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MAGNESIUM OXIDE USE AS A

PIGMENT IN COATING FORMULATIONS

by

Stephen O'Driscoll

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

Western Michigan University Kalamazoo, Michigan

April, 1996

ABSTRACT

The objective of this project was to evaluate the performance of magnesium oxide as a possible pigment for use in paper coatings. The use of magnesium oxide as a pigment in coatings is not currently nor has it ever been practiced in a mill situation. The principle goal of using magnesium oxide was to increase brightness and opacity in coatings at the level of performance found with titanium dioxide at a far lower cost. The evaluation was done on the basis of rheological performance as well as optical testing. Coatings of various levels of magnesium oxide and titanium dioxide were applied to a recycled base sheet similar to that of a topliner of a coated board. The coatings were applied on the Dow Laboratory coater at low speeds with a blade applicator. All coatings were evaluated by the Brookfield viscometer as well as the Hercules high shear viscometer before coating was done so coating could be analyzed at similar viscosities.

The results during experimentation have given the following conclusions. Some shear thinning occurred with the substitution of magnesium oxide for titanium dioxide at high shear rates. Dispersion without any dispersant caused rheological problems with the magnesium oxide. At similar coat weights, the optical objectives of the experiment were satisfied with these results: equal brightness with 100% substitution of magnesium oxide for titanium dioxide, higher gardner gloss with magnesium oxide than titanium dioxide, equal opacity for pure substitution, with optimum opacity coming at an even 50/50 split of the pigments, improved smoothness with total replacement, as measured by Parker Print Surf, and a higher ink absorbtivity in the magnesium oxide coatings due to less dense coating structure containing more pores.

Overall, the use of magnesium oxide is warranted if the problems of dispersion and coater rheology are overcome or managed with the use of a dispersant. The unknowns to be explored with further study are the runnability on a machine coater and the abrasiveness of the magnesium oxide on equipment.

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INTRODUCTION

In the paper industry, anything new is evaluated based on performance versus cost. In the coated board industry there exists a need to cover a recycled, low brightness basesheet with a coating that is able to be printed with an increasingly demanding amount of detail. With relatively no other alternative, these mills have used titanium dioxide to meet the brightness and opacity standards set out in their customer specifications. The extreme cost of this method has lead to the search for cheaper alternatives to the titanium dioxide.¹

In the paper coating field, much progress has been made in the use of clays and different calcium carbonate products to produce the desired coating. Some fine carbonate products have shown to be a satisfactory substitute for titanium dioxide.¹ What titanium dioxide has, that the calcium carbonate does not, is a high refractive index of the particle itself. It also has whiter color than other fillers, which improves brightness. With a small addition of titanium dioxide, problems with optical properties have been taken care of in the past. It is this reliance on the expensive titanium dioxide that is desired to be eliminated.

With a refractive index much higher than the other fillers that replace titanium dioxide and a clear white color, magnesium oxide seems to be a reasonable alternative. The replacement of titanium dioxide requires the replacement of its performance. The focus of this work was to evaluate the performance of magnesium oxide side by side and in combination with titanium dioxide to determine the feasibility of replacing the high priced titanium dioxide with magnesium oxide either in part or in full replacement for a better price.

THEORETICAL BACKGROUND

The current trend in the coating industry points to more and more use of titanium dioxide in the formulations.¹ This is due to the greater demands for better appearing surfaces with the same coat weight. Titanium dioxide is used to improve coatings because of its small particle size to fill voids, high brightness, and high refractive index for improving opacity. The main drawback to the use of titanium dioxide is the cost. Many other pigments are being developed to replace TiO_2 . There are efforts to use calcium carbonate of small particle sizes to replicate the results found with titanium dioxide. The use of magnesium oxide for replacing titanium dioxide had not been extensively investigated before this project was proposed.

Through the guidance of Patrick Theut of Packaging Corporation of America, this project was put into motion with the hopes of developing a new pigment for use in PCA's coated board facility. He expressed a desire to reduce the cost of coating pigments through the reduction or elimination of the titanium dioxide while still meeting specifications.² Martin Marietta of Filer City, Michigan provided the magnesium oxide and **he**lpful information is designing a trial of the pigment. PCA enlisted the help of GenCorp - Specialty Polymers Division to research the possible drawbacks of using the magnesium oxide in coating formulations. Their preliminary work helped tremendously in designing this project to deal with the problems that they encountered.

Magnesium oxide is a powdered product that is commonly used in the manufacturing of rubber, plastics, and detergents. It is also used in pH and odor control as well as a middle ground to producing other magnesium compounds.^{3,4,5} Magnesium oxide itself is a white powder with very little other color. Magnesium oxide obtained from mining tends to be yellow in color, but most mined MgO is from China. In the United States at Martin Marietta, Magnesium oxide is manufactured. The process involves the calcining of salt brine and magnesite which are found as natural minerals. The dead

burned product of the 2000 K temperatures is called periclase, which is magnesium oxide.⁶ The end product is the bright white powder that the paper industry actually presses together and uses as brightness standards for calibrating brightness meters.

In preliminary investigation, the comparison of the pigments themselves and all their attributes was a basic and necessary place to start. Even though the only pigments being varied were the titanium dioxide and the magnesium oxide, data is included for calcium carbonate and clay to illustrate magnesium oxide's possible advantages. The data taken from the CRC Handbook is listed below:

Table 1 - Pigment Comparisons⁷

Pigment	shape	refractive index	hardness	specific gravity	<u>solubility</u>
calcium carbonate (calcite)	hexagonal	1.550	3.5	2.711	.0014
aluminum silicate (kaolin clay)	rhombohexagonal	1.555	-	2.580	-
titanium dioxide (anatase)	tetragonal	2.550	5.8	4.230	-
magnesium oxide (periclase)	cubic	1.736	5.5	3.650	.00062

The following data summarizes information supplied by Martin Marietta about the cost and size of the magnesium oxide particles in the sample. Cost analysis is based on pricing quoted from the fall of 1994. Several grades are available varying in particle size and purity at different prices. The sample used was the Magchem 53A, 2 micron sized particles which was a product of fair quality but not the finest product available.

	Average Size	Cost / lb	Use in Coatings
Titanium Dioxide	0.25 microns	\$1.00	Increases brightness, opacity, and density of coatings.
Magnesium Oxide Magchem 53A	2.0 microns	\$0.57	Has not been used in coatings.
Magnesium Oxide Magchem 50M	0.8 microns	\$0.55	Has not been used in coatings.
Magnesium Oxide Magchem 50	5.0 microns	\$0.25	Has not been used in coatings.

Table 2 - Magnesium Oxide vs Titanium Dioxide^{3,4,5,8}

The GenCorp company did a pigment evaluation for PCA on the replacement of titanium dioxide with magnesium oxide. The evaluation was the groundwork for this thesis. The general conclusions of the study were that the pigment dispersion problems as well as a lack of comparable performance to titanium dioxide warranted a rejection of magnesium oxide as a coating pigment. One of the recommendations of the study was to explore the use of a smaller pigment size of the magnesium oxide. The GenCorp study used a product that was an average of 11 microns in size. This size was considered too large for use in coatings, especially for use in replacing the small particle size of the titanium dioxide. The major rheology problems encountered in the GenCorp study were as the result of the attempt to disperse the magnesium oxide into a slurry at 50% solids. Another recommendation was to deal with the rheology problems of dispersion or use a product in slurry form.⁹

Other projects exploring the use of industrial gypsum and talc in coatings had similar dispersion problems to that of the GenCorp study on magnesium oxide. In the gypsum use study, the reduction of particle size was beneficial in reducing the dispersion

problems associated with that pigment.¹⁰ In the talc study, success was found when dispersant was used along with the addition of the talc to the clay slurry in amounts less than thirty parts.¹¹ In designing an experiment for using magnesium oxide, both of these improvements in dealing with dispersion were helpful.

EXPERIMENTAL DESIGN

The main design consideration in this project was the controlled direct comparison of two coating pigments without influence from any other outside factor. Therefore, all of the additions to the formulation were held constant except for the pigments. Due to the exploratory nature of the project, there was also a control run without either titanium dioxide or magnesium oxide added. The variations during the six runs are shown in Table 3 below.

Table 3 - Pigments Varied During Coating Runs

<u>#2 Clay</u>	<u>Titanium dioxide</u>	Magnesium Oxide
100	0	0
80	20	0
80	15	5
80	10	10
80	5	15
80	0	20

All coatings were simple pigment / latex systems. The latex was only added to build coat weight. All pigments were dispersed in water using a laboratory mixer. The formulations began with the mixing of the clay control formulation.

The clay control formulation was the base for all other formulations, with the titanium dioxide and magnesium oxide being added to it in the required amounts to create the six pigment ratios. The control coating consisted of Hydrasperse #2 clay. Twenty parts Dow 640 NA SBR Latex was added to the clay as well as 0.3% Dispex N-40 dispersant. The addition rate of dispersant was obtained from other projects using new pigments with dilatant behavior.^{10,11} The initial clay base formulation was made up at 60% solids. With the addition of the titanium dioxide and magnesium oxide, the coatings were made down to approximately 63%.

The clay was added to the necessary volume of water with the dispersant being added simultaneously. The latex was added after the clay was fully dispersed. A sample of this coating was taken out to be the control for the experiment. The remaining clay dispersion was divided into five equal amounts. Into each of these five different containers the required amounts of titanium dioxide and magnesium oxide were added to make them up to the other five coatings for the experiment. As the additional pigment was added, 0.3% dispersant was added along with it based on the dry pigment weight.

The resulting six coatings were tested for their rheological behavior. At the makedown solids content the coatings were tested on the Hercules high shear viscometer. The coatings were then made up to the approximate 66% solids content for the coating runs. These coatings were tested for Brookfield viscosity according to TAPPI standard T-648.¹² The coatings were tested at both 10 RPM and 100 RPM.

The coatings were then applied to the base sheet on the Dow laboratory coater. The base sheet contained 50% softwood pulp and 50% mixed office waste that has not been deinked to simulate the conditions in the topliner of boxboard. The Dow coater was run at approximately 10 feet per minute. The coatings were applied with a blade to approximately 11 g / m^2 coat weight.

The coating runs were then conditioned to standard test conditions. Since the main objective of this project was to sustain the optical qualities of the titanium coatings with the use of cheaper magnesium oxide, the following tests were performed: brightness, opacity, gardner gloss, K&N ink reduction, and parker print surface. The brightness, opacity and gloss were run according to TAPPI standards.^{13,14,15} K&N and parker procedures are described below.

The K&N ink reduction test measures the ink absorption of the surface by testing the reduction in brightness caused by the application then removal ink. The specified ink is applied to the sample with a rubber roller. The ink is left on the sample for one minute then rubbed off with paper toweling. The resulting area is tested again for brightness according to the standard. The result reported is the difference between the two

brightness values.

The parker print surface test is done automatically by equipment with the computation of the data done internally by computer analysis. The test machine does various tests making it necessary for using a different head for reporting the roughness of the surface in units of microns. The results, actually indicative of roughness, make the analysis of smoothness as it is discussed in this paper the opposite of the graphed trend.

DISCUSSION OF RESULTS

Effects of Magnesium Oxide on Rheology of Coatings

The first objective of this project was to evaluate the coatings' rheology at a known constant solids content. This was done at a target solids of 63% for the high shear viscosity and 66% for the low shear viscosity and coating runs. In Table 4 below, the exact solids content during rheology testing and coating runs for all coatings is listed.

Table 4 - Solids Content During Rheology Testing

<u>MgO parts / TiO₂ parts</u> 0 / 0	High Shear <u>Solids %</u> 60.36	Low Shear Solids % (Coating Runs) 67.5
0 / 20	62.80	66.7
5 / 15	62.44	65.1
10 / 10	63.06	66.9
15/5	63.25	65.7
20/0	63.48	65.6

Aside from the control coating, the solids contents were all in a close range for the high shear testing. The coating run solids contents were all within 1.7% of the 66% goal making for a good comparison of low shear viscosities. During the makedown of the coatings, there was an increased need for the dispersant and slow addition of the pigments as the magnesium oxide content was increased. The coatings were made down with the magnesium oxide being dispersed first to avoid the shear blocking that it would cause when added later without a dispersant. The use of the dispersant made the magnesium oxide manageable for the project. It is apparent, by the low shear viscosities, that the magnesium oxide coatings were tolerable once they were dispersed.

Once the coatings were fully dispersed, they were tested for viscosity at three different levels: 4400 RPM at high shear, 100 RPM at low shear, and 10 RPM at low

shear. The viscosity most important to this experiment was the Brookfield 100 RPM low shear reading. This viscosity represented the shear stresses that are of concern to board mills and of concern to the operation of this experiment. The 10 RPM reading was done for low shear comparison. The high shear viscosity was done on the Hercules High Shear Viscometer for the purposes of pushing the coatings to their limits and determining a potential weakness. Figure 1 below shows the two low shear viscosity trends as the coating formulations changed.



Centipoise (Thousands)

2.8 2.6 2.4 2.2 P +-1 2 1.8 1.6 1.4 1.2 1 0.8 0.6 0.4 0.2 0 20 / 0 15 / 5 0 / 20 5 / 15 10 / 10 0/0 Magnesium Oxide / Titanium Dioxide 10 RPM Viscosity + 100 RPM Viscosity

Low Shear Viscosity vs Pigment Ratio

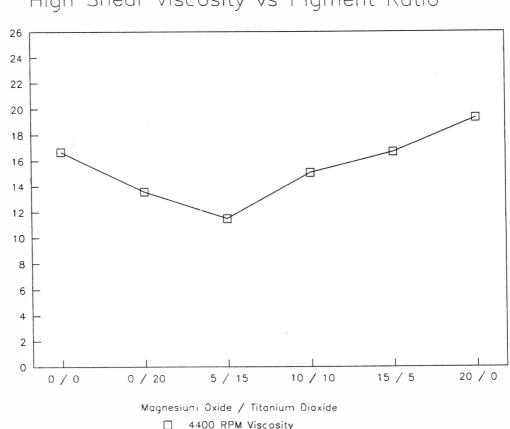
The figure above shows that, at equal solids content, the coatings with magnesium oxide performed equal to the control and the titanium dioxide coating at 100 RPM. A small spike of higher viscosity was seen at 10 RPM for the titanium dioxide coating. This was neglected due to the smooth 100 RPM trend. These results established that these

coatings could be applied to a sheet at equal conditions and produce six runs of relatively equal coat weight. The discussion of the resulting coat weights will come later with the surface characteristic analysis.

As stated before, the Hercules high shear was run to determine some of the fine differences between the use of titanium dioxide and magnesium dioxide. The running conditions in this experiment did not create enough shear for this to be of concern. Conditions encountered in the industry do reach these shear levels, so the reactions of the coatings were of interest. The viscosities at 4400 RPM are shown below in Figure 2.

Figure 2

Centipoise

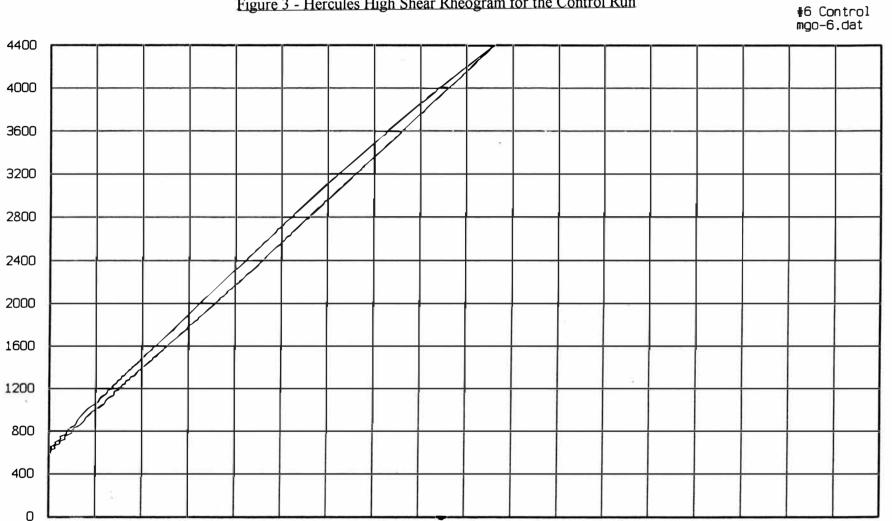


High Shear Viscosity vs Pigment Ratio

The figure above shows that, at equal solids content, the coatings with magnesium oxide have increasing viscosity values with the increase in magnesium oxide. This rise in

viscosity is the main point of concern with the magnesium oxide coatings. The magnitude of concern is lessened by the use of the dispersant to keep the viscosity down for practical use. The following six pages contain rheograms for the coatings run.

Figure 3 - Hercules High Shear Rheogram for the Control Run



RPM

BOE

Ο

TORQUE Kilodyne-cm

Figure 4 - Hercules High Shear Rheogram for 20 parts TiO₂



RPM BOE

TORQUE Kilodyne-cm

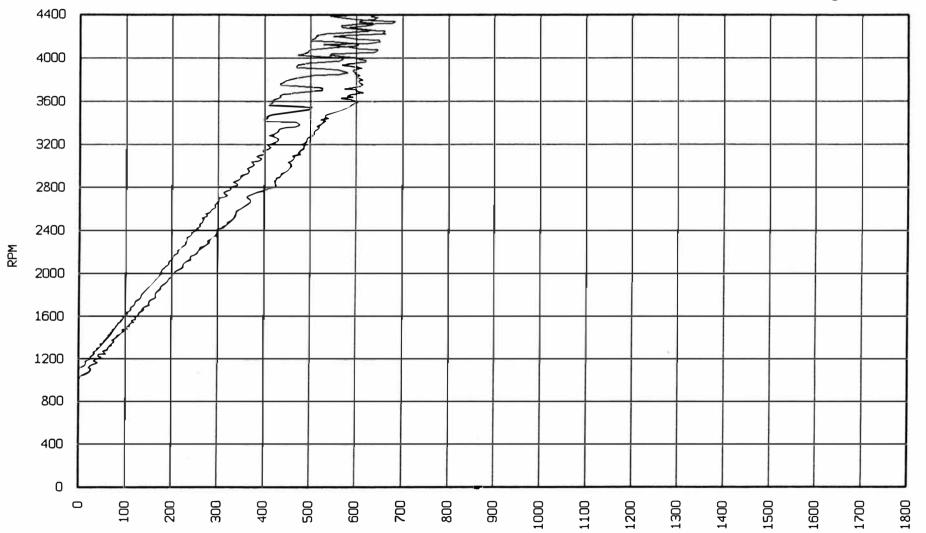


Figure 5 - Hercules High Shear Rheogram for 5 parts MgO / 15 parts TiO2

#4 15 TiO2 5 M mgo-4-1.dat

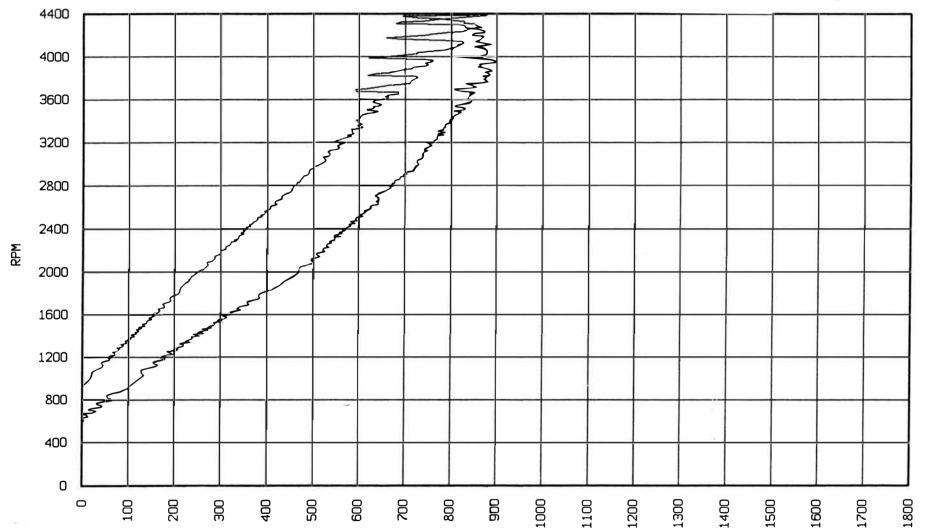
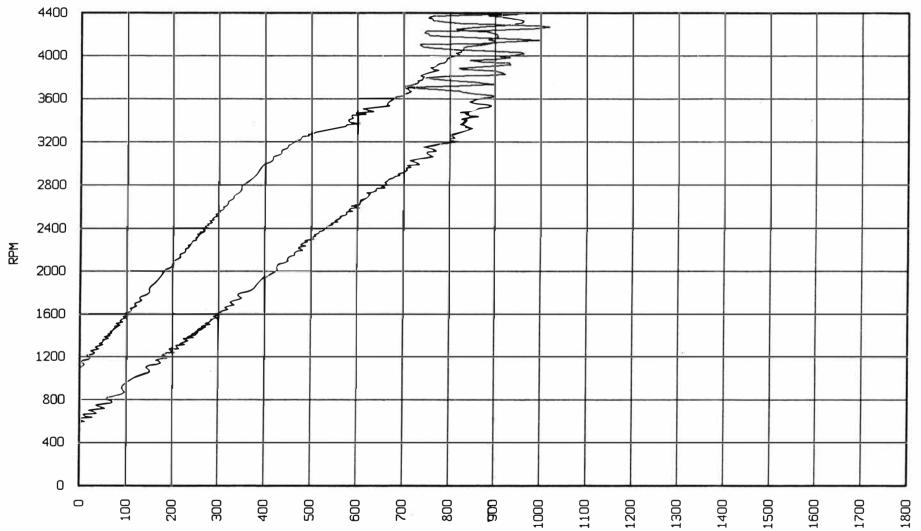


Figure 6 - Hercules High Shear Rheogram for 10 parts MgO / 10 parts TiO₂

#3 10 TiO2 10 | mgo-3-1.dat

TORQUE Kilodyne-cm

18

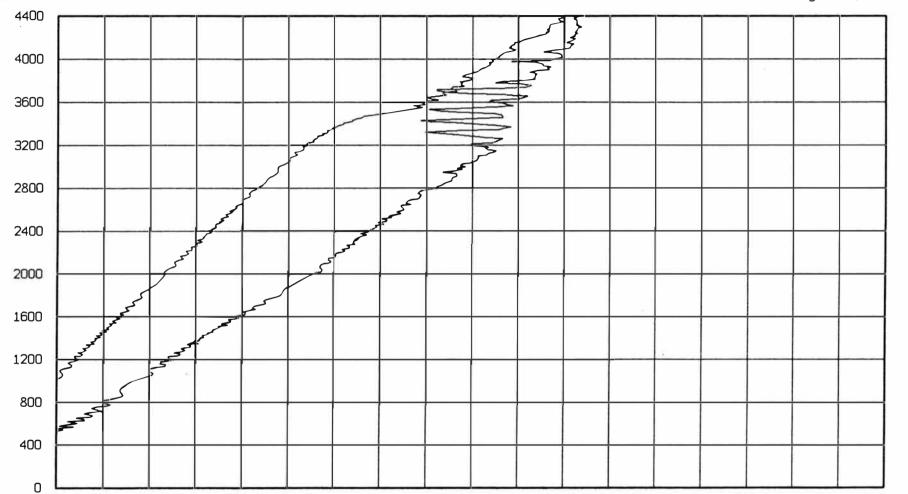


19

Figure 7 - Hercules High Shear Rheogram for 15 parts MgO / 5 parts TiO₂

#2 5 TiO2 15 M₁ mgo-2-1.dat

Figure 8 - Hercules High Shear Rheogram for 20 parts MgO



RPM

#1 20 Mg0 mgo-1-1.dat

TORQUE Kilodyne-cm

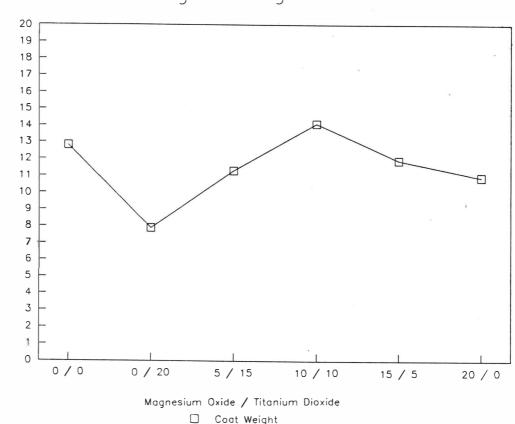
As the rheograms show, there are distinct differences between the coatings in this experiment. The control coating, shown in Figure 3 ran as expected with a near Newtonian graph with a slight thixotropic hysteresis.¹⁶ With the addition of the 20 parts of titanium dioxide, the next coating in Figure 4 changed very little. The result was actually a reduction in the viscosity at the maximum RPM. This reduction was also true of the coating with 5 parts magnesium oxide along with 15 parts titanium dioxide in Figure 5. There was a distinct increase in the hysteresis area of this rheogram over the previous two though. This indicates a greater change in the microstructure of the coating. This trend continues with the greater substitution of magnesium oxide for titanium dioxide. Both the viscosity at 4400 RPM and the hysteresis area increase for the even split of the TiO_2 and MgO in Figure 6. Due to air bubbles in the coating during the high shear runs, the rheograms have a jagged structure to them at the higher RPM ranges. Due to this inconvenience, the hysteresis area was not quantified as well as possible. There exists an obvious trend though, that the area represented by the thixotropy in the coating increases with the use of magnesium oxide. This is possibly due to the fact that the product used was not ground down to its characteristic particle size, instead it was composed of clumps of particles that break down under the high shear. Additional data including the viscosity values used and the hysteresis areas from the Hercules high shear viscometer can be found in Appendix I.

Effects of Magnesium Oxide on Optical and Surface Properties of Coatings

The principle goal of this thesis was to match the optical properties exhibited by the use of titanium dioxide by using magnesium oxide in its place. The major properties of comparison were the opacity and the brightness. These were most important since these are the two most common reasons for adding titanium dioxide to a coating formulation. The other parameters used for comparing the performance of the coatings were: Gardner gloss, Parker print surface, and K&N ink reduction. The relatively low

amount of tests for evaluation is due to the need to only evaluate the coated product for optical and surface properties only. Before comparing the six runs against each other as equals, it must be established that all six have coat weights that are within a close range. In Figure 9 below it is shown that outside of the possible low coat weight of the 20 parts titanium dioxide run and the slightly high coat weight of the 10 parts magnesium oxide, 10 parts titanium dioxide, the weights stayed near $11 \text{ g} / \text{m}^2$.

Figure 9



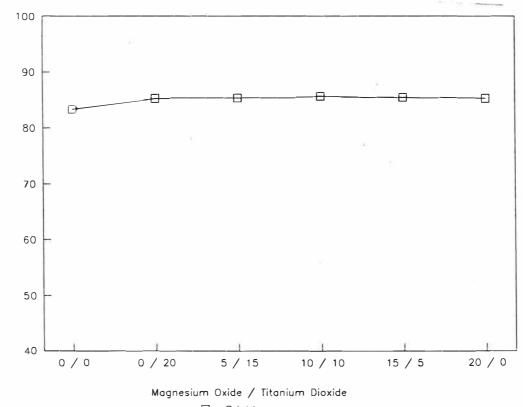
Coat Weight vs Pigment Ratio

The addition of titanium dioxide to coatings for the purpose of increasing brightness is very effective and done regularly. The need to hold up to any standard of brightness created by the coating with 20 parts titanium dioxide was paramount in this experiment. As seen in Figure 10, there is no significant difference in brightness between

Grams / Sq. Meter

the titanium dioxide and the magnesium oxide coatings. The only difference is the expected drop to the control coating that had neither of the brighteners. This match of quality was considered a positive outcome of the project.

Figure 10

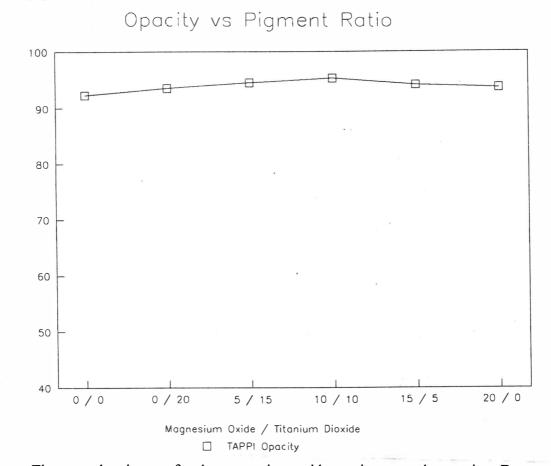


Brightness vs Pigment Ratio

Brightness

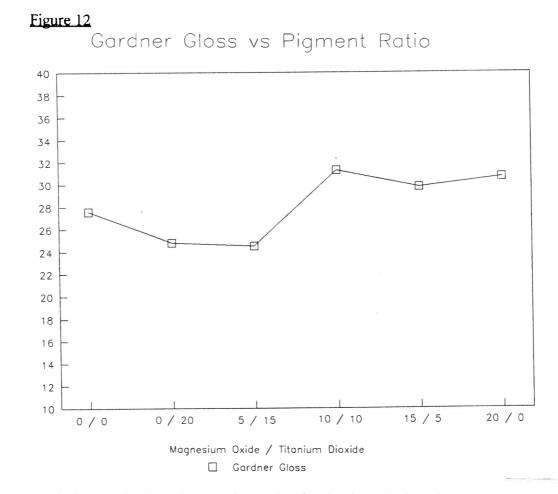
Percent





The second major test for the magnesium oxide coatings was the opacity. Due to the high refractive index of the titanium dioxide, it was not expected that the magnesium oxide would accomplish equal numbers with one to one replacement. As seen above in Figure 11, the opacity was high for all of the coatings, with the highest opacity being recorded on the even split of 10 parts titanium dioxide and 10 parts magnesium oxide. The combination of the two pigments, with their different sizes and shapes, resulted in the best light scattering of all of the coatings. This accomplishment is as significant as the brightness in that a coating covering recycled board must have the opacity to cover the inconsistencies of dirt and ink that appear in the topliner of the board being coated.

Percent

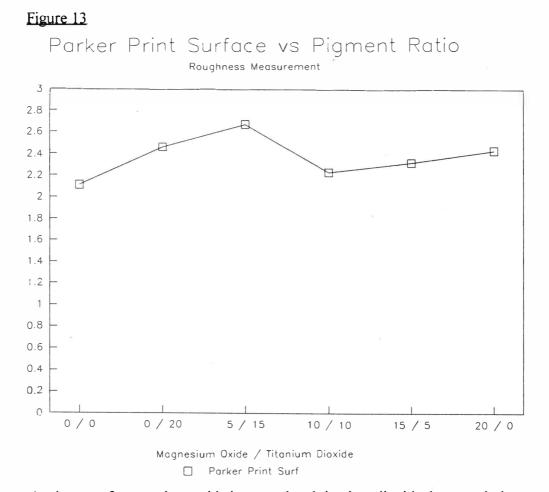


With the substitution of magnesium oxide for titanium dioxide, there was an increase in gloss of the coatings. As seen above in Figure 12, the gloss results for the last three runs with magnesium oxide at 10 parts and higher is far greater than the coatings with the predominant titanium dioxide addition. The only supposition is that the particle shape of the magnesium oxide is flatter than the titanium dioxide resulting in the high gloss values.

With an increase in gloss, it was expected that an improvement in smoothness would also occur. The Parker print surface test actually tests the roughness of the sheet with the increasing microns value meaning an increase in roughness or a loss of smoothness. The results in Figure 13 below indicate that the run with 10 parts of MgO

Gardner Gloss Units

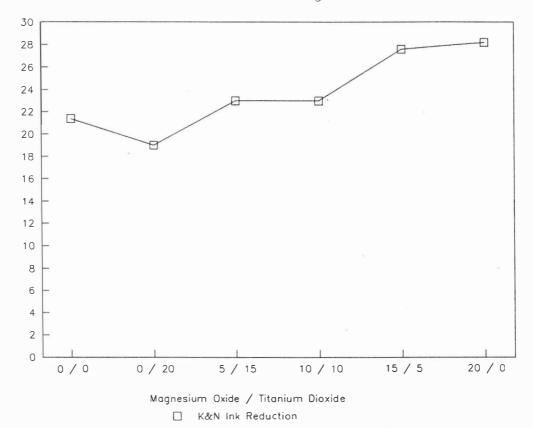
and 10 parts TiO_2 and the run with 15 parts of MgO and 5 parts TiO_2 have a smoother surface than the runs with 15 and 20 parts titanium dioxide. The results indicate that by using magnesium oxide instead of titanium dioxide, a smoother coating will be produced.



As the use of magnesium oxide increased and titanium dioxide decreased, the K&N ink reduction percentage increased. The final test of the coatings was the ink absorptivity of the surface. As seen below in Figure 14, the ink absorption after one minute increases with the content of magnesium oxide and decreases with the use of titanium dioxide in the coatings.

microns

Figure 14



K&N Ink Reduction vs Pigment Ratio

The final evaluation of the substitution for titanium dioxide with magnesium oxide is the cost analysis. The pure cost of the raw material indicates that magnesium oxide is a viable alternative, but more factors enter into the decision than the raw material price. At this point, no conclusion can be made as to whether it would be cost effective to use this or any other magnesium oxide product in an actual production environment. Further study may prove the justification for this use or some other use in the paper industry.

Percent

CONCLUSIONS

The data in this thesis indicated that the rheology of magnesium oxide slurries was inferior to that of titanium dioxide coating slurries. Some degree of shear thinning was observed along with major thixotropic behavior in the high shear rheograms of the magnesium oxide coatings. The rheological problems of dispersing the dry powdered magnesium oxide in water required the aid of a dispersant. Overall, the rheological problems with magnesium oxide were overcome to enough satisfaction with the use of the dispersant to be able to accomplish all of the runs in this project.

At similar coat weights, the optical objectives were satisfied with the following results:

- Equal brightness at every level of substitution of magnesium oxide for titanium dioxide.
- Equal opacity at levels of pure substitution, with maximum opacity coming at a level of 10 parts magnesium oxide and 10 parts titanium dioxide.
- Higher Gardner gloss values for magnesium oxide than titanium dioxide coatings.
- Improved smoothness with levels of magnesium oxide of 10 parts and 15 parts.
- Increased ink absorptivity with the increase in magnesium oxide substitution for titanium dioxide.

RECOMMENDATIONS

The multitude of different directions that the use of magnesium oxide could apply is staggering. This project was done with the coated board industry in mind as the party with the most interest in its use. After completing the work, it is evident that the entire project should have used a sub-micron magnesium oxide product. There exists a need to evaluate the entire available product line of powdered magnesium oxides from Martin Marietta. There is also a need to examine the high shear viscosity relationships of magnesium oxide with actual runnability on a high speed coater.¹⁷ In addition to the coating field, there may be extreme potential for the use of magnesium oxide as a filler for brightening a base sheet where titanium is used.¹⁸The consensus of these ideas illustrates that this paper is only an introduction of magnesium oxide into its possibilities in the paper industry.

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Appendix II

Hercules High Shear Viscosity Data

20 Parts TiO₂

Test Data filename mg0-5-1.dat Fri Apr 07 17:12 1995 Date Steve ODriscoll Operator Name Sample Number #5 20 part TiO2 Percent Solids 62.8 Initial Sample Temp (deg C) 25 Sample Density (g/ml) 1.23 Bob Size Bob E 4399 Maximum RPM Ramp Time (sec) 21 784 Kilodyne-cm (7.8 cm) Maximum Torque Bob Data Cup Radius R2 2.00 cm. Bob Radius 1.98 cm. R1 Bob Height 5.00 cm. Н Gap Clearance XO 0.02 cm. s_factor 0.00008 G_factor 0.00042 Shear Stress factor 0.0081 Shear Rate factor 10.52 CALCULATED VALUES viscosity at maximum RPM 13.6 Centipoise Shear Rate at maximum RPM 46277.48 /sec Shear Stress at maximum RPM 6350.40 dynes/cm^2 Reynolds Number at maximum RPM 16.69 Peak Mechanical Energy 6123703296.00 Ergs/cm^3 Rheogram hysteresis area 325288.00 RPM-Kilodyne-cm Kinematic Viscosity 11.07 CentiStokes

Appendix III

Hercules High Shear Viscosity Data

15 Parts TiO₂ / 5 Parts MgO

Test Data filename mgo-4-1.dat Date Fri Apr 07 18:44 1995 Operator Name Steve ODriscoll Sample Number #4 15 TiO2 5 MgO Percent Solids 62.44 Initial Sample Temp (deg C) 25 Sample Density (g/ml) 1.3 Bob Size Bob E Maximum RPM 4399 Ramp Time (sec) 21 Maximum Torque 663 Kilodyne-cm (6.6 cm) Bob Data Cup Radius 2.00 cm. R2 Bob Radius R1 1.98 cm. Bob Height Н 5.00 cm. Gap Clearance XO 0.02 cm. s_factor 0.00008

Shear Stress factor0.0081Shear Rate factor10.52CALCULATED VALUESviscosity at maximum RPM11.5 CentipoiseShear Rate at maximum RPM46277.48 /secShear Rate at maximum RPM46277.48 /secShear Stress at maximum RPM5370.30 dynes/cm²Reynolds Number at maximum RPM 20.86Peak Mechanical Energy 5178591232.00 Ergs/cm³Rheogram hysteresis area 1762760.00 RPM-Kilodyne-cmKinematic Viscosity8.86 Centistokes

0.00042

G_factor

Appendix IV

Hercules High Shear Viscosity Data

10 Parts TiO₂ / 10 Parts MgO

Test Data filename mgo-3-1.dat Date Fri Apr 07 20:14 1995 Operator Name Steve ODriscoll Sample Number #3 10 TiO2 10 Mg0 Percent Solids 63.06 Initial Sample Temp (deg C) 25 Sample Density (g/ml) 1.3 Bob Size Bob E Maximum RPM 4399 Ramp Time (sec) 21 Maximum Torque 872 Kilodyne-cm (8.7 cm) Bob Data Cup Radius R2 2.00 cm. Bob Radius R1 1.98 cm. Bob Height 5.00 cm. н Gap Clearance X0 0.02 cm. s_factor 0.00008 G_factor 0.00042

CALCULATED VALUES viscosity at maximum RPM 15.1 Centipoise Shear Rate at maximum RPM 46277.48 /sec Shear Stress at maximum RPM 7063.20 dynes/cm² Reynolds Number at maximum RPM 15.86 Peak Mechanical Energy 6811058176.00 Ergs/cm³ Rheogram hysteresis area 7416652.00 RPM-Kilodyne-cm Kinematic Viscosity 11.65 CentiStokes

0.0081

10.52

Shear Stress factor

Shear Rate factor

Appendix V

Hercules High Shear Viscosity Data

5 Parts TiO₂ / 15 Parts MgO

Test Data filename

Date Operator Name Sample Number Percent Solids Initial Sample Temp (deg C) Sample Density (g/ml) Bob Size Maximum RPM Ramp Time (sec) Maximum Torque

Bob Data Cup Radius R2 Bob Radius R1 Bob Height H Gap Clearance X0 s_factor G_factor Shear Stress factor Shear Rate factor

Fri Apr 07 21:56 1995 Steve ODriscoll #2 5 TiO2 15 MgO 63.25 25 1.3 Bob E 4399 21 964 Kilodyne-cm (9.6 cm) 2.00 cm. 1.98 cm. 5.00 cm. 0.02 cm. 0.00008 0.00042 0.0081

mgo-2-1.dat

CALCULATED VALUES viscosity at maximum RPM 16.7 Centipoise Shear Rate at maximum RPM 46277.48 /sec Shear Stress at maximum RPM 7808.40 dynes/cm² Reynolds Number at maximum RPM 14.35 Peak Mechanical Energy 7529655808.00 Ergs/cm³ Rheogram hysteresis area 4911132.00 RPM-Kilodyne-cm Kinematic Viscosity 12.88 CentiStokes

10.52

Appendix VI

Hercules High Shear Viscosity Data

20 Parts MgO

Test Data filename mgo-1-1.dat Date Fri Apr 07 23:21 1995 Steve ODriscoll Operator Name #1 20 Mg0 Sample Number Percent Solids 63.48 Initial Sample Temp (deg C) 25 Sample Density (g/ml) 1.3 Bob E Bob Size 4401 Maximum RPM Ramp Time (sec) 21 Maximum Torque 1113 Kilodyne-cm (11.1 cm) Bob Data 2.00 cm. Cup Radius R2 Bob Radius R1 1.98 cm. Bob Height Н 5.00 cm. Gap Clearance XO 0.02 cm. 0.00008 s_factor G_factor 0.00042 Shear Stress factor 0.0081 Shear Rate factor 10.52

CALCULATED VALUES viscosity at maximum RPM 19.3 Centipoise Shear Rate at maximum RPM 46298.52 /sec Shear Stress at maximum RPM 9015.30 dynes/cm² Reynolds Number at maximum RPM 12.44 Peak Mechanical Energy 8697425920.00 Ergs/cm³ Rheogram hysteresis area 6420328.00 RPM-Kilodyne-cm Kinematic Viscosity 14.86 CentiStokes