubic})} \Delta T \tag{3}$$

For a given tablet substrate and film coating system, ν and $\Delta\alpha$ are constant, thus P_T would vary only as a function of E and ΔT. In this study, the linear expansion coefficients of the film and the maize starch tablet were determined as 1.0×10^{-4} and 0.52×10^{-4} °C^{-1}, respectively, giving to the first approximation volumetric (cubic) coefficients of 3×10^{-4} and 1.56×10^{-4} °C^{-1}, respectively, and thus a $\Delta\alpha_{(\text{cubic})}$ of 1.44×10^{-4} °C^{-1}. The effects of the temperature

Table 3
Calculation of internal stess (P_T) resulting from thermal effects

Coating temperature and humidity	Storage time at 25°C and 50% RH (h)	$\Delta\alpha_{\mathrm{cubic}}$ (°C^{-1})	Glass transition temperature (T_g) (°C)	Temperature of film on tablet (T) (°C)	ΔT ($T_g - T$) (°C)	P_T (Eq. 3) (MPa)
30°C and 45% RH	0 [a]	1.44×10^{-4}	133	30	83	5.01
	6–48	1.44×10^{-4}	133	25	88	5.61
45°C and 20% RH	0 [a]	1.44×10^{-4}	118	45	73	5.01
	6–48	1.44×10^{-4}	118	25	93	5.65

[a] 'Zero-time' evaluations were performed on films immediately after removal from the simulated coating conditions.

and humidity of storage on the glass transition temperature and calculated P_T values of films cast at 30°C/45% RH and at 45°C/20% RH are listed in Table 3.

The measured glass transition temperature of the film samples was influenced by the film formation conditions but was unaffected by subsequent ambient storage. Table 3 shows that less internal stress, P_T, was generated in the films when they were freshly formed. Storage under ambient conditions resulted in the further development of P_T in these films. When the film was freshly formed, the coating temperature was higher than ambient, thus closer to the T_g of the film. Although the elastic modulus of the film was the highest at that point (Table 1), the ΔT was much lower than it was during the ambient storage period; this results in the smaller P_T generation within fresh films.

Analysis of Table 3 shows close similarity between calculated P_T values for films formed at 30°C/45% RH and at 45°C/20% RH. This can be explained by the counteracting mechanisms affecting both T_g (and hence ΔT) and modulus of elasticity of the films. In the case of 30°C/45% RH films, as the drying temperature is close to ambient, changes in ΔT on subsequent ambient storage are small. These films possess a low elastic modulus due to the presence of greater amounts of retained solvent. In the case of films formed at 45°C/20% RH, these mechanisms work in the opposite direction and the resulting P_T values coincided with each other.

3.5. Estimation of internal stress resulting from expansion of the tablet core (P_V)

Estimation of internal stress created by expansion of the tablet core (P_V) can be made by use of Eq. 4 (Rowe, 1983):

$$P_V = \frac{E}{3(1-\nu)} \cdot \Delta V/V \qquad (4)$$

The results of a previous study (Okutgen et al., 1991b) showed that tablets undergo substantial

Table 4
Calculation of internal stress (P_V) resulting from volumetric changes in the core

Coating temperature and humidity	Storage time at 25°C and 50% RH (h)	Film thickness change (Δt) (%)	Tablet core volumetric strain ($\Delta V/V$)	P_V (Eq. 4) (MPa)
30°C and 45% RH	0 [a]	0	14.2×10^{-3}	5.95
	0.75	–	3.7×10^{-3}	1.56
	6	2.31	7.5×10^{-3}	3.21
	24	2.54	12.4×10^{-3}	5.50
	48	2.43	13.0×10^{-3}	5.94
45°C and 20% RH	0 [a]	0	-6.1×10^{-3}	−2.91
	0.25	–	-7.5×10^{-3}	−3.58
	6	4.48	16.1×10^{-3}	6.92
	24	4.93	32.2×10^{-3}	13.73
	48	5.80	37.6×10^{-3}	15.62

[a] 'Zero-time' evaluations were performed at the end of the storage period under the simulated coating conditions.

amounts of dimensional change both during and after film coating. The magnitude of these changes is influenced by air temperature and humidity variations occurring at different stages of the process. These dimensional changes for maize starch tablet cores are summarised in Table 4. The volumetric expansion of the substrate as a result of moisture sorption and stress (and therefore strain) relaxation during ambient storage will cause P_V development within the film coat. There will be some relief in this stress due to the simultaneous swelling (and therefore volumetric expansion) of the coat itself (Table 4). This, however, is already taken into account within the calculations of P_S. These calculations (Table 2) indicate reduced internal stress as the amount of water included in the film increases.

It has been observed previously (Okutgen et al., 1991b) that as soon as tablets of maize starch, which had been warmed by the coating process, are subsequently exposed to ambient temperature and humidity conditions they undergo an abrupt contraction prior to their long-term expansion. This contraction takes place over approx. 45 min for the tablets exposed to 30°C/45% RH and over about 15 min for those exposed to 45°C/20% RH coating conditions. Since this decrease in volume would release some of the already developed P_V, this factor is also taken into account in calculations.

During exposure of the maize starch tablets to 30°C/45% RH, they expanded in both their axial and radial dimensions (Okutgen et al., 1991b). In fact, this expansion of tablets during simulated coating conditions was greater than subsequent expansion of the tablets during their ambient storage, thus more stress was generated in the fresh films compared to the stress development during storage (Table 4).

In comparison, the maize starch tablets exposed to 45°C/20% RH conditions for 1 h undergo contraction (Okutgen et al., 1991b). This contraction, together with the additional contraction occurred on immediate exposure of tablets to ambient conditions, resulting in negative volumetric changes and a negative P_V (Table 4). If these tablets were coated with the film studied here, this would cause a stress relief of up to 3.58 MPa within the coat. During ambient storage, however, the expansion of tablets created increasing amounts of P_V with time. In Table 4, P_V increased in the film despite the decrease in E resulting from ambient storage (Table 1). This suggests that the changes occurring in the volume of the tablet core were a more efficient factor controlling the magnitude of P_V than the elasticity of the film.

3.6. The total internal stress (P)

Table 5 tabulates the total internal stress from a summation of the contributions of P_S, P_T and P_V.

The maximum stress was generated in each case in the freshly formed films. The major part of this stress was built up as a result of shrinkage of the film after its solidification point during solvent evaporation (P_S). This stress was reduced

Table 5

Coating of total internal stress (P) within a film coat around the tablet core summated from the contribution of P_S, P_T and P_V

Coating temperature and humidity	Storage time at 25°C and 50% RH (h)	Calculated internal stress (MPa)			
		P_S	P_T	P_V	Total P
30°C and 45% RH	0 [a]	15.69	5.01	5.95	26.64
	6	7.06	5.61	3.21	15.88
	24	7.06	5.61	5.50	18.17
	48	7.06	5.61	5.94	18.61
45°C and 20% RH	0 [a]	45.44	5.01	−2.91	47.54
	6	15.94	5.65	6.92	28.51
	24	15.94	5.65	13.73	35.32
	48	15.94	5.65	15.62	37.21

[a] 'Zero-time' tensile testing and other evaluations were performed under the simulated coating conditions.

significantly on early exposure of the product to ambient storage by moisture uptake into the film structure which increased the volume of the film. However, on extended storage, as a result of the expansion of the tablet core, P_V became the dominant factor in the development of the internal stress, P. Internal stress generated by thermal strain appears to have a moderate influence on the generation of P. The results show that the magnitude of P expected to develop in the films was greatly influenced by the conditions in which the films were formed and this magnitude changes significantly on exposure of the product to subsequent ambient storage conditions.

3.7. Comparison of the measured mechanical strength of the polymer films (σ and σ / E) and the estimated total internal stress (P)

If the internal stress within the film is equal to or greater than the mechanical strength of the film (i.e., $P \geq \sigma$) it will probably crack and show other defects. Table 6 presents a comparison between the total internal stress (P) expected to develop in fresh films and during their ambient storage and the corresponding measured tensile strength (σ) of the film. A film produced with this set of properties would undergo a severe cohesive failure if it had been manufactured under tablet bed conditions of 45°C/20% RH; all $P - \sigma$ values listed in Table 6 are significantly positive indicating an excess internal stress. In the case of films produced under tablet bed conditions of 30°C/45% RH, however, these values are negative indicating that the mechanical strength of the film (σ) exceeds the internal stress and thus the film coat is capable of tolerating the moderately developed internal stresses.

The use of the $P \geq \sigma$ relationship discussed above to assess whether the internal stress within the film is equal to or greater than its mechanical strength requires quantitative knowledge of all the factors comprising Eq. 1. Because of the difficulty in routinely measuring these components, Eq. 1 can be written in the following rearranged form (Eq. 5, Okhamafe and York, 1985):

$$\frac{\sigma}{E} \leq \frac{1}{3(1-\nu)} \cdot \left[\left(\frac{(\phi_S - \phi_R)}{(1-\phi_R} \right) + \Delta\alpha_{(\mathrm{cubic})} \Delta T + \Delta V / V \right] \qquad (5)$$

The quotient of the tensile strength of the film divided by its modulus of elasticity, the so-called σ/E value, has been shown to be a useful predictor of film performance (Rowe, 1982; Okhamafe and York, 1985). In this work however, the value of σ/E was found to change very little with changes in coating and storage conditions (Table 6).

Table 6 lists values for the right-hand side of Eq. 5 (referred to as A in Table 6) which have

Table 6
Calculation of differences in film strength and calculated total internal stress (P) within a film coat

Coating temperature and humidity	Storage time at 25°C/50% RH (h)	Total internal stress (P) (MPa)	Tensile strength of film (σ) (MPa)	Excess internal stress ($P - \sigma$) [b] (MPa)	σ/E	'A' (right side of Eq. 5)	Difference ($A - \sigma/E$) [b]
30°C and 45% RH	0 [a]	26.64	27.45	−0.80	0.034	0.033	−0.001
	6	15.88	27.39	−11.23	0.032	0.019	−0.013
	24	18.17	27.39	−8.94	0.032	0.021	−0.011
	48	18.61	27.39	−8.50	0.032	0.022	−0.010
45°C and 20% RH	0 [a]	47.54	30.35	+17.19	0.033	0.051	+0.018
	6	28.51	26.53	+2.27	0.032	0.034	+0.002
	24	35.32	26.53	+9.08	0.032	0.043	+0.011
	48	37.21	26.53	+10.97	0.032	0.045	+0.013

[a] 'Zero-time' tensile and other evaluations were performed under the simulated coating conditions.

[b] Thus positive values indicate an internal stress in excess of the measured mechanical properties of the film.

been calculated from the experimental data generated in this work. It also lists the differences between this value and σ/E values. Negative values for $(A - \sigma/E)$ (as is the case for the films coated at 30°C/45% RH) indicate that the strength of the film in general exceeds the potential internal stress and thus these films should have satisfactory performance. Positive values (as with films formed at 45°C/20% RH) may be an indication of possible defects.

The comparison of total internal stress with the adhesion of the films is more complicated since the measured adhesion depends to a great extent on many other factors (e.g., nature of the tablet core excipients, porosity and surface roughness of tablet cores, film thickness, additives incorporated into the film, storage conditions). The measured adhesion of HPMC films is between the limits of 25 and 250 kPa for different film coating formulations applied on different types of tablet cores (Fisher and Rowe, 1976; Rowe and Forse, 1980; Porter, 1982). Hence, it is possible to suggest that an adhesive failure of the film due to internal stress generation may also occur.

4. Conclusions

The magnitude of the final internal stress developed within freshly formed and stored films was found to depend upon the interrelationship between many parameters related to both the coating and the tablet substrate (each of which is influenced by both formulation and processing). Internal stress caused by the volumetric expansion of tablet cores during ambient storage was found to be a significant parameter amongst other well-established mechanisms. The internal stress calculations using the parameters obtained in this study have shown that selection of elevated temperature and low relative humidity tablet bed conditions exacerbate the internal stress development in film coats.

The possibility of defects such as cracking and edge splitting occurring depends on whether the strength of the film coat can withstand the internal stress created at any phase of the film coating process. Films usually have a higher tensile strength when they are freshly made and they tend to become weaker as they absorb atmospheric moisture.

These findings emphasize the importance of the effect of film coating conditions and storage environment on the internal stress generation in the film coat and subsequent film coating defects.

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