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## Pigment Particle Packing and How It Effects Properties of the Coated Sheet

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PIGMENT PARTICLE PACKING  
AND HOW IT EFFECTS PROPERTIES OF THE  
COATED SHEET \

by

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A Thesis submitted to the  
Faculty of the Department of Paper Technology  
in partial fulfillment  
of the  
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Western Michigan University

Kalamazoo, Michigan

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## ABSTRACT

This study shows the relatively small effect that pigment particle packing has on properties of the coated sheet. An attempt was made to vary the degree of flocculation of the coating without effecting its other properties. The tests run on the coated sheets were brightness, opacity, gloss and K & N ink absorption. The results showed no change in both brightness and opacity with an increase in flocculation, and only slight trends in decrease in gloss and K & N ink absorption as the flocculation increased.

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## BACKGROUND AND DEVELOPMENT OF THE PROBLEM

In this study an attempt has been made to determine the effect of pigment particle packing on coated sheet properties. Particle packing refers to the way in which a particle arranges itself with respect to another particle. It includes the distance of separation as well as whether the packing is orderly or random. The study actually had two main objectives. First, to prepare coatings that when applied to a sheet will be different with respect to particle packing. Secondly, to determine whether the difference in particle packing had any effect on the characteristics of the coated sheet. If a relationship does exist between coated sheet properties and pigment particle packing, then it may be advisable to do more work in this area in an attempt to improve on coated sheet properties.

The amount of pigment particle packing can be determined by use of the relative sediment volume (RSV) measurement. The RSV is the ratio of the volume occupied by the sediment to the volume of the solid particles. RSV is defined as:

$$RSV = \frac{V_s + V_v}{V_s}$$

$V_s$  = Volume of the solid in the sediment

$V_v$  = Void volume in the sediment

$V_s + V_v$  = Total volume of the sediment

RSV differs from specific volume because it is independent of the density of the solid. The means of determining RSV of a solid liquid dispersion is to separate the solid phase from



the liquid phase. The techniques available are sedimentation, evaporation, or filtration.

From the definition it can be seen that if the solid volume is held constant, the only way to change RSV is to change the void volume. Since the void volume is directly related to the amount of flocculation of the system, it is easily deduced that changing the degree of dispersion will change the RSV and similarly the packing characteristics of the particles. Kline<sup>1</sup> showed that the measurement of degree of flocculation by the RSV method produced a curve ~~very~~ similar to the curve produced using a Brookfield viscometer to measure the degree of flocculation. In fact, the Brookfield viscometer was shown to be more sensitive to changes in flocculation than was the RSV technique. Therefore, it would seem that Brookfield viscosity is a faster, more accurate method to detect changes in the pigment particle packing.

One problem was to change the flocculation characteristics of the coating without changing any of the other properties of the coating. Chalfin<sup>2</sup> attempted to change the amount of flocculation of a coating by varying the percentage of dispersing agent. NaOH was used as the electrolyte in percentages from 0.0 - 2.1% based on clay solids. His results reconfirmed that RSV was an acceptable method for measuring the amount of flocculation in a clay-water dispersion, but was completely unsatisfactory when adhesive levels were greater than 5 pph. No

correlation was found between RSV and sheet properties. This lack of correlation can possibly be associated to the wide range of PH values incurred by varying the amount of electrolyte. Acid hydrolysis of the starch adhesive was indicated below PH 7. From this study it seems very possible that the lack of correlation between RSV and sheet properties could have been due to changes in the adhesive properties with change in PH of the system. Thus, one of the problems is to prepare a coating of various degrees of flocculation while keeping the PH constant, and thereby reduce the effect of PH on adhesive properties. No other literature was found relating RSV or pigment particle packing and sheet properties.

The theory behind the dispersion of clay is of prime importance for this study. The "Double Layer Theory" as explained by Asdell<sup>3</sup> is the most widely accepted theory for clay dispersion. The essentials of this theory are as follows. The clay particle is surrounded by an adsorbed water layer and bears a net negative charge and reacts as a negative ion and adsorbs positive ions from the surroundings to balance the charge. In water, hydrogen ion reacts with the clay to balance the clay charge forming a water hull around the clay. But, because of the small equilibrium distance that hydrogen ion has, the clay remains in a flocculated state. In this state large agglomerates are present and a great mechanical force is necessary to produce shear. The hydrogen ion can be replaced by other positive ions by a base

exchange reaction. The electrolyte must contain hydroxyl ions to neutralize the replaced hydrogen ions. Neutral salts tend to form acids that reverse the reaction. If sodium ion is used to replace the hydrogen ion of the clay, sodium clay is produced which has a large equilibrium distance and thus has negated the effect of attractive forces between the particles. Very little mechanical force is required to produce shear. If a large excess of electrolyte is added the size of the water hull is reduced and the attractive forces are again effective. The clay system will then begin to reflocculate.

On the basis of the limited amount of previous study and the double layer theory of dispersion, it should be possible to study the effect of pigment particle packing on sheet properties.



## EXPERIMENTAL SECTION

So as to be able to control the dispersion of the clay, an undispersed English Star clay was chosen as the pigment. The dry clay was screened through a #30 screen, with 590 micron openings so as to eliminate large agglomerates. The clay was dispersed at 40% solids using .3% TSPP as the dispersing agent in a Hamilton Beach high speed mixer for ten minutes.

By using a neutral salt, sodium chloride, an excess of sodium ions was introduced to the dispersed sodium clay. The sodium chloride was added to the clay dispersion in solution, in amounts varying from 0.0 - 3.0% dry sodium chloride based on clay solids. It should be noted that a visible thickening of the dispersion was observed on addition of the sodium chloride.

The adhesive chosen was a Stayco M oxidized starch. The starch was cooked at 15% solids in a double boiler for twenty minutes at 190°F. The starch was cooled to 130°F and added to the clay system in amounts of 15% of the clay solids. The coating was mixed in the Hamilton Beach mixer for five minutes at a speed slow enough so air did not become trapped in the coating.

Brookfield viscosity of the coating was measured at 100 rpm using a number 1 or 2 spindle and converted to centipoise using the chart accompanying the instrument. The PH was also measured for each coating. Drawdowns were made with each coating using a #11 Meyer rod. The sheets were allowed to air dry and then were conditioned to Tappi Standard Conditions prior to testing.

The tests that were run on the sheets were brightness, opacity, 75° gloss and K & N ink absorption. After these tests were completed the sheets were calendered with the WMU lab super-calender using 40 psi nip pressure and retested for gloss.

## DISCUSSION OF EXPERIMENTAL RESULTS

From the data displayed in Table I, relating the amount of sodium chloride added and the corresponding change in Brookfield viscosity, it is shown that the flocculation characteristics of the coating are being affected. The viscosity in at least one case more than doubled. From the assumption that viscosity is a realistic method for measuring if the pigment particle packing is affected, then the sodium chloride is truly changing the pigment particle packing. It seems reasonable to assume that by increasing the viscosity the particles are agglomerating together to form larger particles and therefore could possibly be expected to show up in the coated sheet as differences in the sheet properties.

The PH data in Table I shows a trend of a decrease in PH as the amount of sodium chloride added increased. This would indicate that the sodium ions from the TSPP were not completely replacing the hydrogen ions on the clay. Therefore, when the sodium chloride was added the remaining hydrogen ions on the clay were replaced by the sodium ions, liberating the hydrogens to form hydrochloric acid and cause a drop in PH.

The opacity data from Table I shows very little change in opacity if any. It would be impossible to say that a trend one way or the other was indicated. It could have been expected that the opacity would have increased with the increase in flocculation.

This would have been thought due to less surface contact between clay particles and therefore more clay-air interfaces. The clay-air interfaces would provide more potential scattering sites and thus an increase in opacity could be expected. By observing no apparent change in opacity, it can be assumed that the amount of clay-air interface present was not significantly different in the dispersed state and in the flocculated state, if the dispersed and flocculated states were truly present.

The data in Table I shows very little difference in the IPC brightness of the various samples. This was as expected because the relatively small change in particle size would not effect the average amount of reflectance significantly.

The K & N ink absorption data from Table I shows that a trend could possibly exist. This trend is quite small and if present at all is in a decrease in K & N ink absorption as the amount of flocculation of the particles increases. This trend can be explained as follows. If the amount of surface area is decreased by the agglomeration of the particle, and if ink absorption is due mainly to absorption on the clay surfaces, a smaller amount of surface area brought about by flocculation would lead to an ink absorption decrease. Also, because of the decreased area available for the adhesive, the coating will contain a greater excess of adhesive. It has been shown that as the amount of adhesive increases, the ink absorption decreases. Another important effect that enters into the ink absorption is the capillary action of the pores. The

larger pores that are dominant in the flocculated system would tend to produce less capillary effect on the ink and thus would have less ink drawn within it than would the more numerous, smaller pores of the dispersed system. Which ever mechanism may actually apply, at least there is a small indication that something has happened.

The data from Table II is of most interest because of the more widespread belief that gloss is very much dependant on the dispersion characteristics of the pigment. From the data for both the uncalendered and for the various degrees of calendering, there may possibly be a slight trend present. This trend would seem to be that as the flocculation of the system increased the gloss apparently decreases to a slight extent. This would follow logically from the thinking that as the particles agglomerated together and begin packing in a less orderly more bulky manner, that the amount of glossy surfaces will decrease and therefore decrease the overall gloss of the sheet. Although the trend displayed is not in contradiction with the expected, it is less.

One point that should be brought out concerning the procedure and the results is that the sheets were allowed to air dry. This could conceivably produce an effect that would be entirely different than if the sheets were flash dried in a conventional manner.



Table I. Summary Of Data, For Uncalendered Sheets.

% NaCl Added	Coating Viscosity*	PH	B&L Opacity	IPC Brightness	K&N ink**
0.0	84.5	6.95	93.0	85.4	
0.4	96.3	6.75	92.6	85.3	
0.6	132	6.70	92.7	85.1	
0.0	90	6.95	93.6	84.9	49.4
0.5	94	6.65	93.7	85.0	47.8
1.0	116	6.55	93.0	84.9	45.5
1.5	165	6.45	93.2	84.8	45.2
0.0	84	6.55	93.0	85.0	57.7
1.0	104	6.40	92.9	84.9	56.3
2.0	120	6.25	92.6	84.9	56.6
3.0	190	6.20	93.1	84.9	56.0

\*Viscosity expressed in centipoise, measured at 100 rpm using the Brookfield Viscosimeter.

\*\*K&N ink absorption measured by recording the brightness of the sheet; applying ink and allowing to absorb for two minutes; wiping off the ink and again recording brightness. The K&N ink absorption is the difference in brightness divided by the original brightness and expressed in percent.

Table II Gloss Of Coated Sheets\*

% NaCl	0 Nips	2 Nips	4 Nips	8 Nips
0.0	5.8	34.2	40.5	46.6
0.4	5.1	32.7	38.4	46.2
0.6	4.7	31.5	37.4	44.3
0.0	6.5	36.8	44.8	51.8
0.5	5.5	35.0	42.8	50.7
1.0	5.4	34.3	40.6	49.0
1.5	5.4	35.0	41.7	49.3
0.0	5.4	40.7	46.7	54.4
1.0	4.8	39.1	46.0	53.1
2.0	4.6	36.5	42.7	50.6
3.0	4.7	38.3	43.7	51.4

\*Measured at 75° with Hunter Multipurpose Glossmeter

## CONCLUSIONS

This study was undertaken to determine how variation in pigment particle packing influences coated sheet properties. From the rise in viscosity upon addition of sodium chloride to the dispersed clay, it seems safe to assume that this method was successful in changing the pigment particle packing characteristics.

There were indications that a trend in certain properties such as gloss and K & N ink absorption was observed and these trends could be explained in terms of increased flocculation. The opacity did not change which could only lead one to believe that flocculation does not effect the coating as much as might be expected.

From this approach to the pigment particle packing question and from the very slight differences observed in the different samples, it would seem that the dispersion characteristics, within the workable range of coater application, is not highly critical to sheet characteristics.

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