integration characteristics of gelatin in mixtures of the two substances. This finding suggests that the change in disintegration of gelatin derives from a physical change within the tablet subcoat, and not from a chemical reaction between calcium ion and gelatin.

Theimer (1960) has demonstrated by electron microscopy, changes in gelatin fibre structure correlated with ageing. The observed heat-induced changes in the tablet subcoat could involve an initial expansion of the gelatin fibre network that results in trapping of calcium carbonate to form an insoluble matrix. Although this process probably also occurs in the presence of soluble salts, the latter would effect water transport through the matrix and prevent poor disintegration.

Batches of tablets prepared with the original gelatin-acacia-sucrose syrup, but combined with an alternative chalk-free dusting powder, have shown satisfactory disintegration times after accelerated storage.

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Microindentation - a method for measuring the elastic properties and hardness of films on conventionally coated tablets

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A knowledge of the stress-strain characteristics of films is useful in the development of suitable film coatings for tablets especially in comparing film samples as a function of formulation variables, e.g. polymer type, plasticizer type and concentration, solvent system and the effect of fillers and colouring agents. These data are usually determined by measuring the elongation of a strip of film under increasing load forces using a tensile tester (Munden, De Kay & Banker, 1964; Allen, De Marco & Kwan, 1972). The technique involves the casting or spraying of films onto various substrates (e.g. mercury or polytetrafluoroethylene) from which they are removed before testing. Ridgway, Aulton & Rosser (1970) described a pneumatic microindentation apparatus (Research Equipment (London) Limited) which could measure both the elastic modulus and surface hardness of a sample the size of a tablet. Since the apparatus was originally developed for the testing of paint films it was decided to evaluate its potential for use in measuring the elastic modulus and Brinell hardness number of films on conventionally coated tablets.

The apparatus used was essentially the same as that used by Ridgway & others (1970) and consisted of a spherical indenter, diameter 1·55 mm, which could be lowered on to the test surface under a selected load of a few grams. The depth of indentation and the recovery when the load was removed was measured by a double pneumatic recorder with a full scale deflection corresponding to a 6 μm movement in the indenter. The results were calculated using the equations derived by Ridgway & others (1970). Since in their derivation of the equation for the modulus of elasticity, the authors used a value of 0·3 for Poisson's ratio (an unknown variable for cellulose based coatings), the values for this modulus are only comparable within a series of similar materials and cannot be considered absolute. The individual results, therefore, cannot be directly compared with those obtained from stress-strain data but the trends should be similar.

Flat faced tablets 11·1 mm diameter were coated using a formulation containing hydroxypropyl methylcellulose (Shinetsu Chemical Company, Japan) and variable amounts of plasticizer dissolved in a dichloromethane–methanol solvent mixture. The formulation was applied to the tablets in a 15 cm diameter Wurster column. The tablets were dried and stored at room temperature and 50° R.H. for two weeks before testing. Measurements were made on 20 tablets using an indentation load of 5 g.

As a preliminary study indentations were made on films cast on a glass substrate and compared with those made on the film coated tablet. No significant difference could be found between the measurements, indicating that there was no substrate interference with a coating some 30–40 μm in thickness.

Table 1. *The effect of the concentration of glycerol and propylene glycol on the elasticity and hardness of a hydroxypropyl methylcellulose film.*

Plasticizer	Concentration in film % w/w	Young's modulus of elasticity MPa	Brinell hardness number MPa
Unplasticized film	—	135·7	3·53
Glycerol	5	125·5	3·53
Glycerol	10	116·0	3·33
Glycerol	20	99·1	3·04
Propylene glycol	5	114·0	3·04
Propylene glycol	10	108·1	3·04
Propylene glycol	20	102·5	2·84

Some results showing the effect of increasing amounts of the plasticizers, glycerol, and propylene glycol and the effect of increasing the molecular weight of the plasticizer polyethylene glycol on the elastic modulus and Brinell hardness number are shown in Tables 1 and 2. The coefficient of variation of the results (20–25%) is comparable with that reported by Ridgway & others (1970) but as pointed out by them, the measurement technique is more precise than such a variation indicates and almost all the experimental scatter is caused by the intrinsic variability in the sample.

It can be seen from the results that for the plasticizers propylene glycol and glycerol, increasing the concentration in the film leads to a softer more elastic film.

This is to be expected, since both materials contain hydroxyl groups and can therefore associate with the hydroxyl groups of the hydroxypropyl methylcellulose there by decreasing the forces of attraction between the polymer chains and effectively extending and softening the structure. As the concentration of the plasticizer is increased, more hydroxyl groups on the hydoxypropyl methylcellulose will be associated with the plasticizer and the film will become more elastic. A similar effect will occur with the low molecular weight polyethylene glycols (Table 2), but as the molecular weight and the size of the polyethylene glycol is increased the mole fraction of the hydroxyl groups available to associate with the hydroxyl groups of the hydroxypropyl methylcellulose will decrease and the film will become less elastic. The molecular weight of the polyethylene glycol does not appear to have any significant effect on the hardness of the film but in all cases the hardness is less than that of the unplasticized film.

Table 2. *The effect of the molecular weight of polyethylene glycol on the elasticity and hardness of a hydroxypropyl methylcellulose film.* The plasticizer concentration was 20% w/w.

Molecular weight	Young's modulus of elasticity MPa	Brinell hardness number MPa
200	91·0	2·94
300	94·8	2·84
400	100·6	2·45
600	102·1	2·75
1000	103·6	2·75
4000	119·5	3·43
6000	120·3	2·64
20000	141·0	2·94

The results show that differences in the elastic properties of films of different formulations can be highlighted by this method. Although the results have been calculated as a Young's modulus of elasticity, it is possible to compare a series of related samples just in terms of the indentation values obtained under some fixed condition of measurement.

This method offers several advantages over the tensile testing used by other workers: 1. The film to be tested is laid down under conventional coating conditions (e.g. spraying) and need not be removed from the substrate. Although flat faced tablets have been used in this work, biconvex tablets can be accommodated provided a suitable holder is used. 2. The apparatus is small enough to be enclosed in a glove box and measurements can be made at various temperatures and humidities, i.e. the thermomechanical properties of the films can be studied. This could well be of use in predicting the stability of the film formulation from measurements under accelerated storage conditions.

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