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Gloss measurement on film coated tablets

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The gloss of film coated tablets has been assessed using a commercially available specular glossmeter modified to measure the specular reflectance of individual flat-faced, film-coated tablets. For all the pigmented film formulations studied, there was a decrease in gloss with increasing pigment concentration, the magnitude of the decrease being dependent on the pigment used. Film coatings prepared using an aqueous coating system had a much lower gloss reading than those prepared using an organic solvent coating system. Increasing the mean particle size of an inert filler particle resulted in a minimum gloss reading at intermediate particle sizes, while increasing the polymer concentration resulted in a significant decrease in gloss. In the latter case a power law equation was found to fit the data relating the percentage decrease in gloss with the percentage increase in roughness. The results are consistent with the known theories of specular reflectance and illustrate the potential of this rapid and simple technique in the optimization of film formulation and process variables during product development.

The appearance attributes of a film coated tablet—colour, film opacity and gloss—although offering no particular therapeutic advantages, are of considerable psychological importance especially in securing patient cooperation in taking the medicine. They are also extremely valuable in assessing the efficiency of both the film formulation, especially with regard to the stability of the product on storage, and the film coating process, especially with regard to the optimization of the processing variables. However, although the eye is a sensitive and discriminating sensor, it cannot make quantitative measurements and these are fundamentally important especially for predictive purposes. Since recent work on the measurement of colour and film opacity (Rowe 1984a, b, c, d) has demonstrated the potential of both techniques in the optimization of film formulation during product development, it would appear pertinent to study the third appearance attribute mentioned i.e. that of the glossiness of the film coating.

Gloss can be defined as that attribute of a surface that causes it to have a shiny or lustrous appearance. In physical terms gloss can be ascribed to the specular reflection of light by a surface and it is this property that is measured to assess gloss. However, since specular reflection can vary from one surface to another in the fraction of light reflected, in the manner and extent of the scatter either side of the specular direction and in the specular angle, there is no single reflectance scale that yields gloss values that correlate with the perceived glossy appearance of all types of surface. In this work a commercially available specular glossmeter, normally used to measure the gloss of plastics and paints, has been modified to measure the gloss of individual flat-faced, film-coated tablets.

MATERIALS AND METHODS

Gloss measurements were made by measuring the light specularly reflected at 60° by a 11·11 mm flat-faced, film-coated tablet using a photodetector coupled to a signal processor (model D48-7 glossmeter, Hunter Associates Laboratory Inc., Virginia, USA). The rectangular viewing port of the original instrument was modified to that of a circle 10·0 mm in diameter by means of a black painted metal insert, care being taken to ensure that the tablet surface was in the plane of the viewing window and that the reflected image was in the centre of the receptor window. The instrument, without the insert, was initially calibrated using standard ceramic tiles of known gloss and the insert replaced. The calibration was then checked using a secondary standard consisting of a 12·5 mm flat-faced Perspex "tablet" coated with a red PVC tape of known (previously measured) gloss. The gloss readings reported are the means and standard deviations calculated from ten individual tablets.

Three variables were studied in three separate experiments; firstly the effect of pigment concentration using titanium dioxide and FD and C Yellow 5 Lake as representative pigments, the former being applied in both an aqueous and organic solvent coating system; secondly, the effect of the particle size of an inert filler using a white dolomite (Microdol, A/S Norwegian Talc, Norway—see Rowe 1981) as a model pigment applied in an organic solvent coating system, and thirdly, the effect of polymer concentration in an organic solvent coating system. All the tablets used were coated with a film formulation consisting of either a mixture of four parts of hydroxypropyl methylcellulose (Pharmacoat 606—Shin-Etsu Chemical Co., Japan) and one part of ethylcellulose (Grade N7, Hercules Inc., USA) or hydroxypropyl methycellulose alone, both formulations containing glycerol (20% w/w based on polymer) as plasticizer.

For the organic solvent coating system a dichloromethane–methanol (70:30% v/v) solvent mixture with a polymer concentration of 2·0% w/v was used while for the aqueous system a polymer concentration of 5·0% w/v was used. All formulations were applied using either a 6 in Wurster column or 24 in diameter Accela-Cota (Manesty Machines Ltd, Liverpool) the latter being fitted with either an airless spray gun for the organic solvent coating system or an airborne spray gun for the aqueous coating system.

RESULTS

The gloss readings measured can be placed in perspective by comparing them to the value obtained for a polished black ceramic tile of refractive index 1·54, viz. 94·0. Hence in terms of an approximate visual rating, films with values of between 10 and 40 can be designated 'eggshell', while films with values below 10 can be designated 'matte' (Fig. 1). Bearing this in mind, it can be seen that the only film with a distinctive gloss was the one prepared using the lowest polymer concentration (Fig. 3).

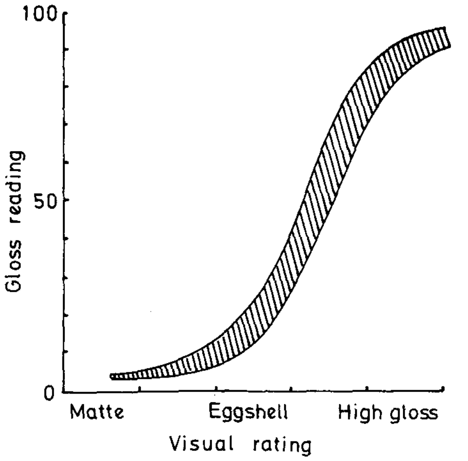

FIG. 1. The relation between the numerical gloss reading and a visual rating of gloss for a 60° incident angle (after Hunter 1975).

The effect of pigment type and concentration on the gloss of both aqueous and organic solvent coating systems is shown in Fig. 2. In all cases there is a decrease in gloss as the pigment concentration is increased. However, the decrease is not linear, occurring rapidly at low pigment concentrations and then more slowly at higher pigment concentrations. There is a marked inflection in the gloss reading for films pigmented with the FD and C Yellow 5 Lake occurring at a pigment volume concentration of 13–15% v/v. This is believed to be the critical pigment concentration of the film (Guillaume 1969).

The magnitude of the decrease in gloss with increasing pigment concentration is far greater for the yellow lake pigment than for the titanium dioxide even though both pigments were applied using the same organic solvent coating system. Also, the relative decrease on increasing the titanium dioxide concentration to 16·6% w/w in the films prepared from both the aqueous and the organic solvent coating system is the same despite the fact that the initial gloss readings of the unpigmented films were different. This initial difference is thought to be due to the differences in the spraying conditions (aqueous—airborne spray, organic solvent—airless spray) and in the viscosities of the coating solutions (aqueous—50 mPas, organic solvent—5 mPas).

On increasing the mean particle size of the dolomite inert filler over the range 2–18 μm, a minimum in gloss is seen to occur (Fig. 3) with intermediate sizes of filler present at all pigment concentrations, although this is increasingly less pronounced at high pigment concentrations. Increasing the polymer concentration and hence the viscosity in an organic solvent coating system significantly affects the gloss of the resultant film coatings (Fig. 4). The viscosity of a 7% w/v solution in this experiment was similar to that of the aqueous coating system used above and both solutions produced films with similar gloss readings (Fig. 2).

DISCUSSION

Before discussing the results in detail it is pertinent to consider the physical factors governing the specular reflection of light by surfaces. For an optically smooth surface the amount of light reflected (R) is a function of the refractive indices of both the surface itself (n_2) and the surrounding medium (n_1) and the

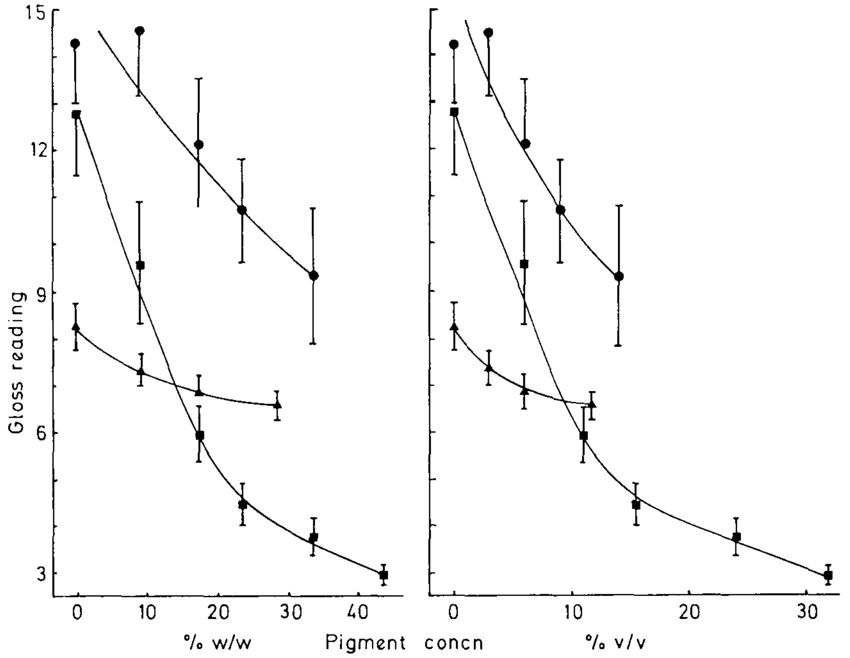

FIG. 2. The effect of pigment concentration on the gloss reading: ● Titanium dioxide—organic solvent coating system; ▲ Titanium dioxide—aqueous coating system; ■ FD and C Yellow 5 Lake—organic solvent coating system.

angle of the incident light (i). In general terms R can be described by the Fresnel equation:

$$R = \left[\frac{(n_2/n_1)^2\cos i - [(n_2/n_1)^2 - \sin^2 i]^{\frac{1}{2}}}{(n_2/n_1)^2\cos i + [(n_2/n_1)^2 - \sin^2 i]^{\frac{1}{2}}}\right]^2 + \left[\frac{\cos i - [(n_2/n_1)^2 - \sin^2 i]^{\frac{1}{2}}}{\cos i + [(n_2/n_1)^2 - \sin^2 i]^{\frac{1}{2}}}\right]^2 \qquad (1)$$

In this study this equation can be simplified since i is 60° and n_1 is unity—the surrounding medium is air. However, very few surfaces, and certainly not the surface of a film coated tablet, can be regarded as optically smooth. All have an inherent roughness which can affect the total amount of reflected light. In the case of very smooth surfaces it is known that a roughness of the order of 0·1 μm can produce a perceptible reduction in the specularly reflected light (Morse 1973). In the case of rough surfaces, the maximum average height of the surface irregularities (h) that will still permit any perceptible gloss can be calculated from the application of the microfacet theory (a theory that assumes that a rough surface can be treated as a collection of statistically distributed irregularities) using the formula (Billmeyer et al 1971):

$$h = \lambda/\cos i \qquad (2)$$

where λ is the wavelength of the incident light. For an incident angle of 60° and a wavelength of 540 nm, h will be of the value of 1·1 μm.

The relevance of these concepts to the gloss readings on film-coated tablets can be most easily

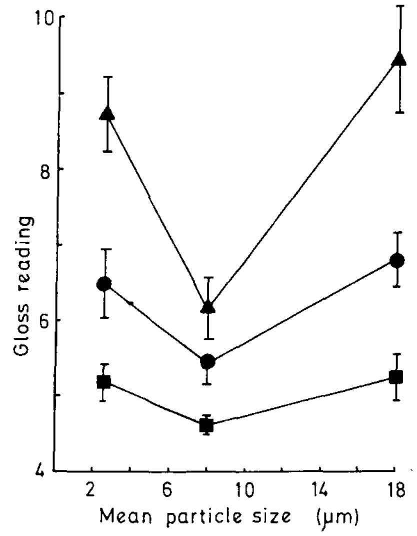

FIG. 3. The effect of the mean particle size of dolomite on the gloss reading: ▲ 14% v/v concentration; ● 24% v/v concentration; ■ 32% v/v concentration.

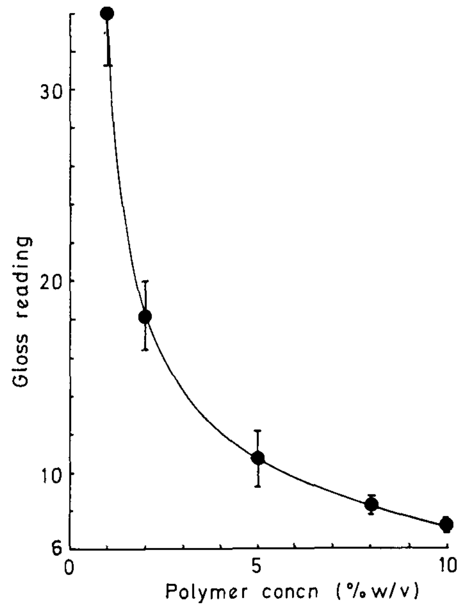

FIG. 4. The effect of polymer concentration (organic solvent system) on the gloss reading.

discussed by reference to the results on the effect of polymer concentration (Fig. 4) since in this system any variation in the specularly reflected light will be due to changes in the surface roughness and these have already been measured (Rowe 1979). A comparison of the relative changes in both the gloss readings and surface roughness of the film coatings (Table 1) shows that there is not a direct linear relation between them. However, the data do appear to fit a power law equation of the form:

$$y = kx^n \qquad (3)$$

where y is the percentage decrease in gloss, x is the percentage increase in roughness, and k and n are constants calculated from the data using linear regression analysis as being 32·63 and 0·152, respectively (correlation coefficient 0·9826, $P > 0·015$).

Table 1. Relative changes in the surface roughness and gloss for film coatings prepared at increasing polymer concentration.

Polymer concn (% w/v)	Decrease in gloss (%)	Increase in roughness (%)
1	0	0
2	46·7	12·7
5	68·5	85·7
8	75·6	285·7
10	78·6	375·4

Such a correlation does not apply in the case of pigmented films (Table 2) since in this case the effect will be further complicated by the changes in the effective refractive index of the surface due to pigment particles either very close to the surface or even protruding from the surface. Another factor to be considered is the inherent randomness of any protrusions especially at low or intermediate pigment concentrations. Roughness measurements, especially those made using a stylus instrument, are invariably taken over small traverse lengths usually across the diameter of the tablets (Rowe 1978) and hence are only likely to pick up protrusions at very high pigment concentrations. On the other hand, gloss is an integrated function over the total face of the tablet and hence will pick up even the most random of protrusions at all pigment concentrations. This fact is particularly important in explaining the initial rapid fall off in gloss with increasing pigment concentration without any apparent change in surface roughness (Fig. 2, Table 2). At pigment concentrations in excess of the critical pigment volume concentration, the film coatings will be rough with little discernible gloss.

Table 2. Relative changes in the surface roughness and gloss for film coatings containing FD and C Yellow 5 Lake.

Pigment concn (% w/v)	Pigment concn (% v/v)	Decrease in gloss (%)	Increase in roughness (%)
0	0	0	0
9·1	5·9	24·8	6·9
16·6	11·1	53·5	6·9
23·1	15·7	65·2	6·9
33·3	23·8	70·4	9·7
42·9	31·9	77·4	24·8

The refractive index contribution is best seen at pigment concentrations in excess of the critical pigment concentration. It can be seen (Figs 2, 3) that the gloss readings at these high pigment concentrations for the organic solvent coating system decrease titanium dioxide pigmented film > dolomite pigmented film > yellow lake pigment film. All film coatings had similar measured surface roughness values and hence the differences can only be explained in the differences in the refractive indices of these pigments —titanium dioxide, 2·52; dolomite, 1·62; yellow lake, 1·52—affecting the intensity of the specularly reflected light—equation 1.

The effect of the particle size of a pigment or filler on the gloss of the film coating is an important factor to be considered by the formulator. Experience in the paint industry has shown little change in gloss if the pigment particles or aggregates are no coarser than 0·3–0·4 μm (Miller 1962) but at larger particle sizes—between 2 and 5 μm there is rapid deterioration in gloss (Miller 1962; Delfosse 1965). Matte coatings with gloss readings of less than 10 are invariably produced with pigments or fillers of mean particle sizes in excess of 10 μm (Delfosse 1965). The results reported here are not at variance with the paint film data since a comparison of the particle size distribution of the three grades of dolomite used showed that the grade with the largest mean particle size also had the widest size distribution (Rowe 1981), and hence is more likely to pack more densely neutralizing the effect of the particle size change.

The overall results show that differences in the gloss of tablet film coatings can be accurately and reproducibly assessed by the method proposed. The measurements are relatively easy and rapid to make and are consistent with the known theories of specular reflectance. A disadvantage of the technique is that only flat-faced tablets can be used but this has not proved to be too much of a problem. The technique as described offers a further objective measurement which in combination with both measurements for colour and opacity can be used to fully describe the appearance attributes of a film coated tablet and thus has potential in the optimization of film formulation and process conditions during product development.

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