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AQUEOUS GLOSS SOLUTIONS: FORMULA AND PROCESS VARIABLES EFFECTS ON THE SURFACE TEXTURE OF FILM COATED TABLETS*

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ABSTRACT

A specially designed spray box was constructed that allows spraying aqueous based film coating formulas under carefully controlled and reproducible conditions. The process variables studied included: spray distance, drying temperature, air cap, atomizing air pressure, and spray rate. Aqueous film coating gloss solutions were prepared using hydroxypropyl methylcellulose. The viscosity grade, surface tension and concentration of polymer in solution were the formula variables studied. The surface texture of film coated tablets before and after spraying with the gloss solutions was analyzed with a stylus type surface roughness analyzer. The effect of the formula and process variables was also assessed by measurement of the tristimulus color difference obtained from colorimeter readings on the tablets, excluding versus including the specularly reflected light from the glossed surface. Within the ranges of the variables studied, the most

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significant effects on the surface texture were from the nozzle set up used and the spray distance.

INTRODUCTION

Film coating has largely replaced sugar coating as the preferred choice when uncoated tablets are unsatisfactory for aesthetic or functional reasons. The original film coated dosage forms were from organic based solvent systems. More recently, there has been a changeover underway to aqueous based systems primarily for economic and environmental reasons. Although numerous advantages for film coating versus sugar coating can be claimed, the elegance or luster of the final product is generally not one of them.[1] For this reason, a gloss solution application as a final coat in film coating in order to optimize the appearance of the dosage form is commonly needed. Besides enhancing the elegance of the dosage form, gloss solution application to film coated tablets can greatly facilitate the printing of dosage form identification markings. The purpose of this work was to investigate some formula and process variables effects on the surface texture of aqueous gloss film coated tablets to provide a rational basis for potential optimization of gloss solution application.

The roughness of a surface is highly correlated with the observed gloss.[2] Stylus type surface roughness analyzers provide a convenient means of quantitating changes in surface texture.[3-5] The arithmetic mean roughness, R_a, is almost universally used in parametric estimations of surface texture. A graphical description of R_a is illustrated in Figure 1.[6] The roughness of a surface such as that on a compressed tablet obviously cannot be totally described with a single number. Some additional measureable parameters of interest include R_{pm} and R_{tm}. R_{pm} is the mean value of the maximum profile height from the profile center line over five sampling lengths (See Figure 2).[7] R_{tm} is the average, measured over five consecutive sampling lengths, of the maximum peak-to-valley height in each sampling length (See Figure 3).[8] R_{pm} can be considered a peak height parameter and R_{tm} a peak-to-valley or

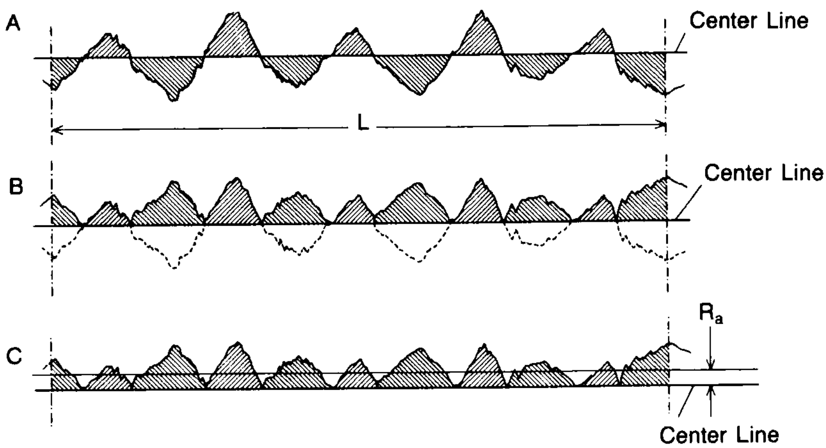

FIGURE 1.
GRAPHICAL DERIVATION OF R_a
(A) Profile with center line
(B) Lower portions of profile inverted
(C) R_a is the mean height of the profile

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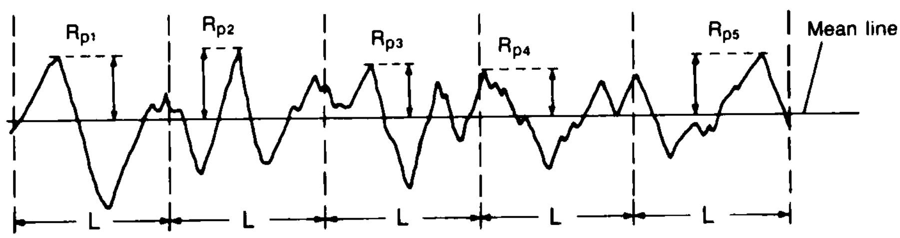

$$R_{pm} = \frac{R_{p1} + R_{p2} + R_{p3} + R_{p4} + R_{p5}}{5} = \frac{1}{5}\sum_{i=1}^{i=5} R_{p1}$$

FIGURE 2.
R_{pm}, PEAK HEIGHT PARAMETER

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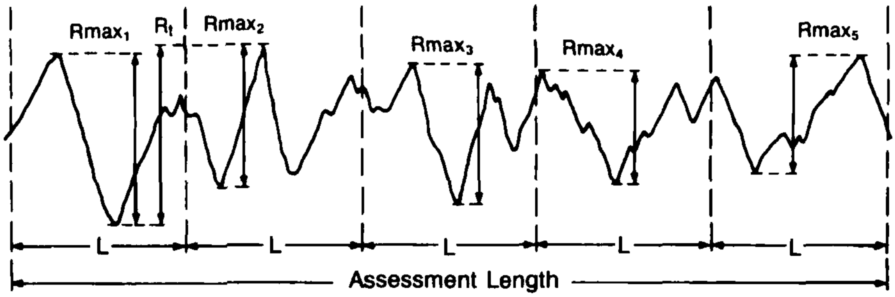

$$Rtm = \frac{Rmax_1 + Rmax_2 + Rmax_3 + Rmax_4 + Rmax_5}{5}$$

$$= \frac{1}{5} \sum_{i=1}^{i=5} Rmax_1$$

FIGURE 3.
Rtm, PEAK-TO-VALLEY PARAMETER

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maximum distance indicator. Rowe has attempted to assign additional meaning to selected ratios between surface roughness parameters.[9] Changes in surface texture on film coated tablets as a result of application of glossing solutions would be expected to decrease the R_a, R_{pm}, and R_{tm} parameters.

In addition to surface roughness analysis, optical methods based on the reflectance properties of a surface can be particularly useful when the visual appearance of a surface is important. The principle of a gloss meter is to determine how a parallel beam of light incident at an angle on the surface is reflected. When the surface is perfectly reflective all the light is specularly reflected (i.e., angle of reflection equals angle of incidence).[10] In this study both roughness and reflectivity of the gloss coated surface were assessed to quantitate effects of the formula and process variables on surface texture.

EXPERIMENTAL

To facilitate the study of the influence of formula and process conditions on characteristics of film coated tablets, a special

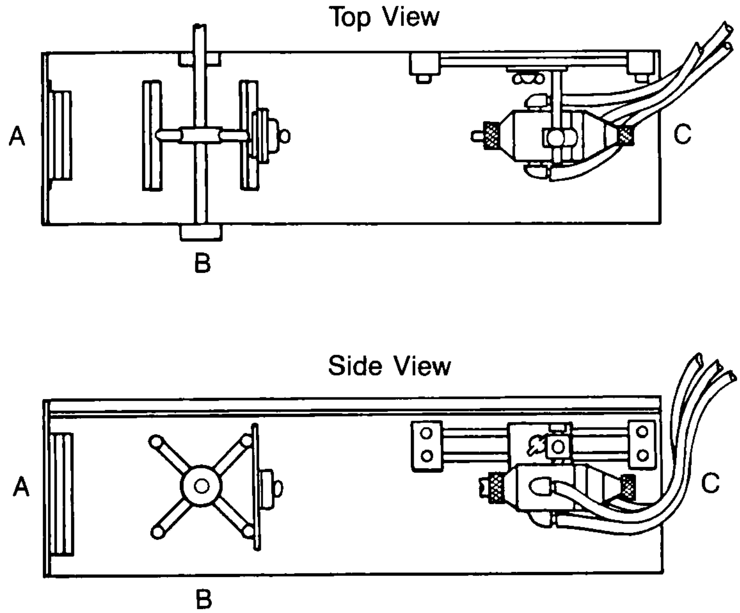

FIGURE 4.
SCHEMATIC DRAWING OF SPRAY BOX
(A) Exhaust filter;
(B) Rotating sample holder;
(C) Spray gun assembly.

spray box was constructed. The spray box is shown schematically in Figure 4. The box was constructed out of stainless steel with a removable plexiglass top plate for viewing of the spray process. Any desired spray gun and nozzle set-up can be easily mounted on a moveable track used to adjust the spraying distance. A Spraying Systems Model 1/4 JAUCO spray gun was used for all gloss solution applications. Heated inlet air, converted from a pan pour coater operation, enters the front of the box. A removeable mesh filter covers the opening to the exhaust duct work. Temperature and air velocity control in the range of interest to tablet film coating are achievable and reproducible. A Masterflex Servodyne Controller was used to revolve the sample holding assembly at 30 RPM. This revolving speed produces a calculated exposure to the flat spray pattern for approximately the same amount of time as that seen for a tablet in a 24 inch Accela-Cota (based on the 0.12 second exposure time reported by Prater, et. al.).[11] To simulate the intermittent exposure of a tablet surface in a perforated pan type coating system, a Texas Instruments PM 510 Programmable Controller was used to control the spraying process. The spraying was started before the tablet samples passed through it to closely mimic the spraying mode actually used in tablet film coating. After each spray a programmed dry cycle was executed. The total number of spray-dry cycles included in the controller program determined the coating duration. Tablet to tablet contact is not simulated in the spray box model.

The independent variables in an initial screening study using a twenty run Plackett-Burman experimental design are shown in Table 1.[12] The viscosity of the coating liquids was measured with a Brookfield viscometer. Surface tensions were determined using a Rosano surface tensiometer. All gloss coatings were applied to 5/8" round flat-faced placebo tablets previously coated in a 24 inch Accela-Cota with an aqueous hydroxypropyl methylcellulose based coating. The twenty run Plackett-Burman design was chosen because initially it was felt all of the listed independent variables could potentially be important and sufficient degrees of freedom for estimating experimental error were desired. A gloss coated tablet from each of these twenty runs was analyzed for its surface roughness and reflectivity. A Taylor-Hobson Surtronic 3 Surface Roughness Analyzer and attached Parameter Module were used to obtain R_a, R_{pm}, and R_{tm} surface roughness parameters. At least six measurements were taken on each tablet using an instrument cut-off value of 0.8 microns. The cut-off value determines the actual size of the sampling length on the tablet surface.

A measure of the reflectivity of the gloss coated tablets was obtained using a MacBeth 1500 Color Measurement System. The tri-stimulus color values using the CIE LAB color coordinate system were measured on each tablet, first excluding then including the specularly reflected light component. Since each tablet serves as its own color control the color difference value obtained between the two measurements is exclusively due to reflected light. The total color difference, ΔE, obtained from measuring a flat tablet in this manner was a very reproducible indication of the reflectivity of the tablet surface.

TABLE 1

Plackett-Burman Experimental Design Variables

Independent Variable	Low Level	High Level
1. Hydroxypropyl Methycellulose Viscosity Grade (2% @ 20° C)	5 cps	15 cps
2. Concentration of Film Former	2% (w/v)	5% (w/v)
3. Spray Distance	6"	10"
4. Drying Temperature	40° C	60° C
5. Air Cap*	67228-45	134255-45
6. Atomizing Air Pressure	20 PSI	50 PSI
7. Spray Rate	50 ml/min	100 ml/min

*Spraying Systems, Inc. (2850 Fluid Nozzle was used in all runs.)

Table 2 contains the independent variables and levels used in a replicated $2^{(5-1)}$ half fractional factorial study to further define significant variables.[13] This experimental design was chosen to provide estimates of two factor interaction effects that may be important in the gloss solution spraying. In this portion of the study a fixed combination of fluid nozzle and air cap was used that, based on the screening study results, was expected to facilitate spraying the glossiest films possible. The concentra- of hydroxypropyl methylcellulose (HPMC) was held constant at 3% (w/v), but the viscosity grade of polymer was again investigated as in the screening study. In addition, the effect of varying the surface tension of the coating liquid was studied by including the presence of 0.1% Brij 30 surfactant as a formula variable. This surfactant produced coating liquids with surface tensions in the range of 26-29 dynes/cm^2.

Film coated tablets were also gloss coated using solution concentrations of HPMC ranging from 1 to 8% (w/v) to examine more closely the effect of polymer concentration over this wide range. Simple solutions of the 5 cps viscosity grade of HPMC in distilled water were sprayed under identical conditions in the spray box

TABLE 2

Half Fractional Factorial Study

Independent Variable	Low Level	High Level
1. Drying Temperature	45^{o} C	60^{o} C
2. Spray Rate	75 ml/min	150 ml/min
3. Hydroxypropyl Methylcellulose Viscosity Grade (2% @ 20^{o} C)*	5 cps	15 cps
4. Spray Distance	6.5 inches	10 inches
5. Brij 30 Surfactant Level	0%	0.1%

* 3% (w/v) aqueous solutions were used in all trials.

onto film coated placebo tablets and R_a values measured. The spraying conditions used were 150 ml/min spray rate, 60^{o} C drying air, 60 PSI atomizing air pressure, 6.5 inch spray distance, and a Spraying Systems 35100 fluid nozzle with a 134255-45 deg. air cap.

RESULTS AND DISCUSSION

The results of the Plackett-Burman experimental design using each of the roughness parameters and the color difference, ΔE, as the dependent variables are summarized in Table 3. Roughness analysis and reflectivity measurements agreed quite closely. Air cap, spray distance, and drying temperature were the three variables calculated to be significant at the 90% confidence level. The effect on tablet gloss, as measured by ΔE, is negative compared to the effect on surface roughness parameters. This, of course, results from ΔE increasing with increasing smoothness, while the stylus measured parameters get smaller in magnitude under similar conditions.

Surface profiles from a color film coated unglossed tablet and two of the glossed tablets from the initial Plackett-Burman experimental design are shown in Figure 5. Profile A is representative of the surface texture present on the film coated tablets before any of the gloss solution applications were applied. The tablet from

TABLE 3

Plackett-Burman Results

Summary of Effects on Surface Texture

Independent Variable	Effect on Surface Parameter: Ra	Rpm	Rtm	ΔE
1. Viscosity Grade	0.01	0.29	0.66	-0.01
2. Concentration	0.06	0.47	1.15	-0.04
3. Spray Distance	0.21*	0.56	1.26*	-0.38*
4. Drying Temperature	0.19*	0.63*	1.01	-0.25*
5. Air Cap	-0.45*	-1.43*	-1.89*	0.79*
6. Atomizing Air Pressure	-0.02	0.02	0.08	0.17
7. Spray Rate	-0.06	-0.19	0.84	0.15

*Indicates a calculated statistically significant result at the 90% confidence level.

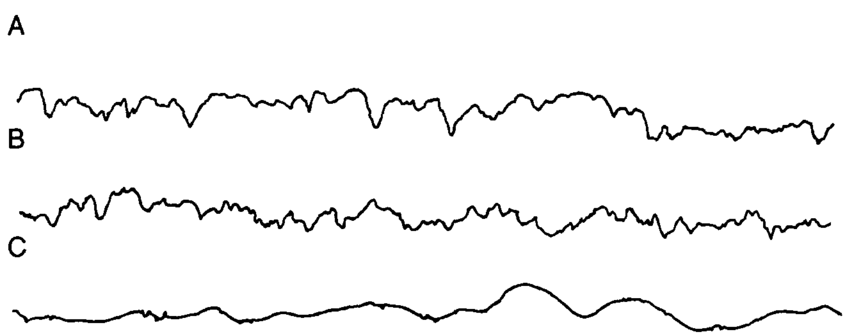

FIGURE 5.
SURFACE PROFILES
(A) Unglossed tablet;
(B) Gloss coated tablet with dull appearance;
(C) Shiniest gloss coated tablet.

profile B actually appeared just as dull after gloss solution application as it did initially. Profile C shows the desired smoothing effect on the surface texture expected from gloss solution application. The R_a values measured from these 3 profiles were 2.8, 2.3, and 1.6 microns for profiles A, B, and C respectively.

Air cap was the most significant variable in producing a smooth, shiny gloss coating. Of the two air caps examined the 134255-45 air cap has a much larger annular orifice that would result in a higher air volume and velocity at a given atomizing air pressure. The larger ratio of air to coating liquid provided by the bigger annular orifice would produce smaller sized droplets that impact the tablet surface at higher velocities. Another significant factor due to the air caps would be seen in the resulting spray patterns. Although not investigated in this study, the larger annular orifice would be expected to produce a narrower more concentrated spray pattern. Figure 6 shows scanning electron photomicrographs corresponding to the surfaces that produced the profiles seen in Figure 5. The smoothest surface was obtained from the largest annular orifice air cap at the shortest spray distance and lowest drying temperature used for spraying. The dullest tablet was obtained with the smaller orifice air cap at higher drying temperature and a longer spray distance. These latter conditions are those most conducive to spray drying. For minimum surface roughness (maximum glossiness), it appears that it is necessary to get the coating droplets to the tablet surface and allow the droplets to spread and coalesce into a uniform film before the solvent is totally evaporated.

It is interesting to note that neither of the formula variables (viscosity grade of HPMC and concentration of HPMC) was found to be significant in the ranges studied. The coating liquids investigated in the screening study differed significantly in viscosity. The viscosity of the 2% HPMC 5 cps coating liquid was about 20-30 cps. At the other extreme, a 5% concentration of HPMC 15 cps coating liquid has a viscosity approximately 10 times higher. The surface tension of all the coating liquids in the screening study were in the range of 40 to 50 dynes/cm^2. The lack of significance of any of the formula variables was not expected.

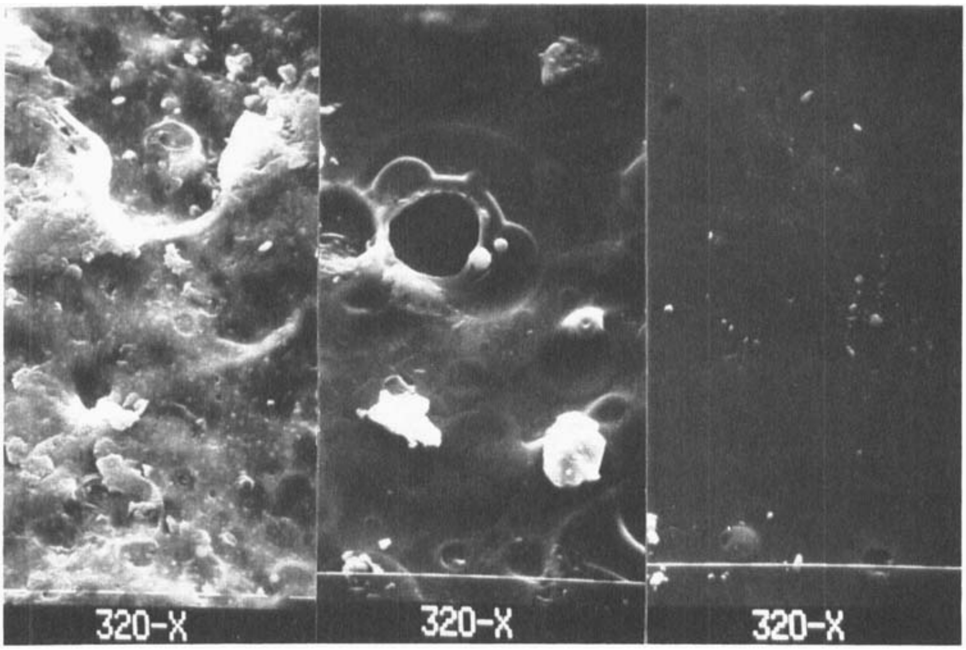

FIGURE 6.
SCANNING ELECTRON PHOTOMICROGRAPHS
(A) Unglossed tablet;
(B) Gloss coated tablet with dull appearance;
(C) Shiniest gloss coated tablet.

To further investigate the primary effects of the formula and process variables the replicated $2^{(5-1)}$ half fractional factorial study was performed. The results for the half-fractional factorial study are shown in Table 4. The experimental design provided the capability of estimating the significance of two factor interactions as well as the primary effects of the variables studied. The effect of spray distance in the factorial study was the only statistically significant variable on both surface roughness and reflectivity measurements on the glossed tablets. Drying temperature and spray rate were also significant when ΔE was the dependent variable measured. There is inherently more variation in the measurement of surface roughness parameters than in the measurement of ΔE with the colorimeter. The roughness parameters are determined from a stylus

TABLE 4

Half Fraction Factorial Results

Independent Variables	Effect on Surface Parameter: Ra	ΔE
1. Drying Temperature	-0.10	-0.51*
2. Spray Rate	-0.08	0.41*
3. HPMC Viscosity Grade	0.03	-0.06
4. Spray Distance	0.56*	-0.89*
5. Brij 30 Surfactant Level	-0.06	0.09
Two Factor Interactions		
Temperature x Spray Distance	0.18	-0.35*
Spray Rate x Spray Distance	0.04	0.27*

*Indicates a calculated statistically significant result at the 95% confidence level.

type pickup drawn in a single line across the surface and would be expected to vary somewhat across the face of a tablet. The specular reflection measured from the flat surface of a tablet is a result of the entire reflecting surface exposed for measurement. The greater precision afforded by the colorimeter readings could account for the detection of more significant variables when ΔE is the dependent variable.

Two significant interaction effects were detected (p=0.05) in the replicated factorial study when ΔE was used as the dependent variable. The interactions between spray distance and temperature and spray rate and temperature were calculated to be significant. As is often the case, the important interaction effects involved the same variables that demonstrated the greatest primary effects as well. The interpretation of the primary effects when significant interactions are detected with the same variables must be made cautiously.[14] The spray distance x temperature interaction is an indication of some degree of spray drying. The other important interaction between spray rate and distance indicates the beneficial effect of higher spray rates to produce glossier tablets was more pronounced at the longer spray distance.

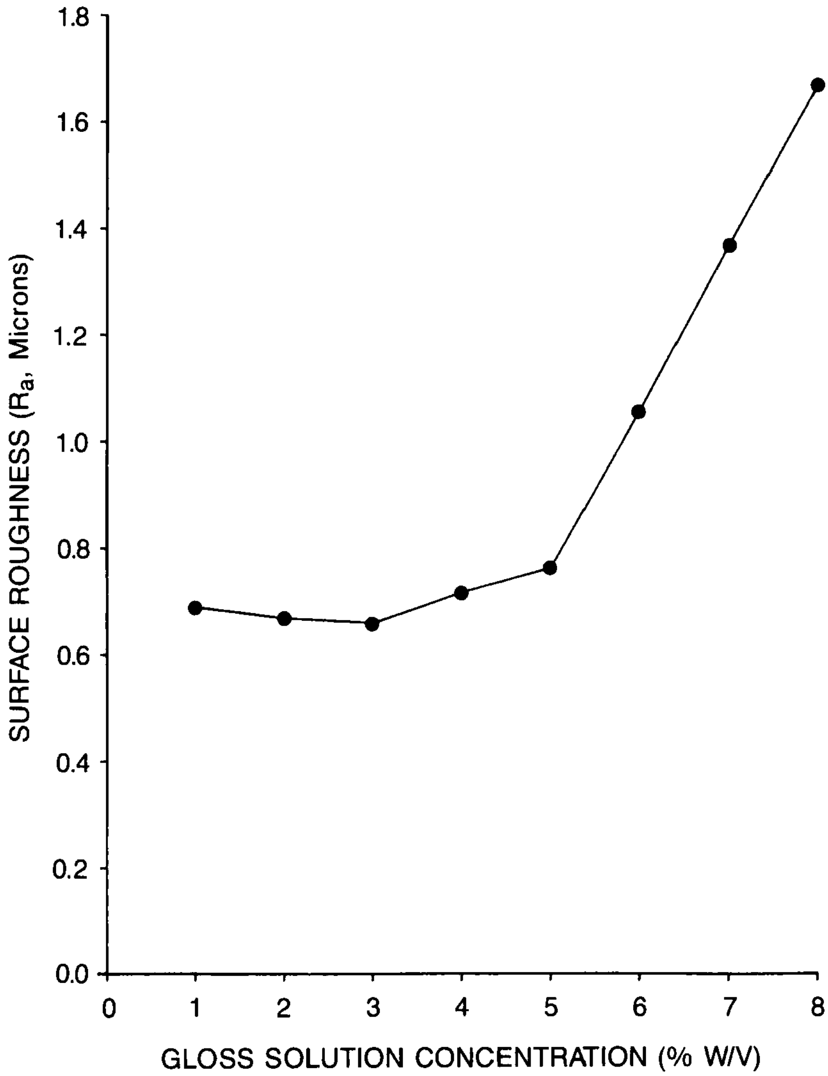

FIGURE 7.
ROUGHNESS VERSUS GLOSS
SOLUTION CONCENTRATION

The results of the half fractional factorial study failed to demonstrate the effect of any formula variables examined on the surface texture of the glossed tablets. This was in agreement with the initial screening study. The effect of increasing concentrations of the HPMC over the broad range of 1 to 8% (w/v) was examined to attempt to determine at what level of polymer the coating liquid formula would become an important factor in the resulting surface texture on gloss coated tablets. Figure 7 shows graphically the response of surface roughness (R_a) to the increasing concentration of HPMC in solution. The roughness is not detectably different over the concentration range from 1 to 5% (w/v) HPMC, which probably covers the range considered for practical applications, but rises sharply at higher concentrations. It is clear that if a wider concentration range had been studied in the screening and factorial studies the effect of concentration would have demonstrated more importance.

No attempt was made in this study to include the complicating influence of tablet to tablet contact that is present to all types of production coating equipment. The results presented here indicate that coating conditions that avoid the extremely rapid drying of the sprayed droplets of coating liquid tend to produce the most elegant appearing tablets. However, these coating conditions which enhance the possible appearance of the isolated individual tablets are the same conditions that could generate potential complications with sticking and picking between tablets in a coating pan. The practical operating range for optimal aqueous gloss coating liquid application may be quite narrow.

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