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Mark K. Phiscator Western Michigan University

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### An Investigation Into the Parameters Affecting Vehicle Migration During the Redistribution of Aqueous Coatings

by Mark K. Phiscator

A thesis submitted in partial fulfillment of the course requirements for The Bachelor of Science Degree

Western Michigan University Kalamazoo, Michigan April 1984

#### Abstract

The purpose of this study was to determine what effect shear has upon the vehicle migration in aqueous coatings. The roll inclined-plane apparatus was utilized to generate various ranges of shear rate. An immobilization factor was then calculated for these different rates of shear indicating the degree of vehicle penetration. The particle size of the binder and the particle size of the pigment were varied to determine their effect on the penetration into the base substrate.

It was concluded that high shear rates <u>pack</u> the particles, either binder or pigment, into the pores of the base substrate. This phenomenon prevents the migration of the liquid phase.

Keywords:	Binder mig	rati	ion .	Binders (A	Adhesives)	•		Blade
	coating	•	Blade j	pressure .	Capillary		•	
	Coating	•	Colors	(Materials)	. Nip	•		Pene-
	tration	•	Shear	• Surfac	e properties		•	
	Water migr	atio	on .	Wetting				

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#### Introduction

During the last few years it has become necessary to increase the profit potentials and therefore the efficiency of paper coating equipment. This was accomplished by increasing the speed on existing machines and installing new machines with the highest speed capability. With the increased speed and resulting tonnage, emphasis was placed on end-use properties of the coated sheet such as smoothness, gloss, opacity, pick strength, ink receptivity, and printability. Although the evaluation of these properties is important from a quality control standpoint and has been useful in the development and testing of new products, the information obtained from such tests is necessarily restricted in its interpretation. It has become increasingly evident that more fundamental information is required to gain a better understanding of the complex processes involved in the coating of This thesis will explore the effects of the coating color as paper. it passes through or under the redistribution nip and the significance of this action upon the end-use properties of the coated sheet. The redistribution nip is defined as a blade, roll, or rod nip commonly used in industrial coating applications.

Many of the above properties which are commonly used to evaluate coated papers are related to the physical structure of the pigment coatings and the distribution/penetration of binder in the dryed sheet. This distribution of binder determines the strength of the coating layer, whereas the penetration determines the degree of bonding to the base sheet. These two parameters of the coating color slip and what influences them will form the backbone of my investigation.

#### Theoretical Background

The movement of material (ie. water, adhesive, or pigment particles) occuring during the redistribution of coating color from the coating pan to the sheet surface can be analyzed as a competition between two porus systems. The cellulose fibers and air-filled interstices forms one system; the pigment and adhesive solution forms the second. The vehicle migration occuring during this redistribution takes place during the interaction between these two systems.

Within this double system, three successive stages of material transport can be distinguished, 1) application, 2) distribution, and 3) drying. (1, 2)

1) <u>Application</u>: Initially, surface irregualrities in the paper are filled up with coating color without separation of the color constituents. This is the necessary first step in coating, but is not referred to as migration (1).

2) <u>Distribution</u>: Contact of paper fibers and coating color transfers liquid (binder plus water) from the color to the paper by a process known to obey the laws of capillary attraction (1-3). Also during the coating process liquid in the slip penetrates more or less quickly into the paper, according to its absorbency. This initial part of migration is frequently called penetration (3). Paperchromatography is an ideal example to illustrate this process. When the end of a strip of paper is dipped into a watery solution or various chemical substances a migration results making it possible to separate even very similar substances. Water and binder migrate in a fashion similar to that discussed above from the coating into the paper. This phenomenon was first investigated by Dappan, who noted the following

facts (2, 4).

As in the example of chromatography, the water migrates quicker than the binder, causing a higher dry content or binder content in the zone of separation between these two liquids. The fibrous material of the coating base tends to absorb the binder selectively and this promotes an increased concentration of the binder in the zone of separation and simultaneously a minimum concentration in the coating. This may only happen with starch if retrogradation has set in, while with casein the lower molecular weight fraction may migrate with the water while the higher molecular weight fraction remains in the color (1, 5, 6)

Migration of pigment must also occur, but since the pigment particles sizes are approximately the same size as the pores into which they must migrate, and possible much larger, pigment migration should occur to a limited extent only (1, 7). Note: it has been assumed here that migration within this two system model is soley a transfer of liquid from one porus system to another, there being no other material transport.

3) <u>Drying</u>: Coating application has been complete and the sheet enters the third stage. The third stage of material transportation occurs when the sheet is being dryed. The direction in which the material moves can now change towards the direction in which the water is taken away. For instance, assuming that this occurs only on the coated side, the amount of binder will become greater at the surface through further supply of binder from the inside of the coating dependent upon the speed of drying (8, 9).

As water migrates to the atmosphere (evaporation) the total solids of the color nearest the base will increase; this

may make the color being doctored so dilatent or viscoelastic, or both, as to render the finished coating unsatisfactory (4, 9). Additionally, although not necessarily a part of the third stage, color recirculation may increase the total solids of the color fed to the machine (1). This increased total solids level at the coating redistribution nip should be considered in mill operations, however, it is not significant for this research proposal.

The literature reports drying as an internal mechanism of liquid flow which may occur by one or more of several mechanisms. Some of these are listed as diffusion, capillary flow, flow caused by shrinkage, pressure gradients, etc (11). Gardner (12) reports it as a mass transport phenomena.

It would seem that as the drying rate is increased, there is a corresponding increase in the kinetic energy of the water molecules that rush to the coating surface to replace water lost by evaporation. These moving water molecules will then transfer some of their kinetic energy to the other ingredients in the coating (13). As stated earlier, since binders are of lower density than pigments, there is less inertial mass to overcome and the binders are more easily moved along by the flowing water to the coating surface.

There are essentially three different regions of migration occuring during the second stage of material transport which may be distinguished during the coating operation (1). Consider a pond type blade coater. Before the base sheet can reach the blade nip, the coating liquid will migrate into the sheet by capillary attraction. This region may therefore be called the "capillary region." This region of migration is thus time dependent, the variable being pond depth and machine speed. An inverted blade

system would extend this dwell time even more. Secondly, at the blade nip, hydrodynamic and viscoelastic forces subject the color to pressures which may exceed 1000 psi and act for only a few microseconds (1). This is denoted as the "pressure region." Thirdly, between the nip and first drying stage, the color is subjected to a second period of capillary migration. This third region, however, does not affect the nature of the color presented to the blade.

For example Figure 1 is a typical pond type or trailing blade coating configuration. "C<sub>1</sub>" indicates the first capillary region, "P" the pressure region, and "C<sub>2</sub>" the second capillary region. Other coating redistribution methods may have more areas that each of these regions occur in. Refer now to Figure 2. In the typical inverted blade or flooded nip coating configuration, "C<sub>1</sub>" the first capillary region and "P" the first pressure region occur at the application roll. Following this initial capillary and redistribution period, the color slip is subjected to an additional period of capillary migration "C<sub>1</sub>" and a second pressure migration region "P'". The second capillary region "C<sub>2</sub>" follows these regions before the sheet enters the dryer sections

Studies indicate that soluble binders migrate to the greatest degree and that particulate binders migrate to a degree that is inversly proportional to their size (13). Some degree of migration is desired however, because with insufficient binder migration, the color is inadequately bonded to the base sheet, whereas excessive binder migration weakens the finished coating layer. But whatever the reason for, or the causes of migration, the final result can be a change in coating properties. Industry has observed this in the form of "railroad tracking", mottle and other changes in ink absorption or receptivity.

This investigation will deal speciffically with the second region



of migration, the pressure region. The purpose is to develop a better understanding of the effect of vehicle migration on the morphological structure of the coating and to find its relationship to the physical properties mentioned earlier.

#### Roll Inclined-Plane

Liquid migration due to hydrodynamic forces occurring on redistribution of the color slip is a very difficult parameter to measure. Experimental results reported in previous papers indicate that the preferential migration of coating color components which occurred when a coating color was brought into contact with new stock greatly influenced the pigment adhesive demand. The following work was carried out in order to obtain a clearer understanding of the factors which affect preferential adhesive migration. It was decided that the roll inclined-plane should be used for such studies since the experimental conditions which exist during the spreading of the drop so closely simulated those which are theorized to exist in the nip during the commercial coating operation. This paper must therefore be considered as a progress report on the development of a new method for studying the penetration of coating color components.

During the coating operation, preferential vehicle migration is brought about by a combination of two types of forces. One of these is the mechanical force exerted on the coating color by the method of application or redistribution to the raw stock. The other is the force of capillarity which pulls the coating color into the pores of the raw stock. These types of force were discussed earlier. The relative importance of these types of force is dependent upon the coating conditions. The coating condition to be considered in this research is the effect of shear on vehicle migration.

As mentioned earlier, the roll inclined-plane technique will be used to develop shear. The technique was first used by Arnold

in 1942 for measuring the flow properties of coating color slips at rates of shear in the range of 8.0 x  $10^4$  reciprocal seconds, which is in the range of shear produced in high-speed coating machines. (2) During the measurement, the slip is also subjected to pressures comparable to those in the actual coating nip. The testing device used consisted of an inclined glass plate, down which a smooth cylinder is allowed to roll. The roll inclinedplane apparatus used in his work is shown in Figure 3. The plane consisted of a 6 ft. by 1 ft. by  $\frac{1}{2}$  in. sheet of plate glass mounted on a 2 in. plank. One end of the plank is adjustable so that it may be raised or lowered to produce any desired angle of inclination of the glass plate. The roll was 10 cm long and 11.1 cm in diameter and weighed approximately 17 pounds. A very accurately measured quantity of coating color was placed on the incline plane and the cylinder was allowed to roll over the drop of coating color. By measuring the spread area of the tear drop pattern after passage of the roll a value of film thickness can be determined. By relating this to the speed of the roll and the roll weight, viscosity can be determined. Using the roll inclined-plane, Hemstock and Swanson (5) measured fluid penetration characteristics of coating color by what they termed the immobilization factor. The immobilization factor is defined as "the ratio of the spread area created by a bare steel roll and that created by a roll wrapped with raw stock and rolled over the drop at the same speed." (11) Hence, an immobilization factor of unity would indicate neither fluid penetration nor paper roughness. The closer the values approach unity, the slower the release from the coating color.

This report is essentially a continuation of this work. Using the roll inclined-plane technique to develop different rates



of shear, an immobilization factor for each shear rate can be determined. This changing immobilization factor will be used to evaluate what effect shear has upon the vehicle migration of coating color.

#### Materials and Methods

The roll-inclined plane apparatus used in this work was constructed as shown in Figure 3. The plane consisted of a 6 ft. by 1 ft. by  $\frac{1}{4}$  in. sheet of plate glass mounted on a 2 in. plank and inclined at a convenient angle. The roll was machined from a steel block with a highly polished surface. The roll used in this work was 4 in. in diameter, 4 in. long and weighed 8.3 lbs.

For studies of the penetration of coating color components into raw stock, the roll was wrapped with a raw stock consisting of 100% groundwood. Drops of coating color were spread between the wire side of the raw stock and the glass plane. On each series of test, five drops were spread between the wrapped roll and glass plane and five between the unwrapped roll and plane and the area of each pattern obtained. In general, the pattern areas between the plane and raw stock were somewhat more precise then the areas formed by the glass plane and the unwrapped roll. (Appendix A)

To represent the wide range of coating elements presently used in the industry, I have chosen common color components in varying grades of particle sizes. The binder chosen is latex due to its predominant industrial use. The particle sizes are: 1) 0.06 microns, 2) 0.18 microns, and 3) 0.35 microns diameter spheres. the coating pigment used will vary in particle size as follows: 1) 15% < 1 micron, 2) 30% < 1 micron, and 3) 65% < 1 micron.

By paring each binder with each pigment, nine resulting combinations were obtained. Each of these nine formulations were subjected to four ranges of shear, both eith and without a base stock substrate. These formulations were prepared at 50% total solids and 10% binder based on the toatl. The composition was purposely designed to be void of any defoamers or chemical addi-

tives that may interfere with the variable studied. A blue dye was added to color the composition to aid in determining the spread area of the coating drop. This will be discussed later in greater detail. Table 1 summarizes the conditions described above.

The size distribution of the pigment particles was determined from the 1982 Pulp & Paper Buyers guide (see Appendix B). The binder particulate spherical diameters were obtained directly from the supplier, Dow Chemical Company.

The viscosity of each color combination was determined on the Hercules High Shear viscometer. Rheograms and calculations can be found in Appendix C.

Table 1			
Base Stock	- 100% ground wo	bod	
Pigment	- No. 1 CaCO3 H	lydra Carb 65 - 65*	
	No. 2 CaCO3 F	Iydra Carb 30 - 30*	
	No. 3 CaCO3	Vicron 15-15 - 15*	
	* % less than	l micron	х. х
Binder	- Latex l - Dow	(SBR) 615A - 0.18 mi	cron dia. spheres
	Latex 2 - Dow	(SBR) 620A - 0.06 mi	cron "
	Latex 3 - Dow	(Styrene) 722 - 0.35	micron dia. spheres
Composition	- 50% total soli	ids .	0.1 grams Dispex
	10 pph binder		
*****	0.1 grams of	prusian blue dye	************
	Paired	Combinations	
<u>Color lL/1C</u>	<u>Color 2L/1C</u>	Color 3L/1C	<u>Color 1L/2C</u>
No. 1 CaC03	No. 1 CaCO3	No. 1 CaCC3	No. 2 CaCO3
Latex 1	Latex 2	Latex 3	Latex l
Color 2L/2C	Color 3L/2C	Color 1L/3C	Color 2L/3C
No. 2 CaC03	No. 2 CaCO3	No. 3 CaCO3	No. 3 CaCO3
Latex 2	Latex 3	Latex 1	Latex 2

Color 3L/3C

No. 3 CaCO3

Latex 3

#### Procedure:

The speed " $\overline{V}$ " obtained by the rolling cylinder neglecting friction can be related to the verticle height through which it moves by the formula

 $\overline{v} = \sqrt{4/3 \text{ gh}}$  (1) where: h = the vertical height, m g = acceleration of gravity, 9.807 m/s<sup>2</sup>

It should be noted that the velocity will change as the roll travels through the coating. However, the small volume of coating color used should not change this velocity appreciably.

In measuring the flow properties of coating slips or other fluids of high viscosity, a complete line of contact must be maintained between the roll and glass plane. As the roll passes over the droplet of fluid, the latter is rolled into a tear-dropped symmetrical pattern. The area of the fluid pattern can be calculated by tracing the outline with tracing paper. This tracing is then cut out and weighed. The area is then obtained by the following relationship:

 $Ag = M_{t} \quad (2)$ where:  $M_{t} = mass$  of tracing cutout, g  $(B.W.)_{t} = basis weight of tracing paper, g/m2$  Ag = spread area obtained between steelroll and glass plane, m<sup>2</sup>

This same procedure is used to determine the area of the tear shaped pattern obtained when the base stock is wrapped around the steel roll. However, no tracing is necessary. The cutout of the pattern on the raw stock is used.

$$Ap = M_p \qquad (3)$$
$$\frac{M_p}{(B.W.)_p}$$

where: M<sub>D</sub> = mass of cutout - mass of coating, g

$$(B.W.)_n = basis weight of raw stock, g/m^2$$

Ap = spread area obtained between raw stock and glass plane, m<sup>2</sup>

The coating color was dyed with prussian blue dye to aid in tracing the pattern onto the tracing paper and to define the pattern when cuttine it out on the raw stock.

The film thickness is calculated by dividing the volume of fluid used by the area of the pattern obtained. For this work a fluid volume of 0.05 cc was found to be satisfactory. Thus the film thickness is calculated as follows:

Wg = 
$$\frac{v}{Ag}$$
 (4)  
where: v = volume of coating, m<sup>3</sup>  
Ag = glass plane/steel roll spread area, m<sup>2</sup>  
Wg = film thickness of spread coating between  
the steel roll and glass plane, m

Due to its importance in the calculations, an accurate, reproducible method of measuring drop volume was necessary. This was accomplished by measuring the coating color with a hypodermic syringe. With practice and care in the complete removal of air from the barrel of the syringe, volumes can be measured within  $\mp$  0.0005 cu. cm.

By changing the height or placement of the steel cylinder the velocity will vary and hence, the corresponding shear (7). The shear rate is determined by the relationship

$$\mathcal{T}_{max} = \overline{\underline{V}}_{Wg}$$
 (5)  
where: Wg = film thickness of the spread coating drop  
between the glass plane and the steel  
cylinder, m  
 $\overline{V}$  = attained velocity of the steel roll, m/sec  
 $\overline{\mathcal{T}}_{max}$  = shear rate, l/sec.

Actually, the plane of greatest shear in the coating droplet will occur immediately ahead of the actual nip, so that the distance over which the velocity gradient extends in this plane is slightly greater than the average film thickness (14). However, it is the range of the rate of shear which is of interest rather than the exact values.

In reference to Figure 4, the steel cylinder positions are indicated as well as the coating drop position. To determine the velocity attained at each of these positions and the corresponding shear the verticle height at each of these position was measured. The angle  $\propto$  is determined as follows:

$$\propto = \text{sine}^{-1} \frac{1.375"}{48"} = 1.640$$

The other verticle positions are determined as follows:

	position l	$h_1 = 1.375"$
(48-12) sine $1.64^\circ = h_2$	2	$h_2 = 1.032"$
$(48-24)$ sine $1.64^{\circ} = h_{3}$	3	$h_3 = 0.688"$
$(48-36)$ sine $1.64^\circ = h_4$	. 4	h4 = 0.344"

From equation (1) the attained velocity at each of these positions can be determined.

 $V = \sqrt{4/3 \text{ gh}}$  (1) Thus: position 1  $V_1 = 67.67 \text{ cm/sec}$ 2  $V_2 = 58.52 \text{ cm/sec}$ 3  $V_3 = 47.85 \text{ cm/sec}$ 4  $V_4 = 33.83 \text{ cm/sec}$ 

From equation (5) the shear rates can be calculated for each position of the roll.

$$\overline{C} = \frac{\overline{v}}{\overline{w}g}$$
 (5)





FIGURE 4

In calculating the shear rate for each position of the steel roll, an average film thickness for each position is used. Although this shear rate will vary somewhat between color combination it is the range of shear produced and not exact values desired. Thus for each position the following ranges of shear rate were produced:

position: 1..... 1.5 x 10<sup>6</sup> 1/sec 2..... 1.4 x 10<sup>6</sup> 1/sec 3..... 1.3 x 10<sup>6</sup> 1/sec 4... 0.9 x 10<sup>6</sup> 1/sec

As discussed earlier, the immobilization factor "I" defined as the volume of fluid which migrates into the raw stock per unit area of fluid contact (expressed in cubic meters per square meter) will be utlized (25). The relationship is as follows:

 $I = \frac{(Ag - Ap) Wg}{Ap}$ (6)

where: Ag = spread area of drop between glass plane and steel roll,  $m^2$ 

- Ap <u>-</u> spread area of drop between glass plane and steel roll wrapped with raw stock, m<sup>2</sup>
- Wg = film thickness of spread drop between glass plane and steel roll, m

Raw data and calculations can be found in Appendix A.

#### <u>Results and Discussion:</u>

The relationships considered above suggest that the rate of migration of a fluid into a raw stock should be related to the hydrodynamic force which occrus on redistribution of the coating color. It was therefore of interest to determine whether or not the amount of fluid migration which occurred during the relatively short "dwell time" of the roll inclined-plane technique could be related to the particle sizes of the pigment and/or binder. To do so the color compositions discussed earlier prepared at 50% total solids. At this solid level the effect of viscosity variations would be minimized.

Each of the color combinations were subjected to the four shear rates discussed earlier. At each of these shear rates an immobilization factor "I" was calculated. In calculating the immobilization factor it is assumed that the color slip migrates as a one component system. However, some of the vehicle will migrate into the sheet leaving clay particles on the surface of the raw stock. When the color slip is subjected to the above high shear rates a situation will exist in which both particle plugging of the substrate pores and vehicle migration will occur.

In Figure 5, 6, and 7 graphical representation of test results is depicted. In all cases the effect of increasing shear rate is plotted against the immobilization factor "I". By graphing different pigment particle size suspensions and maintaining the binder particulate size constant, all conclusions can be drawn from these plots.

Figure 5 which depicts the smallest binder particulate size (1C) indicates that as the shear rate increases, the immobilization factor decreases. This occurs because at a zero rate of shear



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(corresponding to a unity immobilization factor) followed by the onset of low shear (position 4) the liquid in the coating drop immediately migrates into the base stock. This is indicated by the high immobilization factor or large volume of mobilized liquid. At high rates of shear (position 1) the particles, either binder or pigment, are forced into the substrate pores, preventing the movement of liquid. This is evident from a low immobilization factor or a low volume of mobilized liquid.

Also apparent is that the greatest volume of migrating liquid occurs with the smallest particulate size (1C). This is because the small binder particulate size and small pigment particle size facilitates the movement of liquid. The largest pigment size (3C) results in the smallest amount of mobilized liquid. The small particulate binder particles may be carrying the larger pigment particles into the substrate pores, plugging them. In the case of the medium-sized range of pigment particles (2C), one of these things may have occurred. One, the immobilization factor is still increasing with an increasing shear rate. Two, the data at the high shear rate position may be inaccurate. Or, three, this particular color composition is not affected by the generated shear.

In reference to Figure 6, the following conclusions can be drawn. Keep in mind that this binder was chosen to act as a buffer to the extreme situations of Figures 5 and 7.

In contrast to the conclusions theorized from Figure 5, it appears that the larger the pigment particle size the higher the immobilization factor. The larger binder particulate size results in a much higher immobilization factor for the large pigment particles of this suspension than shown in Figure 5. The



other suspensions do not appear to be as greatly affected by the varying rates of shear for this binder particulate size. This may be because the small particle size of these pigment suspensions is negated by the larger binder particulates.

The difference between Figure 5's results and those of Figure 6 are due to the larger binder particles and hence fewer total particulates available. Based on 10 pph, there will be three times as many binder particulates present in color suspensions involving the binder used in Figure 3 (binder 1L). Thus with fewer particulates present, these particulates will not be able to contribute to pore plugging. Hence, the smaller immobilization factor for the smaller pigment suspension (2C and 1C). In the case of the larger pigment particles (3C) the immobilization factor is extremely high. This indicates that a large volume of water is mobilized or is migrating into the substrate.

In Figure 7 the largest binder particulate size is represented. The larger pigment particles of suspension (3C) have a low immobilization factor indicating very little fluid penetration. Again, this is due to pore plugging from both the binder particulates and the pigment particles. The smallestpigment particles (1C) have a high immobilization factor that decreases rapidly as the shear rate increases. This behavior was also shwon in Figure 5, containing the smaller binder particulates. This phenomenon will be discussed in conclusions.

The middle ranged pigment suspensions (2C) have a low immobilization factor at a low shear with an increasing factor with increasing shear. This may be caused by the binder particulates plugging the substrate pores as the shear rate is increased. The



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The possible reason for this not occurring in the 1C suspension is that the very small particles overcome the hydrodynamic force and migrate into the base sheet at the high shear instead of plugging.

#### Conclusions

Based on the above discussion of results the following conclusions can be drawn. The most apparent conclusion is that aqueous coating slips are affected by shear. They are affected in such a way that shear causes the migration of liquid into the substrate. Pressure migration is partially determined by the viscosity of the liquid phase migrating and the particle sizes of the pigment and binder components. However, these being major, other factors must be cited as contributing in some way to the rate and extent of migration (both pressure and capillary).

- 1) the ability of the liquid phase to wet the color pigment
- 2) the ability of the liquid phase to wet the paper fibers
- 3) the interaction of the binder with the pigment and the ability of pigment-binder bonds to be broken under pressure
- 4) the pore size of the paper and the way it changes under pressure
- 5) the pore size of the pigment system and the way it changes (by changing packing) under pressure
- 6) the extent to which liquid is absorbed by the pigment, which determines how much "free" water is in the system available for migration

It is apparent that the smallest pigment particles cause the immobilization factor to begin high and decrease rapidly. This indicates that small pigment particles are more easily transported with the moving liquid. These small pigment particles cause the penetration of liquid into the base stock to cease. Pressure packs the particles more closely, tending to strip off some of the absorobed water, and forces the liquid phase through the pigment pores at a rate determined by the pigment particle size and

viscosity of the moving liquid phase. (Appendix B & C) This phenomenon may be desired in industrial applications because the binder will not be carried into the substrate resulting in better print quality.

It should be emphasized that although work of this nature enables fairly broadly based conclusions to be drawn, the fact that exceptions to those conclusions exist mean that in any one case, migration work should always be related to the specific paper, color pigment, and color binder involved. Work of the nature undertaken in this thesis is of a new nature and should be considered as a ground breaking for future work. The roll inclined-plane technique used in determining the concepts discussed in this paper may also be utilized for studies involving WRV's, viscosity theories, and numerous other areas. This apparatus can simulate theorized industrial operations more closely than other laboratory techniques and will provide for much future research.

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### APPENDIX A

# Experimental Raw Data

1L/1C

1	2	3	<sup>32</sup> 4
Ag Ap 0.113 0.115 0.119 0.94 0.115 0.115 0.111 0.111	Ag Ap 0.141 0.119 0.181 0.122 0.148 0.101 0.132 0.123	Ag Ap 0.181 0.108 0.247 0.134 0.248 0.128	Ag Ap 0.252 0.115 0.262 0.120 0.253 0.145 0.261 0.142
$\begin{array}{c} 0.117 & 0.108 \\ 0.115 & 0.109 \end{array}$	$\begin{array}{c} 0.131 \\ 0.131 \\ 0.148 \\ 0.117 \end{array}$	$\begin{array}{c} 0.191 & 0.190 \\ 0.221 & 0.125 \\ 0.218 & 0.125 \end{array}$	$\begin{array}{c} 0.251 & 0.147 \\ \underline{0.259} & 0.139 \\ 0.257 & 0.133 \end{array}$
$Wg_1 = 4.35 \times 10^{-10}$	10-7	$I_1 = 23.94 \times 1$	0-9
$Wg_2 = 3.38 \times 10^{-10}$	10-7	$I_2 = 89.56 \times 1$	0-9
$Wg_3 = 2.29 x 2$	10-7	$I_3 = 170.4 \times 1$	0-9
Wg4 = 1.95 x 1	10-7	I4 = 181.8 x 1	0-9
	4		

1L/2C

1 2		3	4	
Ag Ap	Ağ         Ap           0.104         0.098           0.100         0.089           0.104         0.086           0.102         0.109           0.103         0.101           0.103         0.097	Ag Ap	Ag Ap	
0.108 0.107		0.115 0.098	0.137 0.097	
0.097 0.085		0.108 0.109	0.116 0.095	
0.106 0.106		0.122 0.116	0.126 0.094	
0.100 0.085		0.117 0.094	0.130 0.105	
0.088 0.097		0.121 0.104	0.123 0.109	
0.102 0.096		0.117 0.104	0.126 0.100	
Wg1 = 4.90 x	10-7	$I_1 = 30.63 x$	10 <sup>-9</sup>	
Wg2 = 5.15 x	10-7	$I_2 = 31.86 x$	10 <sup>-9</sup>	
Wg3 = 4.27 x	10-7	$I_3 = 53.38 x$	10 <sup>-9</sup>	
Wg4 = 3.97 x	10-7	$I_4 = 103.2 x$	10-9	

1L/3C

1	2	3	4
$\frac{Ag}{0.126} \frac{Ap}{0.025}$	$\frac{\text{Ag}}{0 110 0 004}$	Ag Ap	Ag $Ap$
0.121 0.082	0.114 0.088	0.120 0.086	0.132 0.095
0.110 0.100	0.120 0.091	0.109 0.098	0.135 0.098
0.122 0.071	0.117 0.079	0.168 0.088	0.139 0.103
0.108 0.073	0.130 0.087	$0.141 \ 0.097$	$\frac{0.137}{0.101}$
0.117 0.000	0.120 0.080	0.130 0.091	0.139 0.098
Wg <sub>l</sub> = 4.27 x	10-7	$I_1 = 197.5 x$	10-9
$Wg_2 = 4.17 x$	10-7	$I_2 = 151.6 x$	10-9
Wg3 = 3.85 x	10-7	I3 = 165.0 x	10-9
Wg4 = 3.60 x	10-7	I4 = 150.6 x	10-9

21/1C

1	1 2		2	3		4	
<u>Ag</u> 0.141 ( 0.115 ( 0.171 ( 0.117 ( 0.131 ( 0.135 (	<u>Ap</u> 0.106 0.103 0.106 0.102 0.101 0.104	Ag 0.129 0.179 0.166 0.115 0.105 0.139	Ap 0.108 0.118 0.106 0.115 0.117 0.113	Ag 0.160 0.141 0.162 0.177 0.165 0.161	Ap 0.108 0.120 0.109 0.121 0.119 0.115	Ag 0.172 0.192 0.120 0.137 0.181 0.160	Ap 0.117 0.126 0.131 0.128 0.199 0.140
Wg <sub>l</sub> = 1	3.70 x 10	0-7		Il = ]	L10.3 x 1	10-9	
Wg <sub>2</sub> =	3.60 x 10	0-7		I <sub>2</sub> = 8	85 <b>.</b> 13 x 3	10 <sup>-9</sup>	
Wg3 =	3.11 x 1	0-7		I <sub>3</sub> = 1	L24.4 x :	10-9	
Wg4 =	3.13 x 10	0-7		I4 = 4	44.71 x 1	10-9	

2L/2C

]	L	2	3	4	
Ag 0.108 0.122 0.082 0.097 0.101 0.102	Ap 0.078 0.102 0.109 0.081 0.089 0.092	Ag Ap 0.116 0.128 0.115 0.106 0.130 0.094 0.107 0.101 0.103 0.118 0.114 0.109	Ag Ap 0.125 0.118 0.108 0.105 0.122 0.114 0.111 0.120 0.109 0.106 0.115 0.113	Ag Ar 0.113 0.11 0.126 0.11 0.124 0.12 0.135 0.11 0.133 0.12 0.126 0.11	826518
Wgl =	4.90 x 1	0-7	$I_1 = 53.26 x$	10-9	
Wg2 =	4.39 x 1	0-7	$I_2 = 20.14 x$	10-9	
Wg3 =	4.35 x 1	0-7	$I_3 = 7.70 x$	10-9	
Wg4 =	3.96 x 1	.0-7	$I_4 = 26.92 x$	10-9	

2L**/**3C

Ag 0.140 0.142 0.127 0.126 0.139 0.135	Ap 0.078 0.061 0.070 0.065 0.069 0.069	Ag Ap 0.128 0.077 0.115 0.065 0.137 0.069 0.139 0.071 0.136 0.072 0.131 0.071	Ag Ap 0.083 0.073 0.130 0.076 0.121 0.073 0.126 0.077 0.119 0.075 0.116 0.073	$\begin{array}{c c} Ag & Ap \\ \hline 0.114 & 0.065 \\ 0.082 & 0.067 \\ 0.082 & 0.075 \\ 0.095 & 0.076 \\ \hline 0.106 & 0.083 \\ \hline 0.096 & 0.073 \\ \end{array}$
Wgl =	3.70 x 10	-7	I <sub>1</sub> = 353.9 x 1	0-9
Wg <sub>2</sub> =	3.82 x 10	-7	$I_2 = 322.8 \times 1$	0-9
<sup>Wg</sup> 3 =	4.00 x 10	-7	$I_3 = 235.6 \times 1$	0-9
Wg4 <u>=</u>	5.00 x 10	-7	$I_4 = 157.5 \times 1$	0 <sup>-9</sup>

3L/1C

]	L	2		3	4
Ag 0.107 0.132 0.101 0.085 0.112 0.107	Ap 0.107 0.084 0.102 0.101 0.095 0.098	<u>Ag</u> <u>Ap</u> 0.145 0.095 0.103 0.082 0.105 0.124 0.130 0.115 0.141 0.103 0.125 0.104	1	Ag Ap 0.129 0.094 0.179 0.106 0.162 0.082 0.157 0.109 0.143 0.117 0.154 0.102	Ag Ap 0.174 0.099 0.182 0.109 0.187 0.090 0.175 0.103 0.180 0.105 0.175 0.101
Wg <sub>l</sub> =	4.67 x	10-7		$I_1 = 42.89 x$	10-9
Wg <sub>2</sub> =	4.00 x	10-7		$I_2 = 80.77 x$	10-9
<sup>wg</sup> 3 =	3.25 x	10-7		$I_3 = 165.7 x$	10-9
Wg <sub>4</sub> =	2.86 x	10-7		$I_4 = 209.5 x$	10-9

3L/2C

1		2	2		3		4	
Ag	Ap_	Ag_	Ap		Ag	Ap_	Ag	Ap_
0.116	0.089	0.134	0.104		0.134	0.111	0.135	0.120
0.088	0.095	0.124	0.100		0.111	0.093	0.111	0.104
0.102	0.107	0.122	0.097		0.122	0.105	0.121	0.100
0.136	0.110	0.127	0.112		0.132	0.117	0.137	0.118
0.114	0.100	0.125	0.109		0.135	0.115	0.135	0.117
0.111	0.100	0.126	0.104		0.127	0.108	0.128	0.112
Wg <sub>1</sub> = Wg <sub>2</sub> = Wg <sub>3</sub> = Wg4 =	4.50 x 1 3.97 x 1 3.94 x 1 3.91 x 1	LO-7 LO-7 LO-7 LO-7			$I_1 = 4$ $I_2 = 8$ $I_3 = 6$ $I_4 = 5$	49.50 x 83.98 x 59.31 x 55.86 x	10 <sup>-9</sup> 10-9 10-9 10-9	

1		2		3		4	
Ag 0.092 0.091 0.087 0.090 0.084 0.089	Ap 0.060 0.058 0.065 0.057 0.061 0.060	Ag 0.090 0. 0.088 0. 0.092 0. 0.090 0. 0.093 0. 0.091 0.	Ap .070 .081 .083 .055 .075 .073	$\begin{array}{c c} Ag & A1\\ \hline 0.110 & 0.08\\ 0.084 & 0.06\\ 0.101 & 0.08\\ 0.103 & 0.08\\ 0.099 & 0.08\\ \hline 0.099 & 0.08\\ \hline 0.099 & 0.08\\ \hline \end{array}$	231 28 29 29 29 29 29 29 29 29 29 29 29 29 29	Ag 0.105 0.110 0.095 0.114 0.107 0.106	Ap 0.095 0.084 0.097 0.096 0.089 0.092
Wg <sub>1</sub> = Wg <sub>2</sub> = Wg <sub>3</sub> = Wg <sub>4</sub> =	5.62 x 10 5.49 x 10 5.05 x 10 4.72 x 10	)-7 )-7 )-7 )-7 )-7		$I_{1} = 271.0$ $I_{2} = 135.4$ $I_{3} = 76.40$ $I_{4} = 71.82$	6 x 10 4 x 10 0 x 10 3 x 10	)-9 )-9 )-9 )-9	



10	Hydrocarb	65 -	65%	Κ	1	micron
20	Hydrocarb	30 -	30%	<	l	micron
3C	Vicron 15-	-15 -	15%	<	1	micron

## APPENDIX C

Viscosity	y Determination
(Hercules	B High Shear Viscometer)
	77
ll/lC	$5.00 \times 10^{-2} P$
1L/2C	$4.59 \times 10^{-2} P$
1L/3C	$4.85 \times 10^{-2} P$
Average	4.81 x $10^{-2}$ P
S. Dev.	.28
******	***
	η
2L/1C	$4.00 \times 10^{-2} P$
2L/2C	$3.90 \times 10^{-2} P$
2L/3C	$5.00 \times 10^{-2} P$
Average	$4.30 \times 10^{-2} P$
S. Dev.	.61
******	***************************************
	17
3L/1C	$4.00 \times 10^{-2} P$
3L/2C	$4.33 \times 10^{-2} P$
3L/3C	$4.00 \times 10^{-2} P$
Average	4.11 x $10^{-2}$ P
S. Dev.	.19
*******	***************************************
Total Ave	erage 4.41 x 10 <sup>-2</sup> P
Total S.	Dev. .46

















