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The mechanical properties of some binders used in tableting

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Films of acacia, gelatin, methylhydroxyethyl cellulose, polyvinylpyrrolidone (PVP) and starch have been prepared with moisture levels of 2·0 to 20·0% w/w obtained by equilibration at relative humidities from 0 to 90%. Tensile strengths of 60 mm × 2 mm strips of these films have been determined by stressing to failure at a constant rate of $6{\cdot}6 \times 10^{-5}$ ms^{-1} in a tensile strength apparatus. From the stress-strain curves the modulus of elasticity, yield strength and the work done in breaking unit cross-sectional area of the films have been calculated. This has given an indication of the binders' mechanical behaviour over the range of moisture contents likely to be encountered in tableting.

Many materials have been considered for their use as binders in compressed tablets. Willis, Banker & DeKay (1965) investigated a variety of polymers as suitable binders in the granulation of powders. Other workers (Holstius & DeKay, 1952; Nelson, Arndt & Busse, 1957; Lehrman & Skauen, 1958) have evaluated binders by considering the properties of tablets produced using various binding agents. Mendes (1968) assessed the characteristics of eight binders by a "hardness-friabrasion" index of tablets produced using a given agent. He considered that artificial methods for evaluating binders outside tablet systems did not produce satisfactory conclusions, although no experimental evidence was presented. Little work has been reported on the mechanical properties of binders, but Schott (1970) measured the tensile strength of mixed films of cellulose and bentonite when studying the interaction between clays and cellulose. The purpose of this investigation has been to measure various mechanical properties of binders in film form over a range of moisture contents that would be expected to be found in pharmaceutical granules.

MATERIALS AND METHODS

The binders used were: Acacia B.P.; Gelatin (Cheshire Gelatins) lime treated, bloom strength 154 and B.P. quality; methylhydroxyethylcellulose, Tylose MH50 (Hoechst A.G.); polyvinylpyrrolidone (PVP), Plasdone K29–32 (G.A.F. Corp.); starch (maize) B.P.

An air-free solution of each binder was prepared. Films were cast by pouring the solution on to sheets of aluminium foil supported on glass plates held at an angle of 5–10° to the horizontal. The concentrations of the solutions used were acacia 40% w/w, gelatin 25% w/w, methylhydroxyethyl cellulose 4% w/w, PVP 50% w/w and starch 2·5% w/w. The films were dried at room temperature and the foil backing removed. This procedure consistently produced films of thicknesses between 30 and 85 μm, each with a constant thickness to ± 5 μm. Strips 60 mm × 2 mm were cut

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from the films with a scalpel. The strips were mounted on card supports with epoxy adhesive (Fig. 1). To prevent shear stressing when subsequently tested, the strips were carefully aligned with the punched holes in the card. To enable the cross-sectional area of each strip to be calculated, the strip widths were measured with a travelling microscope and their thicknesses were determined with a micrometer screw gauge. In addition, films were examined microscopically and any with flaws rejected. Different film moisture contents were obtained by allowing samples of 15 supported strips to equilibrate for 3 days in desiccators under relative humidities ranging from 0 to 90%. The desiccators were kept in an incubator at 25° ± 0·1°. Moisture contents were determined by drying to constant weight at 110°.

Tensile strength measurements were carried out by clamping a supported film strip to the tensile strength apparatus, Fig. 2. The apparatus consists of a heavy gauge frame supporting a calibrated load cell (Schaevitz, U.S.A.). A bracket having a

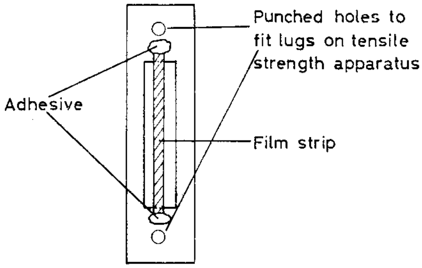

FIG. 1. Cardboard film support.

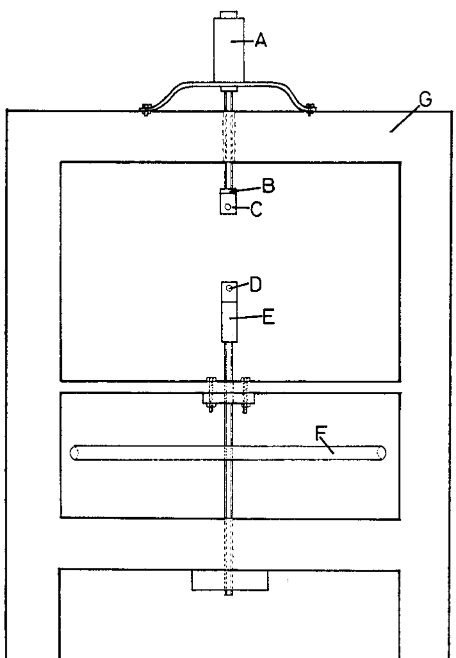

FIG. 2. The tensile strength apparatus. A, load cell. B, upper jaw. C, upper lug. D, lower lug. E, lower jaw. F, pulley. G, rigid frame.

short cylindrical lug is connected to the core of the load cell to form the upper jaw of the apparatus. Output from the load cell is amplified and fed to a chart recorder (Telsec Instruments Ltd., Oxford). The lower jaw has a similar lug but is connected to a belt-driven pulley and induction motor. Rotation of this pulley moves the lower jaw downwards. To effect a measurement with the apparatus, the punched holes at the ends of the card support were located over the two lugs, the sides of the card supports were cut away and the lower jaw driven downwards at a constant speed of $6{\cdot}6 \times 10^{-5}$ ms^{-1}. This movement strained the film at a constant rate and the stress-strain curve was plotted directly on the recorder chart. The ultimate tensile strength was calculated as the maximum load before fracture divided by the initial cross-sectional area of the strip. The work done in breaking unit cross-sectional area of the film was calculated from the stress-strain curve by determining the area under the curve with a planimeter and dividing this value by the initial cross-sectional area of the film. Young's modulus was determined from the initial slope of the stress-strain line and yield strengths were found from the stress at which deviation from linearity first occurred.

RESULTS AND DISCUSSION

The relations between % w/w moisture content of the binder films and relative humidity are shown in Fig. 3. PVP absorbs more water than starch, and starch more than methylhydroxyethyl cellulose at all humidities. At low humidities, all binders showed an approximately linear relation between moisture content and relative humidity. These regions probably correspond to the uptake of bound moisture only.

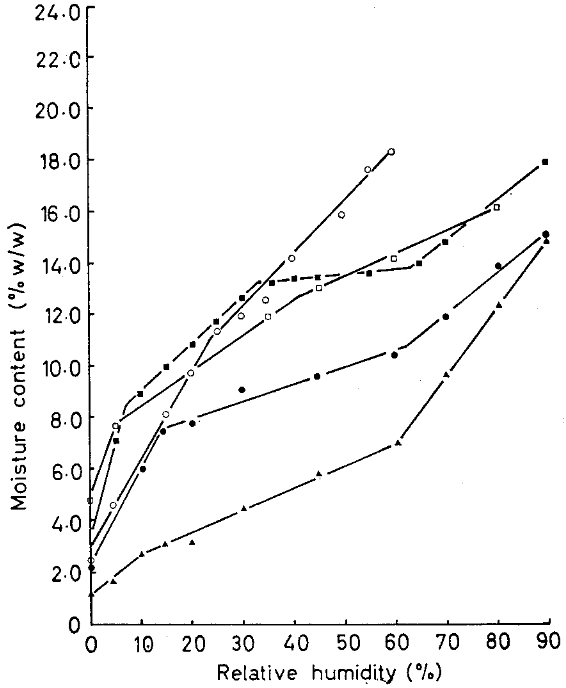

FIG. 3. The variation of moisture content of binder films with relative humidity. ▲ Methylhydroxyethylcellulose. ● Starch. □ Acacia. ○ PVP. ■ Gelatin.

The bound moisture sites will eventually become saturated however, so that the water absorbed at high humidities will be mainly free moisture. Inflections were found in the curves at the transitions between uptake of purely bound and purely free moisture. The points of inflection depict the apparent limits of free and bound moisture and these values are shown in Table 1. From this Table it can be seen that in the case of

Table 1. *The mechanical properties of binder films.*

Binder	Lower limit of free moisture %	Upper limit of bound moisture %	Relative humidity %	Moisture content % w/w	Young's modulus E MN m^{-2}	Yield strength MN m^{-2}	Ultimate tensile strength MN m^{-2}	Work done in breaking unit cross-sectional area of film Joules cm^{-2}
Acacia	45	5	20	9·8	2020	—	30·8	1·41
			45	13·2	1880	—	27·1	1·31
			70*	16·3	1410	—	12·9	0·36
Gelatin	65	6	20	10·8	2680	75·2	85·0	12·0
			45	13·5	2450	63·3	74·3	7·2
			70	15·0	2490	71·7	74·8	6·9
Methylhydroxyethyl cellulose	63	10	20	3·1	1540	29·9	50·0	34·6
			45	5·7	1680	31·5	47·6	32·2
			70	9·6	1350	21·8	52·1	34·3
PVP	25	25	20	10·4	930	—	19·5	1·02
			45	15·2	840	—	14·6	0·95
			65*	17·9	460	—	12·1	2·24
Starch	61	14	20	8·1	1910	28·4	51·9	18·5
			45	9·7	2030	33·1	50·7	14·3
			70	11·9	1740	28·1	46·0	11·2

Each value calculated from the stress-strain graphs is the mean of 15 determinations.

* Acacia and PVP would not form coherent films above 80 and 65% relative humidities respectively.

methylhydroxyethyl cellulose, free moisture uptake occurs only at humidities greater than 63%. By contrast for PVP films, free moisture will exist at humidities down to 25%. Table 1 also shows values of Young's modulus, yield strength, ultimate tensile strength and work done in breaking unit cross-sectional area of film at relative humidities of 20, 45 and 70%.

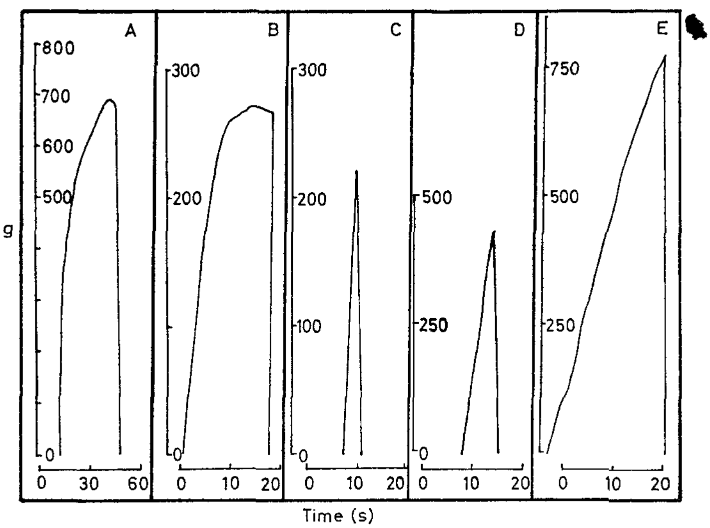

FIG. 4. Typical stress-strain graphs. A, Methylhydroxyethylcellulose. B, Starch. C, Acacia. D, PVP. E, Gelatin.

Typical stress-strain curves obtained for the films are shown in Fig. 4. Acacia and PVP showed no yield points in contrast to the other three binders. The PVP and acacia curves were characteristic of weak materials, gelatin of hard strong, and methylhydroxyethyl cellulose and starch of hard tough materials according to the classification of Carswell & Nason (1944). The relations between the tensile strengths of the binder films and moisture content are shown in Fig. 5. Acacia was found to be a weak material possessing a low tensile strength. Consequently low values were found for the work done in fracturing unit cross-sectional area of film, particularly at the 70% humidity value. The tensile strength of acacia remained approximately

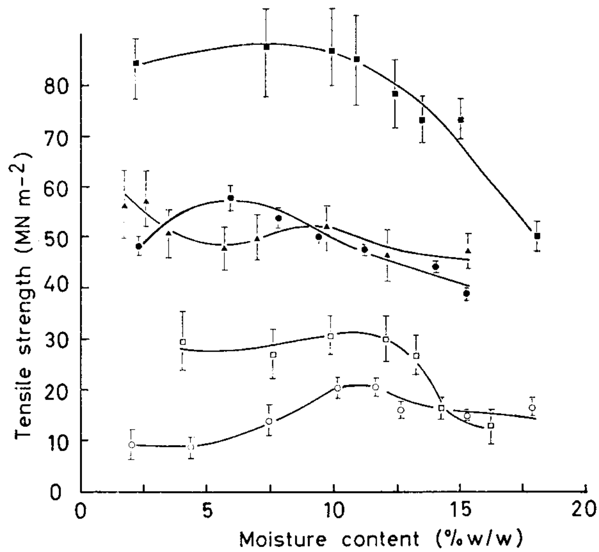

FIG. 5. The effect of moisture content on the ultimate tensile strength of binder films. ■ Gelatin ▲ Methylhydroxyethylcellulose. ● Starch. □ Acacia. ○ PVP. The vertical bars show the limits of error of the means at $P = 0{\cdot}95$.

constant at 30 MN m^{-2} over the moisture range 4 to 12% w/w, dropping steeply to 12·9 MN m^{-2} as the moisture content increased beyond 12% w/w. The point at which the tensile strength no longer remained constant (about 12·6% w/w moisture content) corresponded to the lower limit of free moisture for acacia, found from Fig. 3. Thus the tensile strength of acacia decreases sharply as free moisture uptake occurs.

Low values for work done and tensile strength were recorded for PVP which showed it to be the weakest of the five materials studied. In addition, PVP also exhibited low values for Young's modulus (Table 1) showing it to be the most readily deformable of these binders as well as having low strength. Its value as a good tablet binder may in part be attributed to this high deformability which will aid consolidation during compaction. In contrast to acacia the lower limit of free moisture (about 10·7% w/w) corresponds with the maximum value of tensile strength recorded for PVP.

Gelatin possessed the highest values of Young's modulus and tensile strength, although this binder was again somewhat brittle, producing only a little yielding before fracture. Its usefulness as a binder may be limited because its high elastic modulus and low plasticity will resist consolidation. In addition, because of its brittleness, granules and tablets containing gelatin will be relatively friable. Its high tensile strength, however, corresponds with its known property of producing hard tablets. The tensile strength graph obtained with gelatin was found to be similar in shape to the curve exhibited by acacia. As with acacia a sharp drop in tensile strength was recorded as free moisture uptake occurred.

Starch and methylhydroxyethyl cellulose were much tougher materials. They both exhibited moderately high tensile strengths and withstood considerable strain before failing. This is shown in Table 1 by the high values for work done in breaking unit cross-sectional area of film. Their stress-strain curves showed stress maxima followed by small reductions before eventual failure. This is evidence of necking, or the reduction of area at a relatively weak cross-section of the test specimen, commonly found with ductile materials. The starch films, however, became markedly more brittle at very low moisture contents, that is below 6% w/w. The upper limit of bound moisture for starch (about 6·5% w/w) corresponded with a tensile strength maximum, but the opposite was found with methylhydroxyethyl cellulose. This material exhibited a minimum value of tensile strength at the point at which the limit of bound moisture uptake occurred (about 3·5% w/w).

Although many other factors may be involved, it may in addition be expected from these results that, in general, granules made with acacia or PVP would be relatively friable. Granules containing gelatin or starch would be less friable, and methylhydroxyethyl cellulose would produce the least friable granules.

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