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# THE EFFECTS OF VARIOUS TYPES AND PERCENTAGES OF FILLERS WITH THE MECHANICAL PROPERTIES OF LATEX SATURATED PAPER

By

Jeffrey Alan Kuehn

A Thesis Submitted to the Faculty of The Graduate College in partial fulfillment of the requirements for the Degree of Master of Science Department of Paper Engineering, Chemical Engineering and Imaging

> Western Michigan University Kalamazoo, Michigan April 2005

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#### Jeffrey Alan Kuehn

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#### THE EFFECTS OF VARIOUS TYPES AND PERCENTAGES OF FILLERS WITH THE MECHANICAL PROPERTIES OF LATEX SATURATED PAPER

Jeffrey Alan Kuehn, M.S.

Western Michigan University, 2005

This study examined how the percentage of mineral fillers and their various types, when added to the base paper, affects the mechanical characteristics of latex impregnated paper.

A series of model handsheets were produced and subjected to physical property tests. The basis weight of these handsheets was held constant, as various types and percentages of filler were added. This base paper was then impregnated with a known acrylic emulsion. All variables with the impregnation process were held constant during the impregnation process. Physical properties were tested on the handsheets before and after impregnation. All physical tests on the handsheet samples were carried out in accordance with TAPPI procedures.

Evaluation was completed to determine the effect that different filler types have on the physical properties of impregnated paper, along with the relationship of mineral filled paper grades before and after impregnation. The results indicated moderate increases in smoothness, decreases in air permeability and reductions of all strength properties with the increased addition of mineral fillers to the base sheet of the latex impregnated paper.

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#### CHAPTER 1

#### **INTRODUCTION**

This thesis project involves the replacement of wood fiber in the base sheet of latex impregnated paper with various types and percentages of mineral fillers and examining how this affects the mechanical characteristics of latex impregnated paper. All work was completed at the research and development laboratory of Kimberly Clark Corporation Technical Paper Mill, located in Munising, Michigan.

The replacement of wood fiber in paper with filler can provide numerous benefits for the papermaker, including savings in the cost of raw materials, lower steam consumption, improved optical properties and better print qualities. The increasing cost of wood fiber is the main driver that has caused many paper manufacturers to replace a portion of their wood fiber with a filler substitute such as ground calcium carbonate (GCC), precipitated calcium carbonate (PCC) or clay. There are however, limits to the amount of filler that can be substituted for papermaking fiber. At higher filler content, papers can suffer losses in strength, stiffness and sizing. The mechanism responsible for the decrease of strength properties is the disruption of the fiber to fiber bonds due to the addition of mineral fillers.

Latex saturating of paper is designed to provide improvements in the quality of the paper it self, as measured by the dry or wet tensile strength, the bursting (mullen) strength, the folding endurance and in some instances the tear strength. Paper impregnation is a similar process to surface sizing of paper in both the process and the mechanism of pick-up or impregnation. Varying the paper structure and composition can increase or decrease dry pick-up. Some of the variables that influence sizing solution pick-up are the sizing solution concentration, filler addition percentage and type, internal

sizing and basis weight. The paper pick-up capacity is governed by the bulk properties of paper rather than by its surface roughness. Filler content can strongly influence the porosity and pore size distribution, which govern solution pick-up.

The end use of all paper dictates the overall properties that are required in the final product. Printing papers may require high opacity, excellent formation, little dusting and cost competitiveness. Release liner grades (latex impregnated) often require paper that has additional durability, excellent release, decent strength, average formation and cost competitiveness. The primary objective of latex saturating paper is to increase the physical strength properties, improve the papers liquid wetting resistance and increase the release properties.

The first two chapters will review filler addition and saturating within the paper industry. Chapter 2 specifically discusses the history of base sheet filler usage in paper making. Chapter 3 will discuss acrylic emulsions for paper impregnation along with properties and application of impregnated paper. Chapter 4 provides an in-depth review of the experimental analysis completed for this thesis project. Chapter 5 will discuss the experimental results, which will reference the raw data and graphs located in appendices A & B. Finally, Chapter 6 concludes the experimental results and discusses the benefits or draw-backs to the manufacturing of specialty paper.

#### CHAPTER 2

#### BASE SHEET FILLER USAGE

#### 2.1 History of Base Sheet Filler Usage in Papermaking

The increasing use of calcium carbonate as a filler in fine papers has been a growing trend of recent years. The resultant alkaline sheets are brighter, stronger and are more permanent than sheets that are made under acidic conditions. In addition, the use of calcium carbonate is a means of reducing the furnish costs by the substitution of kraft fiber with inexpensive filler. With these incentives, many papermakers aim to raise the filler content as much as possible, but a limitation is the loss in sheet strength. Filler particles contribute nothing to paper strength and reduce the concentration of load-bearing fibers. In addition, the filler particles accumulate on fiber surfaces, reducing paper strength by interfering with interfiber bonding.

Over the last fifteen years much of the U.S. fine paper industry has moved from acid to alkaline wet end production. This move has been facilitated to a large extent by the availability of precipitated calcium carbonate fillers produced at satellite plants close to the paper mill. The U.S. paper industry however lags behind the European fine paper industry regarding filler content. In Europe ground calcium carbonate fillers and not precipitated calcium carbonate fillers are the norm.

In the process of making paper and paper board, mineral particles such as calcium carbonate, calcium sulphate, kaolin, talc, titanium dioxide or aluminum hydroxide are often used as fillers and pigments. These inorganic materials are incorporated into the fibrous web in order to improve the quality of the resulting product. In the absence of such "fillers" the web or sheet of paper or paperboard can have relatively poor texture due to the discontinuities in the fibrous web. (1)

The use of such fillers as PCC, GCC and clay in the manufacturing of paper can provide numerous benefits, including savings in cost of raw materials, lower steam consumption and improved optical properties. There are however, limits to the amount of filler that can be substituted for papermaking fiber. Higher filler content papers can suffer losses in strength, stiffness and sizing. The web strength of a sheet of paper generally declines as filler is substituted for fiber, so preferred filler would have the least impact on fiber-to-fiber bonds and still maintain the strength of the paper web at high filler levels.

#### Previous Experimental Work

The fact that only a few papers contain more the 30% fillers while some papers contain no fillers at all suggest that fillers can cause problems. Previously published literature provides information concerning "Effects of the paper structure and composition on the surface sizing pick-up" (2), "Effects of fibers and fillers on the optical and mechanical characteristics of paper" (3). Few papers have discussed the relationship between the mechanical strength of latex impregnated papers and the type and percentage of filler additives.

#### 2.2 Common Fillers Used in the Manufacturing of Paper

#### Ground Calcium Carbonate (GCC)

Calcium carbonate deposits, in the form of chalk, limestone, or marble, are found in many countries throughout the world in large quantities and varying qualities. Most of these deposits are composed of the skeletal remains of tiny sea creatures (coccolithophore algae) that were deposited on the ocean floor over 100 million years ago.

There are two basic methods for the production of natural ground calcium

carbonate fillers: dry and wet grinding.

Natural ground calcium carbonates do not exhibit any internal porosity due to the rhombohedral particle shape of calcite and compaction over geological time frames of the various deposits. The specific area of ground calcium carbonate fillers is low, in the order of  $2 - 14 \text{ m}^2/\text{g}$  and directly proportional to the mean particle size of the pigment expressed in microns.

Natural ground calcium carbonate fillers, because of their low specific surface area, disrupt fiber-to-fiber bonds within the sheet of paper less than many other types of filler. Bown (4) compared the impact of filler content on burst strength and sheet bulk for a range of fillers. He concluded that, when compared to a scalenohedral PCC filler, natural GCC fillers have much less impact on sheet strength, develop lower opacity and exhibit less bulk. The conclusions are interrelated, as the high internal porosity of a PCC filler would be expected to develop for opacity and have a greater disrupting effect on the internal bonding of the sheet and produce more bulk.

The absence of internal porosity also means that sheet drainage is improved with the use of GCC fillers when compared to PCC fillers. This together with the relative greater strength of GCC filled sheets means that higher filler loadings can be attained, which in turn saves fiber usage. (5)

#### Precipitated Calcium Carbonate

In the past decade, precipitated calcium carbonate (PCC) has had more influence on paper fillers than any other filling pigment. The primary factor that has created the growth of PCC as a paper filler is the concept of on-site (at the paper mill) plants that have made it an economically attractive brightener and opacifier.

The basic concept to the creation of PCC is to take flue gas  $(CO_2)$  from a pulp mill calciner or a power boiler, clean it up and react it milk-of-lime to form PCC.

The real PCC growth came in uncoated printing and writing papers. The primary drivers were the availability of on-site PCC plants and the ability to substitute filler for fiber when running alkaline conditions without losing strength.

The strength gain comes from the reduction or elimination of alum, a de-bonding agent. There is nothing special about PCC or ground calcium carbonate (GCC) that would provide greater strength. The key is the reduction or elimination of alum.

#### Retention of Fillers

PCC fillers carry a surface charge when dispersed in water. This charge will usually be positive unless some other substance is absorbed on the surface to drive it negative. The most common form of PCC filler exhibits a zeta potential ranging from +3.0mV to +25 mV at a pH of 8.0. It is this positive zeta potential which is responsible for the increased retention of PCC filler over other common types of fillers that typically exhibit a negative charge.

#### 2.3 Well Founded Conclusions with Filler Addition on the Mechanical Properties of Paper

# Fillers lower paper strength and some filler types are more detrimental to paper strengths than others

It seems that since the time of ancient papermaking it has been known that fillers weaken paper. Indeed, the word filler is pejorative with connotations of adding no functionality – the more appropriate term has not been widely adapted in the paper industry. The widespread conversion from clay to calcium carbonate fillers has given the impression that clay is less strength reducing than precipitated calcium carbonate (PCC) or ground calcium carbonate (GCC). When compared at constant total area of filler per mass of paper, the burst strength followed the trend clay > talc > GCC. (6)

Natural ground calcium carbonate (GCC) has a natural rhombohedral particle shape and precipitated calcium carbonate (PCC) typically has a scalenohedral morphology (S-PCC) when used in the papermaking environment.

The scalenohedral form of PCC imparts most opacity and bulk to a sheet of paper by virtue of its morphology, which contains internal voids that scatter light. The scalenohedral PCC filler provides bulk to the sheet of paper and can effectively replace titanium dioxide despite its lower pigment refractive index; however, the internal pore volume of these pigments substantially retards the drainage of the paper web and can result in significant slowing of the production rate of a paper machine. The voluminous nature of this pigment type also significantly weakens the fiber-to-fiber bonds in a paper sheet at higher filler levels. In an attempt to reduce the pigments impact on sheet drainage, "prismatic" or rhombohedral PCC particles can be incorporated into the sheet of paper. A characteristic of precipitated calcium carbonate is their narrow particle size distribution (PSD). The narrow PSD and unique morphologies of PCC's play a major role in explaining their superior optical performance. However, particles with narrow PSD's exhibit less than optimum packing efficiencies, resulting in greater distribution of fiberfiber bonding. By blending PCC's of different particle size, the PSD is broadened, resulting in more efficient particle packing and improved sheet strength.

In addition to the scalenohedral form, PCC can be produced with a prismatic morphology. The prismatic morphology is unique in that it has a "less open" structure than scalenhedral PCC. As a result, for a given average particle size, the prismatic PCC has a lower specific surface area than scalenohedral PCC. The lower surface area of prismatic PCC is desirable in order to minimize demand for sizing chemicals at high filler levels and maximize dewatering and strength. Blends of scalenohedral and "solid particle" PCC can be optimized to achieve a balance of strength, sizing and machine runnability. Manufacture of these "solid particle" precipitated calcium carbonate pigments requires low temperature control of the precipitation process, which results in

expensive pigments. The major limitation of "prismatic" or rhombohedral PCC mineral fillers is that they do not enhance the bulk of a sheet of paper.

An increase in tensile strength can occur when particle size distribution allows for the greatest packing efficiency in which fiber-fiber bonding is less disturbed. Thus depending on requirements for specific paper grades, adjusting the average particle size of scalenohedral PCC can provide an opportunity for moderate increases in filler levels (7).





Scalenohedral(S)-PCC, in the form of single particles of filler, gives a higher bulk than GCC, which creates a relatively high voluminous structure through further interparticle flocculation. The resulting separation between the paper fibers increases, thereby increasing paper thickness and bulk. S-PCC particles increase the separation between fibers and flocs, thus reducing the occurrence of hydrogen bonds. The difference in the tensile strength between S-PCC and GCC is dramatic.

Table 1 gives an example of debonding due to surface area. A 100-gsm paper with 15% GCC filler has 33% less pigment surface area to debond vs. the corresponding PCC

#### Table 1

#### Debonding Due to Surface Area

Filler	Surface Area for m2/g	Calculated Area m2/100g sheet
PCC	8.0	120
Ground Limestone	6.0	90
Fiber Only	2.0	170
PCC	8.0	120
Ground Limestone	6.0	90
Fiber Only	2.0	170

# For a given filler type, the smallest fillers have the most detrimental effect on paper\_strength

Most fillers are available in a range of particle size distributions. Size-dependent effects include light-scattering efficiency, abrasiveness and strength loss. A good indication of the role of particle size is that the loss of burst strength increases with the total surface area of filler (i.e. with decreasing filler specific area) in a sheet made with a constant mass fractionated clay. (6)

Total surface area is also a factor in the retention of a filler in a paper web. There are two basic mechanisms that control retention of a filler in a paper web. These mechanisms are filtration retentions and absorption retention. As filler particle size increases, filtration retention increases and reaches a maximum value, with small or colloidal particles not well retained in the fiber web. Total retention increases as mean particle size increases until the absorption component, which decreases with increasing particle size, reduces total retention. Typically anionic slurries of GCC products are comprised of particles of a broad particle size range, which would not be expected to be retained well in a paper web by either a filtration mechanism or an absorption mechanism to negatively charged wood fibers.

A new range of ground calcium carbonate fillers most effectively use all of the particles present in a high solids dispersed mineral slurry, such that the bulk of the sheet of paper and its opacity are significantly enhanced. The combination of the low specific surface area of the pigment and low internal porosity provides maximum drainage to the fiber web with minimal impact on fiber-to-fiber bonding or sheet strength. This combination of properties minimizes the drying demand of a paper machine while ensuring that high production rates can be maintained.

#### The filler distribution in sheets is rarely uniform in the z (thickness) direction

Even before electron microscopy, ash measurements from split sheets revealed that fillers are not distributed uniformly in the thickness direction of paper. This reflects the complicated influences of former design, machine conditions, wet end chemistry, etc.

# The size distribution with the sheet is unlikely to equal the original size distribution of the filler dispersion

It is generally accepted that fillers often form aggregates before sheet formation, which may include fines. Furthermore, both deposition and filtration retention mechanisms predict that filler retention is particle size dependent.

2.4 Controversial Conclusions with Filler Addition on the Mechanical Properties of Paper

#### Fillers weaken paper by lowering the fiber/fiber-bonded area

This is a very old idea of unknown origin and it is the basis of all existing mathematical theories of filler induced paper strength loss. There is little evidence for this conclusion. Davidson (8) compared the light scattering from unfilled and filled sheets from which the filler had been dissolved. The latter sheet showed higher light scattering, indicating more fiber/air surfaces and thus less bonding. At a similar level of proof, if one subtracts the contribution of filler, the density of most papers decreases with increasing filler content, which suggest less fiber/fiber bonding (9).

As a speculative digression, other mechanisms are possible. One could imagine that filler particles act as flaws causing local areas of stress concentration, which initiate sheet failure. Alternatively, the additional friction caused by small filler particles in the fiber/fiber bond region might destructively interfere with shrinkage during the drying of fiber/fiber bonds. (6)

#### CHAPTER 3

#### CHEMICAL ADDITION DURING PAPERMAKING

3.1 Usage of Chemicals during Papermaking

The world paper industry is a massive user of chemical additives, for several different purposes, in both the wet end and dry ends of the process. The addition of chemicals in papermaking improves the performance, including and increasingly, in both the reduction of effluent for disposal and the quality of the product. A convenient division of the function of these chemicals is:

- (a) Process efficiency-improving chemicals, which improve efficiency of the papermachine, to keep fiber, fines and filler losses to a low level.
- (b) Performance chemicals, which change the properties of the paper, to enhance the physical strength properties of the finished product. In many instances allowing paper to be used in applications it is usually not suited for.

The principal object of addition of chemicals, or polymers, during the process of web formation is to improve the following properties of the resulting paper:

(a) Wet Strength

- (b) Elongation Before Break
- (c) Dry Strength and Toughness
- (d) Puncture Resistance
- (e) Chemical Resistance
- (f) Scuff Resistance

- (g) Tear Strength
- (h) Folding strength
- (i) Sizing
- (j) Embossing and Moulding

Impregnation of paper with acrylic ester polymer lattices can improve fold endurance, bursting strength and dry and wet tensile strength, but decrease the tear strength of the modified paper. The tensile strength of paper impregnated with acrylic lattices increases with increasing hardness of the polymers present.

#### 3.2 Acrylic Emulsions for Paper Impregnation

The addition of polymeric materials to paper has been practiced for many years as a means of improving physical properties such as tensile strength, edge tear, internal tear, elongation and wet strength. First attempts to modify paper with a resin involved precipitation of natural rubber latex with alum. Later, various synthetic lattices were used, either by alum precipitation or by deposition in combination with nitrogenous resins. Resin addition by impregnation, in a paper conversion operation, was then developed and is now a common method.

The range of usefulness of acrylic polymers is as wide as the variation in their physical properties. They have been successfully employed in such products as wallpapers and wall coverings, book covers, tape backings, signs and poster boards (especially those requiring exterior durability), filter papers, decorative and protective overlays, high strength papers and synthetic fiber papers.

Many paper impregnating resins are aqueous emulsions of acrylic polymers. These polymers have outstanding resistance to color change and to degradation caused by heat, light and chemicals.

Acrylic polymer emulsions vary from soft elastomers to hard materials with high tensile strength. They also vary in functionality – the ability of the polymer to crosslink with itself as well as with crosslinking agents.

In the polymerization of acrylic esters, functional monomers (monomers having both a polymerization group and a reactive group in the same molecule) can be introduced. Typical reactive groups include carboxyl, amido, amino, epoxy and hydroxyl. These monomers, usually added in relatively small quantities, copolymerize readily with the principal monomers in the system and impart valuable properties to the finished polymer. Pendant reactive groups in a polymer serve as sites for crosslinking.

In general the functional groups can be categorized as follows; no functional group offering crosslinking, Self crosslinking, Crosslinkable, Thermosetting.

a) Self-crosslinking

Polymers that crosslink with themselves slowly at room temperature and more rapidly at elevated temperatures. Further acceleration can be achieved through the use of acid catalysts

b) Crosslinkable

Polymers that do not respond to acid catalyst, however they crosslink to a lesser degree than the self cross-linking polymers, under the influence of elevated temperatures. They may also react to various external crosslinking agents that combine with the backbone polymer.

c) Thermosetting

Polymers that have a self-cross linking mechanism built into the polymer and reacts similar to Self-crosslinking. However, the crosslinking capacity of these resins is much greater than that of any of the self-crosslinking saturants.

#### 3.3 Properties of Impregnated Paper

The properties of impregnated paper will be a function of the base stock used – type of fiber or fiber blend, stock freeness, absorbency, basis weight and fiber length. The conditions of operation in the saturator will also influence the end result. These include the contact time of the stock with the emulsion bath and diameter, hardness and pressure of the squeeze rolls. Properties will also be dependent on resin pickup and degree of cure.

In general as polymer hardness increases, tensile strength increases and edge tear and elongation are reduced. A relatively hard polymer would therefore be used if high tensile strength or stiffness were desired. A soft polymer would provide high edge tear strength and stretch. If washing and dry cleaning resistance were important, polymers showing good solvent resistance and wet strength would be chosen. These latter properties are directly related to the level of crosslinking functionality.

There is generally an inverse relationship between tensile and edge tear strengths. Resins showing high tensile strength have poor edge tear strength. Delamination resistance property appears to be controlled principally by the internal bonding strength of the acrylic polymer-cellulose combination. The saturants giving the highest delamination resistance are all of intermediate hardness and all are self crosslinking polymers containing highly polar functional groups.

#### 3.4 Applications of Impregnated Paper

Special wrapping papers have been made by impregnating unbleached kraft paper with styrene-butadiene latex. After drying, one side of the paper is coated with a pressuresensitive resin and the other with a silicone dispersion. Paper for heavy duty sacks can be prepared by impregnating with butyl acrylate-vinyl acetate copolymer, with a significant improvement in wet strength and grease resistance. A dense and solvent-resistant paper is comprised from a web impregnated with a latex of polyvinyl acetate, an acrylic copolymer. The impregnant is filled with 20%-65% powered chalk.

Moisture-proof paper, intended for wet applications of sand paper, is impregnated with nitrile latex, followed by an alkylresorcinol-formaldehyde resin, followed with a top coating.

#### **CHAPTER 4**

#### EXPERIMENTAL PROCEDURE

#### 4.1 Overview of Experiment

Handsheets were produced with a target basis weight of  $60 \text{ g/m}^2$  using a Nobeland-Wood-type sheet former. The furnish consisted of a 70:30 blend of bleached softwood and hardwood kraft pulps beaten for 10 minutes at pH 7.0. A total of 13 different handsheet compositions were competed. The filler types and target levels are listed below

#### Table 2

	% Added		% Added		% Added
Filler Type GCC	0% (*)	Filler Type: PCC	-	Filler Type: CLAY	-
Filler Type GCC	2.5(*)	Filler Type: PCC	2.5(*)	Filler Type: CLAY	2.5(*)
Filler Type GCC	5%	Filler Type: PCC	5%	Filler Type: CLAY	5%
Filler Type GCC	7.5%(*)	Filler Type: PCC	7.5%(*)	Filler Type: CLAY	7.5%(*)
Filler Type GCC	10%	Filler Type: PCC	10%	Filler Type: CLAY	10%

#### Filler Addition Type and Percent Added

\* Duplicates created for comparison purposes

To aid in filler retention a high molecular-weight cationic polyacrylamide (C-PAM) retention aid was added to the furnish at a level of 0.05%. The sheets were pressed with a hydraulically loaded plate press at a total pressure of 300 psi., then dried on a stainless steel drying cylinder, heated with 15 psig steam, for 1 minute per side.

Impregnation of the paper was completed with a single nip laboratory saturator.

The velocity (200 Feet/minute), nip pressure (20 pounds per linear inch) and saturant temperature (24° C) was kept constant for all experiments. All sheets were impregnated with the same acrylic emulsion with the following recipe.

#### Table 3

#### Impregnation Formula

INGREDIENTS	DENSITY	% SOLIDS	AMOUNT ADDED
Water	1.0		750 grams
Rhoplex B-20	1.1	45%	600 grams
Ammonia	.9		Add until pH of solution is 8.75

#### 4.2 Handsheet Forming

The pulp furnish selected for this experiment consisted of a 70:30 blend of bleached softwood and hardwood kraft pulp. The softwood pulp was manufactured at Kimberly Clark's Terrace Bay Ontario pulp mill, the dominant wood fiber species in the softwood is Jack Pine. The hardwood pulp was manufactured at the Aracruz Brazilian pulp mill, the dominant wood fiber species in the hardwood is Eucalyptus.

Seven batches of pulp were prepared using a Valley Beater in accordance to TAPPI T 200 "Laboratory processing of pulp (beater method)". Each batch consisted of 252 (o.d.) grams of softwood pulp and 108 (o.d.) grams of hardwood pulp diluted with 23 liters of tap water, pH for each batch was measured as 7.3pH and temperature was 78°F. After soaking overnight, each batch was first disintegrated by hand and then placed in the Valley Beater for 5 minutes. After the 5 minute disintegration process was complete the Valley Beater was loaded with the standard 5500g weight and the pulp slurry was beaten a total of 10 minutes. All seven batches were then intermixed with each other to create a homogenous mixture.

Freeness of the pulp was then tested in accordance to TAPPI T 227 "Freeness of

Pulp", the results are listed in Table 4. For storage purposes and ease of handling, the pulp slurry was dewatered in the Noble and Wood sheet former and refrigerated in 20 separate zip-lock storage bags.

An additional four beater batches were prepared using the same procedures as above. These four batches were blended together, labeled and stored separately to be used in making duplicate samples for comparison purposes.

#### Table 4

#### **Pulp Freeness**

SAMPLE	Consistency	Temp.	Freeness	Freeness	Freeness	Avg.Freeness
Original Pulp	.31%	29° C	700	695	700	698
Duplicate Pulp	.32%	29° C	695	695	700	697

Stock preparation was completed in a 12 gallon plastic container. Each sample group was individually prepared according to the following. Approximately two-thirds of a zip-lock bag containing the prepared pulp, estimated weight 85 o.d. grams, was mixed with 22 liters of water. A trial handsheet was then prepared to determine the consistency of the stock solution. To obtain handsheets with the desired filler percentages of 2.5%, 5%, 7.5% and 10% the following methodology was incorporated. The initial amount of filler that was added to the stock solution was "calculated" filler amount based on stock consistency. Handsheets were then created and ash samples ran to determine the retention of the filler within the handsheets. Based on the ash content the amount of the filler added to the stock solution was adjusted to obtain the required filler content. Actual results are shown in Tables 11-22. To aid in filler retention a high molecular-weight cationic polyacrylamide (C-PAM) retention aid was added to the furnish at a level of 0.05%.

The three types of mineral fillers used for this experiment were, precipitated calcium carbonate (PCC), ultra fine ground calcium carbonate (GCC) and kaolin clay. All

were received as aqueous solutions. The precipitated calcium carbonate with the product name M-60 Spray Dried was supplied by the Mississippi Lime Company. This product had a slurry solids of 55% and a mean particle size of 0.95 micron. The ground calcium carbonate with the product name OMYAFIL<sup>®</sup> 75 was supplied by OMYA Incorporated. This product had a slurry solids of 72% and a mean particle size of 1.00 micron. The kaolin clay with the product name UW-90 was supplied Engelhard Corporation. This product had a slurry solid of 70% and a mean particle size of 0.36 micron.

Handsheets were formed using a Noble-and-Wood type sheet former producing a finished sheet size of  $9.5 \times 11.5$  inches. Target weights for the finished handsheets were between 4.02 and 4.44 grams oven dry, corresponding to a grammage of 60 g/m<sup>2</sup> with a tolerance of 5%.

After handsheets had drained in the sheet former they were couched off with blotting paper, pressed using a hydraulically loaded press plate loaded to 300 psig for 1 minute. The sheets were then dried on a stainless steel drying cylinder, heated with 15 psig steam for 1 minute per side.

A total of 300 handsheets were formed. There were a total of 13 original test groups and 7 repeat test groups of which 15 handsheets were formed for each group. The filler types and target levels are listed below:

#### Table 2

	% Added		% Added		% Added
Filler Type GCC	0% (*)	Filler Type: PCC	-	Filler Type: CLAY	-
Filler Type GCC	2.5(*)	Filler Type: PCC	2.5(*)	Filler Type: CLAY	2.5(*)
Filler Type GCC	5%	Filler Type: PCC	5%	Filler Type: CLAY	5%
Filler Type GCC	7.5%(*)	Filler Type: PCC	7.5%(*)	Filler Type: CLAY	7.5%(*)
Filler Type GCC	10%	Filler Type: PCC	10%	Filler Type: CLAY	10%

#### Filler Addition Type and Percent Added

\* Duplicates created for comparison purposes

#### 4.3 Handsheet Impregnation

The Acrylic Emulsion, Rhoplex B-20, manufactured by Rohm and Haas Company was used as the impregnating resin. Rhoplex B-20 is a non-ionic type emulsion with particle size of less than 0.1 micron and is crosslinkable at elevated temperatures. Rohm and Haas manufactures a line of "Rhoplex" resins that are particularly suited to paper impregnation because of their excellent mechanical stability. This stability is singularly important with the present trend toward faster machine speeds and longer operating times. Rhoplex resins also offer excellent adhesion, not only to cellulosic fibers, but to most of the synthetic fibers being used to modify paper substrates. These types of polymers have outstanding resistance to color change and to degradation caused by heat, light and chemicals.

All sheets were impregnated using the following saturant formula:

#### Table 3

#### Impregnation Formula

INGREDIENTS	DENSITY	% SOLIDS	AMOUNT ADDED
Water	1.0		750 grams
Rhoplex B-20	1.1	45%	600 grams
Ammonia	.9		Add until pH of solution is 8.75

Prior to impregnation the paper samples were equilibrated for 24 hours in a standard controlled humidity room (50% RH, 23° C). Each sample was weighed prior to impregnation so that saturant pick-up could be calculated.

In preparation for the impregnation process, 2 inch wide leaders were taped onto the handsheets. This was completed to help ensure that a uniform impregnation pattern was in place before the handsheet entered the saturator nip. Impregnation of the paper was completed with a single horizontal nip laboratory saturator. The velocity (200 Feet/minute), nip pressure (20 pounds per linear inch), saturant temperature (24° C) and the saturant formula were all kept constant for all experiments.

Impregnated samples were then dried on a stainless steel drying cylinder, heated with 15 psig steam for 30 seconds per side.

#### 4.4 Handsheet Testing

Physical properties of the handsheets were conducted before and after impregnation. Prior to any testing of the impregnated and unimpregnated handsheets all were temperature and humidity conditioned according to TAPPI 402 "Standard Conditioning and Testing Atmosphere for Paper, Board, Pulp, Handsheets and Related Products." All handsheets were then weighed. Five impregnated and unimpregnated handsheets for each filler percentage group were then selected based on physical condition and weight similarity. All physical tests on the handsheets were completed in accordance to the following TAPPI procedures: (deviations from the procedures are noted)

TAPPI T 411 "Thickness (caliper) of paper, paperboard and combined board", five measurements were taken per sheet and then averaged.

TAPPI T 538 "Smoothness of paper and paperboard (Sheffield method)", five wire side measurements were taken per sheet and then averaged.

TAPPI T 460 "Air resistance of paper", Air resistance was measured through (5) five layers, from which (5) five separate samples points were recorded.

TAPPI T 494 "Tensile Breaking Properties of Paper and Paperboard (Using Constant Rate of Elongation Apparatus)", at a rate of separation of the jaws 12.0 inches/min and jaw separation of 4.0 inches.

TAPPI T 414 "Internal Tearing Resistance of Paper", all five sheets cut to the standard widths were used per tear.

TAPPI T 403 "Bursting Strength of Paper"

TAPPI T 511 "Folding Endurance of Paper (MIT Tester)" Fold for the unimpregnated grades was completed with the standard 1 kilogram weight, a 2 kilogram was used for the impregnated samples.

#### CHAPTER 5

#### EXPERIMENTAL RESULTS

#### 5.1 Handsheet Forming and Impregnation Process

The forming and impregnating of the handsheets was completed with very few difficulties. The major issues encountered with forming the handsheets were obtaining the correct filler content and discovering that the proposed higher filler loaded sheets were not practical.

The poor retention of the mineral filler posed a problem with acquiring the desired filler percentage. Retention of the filler varied from 20%-50%, depending on the mineral filler type and the percent loading. To compensate for this poor retention additional filler was added to the pulp slurry. It is believed that the main mechanism for the retention of the mineral fillers was one of mechanical entrapment. The kaolin clay which had a mean particle size of 0.36 micron had about half the retention as the precipitated calcium carbonate and ground calcium carbonate whose mean particle sizes were 0.95 and 1.0 micron respectively.

The original proposed filler additions rates were 5%, 10%, 15% & 20%. Attempts were made to form handsheets at the 15% filler content. The combination of low refining, light pressing and high filler loads these sheets were of so poor strength quality that they would not have been able to survive the impregnation process. The final filler addition rates determined best for this experiment were 2.5%, 5%, 7.5% & 10 %.

The only significant problem that occurred during the impregnation process was

that an occasional handsheet would adhere to the soft nip roll causing the paper to wrap the roll and destroying the handsheet. Having discussed the usage of this equipment with Kimberly Clark laboratory staff I was warned of this potential problem prior to making the handsheets and additional handsheets were made to compensate for this potential loss.

#### 5.2 Handsheet Test Results

#### 5.2.1 Impregnated vs. Unimpregnated

The focus of this analysis was to compare unimpregnated and impregnated handsheets with 0% filler addition. Test data, for impregnated and unimpregnated handsheets with 0% filler, is located in Table 5. Additional graphical representation is available in Appendix B, Figures 8-15.

#### Saturant Pick-up

The low refining and pressing required for impregnated grades create a base sheet with below normal physical strength characteristics. This low refining and pressing creates a base sheet that is open with high bulk. This in turn allows adequate pick-up of the saturant. Average saturant pick-up for the unfilled base sheet was 27.46%.

#### Caliper

The impregnation process decreased the overall caliper of the handsheets from 6.56 to 6.14 mils. The sheets were compressed on average .42 mils.

#### **Smoothness**

With the combination of low press loading of the sheets, using blotter paper, applying minimal amount of pressure to the sheet during the drying process and lack of calendaring, the smoothness of the sheets was bound to be very poor. The unimpregnated
sheets averaged 397.4 Sheffield Units, while the impregnation process seemed to make the sheets a little smoother with average readings of 378.3 Sheffield Units. The saturant must have leveled off the peaks on the valleys of the sheet.

#### Air Resistance

As expected air resistance increased with the impregnation process. The unimpregnated sheets averaged 2.21 Gurley units of air permeability. After impregnation this rose to 4.61 Gurley units. The sheets had sealed-up twice as much.

#### Strength Properties

Acrylic polymer emulsions used for paper impregnation vary from soft elastomers to hard materials with high tensile strength. The parameter used as a criterion for polymer hardness is  $T_{300}$  – the temperature at which the torsional modulus of a polymer film of standard dimensions is 300 kilograms per centimeter (the higher the temperature, the greater the stiffness).

The acrylic emulsion used in this impregnation process, Rhoplex B-20, can be used in paper impregnation in applications such as strippable wall papers, tape stock, sandpaper backing and high endurance papers. Rhoplex B-20 is particularly suitable for paper impregnation because of its excellent mechanical stability and specific adhesion to cellulose fibers. Rhoplex B-20 is a polymer showing intermediate hardness,  $T_{300}$  of -5° C, and as such provides a good blend of properties: good tensile strength, edge tear strength and stretch and excellent folding endurance.

#### Table 5

	Unimpregnated	Impregnated
WEIGHT	4.46	5.60
(oromo)	4.40	5.09
		07.40
		27.40
	0.50	0.44
CALIPER	0.50	6.14
(mils)		
SMOOTHNESS	397.40	378.30
(Sheffield Units)		
POROSITY	2.21	4.61
(Gurley Units)		
TENSILE	3.05	9.51
(kilogram)		
ELONGATION	1.60	7.52
(%)		
TEAR	78.10	74.00
(grams force)		
BURST	4.55	51.34
(psig)		
FOLD	4.35	409.40
(# double folds)		
FOLD (log 10)	0.62	2.59
(# double folds)		
BULK	2.63	1.93
(cm³/g)		

#### Unfilled Unimpregnated Handsheets vs Unfilled Impregnated Handsheets

## <u>Tensile</u>

The tensile strength and tensile elongation both increased dramatically after the impregnation process. Tensile strength increased from an average 3.05 kilograms to 9.51 kilograms. Elongation increased from 1.60% to 7.52%.

### Tear

Elmendorf tear strength decreased after impregnation. A decrease from 78.1 grams force 74 grams force was observed. It is proposed that the work of Elmendorf tear is consumed by two processes: (a) stretching individual fibers until they break in tensile failure and (b) pulling individual fiber out of the network against frictional forces. Almost 60 years ago Van den Akker (10) hypothesized that the work of fiber failure was small

compared with the work of fiber pull-out, since the frictional forces act successively along the entire length of each fiber as it is withdrawn from the sheet. In a recent publication, Seth and Page (11) have proposed a new mechanism for tearing strength. They suggest that the energy released when fibers fail is a much greater part of the tearing energy than had been thought. Below the maximum tear strength, energy is consumed by both the release of energy upon fiber failure and by some other cause, presumably the release of energy when fiber-fiber bonds fail. For sheets bonded beyond the maximum of tear strength, the proportionality of tear strength to the square of fiber strength suggests that the release of energy upon fiber failure accounts for a large part of the work of tear. (12)

The reduction in tear after impregnation is a result of the increased tensile strength. Tear strength decreases with an increase in tensile strength because the tear forces become more concentrated at the tear apex.

### <u>Mullen</u>

As with tensile a dramatic increase was seen with Mullen after impregnation. Mullen increased on average from 4.55 pounds per square inch gauge (psig) to 51.34 psig.

### Folding Endurance

By far the largest increase in physical properties resulting from the impregnation of the paper was in the area of folding endurance. Even with the folding endurance test being modified for the impregnated paper by increasing the load weight from 1 kilograms to 2 kilograms the folding endurance was superior after impregnation. Prior to impregnation the average number of folds was 4.4 double folds when a 1 kilogram weight was applied. After impregnation and applying a 2 kilogram weight the average number of double folds increased to 409.4. Conversion of the number of double folds to the logarithm (base 10) was also completed and displayed in Table 5.

In conclusion, the acrylic emulsion chosen, Rhoplex B-20, had a medium polymer hardness which in turn influenced the physical properties of the final impregnated sheet. The final product was one that had relatively good tensile and elongation values, but also was not too stiff, allowing it to still have excellent folding endurance.

#### 5.2.2 Increasing Mineral Filler Content

The properties of the impregnated paper are a function of the base stock used – type of fiber or fiber blend, stock freeness, absorbency, basis weight and fiber length. The conditions of operation in the saturator might also affect the end result. These include the contact time of the stock with the emulsion bath, solids, viscosity and temperature of the emulsion bath and diameter, hardness and pressure of the squeeze roll. Properties of the impregnated paper will also depend on the resin pick-up and degree of cure.

The focus of this analysis is on comparing the impregnated handsheets with 0%, 2.5%, 5%, 7.5% & 10% filler addition, ground calcium carbonate OMYAFIL-75. Test data is located in Table 6. Additional graphical representation is available in Appendix B, Figures 16-25.

#### Saturant Pick-up

A decreasing trend in saturant pick-up is noticed with increasing ground calcium carbonate (GCC) addition. The variables responsible for the amount of saturant pick-up are many. The core variables include saturant formula make-up, saturator press set-up and base sheet make-up. With the addition of GCC the sheet bulk decreases. The dense non porous GCC fills the voids in this very open sheet and replaces a percentage of fiber which the saturant likes to adhere to.

### Caliper

Caliper decreased with increasing amount of GCC. The randomly-shaped particles along with the high density, allowed the filler to efficiently pack itself into the fiber network.

#### Smoothness

With increased addition of filler the overall trend for smoothness increased, which in turn means a rougher sheet at higher filler concentrations. The increase in smoothness is very subtle.

#### Air Resistance

Air resistance increased with the increased concentrations of ground calcium carbonate. An increase from 4.61 Gurley units to 6.44 Gurley units was seen from 0% filler to 10% filler respectively. With increasing amounts of filler some the voids in this very open sheet started to become filled in.

### Strength Properties

As previously mentioned the properties of the impregnated paper is a function of the base stock used – type of fiber or fiber blend, stock freeness, absorbency, basis weight and fiber length. It is also a well known fact that filler particles contribute nothing to paper strength and reduce the concentration of load-bearing fibers. In addition the filler particles accumulate on fiber surfaces, reducing paper strength by interfering with fiber-to-fiber bonding.

### <u>Tensile</u>

The trend for tensile strength was not consistent with what was expected. From 0% filler to 5% filler concentration the tensile strength increased from 9.51kg to 10.27 kg. From 5% to 10% the tensile strength did decrease as would be expected from 10.27kg to 9.34kg. Elongation on the other hand had a constant decrease as filler concentration increased.

## Tear

Elmendorf tear strength decreased with increased filler concentration. Tear values decreased from 74.6 gm force to 67.0 gm force from 0% filler to 10% filler respectively.

## Mullen

As with tear, a steady decrease with Mullen strength is seen as filler concentration is increased. Mullen decreased on average from 51.34 psig to 45.90 psig from 0% to 10% filler respectively.

## Table 6

GCC Added	0%	2.5%	5%	7.5%	10%
WEIGHT	5.69	5.61	5.54	5.47	5.54
(grams)					
PICK-UP	27.46	27.37	25.92	26.87	26.02
(%)					
CALIPER	6.14	5.85	5.63	5.61	5.62
(mils)					
SMOOTHNESS	378.30	383.70	382.40	375.10	385.60
(Sheffield Units)					
POROSITY	4.61	5.71	6.22	6.22	6.44
(Gurley Units)					
TENSILE	9.51	10.03	10.27	9.46	9.34
(kilogram)					
ELONGATION	7.52	7.45	7.20	6.93	6.79
(%)					
TEAR	74.00	73.80	69.80	71.60	67.00
(grams force)					
BURST	51.34	48.65	49.00	47.75	45.90
(psig)					
FOLD	409.40	170.80	116.20	109.90	104.20
(# double folds)					
FOLD (log 10)	2.59	2.21	2.01	2.01	1.97
(# double folds)					
BULK	1.93	1.87	1.82	1.84	1.82
(cm³/g)					

## Test Data for Ground Calcium Carbonate (GCC) Impregnated Handsheets

## Folding Endurance

By far the biggest loser in physical properties resulting from the addition of filler was in the area of folding endurance. Prior to filler addition, the average number of folds was 409 double folds, with just 2.5% filler addition the number of double folds dropped 50% to 170 and at 10% filler concentration the number of double folds dropped to 104.

In conclusion, with the addition of the ground calcium carbonate mineral filler to the base sheet of latex impregnated paper a decrease in the physical strength properties was noticed. Saturant pick-up varied little with increased filler rates. It is believed that the loss of strength was associated with the decreased fiber to fiber bonds and a weaker bond between the filler and the acrylic emulsion, since saturant pick-up varied little with the increased filler rates.

#### 5.2.3 Influence of Mineral Filler Type

The web strength of a sheet of paper generally declines as filler is substituted for fiber. Previous studies have demonstrated that this loss in strength can somewhat be diminished by the type of mineral filler being used. In most instances, the majority of the differences in physical paper properties generated by different fillers can be explained and practically predicted by the difference in their particle shape, density and size distribution.

The scalenohedral form of precipitated calcium carbonate imparts most bulk to a sheet of paper by virtue if its morphology, which contains internal voids. The voluminous nature of this pigment type significantly weakens fiber to fiber bonds at higher filler levels.

With ground calcium carbonate, the combination of low specific surface area of the pigment and low internal porosity provides minimal impact on fiber to fiber bonding, or sheet strength.

Fillers with finer particle size invariably have greater effect on strength and optical properties at a given loading than fillers with coarser particles, whereas fillers with platier particles reduce permeability and bulk.

The final properties of the impregnated paper are dependent on the base stock used – type of fiber or fiber blend, stock freeness, absorbency, basis weight and fiber length.

The focus of this analysis will be to compare unimpregnated and impregnated handsheets with 0%, 2.5%, 5%, 7.5% & 10% filler addition, of ground calcium carbonate OMYAFIL-75, precipitated calcium carbonate M-60 and kaolin clay UW-90. A summary of the test data for the impregnated sheets is presented in Table 7. Statistical results are available in Tables 8 and 9. Graphical representation of this data is available in Appendix B Figures 26-44.

#### Table 7

Summary of Test Results of Impregnated Handsheets for all Filler Types at Addition Levels from 0% to 10%

FILLER ADDITION	GCC PICK-UP	PCC PICK-UP	CLAY PICK-UP	GCC CALIPER	PCC CALIPER	CLAY CALIPER	GCC SMOOTHNESS	PCC SMOOTHNESS	CLAY SMOOTHNESS	GCC POROSITY	PCC POROSITY	CLAY POROSITY
%	%	%	%	MILS	MILS	MILS	SU	SU	SU	GU	GU	GU
0.00	27.46	27.46	27.46	6.14	6.14	6.14	378.30	378.30	378.30	4.61	4.61	4.61
2.50	27.37	27.27	27.78	5.85	5.83	5.75	383.70	384.70	371.10	5.71	5.88	543
5.00	25.92	27.65	27 41	5.63	5.91	5.74	382.40	378.40	376.20	6.22	4.96	4.89
7.50	26.87	28.35	26.71	5.61	5.76	5.67	375.10	374.60	371.00	6.22	6.13	568
10.00	26.02	29.05	25.73	562	5.79	5.65	385.60	366.40	372.40	6.44	6.27	5.70

FILLER ADDITION	GCC FOLD	PCC FOLD	CLAY FOLD	GCC BURST	PCC BURST	CLAY BURST	GCC TEAR	PCC TEAR	CLAY TEAR	GCC TENSILE	PCC TENSILE	CLAY TENSILE	GCC ELONG	PCC ELONG	CLAY ELONG
%	#	#	#	PSIG	PSIG	PSIG	GF	GF	GF	(kg)	(kg)	(kg)	%	%	%
0.00	409.40	409.40	409.40	51.34	51.34	51.34	74.00	74.00	74.00	9.51	9.51	9.51	7.52	7.52	7.52
2.50	170.80	249.15	285.15	48.65	51.55	49.65	73.80	73.40	78.70	10.03	9.92	9.29	7.45	7.08	6.99
5.00	116.20	176.20	223.80	49.00	46.70	49.50	69.80	72.60	71.20	10.27	9.65	8.85	7.20	7.80	7.41
7.50	109.90	129.95	113.30	47.75	45.45	50.25	71.60	69.10	70.00	9.46	9.27	8.66	6.93	7.07	7.08
10.00	104.20	109.50	77.60	45.90	45.40	45.70	67.00	66.40	66.60	9.34	8.66	8.01	6.79	7.10	7.02

#### Saturant Pick-up

A decreasing trend in saturant pick-up is noticed while increasing the amounts of

kaolin clay (UW-90) and ground calcium carbonate (OMYA). However saturant pick-up increased with increasing amount of precipitated calcium carbonate (M-60), at 2.5% filler the saturant pick-up was 27.27%, while at 10% filler addition the saturant pick-up increased to 29.05%. The PCC imparts most bulk to a sheet of paper by virtue of its morphology, which contains internal voids. The overall decrease and/or increase of saturant pick-up due to the addition of filler was no greater than 6.5% of the overall saturant pick-up for the non-filled paper. Statistical results for the impregnated test results suggest that the increases or decreases in pick-up for all types of fillers with addition levels of 2.5% and 7.5% are not within the 95% confidence level. The minimum saturant pick-up was 25.73% (10% kaolin clay) and the maximum pick-up was 29.05% (10% M-60).

### **Caliper**

The caliper decreased with the addition of all three types of fillers. As the addition rates increased the caliper continued to decrease. The data supports that at equal rates of filler addition the sheets containing precipitated calcium carbonate have the most bulk. Statistical results for the impregnated test results suggest that the decrease in caliper for all types of fillers with addition levels of 2.5% and 7.5% are within the 95% confidence level.

#### Smoothness

Extremely high values were recorded for smoothness with all filled and unfilled unimpregnated sheets. Values ranged from 393.4 to 400 Sheffield Units for the unimpregnated paper and from 366.4 to 386.8 Sheffield Units for the impregnated paper. The only dominant trend appears to be with the addition of the kaolin clay, with a smoother sheet being created both before and after impregnation compared to all the other sheets. Statistical results for the impregnated test results suggest that the increases or decreases in smoothness for all types of fillers with addition levels of 2.5% and 7.5% are

not within the 95% confidence level.

#### Air Resistance

One of the requirements of the base sheet for impregnation grades is to have a rather open sheet. The low refining and light pressing imparts this desired property into the sheet. Air resistance increased with the increased concentrations of all the mineral filler additions. Ground calcium carbonate slightly increases air resistance more than kaolin clay and precipitated calcium carbonate. Statistical results for the impregnated test results suggest that the increases in air resistance for all types of fillers with addition levels of 2.5% and 7.5% are within the 95% confidence level.

#### Strength Properties

The strength properties of latex impregnated paper come from a multitude of inputs, a few of these being; the strength of the base sheet, the type and amount of saturant applied and the curing and aging conditions of the saturant. Out of these the strength of the base sheet and the saturant pick-up are affected by the addition of mineral fillers. Additional variables such as adhesion strength between the fiber, mineral filler and acrylic emulsion may have also influenced the strength properties.

## <u>Tensile</u>

The lowest tensile strength sheets of the unimpregnated paper contained kaolin clay as the filler; the sheets containing the PCC & GCC had slightly higher and similar tensile strength characteristics. Each of the saturated samples displayed decreasing tensile strength with increasing filler addition, as presented in Figure 6. The sheets containing the kaolin clay were of the lowest tensile strength, while those containing the GCC had the highest tensile strength. The tensile strength of the PCC & GCC filled sheets at 2.5% & 5% actually had greater or equal tensile than the non-filled impregnated samples. Elongation for the impregnated sample test results revealed decreasing elongation with

increasing filler addition, as presented in Figure 7. Though a decreasing trend was present with both tensile strength and elongation a confidence level of 95% was not met for impregnated test results with addition levels of 2.5% and 7.5%.

#### <u>Tear</u>

Elmendorf tear strength decreased with increased filler concentration in both unimpregnated and impregnated samples. The unsaturated samples containing the PCC (M-60) were impacted the worst. For those samples containing the GCC (OMYA), from 0% to 10%, the tear values were greater than the unfilled sheets. The tear values of the saturated samples were similar across all filler addition rates. The strength of the saturated samples at 0% filler is 74 grams force and at 10% filler addition the values for all three filler types is between 66.4-67 grams force.

#### <u>Mullen</u>

Extremely low values of Mullen strength was recorded for all unimpregnated grades; one observation worth noting on the unimpregnated grades is that the highest value was that of the grade with 2.5% addition of the kaolin clay (UW-90), as presented in Figure 41.

The test results of the impregnated paper for Mullen strength displayed decreasing strength with increasing filler addition rates. The filler that least impacts the Mullen strength at most addition rates is the kaolin clay (UW-90), as presented in Figure 4.

#### Folding Endurance

As with Mullen, extremely low values were observed with the folding endurance of the unimpregnated paper. At addition rates of 10%, for all filler types, the test samples were unable to complete even 1 double fold. On average the GCC mineral additive demonstrated slightly greater folding endurance than the PCC and kaolin clay, with the kaolin clay being the poorest performer. Figure 43 represents these results

The addition of filler to the base sheet for the impregnated grades severely decreased the folding endurance. The values ranged from 409 double folds with 0% mineral additive, to 77 double folds with 10% kaolin clay. An addition of only 2.5% mineral additive decreases the number of double folds by at least 30%, as seen in Figure 3. At the higher addition rates of 7.5% and 10% PCC has the greatest folding endurance. It is believed that the higher percent pick-up of saturant observed with this filler type promoted the greater folding endurance. Statistical results for the impregnated test results suggest that the decrease in double folds for all types of fillers with addition levels of 2.5% and 7.5% are within the 95% confidence level.

#### 5.2.4 Statistical Comparison of Duplicate Handsheets

Comparison of the duplicate handsheets with the original handsheets was completed by reviewing the similarities with the averages of the tests results for each group of handsheets and by observing the similarities between the standard deviations of the raw data. The margin of error was reviewed by calculating the Z-values and correlating that to the confidence level. Table 8 and 9 located on the next pages present these results.

Only a few dissimilarities were readily apparent when comparing the averages between the original and repeat handsheets. The tear averages of sheets that contained 2.5% UW-90, kaolin clay, were 72.8 (gf) for the original handsheets and 84.6 (gf) for the repeat handsheets. The repeat average is comparably higher than any of the other handsheets produced; in addition standard deviation was excessively high for this data group. Upon further analysis of the data it was noticed that one data point was nearly twice as great as the rest of the data points.

Standard deviations appeared to be low in all test categories except for the Folding Endurance. At first the large standard deviations were somewhat disturbing, but

after further analysis it was realized that standard deviations were consistently high for all filler percentages and filler types. TAPPI test method T 511, "Folding Endurance of Paper (MIT Tester)" explains that the greatest source of test variability is that the folding stresses are applied to a very small area of the paper. Failure occurs at this point and not, as in normal tensile test, at the weakest point in the test strip. Thus, "within sample" variability is great.

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Impregnated Test Results Comparison

		0% FIL	LER	DUP. 0%	FILLER	2.5% C	LAY	DUP. 2.5	% CLAY	7.5% C	LAY	DUP. 7.59	CLAY		2.5% P	сс	DUP. 2.59	6 PCC	7.5% P	сс	DUP. 7.59	6 PCC	
		AVG.	STD	AVG	STD	AVG.	STD	AVG	STD	AVG.	STD	AVG	STD	0 married	AVG.	STD	AVG	STD	AVG.	STD	AVG	STD	and and a second
	WEIGHT	5.73	0.17	5.64	0.03	5.55	0.08	5.54	0.06	5.53	0.08	5.51	0.08	0.09	5.61	0.07	5.54	0.10	5.65	0.11	5.59	0.04	0.10
	PICK-UP	27.44	0.69	27.49	0.49	27.47	2.38	28.10	0.23	26.92	0.00	26.50	0.00	1.04	27.39	0.75	27.16	0.69	28.00	0.28	28.69	1.42	0.80
	CALIPER	6.12	0.17	6.15	0.18	5.77	0.09	5.73	0.11	5.71	0.15	5.64	0.25	0.17	5.87	0.26	5.78	0.11	5.77	0.07	5.74	0.13	0.17
	SMOOTHNESS	378.00	4.36	378.60	3.85	370.40	5.37	371.80	3.11	370.60	7.44	371.40	10.19	6.21	386.80	2.77	382.60	8.68	382.20	4.15	367.00	7.97	5.74
	POROSITY	4.62	0.14	4.61	0.14	5.56	0.19	5.30	0.14	5.60	0.18	5.77	0.18	0.16	6.24	0.28	5.52	0.32	6.08	0.28	6.17	0.38	0.27
	TENSILE	9.37	0.39	9.65	0.54	9.31	0.70	9.28	0.46	8.72	0.43	8.59	0.52	0.52	10.27	0.27	9.58	0.95	9.60	0.24	8.93	0.62	0.56
	ELONGATION	7.52	0.36	7.53	0.47	7.00	0.38	6.98	0.33	7.15	0.44	7.00	0.55	0.43	7.46	0.23	6.69	0.23	7.20	0.44	6.95	0.63	0.42
	TEAR	73.40	2.97	74.60	5.81	72.80	1.30	84.60	27.15	69.60	3.58	70.40	2.51	11.55	72.80	1.30	74.00	1.22	70.40	2.97	67.80	1.64	3.09
	BURST	52.38	2.94	50.30	2.98	49.90	6.01	49.40	4.38	49.50	6.60	51.00	1.82	4.47	53.10	5.34	50.00	4.69	43.70	3.53	47.20	4.47	4.09
ω	FOLD	409.50	103.22	409.30	165.77	274.20	109.64	296.10	72.50	111.90	36.36	114.70	30.54	98.04	276.10	92.47	222.20	92.51	132.80	30.89	127.10	47.65	98.71
9	FOLD (log 10)	2.60	0.13	2.58	0.17	2.40	0.19	2.46	0.11	2.03	0.13	2.04	0.12	0.14	2.42	0.15	2.31	0.21	2.11	0.10	2.08	0.16	0.16

	2.5% G	сс	DUP. 2.5	% GCC	7.5% 0	SCC	DUP. 7.5	% GCC	
	AVG.	STD	AVG	STD	AVG.	STD	AVG	STD	0
WEIGHT	5.68	0.06	5.54	0.06	5.51	0.07	5.43	0.10	0.09
PICK-UP	27.55	0.53	27.19	0.64	27.75	1.01	25.99	0.79	0.71
CALIPER	5.96	0.09	5.73	0.14	5.60	0.17	5.62	0.10	0.15
SMOOTHNESS	385.40	4.28	382.00	7.97	378.00	7.91	372.20	8.98	6.57
POROSITY	5.52	0.07	5.89	0.26	6.06	0.21	6.38	0.59	0.29
TENSILE	10.11	0.66	9.96	0.32	9.45	0.58	9.47	0.73	0.55
ELONGATION	7.56	0.42	7.34	0.32	7.14	0.64	6.72	0.49	0.46
TEAR	74.40	3.91	73.20	3.49	70.20	2.17	73.00	5.20	4.12
BURST	48.90	4.04	48.40	3.75	47.80	4.42	47.70	4.16	3.76
FOLD	146.30	49.31	195.30	63.16	105.40	39.56	114.40	52.28	90.23
FOLD (log 10)	2.14	0.14	2.27	0.13	2.00	0.15	2.02	0.19	0.15

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-	Average 0%	Average 2.5%	Average 7.5%	Z-Value	Z-Value	Average 2.5%	Average 7.5%	Z-Value	Z-Value	Average 2.5%	Average 7.5%	Z-Value	Z-Value
	0% FILLER	2.5% CLAY	7.5% CLAY	2.5%	7.5%	2.5% PCC	7.5% PCC	2.5%	7.5%	2.5% GCC	7.5% GCC	2.5%	7.5%
				1.1									
WEIGHT	5.69	5.55	5.52	1.50	1.82	5.57	5.62	1.16	0.68	5.61	5.47	0.86	2.39
PICK-UP	27.46	27.78	26.71	0.31	0.73	27.27	28.35	0.24	1.10	27.37	26.87	0.13	0.83
CALIPER	6.14	5.75	5.67	2.29	2.75	5.83	5.76	1.88	2.28	5.85	5.61	1.96	3.60
SMOOTHNESS	378.30	371.10	371.00	1.16	1.18	364.70	374.60	1.12	0.64	383.70	375.10	0.82	0.49
POROSITY	4.61	5.43	5.68	4.99	6.53	5.88	6.13	4.66	5.57	5.71	6.22	3.77	5.55
TENSILE	9.51	9.29	8.66	0.42	1.66	9.92	9.27	0.74	0.43	10.03	9.46	0.94	0.10
ELONGATION	7.52	6.99	7.08	1.25	1.04	7.08	7.07	1.07	1.08	7.45	6.93	0.16	1.28
TEAR	74.00	78.70	70.00	0.41	0.35	73.40	69.10	0.19	1.59	73.60	71.60	0.05	0.58
BURST	51.34	49.65	50.25	0.38	0.24	51.55	45.45	0.05	1.44	48.65	47.75	0.71	0.95
FOLD	409.40	285.15	113.30	1.27	3.02	249.15	129.95	1.82	2.83	170.60	109.90	2.64	3.32
FOLD (log 10)	2.59	2.43	2.04	1.09	3.81	2.38	2.09	1.44	3.14	2.21	2.01	2.48	3.76

# Impregnated Test Results Confidence Intervals

#### **CHAPTER 6**

#### CONCLUSIONS

The reasons for adding mineral fillers to paper vary from cost savings to obtaining improved print quality, opacity, brightness and overall appearance. There has never been an argument that the addition of filler will improve the strength properties of paper.

With the introduction of mineral fillers in the base sheet of impregnated paper there is a decrease in the physical strength properties. A summary of the results of the strength tests conducted, Fold, Mullen, Tear and Tensile, are presented in Figures 3-6 and Table 7.

The strength property that decreased the most with the addition of any mineral filler was Folding Endurance. A steep drop of at least 30% was noticed with only 2.5% filler addition. With 0% filler addition the number of double folds was 409, with the addition of 2.5% Kaolin Clay the number of double folds declined to 285, at 2.5% addition of PCC the number of double folds was 249, and at 2.5% GCC the number of double folds was 185. Respectively at a 10% addition rate the number of double folds was 78 for Clay, 109 for PCC and 105 for GCC.

A trend of decreasing strength with increasing amounts of filler was also noticed for Burst, Tear and Tensile. With the maximum addition rate of 10% filler, an average of 11% decrease in strength was measured for Burst, an average of 9.5% decrease in strength was measured for Tear and an average of 8.8% decrease in strength was measured for Tensile.

Each of the mineral fillers impacted the various strength properties differently. For Fold the addition of Kaolin Clay resulted in higher strength paper when compared to impregnated paper filled with equal amounts of PCC & GCC. For Burst the addition of

Kaolin Clay resulted in higher strength paper when compared to impregnated paper filled with equal amounts of PCC & GCC. For tensile the addition of GCC resulted in higher strength paper when compared to impregnated paper filled with equal amounts of PCC & Clay.



Figure 2. Double Fold as a function of Filler Content for Handsheets containing GCC, PCC and Kaolin Clay



Figure 3. Burst as a function of Filler Content for Handsheets containing GCC, PCC and Kaolin Clay



Figure 4. Tear Strength as a function of Filler Content for Handsheets containing GCC, PCC and Kaolin Clay



Figure 5. Tensile Strength as a function of Filler Content for Handsheets containing GCC, PCC and Kaolin Clay



Figure 6. Tensile Elongation as a function of Filler Content for Handsheets containing GCC, PCC and Kaolin Clay

With the increased addition of Clay and PCC the handsheets smoothness improved. The improvements of smoothness were very subtle. The addition of Clay seemed to improve the sheet smoothness the best, but from 0% to 10% addition smoothness minimally increase from 378.3 SU to 372.4SU. Air permeability decreased with both the saturation of the handsheets and the addition of mineral fillers. This was to be expected since the handsheets were formed with long fiber pulps that were lightly refined and pressed, resulting in a very open, rough sheet.

A common manufacturing process for many years has been the impregnation of paper with various types of lattices and acrylic emulsions. Impregnation of paper can improve fold endurance, bursting strength, dry and wet tensile, along with oil and water resistance. Applications of impregnated paper range from special wrapping papers, release liners grades, printed decorative overlay, book cover, sandpaper backing, masking tape paper, jeans labels and veneer backing.

Many of the end-use products of latex impregnated paper require durability and strength. The introduction of mineral fillers into the base sheet retards many of these desired strength properties. The benefits of mineral fillers in traditional papermaking, such as higher opacity, greater brightness, increased bulk, improved print quality and a low cost substitute of fiber may not be as important.

Though there was a reduction in all of the strength properties tested with the addition of mineral fillers to the base sheet of latex impregnated paper this loss in strength may be acceptable for certain applications of latex saturated paper. Applications such as medical packaging grades, release liner grades, and moisture and oil resistance grades, in which properties such as formation, release and porosity are of key importance.

#### CHAPTER 7

#### **RECOMMENDATION FOR FUTURE ANALYSIS**

There are a vast number of areas that additional experimentation may uncover hidden benefits with the addition of mineral fillers to the base sheet of latex impregnated paper. A general observation was made that with the introduction of fillers the sheet appearance improved. Formation testing of the samples would allow accurate trending of this property. Additional testing should be completed for the optical properties brightness and opacity.

With the addition of the calcium carbonate to the base paper the effect of the calcium carbonate filler on the alkalinity of the finished paper was not investigated, but is sometimes a desired property.

The use of mineral filler that allows for optimal packing efficiency may decrease the reduction of strength properties. This experiment was limited to 3 specific fillers and one type of latex. The introduction of different lattices into the experiment may allow one to observe the interactions between the filler and the lattices.

Another area that may be of some interest with latex impregnated paper and the introduction of fillers in the base sheet of latex impregnated paper is being able to accurately control and modify the strength properties of latex saturated paper. In some applications the excessive strengths of latex saturated paper may need to be accurately modified.

APPENDIX A

HANDSHEETS TEST RESULTS - RAW DATA

Ta	ble	10

# Handsheet Testing Raw Data 0% Filler

			GRADE - 0% FILLER				REPE	AT GRADE - 0%		
	WEIGHT (grams)	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE	WEIGHT (grams)	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE
	PICK-UP (%)	4.53 4.5 4.66 4.5	0.07 4.54	5.92 5.62 5.56 5.66 26.88	0.17 5.73	PICK-UP (%)	4.39 4.34 4.33 4.41	0.05 4.38	5.64 5.6 5.66 5.63	0.03 5.64
47	CALIPER (mils)	6.7		27.31 27.15 27.23 28.64 6.28	0.69 27.44	CALIPER (mils)	6.48		28.18 27.56 27.48 26.80	0.49 27.49
	SMOOTHNESS (Sheffield Units)	6.44 6.58 6.48 6.22	0.18 6.48	6.28 5.94 5.94 6.15 375	0.17 6.12	SMOOTHNESS (Sheffield Units)	6.84 6.7 6.56 6.6	0.14 6.64	6.16 5.92 6.26 6.38	0.18 6.15
	POROSITY (Gurley Units)	400 396 394 400	2.83 397.00	377 373 383 382 4.5	4.36 378.00	POROSITY (Gurley Units)	397 400 398 394	2.49 397.80	377 381 383 373	3.85 378.60
	TENSILE (kilogram)	2.3 2.15 2.18 2.22	0.06 2.22	4.45 4.62 4.75 4.76	0.14 4.62	TENSILE (fritogram)	2.14 2.23 2.3 2.18	0.06 2.21	4.5 4.58 4.75 4.45	0.14 4.61
		3.743 2.1 2.643 2.1 2.853 2.1 2.433 2.1	964 606 977 0.40 544 2.92	9.165 9. 9.647 10. 9.684 9.	004 005 499 0.39 9.37	(100021011)	3.199 3.2 3.36 3.5 3.076 3.12 2.544 3.06	0.27 0.27 3.17	9.437 8.96 10.178 10.12 8.696 9.47 10.413 9.76	3 7 5 0.54 9 9.65

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			GRADE - 0% FILLER				REPE	AT GRADE - 0%		
	ELONGATION (%)	UNSATURATED 1.734 1.456 1.462 1.64 2.096 2.006 1.625 1.822 1.625 1.822	Average	SATURATED 7.661 7.023 7.66 7.842 7.294 7.934 7.024 7.751	Average	ELONGATION (%)	UNSATURATED 1.551 1.625 1.368 1.545 1.915 1.37 1.367 1.415	Average 0.20	7.388  7.532    7.114  8.128    7.388  7.869    7.477  7.023	Average 0.47
	TEAR (grams force)	1.278 1.275 78 74 75 74 81	5 1.66 3.05 76.40	72 78 70 74 73	2.97 73.40	TEAR (grams force)	1.461 1.873 83 85 82 77 72	5.26 79.80	8.389 6.945 85 72 72 72 72 72 72	7.53 5.81 74.60
48	BURST (psig)	5 5 3 8 5 2 3	1.29 4.00	50 56 51 45 56 56 55 50	2.94 52.38	BURST (psig)	6 5 6 5 5 5 6 4 5 4	0.74 5.10	48  49    53  50    47  52    48  51    51  56	2.98 50.30
	FOLD (# double folds)	3 4 6 5 7 3 3	5 5 1.55 2 4.20	366 469 403 463 344 556 442 490 345 194	103.22 409.50	FOLD (# double folds)	5 7 4 4 3 4 6 4	1.18 4.50	201 404 506 410 711 336 259 326 645 295	165.77 409.30
	ASH (%)					ASH (%)		4.		
	FOLD (log 10) (# double folds)	0.477 0.602 0.778 0.699 0.602 0.699 0.845 0.477 0.477 0.301	0.17 0.60	2.563 2.689 2.605 2.666 2.537 2.747 2.645 2.690 2.538 2.288	0.13 2.60	FOLD (log 10) (# double folds)	0.699 0.845 0.602 0.602 0.602 0.802 0.602 0.802 0.477 0.602 0.778 0.602	0.11 0.64	2.303 2.606 2.704 2.813 2.852 2.526 2.413 2.513 2.810 2.470	0.17 2.58
	BULK (cm³/g)		2.55		1.91	BULK (cm³/g)		2.71		1.95

Tal	ble	11	

# Handsheet Testing Raw Data 2.5% Clay Filler

	GRA	DE - 2.5% CLAY FILL	ER			REPEAT GR/	ADE - 2.5% CLAY FILL	ER	
WEIGHT	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE	WEIGHT	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE
(grams)	4.33 4.4 4.36 4.29	0.05	5.69 5.54 5.53 5.51	0.08	(grams)	4.32 4.34 4.28 4.34	0.03	5.49 5.63 5.55 5.56 5.49	0.06
PICK-UP (%)			31.41 27.65 25.11	2.20	PICK-UP (%)			28.27 28.25 27.88	0.04
CALIPER O (mils)	<u>6.26</u> 6.3		20.67 26.50 5.74 5.68	27.47	CALIPER (mils)	6.12 6.3		27.82 28.27 5.82 5.8	0.23 28.10
SMOOTHNESS	6.26 6.32 6.34	0.04 6.30	5.84 5.7 5.9	0.09 5.77	SMOOTHNESS	6.18 6.32 6.06	0.11 6.20	5.8 5.58 5.66	0.11 5.73
	393 397 393 392 393	2.00 394.00	379 379 370 367 365	5.37 370.40		399 397 392 392	3.11 394.80	370 371 375 375	3.11 371.80
(Gurley Units)	2.44 2.6 2.6 2.68	0.09	5.7 5.57 5.34 5.8	0.19	(Gurley Units)	2.64 2.64 2.64 2.65 2.65	0.03	5.29 5.43 5.1 5.44	0.14
TENSILE (kilogram)	3.211 3.372 3.236 3.286 3.335 3.296	2.50	8.313 8.0 9.671 9.52 9.82 9.83	5.50 09 23 32	TENSILE (kilogram)	2.38 2.754 2.0 2.952 3.3 3.471 3.2	2.63 643 335 286	9.846 9.67 9.301 9.30 8.757 8.69	5.30 11 13
	3.088 3.446 3.347 3.224	0.10 3.28	9.721 10.17 9.091 8.85	78 0.70 56 9.31		3.644 3.5 3.706 2.6	533  0.42    531  3.20	10.017 9.28 8.98 8.86	9 0.46 9 9.28

## Table 11 -Continued

	GRADE - 2.5% CLAY FILLER		REPEAT GRADE - 2.5% CL	AY FILLER
ELONGATION (%)	UNSATURATED STDEV AVERAGE 1.732 1.732 1.732 1.732 1.915 1.641 1.551 1.915 0.21	SATURATED STDEV AVERAGE 7.114 7.204 7.568 7.477 7.114 6.749 6.567 7.023 0.38	UNSATURATED STDEV AVERAGE (%) 1.551 1.368 1.094 1.458 1.951 1.642 1.913 1.822 0.28	SATURATED  STDEV AVERAGE    7.114  7.387    7.206  6.75    7.114  6.659    7.295  6.93    0.33
TEAR (grams force)	1.278  1.368  1.66    86  78	6.838  6.384  7.00    72  72    75  1.30    73  72.60	1.73  1.277  1.58    TEAR (grams force)  74  78    80  78  80    80  7.13  64  72.40	6.293 7.023 6.98 133 74 75 69 27.15 72
BURST (psig)	4  4    18  15    15  17    5  4  8.14    5  5  9.20	55  50    45  45    60  50    42  57    51  44	BURST 5 5 (psig) 4 4 15 17 15 16 5.69 8 5 9.20	64.60 53 41 50 47 47 56 54 50 4.38 49.40
FOLD (# double folds)	3  3    2  3    3  5    2  3    4  3	374  144    149  335    263  432    144  284    109.64    402  215	FOLD 3 7 (# double folds) 6 5 6 5 2.95 6 6 5 6.50	289  237    331  177    350  227    428  286    341  315    296.10
ASH (%)	2.88 2.39 2.52		ASH 2.88 (%) 2.39 2.44	
FOLD (log 10) (# double folds)	0.477 0.477 0.301 0.477 0.477 0.699 0.301 0.477 0.12 0.602 0.477 0.48	2.573  2.158    2.173  2.525    2.420  2.635    2.158  2.453    0.19  2.604    2.332  2.40	FOLD (log 10) (# double folds) 0.778 0.899 0.778 0.899 0.778 0.899 0.778 0.899 0.78	2.461  2.375    2.520  2.246    2.544  2.356    2.631  2.425    0.11  2.533    2.498  2.48
BULK (cm³/g)	2.59	1.86	BULK 2.57 (cm <sup>3</sup> /g)	1.85

# Table 12

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# Handsheet Testing Raw Data 5% Clay Filler

	GR	ADE - 5% CLAY FILL	ER	
	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE
WEIGHT (grams)	4.29 4.26 4.32	0.02	5.48 5.4 5.55	0.05
PICK-UP	4.29	4.30	5.44	5.46
(%)			26.27 27.36 27.88 28.44	0.82
CALIPER	//		27.10	27.41
(mils)	6.02 5.82 5.84		5.78 5.66 5.86	
SMOOTHNESS	6.16 6.37	0.23 6.04	5.7 5.7	0.08 5.74
(Sheffield Units)	397 386 395		380 375 378	
	394	4.28	372	3.03
	395	393.40	376	376.20
(Gurley Units)	2.38 2.1 2.5		4.93 4.47 5.08	
	2.43	0.15	4.89	0.25
TENSILE	2.42	2.37	5.00	4.09
(kilogram)	2.816 3.47 3.039 2.66	1 B	8.881 8.0 9.239 9.1	04
	2.174 2.14	9 042	8.436 8.6	83 51 0 40
	2.853 2.29	7 2.67	9.19 8.8	19 8.85

## Table 12 -Continued

	UNSATURATED ST AV 1.278 1.734 1.459 1.368	IDEV /ERAGE	SATURATED	STDEV AVERAGE
	1.094 1.388 1.732 1.481 1.275 1.458	0.20 1.42	7.661 7.93 7.204 7.75 6.93 7.02	4 1 0.39 3 7.41
TEAR (grams force)	80 78 76 83 78	2.65 79.00	77 70 69 72 68	3.56 71.20
BURST (psig)	3 3 4 5 4 4 5 5 5 5 3 4	0.82 4.00	55 34 57 44 50 5 49 44 55 55	8 0 1 5 6.60 5 49.50
FOLD (# double folds)	4 2 3 2 4 3 2 3 2 3	0.79 2.80	187  290    141  28    77  150    517  19    169  22	0 1 1 1 121.01 7 223.80
ASH (%)	4.83	4.80		
FOLD (log 10) (# double folds)	0.602 0.301 0.477 0.301 0.602 0.477 0.301 0.477 0.301 0.477	0.12 0.43	2.272 2.462 2.149 2.449 1.886 2.199 2.713 2.28 2.228 2.356	0.22 3 2.30
BULK (cm³/g)		2.52		1.88

GRADE - 5% CLAY FILLER

	Tabl	e 1	3
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# Handsheet Testing Raw Data 7.5% Clay Filler

		GRAD	DE - 7.5% CLAY FILLER				REPEAT GRADE	- 7.5% CLAY FILLER		
	WEIGHT	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE	WEIGHT	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE
	(grams)	4.32 4.38 4.5 4.29 4.4	0.08 4.38	5.63 5.45 5.47 5.6 5.49	0.08 5.53	(grams)	4.3 4.35 4.42 4.35 4.4	0.05 4.36	5.59 5.48 5.47 5.59 5.42	0.08 5.51
	PICK-UP (%)	2		30.02 25.58 23.76 28.74		PICK-UP (%)			29.10 26.27 23.76 28.51	
53	CALIPER (mils)	6.28 6.35		<u>26.50</u> <u>5.77</u> <u>5.63</u>	26.92	CALIPER (mils)	6.35 6.3		24.88 5.65 5.74	26.50
	SMOOTHNESS	6.21 6.3 6.42	0.08 6.31	5.5 5.76 5.9	0.15 5.71	SMOOTHNESS	6.15 6.58 6.49	0.17 6.37	5.35 5.99 5.45	0.25 5.64
		392 393 397 395 393	2.00 394.00	372 383 367 366 365	7.44 370.60		393 399 394 395 396	2.30 395.40	370 381 366 382 358	10.19 371.40
	(Gurley Units)	2.48 2.62 2.6 2.67 2.55	0.07 2.58	5.71 5.6 5.4 5.83 5.44	0.18 5.60	(Gurley Units)	2.56 2.93 2.9 2.58 2.66	0.18 2.73	5.84 5.69 5.9 5.92 5.5	0.18 5.77
	(kilogram)	2.201 2.543 2.358 2.501 3.471 2.102 2.754 2.204 2.45 2.759	0.40 2.53	8.732 7.934 9.201 9.375 8.436 8.425 8.752 9.001 8.889 8.45	0.43 8.72	i Ensile (kilogram)	2.543 2.208 2.445 2.449 3.109 2.305 2.69 2.301 2.56 2.775	0.27 2.54	8.56 9.291 8.001 8.25 8.779	7.934 9.001 8.76 9.315 0.52 8.015 8.59

### Table 13-Continued



# Table 14

# Handsheet Testing Raw Data 10% Clay Filler

	GF	RADE - 10% CLAY FIL	LER		
•					
	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE	
WEIGHT (grams)	4.35 4.4 4.32		5.47 5.44 5.5		
	4.28	0.04	5.6	0.08	
PICK-UP	4.33	4.34	5.63	5.53	
(%)			23.20 24.49 22.49 28.74 29.72	3.29 25.73	
CALIPER					
(mils)	6.25 6.29 6.21 6.24 6.34	0.05 6.27	5.73 5.49 5.58 5.7 5.73	0.11 5.65	
(Sheffield Units)	395 397 399 395 394	2.00 396.00	370 381 376 380 355	10.64 372.40	ũ
POROSITY					
(Gurley Units)	2.52 2.6 2.66 2.7 2.99	0.18 2.69	5.75 5.66 5.84 5.79 5.44	0.16 5.70	
		02	7 401 7 07	ล	
(KIN Graffi)	1.93 2.2 1.479 2.1 1.683 2.1 2.505 1.9	09 01 65 0.32	7.43 7.87 7.892 8.45 7.902 8.2 7.302 7.99	0.48	
	2.443 1.7	63 2.02	7.992 8.97	8.01	

## Table 14-Continued

	GRADE	- 10% CLAY FILLER		
ELONGATION	UNSATURATED ST	IDEV VERAGE	SATURATED	STDEV AVERAGE
(%)	1.221  1.84    1.34  1.207    1.219  1.44    1.309  1.319    1.217  1.308	0.19 1.34	6.901  7.38    6.83  6.749    7.25  7.335    7.001  7.29    6.329  7.183	0.33 7.02
TEAR (grams force)	72 71 68 75 70	2.59 71.20	69 66 70 68 62	3.13 66.60
BURST (psig)	ND ND ND ND ND ND ND ND ND ND	D	44  39    51  40    46  47    42  44    52  52	4.79 45.70
FOLD (# double folds)	ND ND ND ND ND ND ND ND ND ND	D	80  55    78  102    102  78    777  45    69  110	21.60 77.60
ASH (%)	9.48 9.87	9.68		
FOLD (log 10) (# double folds)	N	D	1.778  1.740    1.892  2.009    2.009  1.892    1.686  1.653    1.839  2.041	0.13 1.87
BULK (cm <sup>3</sup> /g)		2.59		1.83

Table 15	
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# Handsheet Testing Raw Data 2.5% PCC Filler

		GRA	DE - 2.5% PCC FILLER				REPEAT GRAD	DE - 2.5% PCC FILLER		
	WEIGHT		STDEV AVERAGE	SATURATED	STDEV AVERAGE	WEIGHT		STDEV AVERAGE	SATURATED	STDEV AVERAGE
	(grams) PICK-UP	4.38 4.39 4.43 4.39 4.43	0.02 4.40	5.68 5.58 5.51 5.63 5.65	0.07 5.61	(grams) PICK-UP	4.21 4.43 4.3 4.42 4.27	0.10 4.33	5.56 5.62 5.41 5.46 5.63	0.10 5.54
	(%)			27.64 28.57 26.67 27.09 26.97	0.75 27.39	(%)			26.65 27.44 27.90 27.57 26.23	0.69 27.16
57	(mils)	6.5 6.8 6.58 6.36 6.58	0.16 6.56	6.06 5.86 5.48 6.16 5.78	0.26 5.87	(mils)	5.96 6.42 6.36 6.2 6.24	0.18 6.24	5.9 5.86 5.64 5.7 5.82	0.11 5.78
	SMOOTHNESS (Shaffield Units)	400 400 400 397 400	1.34 399.40	390 388 383 388 388 385	2.77 386.80	SMOOTHNESS (Sheffield Units)	400 395 397 396 398	1.92 397.20	384 369 385 382 393	8.68 382.60
	POROSITY (Gurley Units)	2.45 2.52 2.57 2.76 2.45	0.13 2.55	6.7 6.03 6.28 6.1 6.07	0.28 6.24	POROSITY (Gurley Units)	2.9 2.36 2.58 2.74 2.41	0.23 2.60	5.8 5.29 5.66 5.77 5.07	0.32 5.52
	(kilogram)	4.237 3.224 3.458 3.854 3.409 3.137 3.841 3.792 3.31 4.014	0.37 3.63	10.017 10.017 9.943 10.104 10.721 10.166 10.314 10.252 10.499 10.635	0.27 10.27	(kilogram)	3.607 3.619 3.878 3.94 3.545 3.94 3.36 3.162 3.742 3.73	0.25 3.65	8.103  9.066    9.128  9.461    11.141  11.154    9.079  9.19    10.005  9.424	0.95 9.58

## Table 15-Continued

	GRADE - 2.5% PCC FILLER				REPEAT GRADE - 2.5% PCC FILLER					
ELONGATION (%)	UNSATURATED 51 2.098 1.732 1.825 2.279 1.822 2.189 2.098 2.189	IDEV VERAGE	SATURATED 7.387 7.20 7.661 7.84 7.478 7.2 7.201 7.20	STDEV AVERAGE	ELONGATION (%)	UNSATURATED 1.825 1.734 1.641 1.642 1.642 1.914 1.369 1.365	STDEV AVERAGE	8.474 6.38 6.75 6.65 7.113 7.02 6.476 6.65	STDEV AVERAGE	
TEAR (grams force)	2.006 2.169 2.096 1.734 88 85 83	2.00	7.244 7.754 7.11 72 72 72 75	14 7.46	TEAR (grams force)	1.308 1.308 1.825 1.642 85 78 75	1.66	6.478 8.83 6.75 6.65 74 72 75	7 0.23 6 6.69	
BURST (psig)	85 83 7 5 5 4 6 4	2.05 84.80	72 73 58 9 52 9 53 4	1.30 72.80 56 57 49	BURST (psig)	77 74 5 5 7 4 5 4	4.32 77.80	74 75 47 4 58 4 50 5	1.22 74.00 7 8 3	
FOLD (# double folds)		1.03 5.20	40 54 54 54 54 54 54 54 554 554 554 554	55 5.34 57 53.10 47 52	FOLD (# double folds)	5 5 4 5 7 11 12 7 13 8	0.88 4.90	45 4 59 5 266 8 183 10 308 21	5 4.69 50.00 8	
ASH (%)	6 4 4 7 2.15 2.6	1.32 4.80		92.47 80 276.10	ASH (%)	13 7 7 12 2.74 2.43	2.71 9.70	243 14 333 34	92.51 5 222.20	
FOLD (log 10)	0.602 0.602	2.38	2.322 2.65	50]	FOLD (log 10)	0.845 1.041	2.59	2.425 1.94	ন	
(# double folds)	0.778 0.778 0.477 0.602 0.778 0.602 0.602 0.845	0.12 0.67	2.487 2.55 2.548 2.42 2.433 2.34 2.187 2.25	59 20 44 0.15 55 2.42	(# double folds)	1.079 0.845 1.114 0.903 1.114 0.645 0.845 1.079	0.12 0.97	2.262 2.02 2.469 2.32 2.366 2.14 2.522 2.53	5 2 8 0.21 8 2.31	
(cm³/g)		2.01		1.87	(Cm <sup>3</sup> /g)		2.30		1.87	

Tal	ble	16
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# Handsheet Testing Raw Data 5% PCC Filler

	GR	ADE - 5% PCC FILLER	२	
	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV
WEIGHT				
(grams)	4.36 4.33 4.29 4.26 4.41	0.06 4.33	5.43 5.64 5.61 5.56 5.46	0.09 5.54
PICK-UP			00.07	
(70)			<u>26.37</u> 28.77 27.50 27.52 26.10	1.03 27.65
CALIPER				
(mils)	6.74 6.4 6.38 6.44 6.3	0.17 6.45	5.76 8.02 5.78 8.04 5.96	0.13 5.91
SMOOTHNESS	400		374	
(Snemeid Units)	400 396 399 400 395	2.35 398.00	374 388 381 378 371	6.58 378.40
POROSITY				
(Gurley Units)	2.33 2.49 2.31 2.41 2.4	0.07 2.39	5.07 4.74 4.64 5.23 5.1	0.25 4.96
TENSILE		-		
(kilogram)	3.174 2.34 2.767 2.95 2.841 3.18	7	9.103 8.8 9.118 9.3 10.795 10.	69 87 81
	3.113 2.63	1 0.34	10.042 9.	98 0.87
	2.248 3.1	5 2.64	9.301 9.2	94 9.65

# Table 16-Continued

	UNSATURATED	STDEV	SATURATED	STDEV
ELONGATION		AVERAGE		AVERAG
(%)	1.641 1.1 1.642 1.3 1.276 1.4 1.732 1.3	185 166 159 168 0.23	7.751 7.3 7.844 7.9 8.027 7.9 7.842 6.3	297 934 361 391 0.3
	1.094 1.7	732 1.45	7.297 7.	935 7.
TEAR	66		78	
(grams force)	66		74	
	62	4.56	70	3.
	67	63.40	72	72.
BURST	ND ND		44	50
(psig)	ND ND		46	44
	ND ND	-	46	45 3.4
	ND ND		44	44 46.
FOLD	2	4	85	99
(# double folds)	5	2	199	145
	5	3 1.16	111	150 79.1
	2	4 3.30	327	143 176.
ASH	4.5			
(%)	4.85		•	
			· ·	
		4.68		
FOLD (log 10)	0.301 0.6	302	1.929 1.9	96
(# double folds)	0.699 0.3	301	2.299 2.	161
	0.477 0.4	177 0.16	2.425 2.3	375 176 0 ·
	0.301 0.6	0.49	2.515 2.	155 2.2
DUBK			0	
BULK		2.67		1.1

GRADE - 5% PCC FILLER

		GRA	ADE - 7.5% PCC FILLER			REPEAT GRADE - 7.5% PCC FILLER					
		UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE		UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE	
	WEIGHT (grams)	4.42 4.4 4.42		5.68 5.76 5.73		WEIGHT (grams)	4.3 4.24 4.24		5.62 5.6 5.63		
	PICK-UP (%)	4.4	0.02 4.42	5.48 5.6	0.11 5.65	PICK-UP (%)	4.31 4.35	0.05 4.29	5.56 5.54 27.73	0.04 5.59	
				28.29 28.19 28.04 27.56	0.28 28.00				27.27 30.93 29.00 28.54	1.42 28.69	
61	(mils)	6.48 6.48 6.62 6.82	0.22	5.75 5.72 5.86 5.89	0.07	(mils)	6.34 8.18 6.42 6.38	0.09	5.84 5.85 5.81 5.6	0.13	
	SMOOTHNESS (Sheffield Units)	6.08 400 395	6.48	5.84 386 381	5.77	SMOOTHNESS (Sheffield Units)	6.33 393 396	6.33	5.61 356 363	5.74	
	POROSITY	397 400 396	2.30 397.60	382 386 376	4.15 382.20	POROSITY	392 392 399	3.05 394.40	367 373 376	7.97 367.00	
	(Gurley Units)	2.36 2.55 2.58 2.75 2.75	0.16 2.60	6.08 6.55 5.85 6.06 5.87	0.28 6.08	(Guney Units)	2.55 2.63 2.62 2.55 2.55	0.04 2.59	6.8 6.22 5.98 6.01 5.84	0.38 6.17	
	(kilogram)	2.829 2.87 2.754 3.00 2.582 2.98 2.83 2.78 2.789 2.75	8 9 5 5 0.12 4 2.82	9.135 9.453 9.676 9.673 9.673 9.344 9.784 9.88 9.511 9.700	3 5 1 0.24 9,60	(kilogram)	2.763 2.44 2.943 2.76 2.88 2.8 3.501 3.01 3.002 3.18	5 4 1 4 0.28 2 2.93	9.499 8.7 9.647 6.4 9.066 9.4 8.856 7.6 8.556 9.4	15 36 19 33 0.62 37 8.93	

# Handsheet Testing Raw Data 7.5% PCC Filler
### Table 17-Continued

	GRADE - 7.5% PCC FILLER		REPEAT GRADE - 7.5%	PCC FILLER
ELONGATION (%)	UNSATURATED STDEV AVERAGE 1.368 1.368 1.675 1.734 1.004 1.305	SATURATED STDEV AVERAGE 7.297 7.661 6.746 7.02 7.387 7.114	UNSATURATED STDEV AVERAC ELONGATION (%) 1.48 1.185 1.551 1.459 1.822 1.549	SATURATED STDEV AVERAGE 7.204 6.75 6.019 7.201 7.445 6.293
TEAR (grams force)	1.342 1.551 0.24 1.776 1.275 1.44	7.023 7.204 0.44 6.474 6.027 7.20 71 75 70	1.733 1.368 0.   1.458 1.622 1.   TEAR 63 1.   (grams force) 58 60	20 6.474 6.001 0.63 54 6.566 7.476 6.95 66 69 67
BURST (psig)	55 3.70   60 59.80   ND ND   ND ND	69 2.97   67 70.40   40 45   45 47   41 50	65 2.   60 61.   BURST ND ND   (psig) ND ND	77 67 1.64 20 70 67.80 48 54 51 50 47 47
FOLD (# double folds)	ND ND   ND ND   1 2   2 3   2 1	44 42 3.53   38 45 43.70   120 188   144 148   95 95	FOLD 1- (# double folds) 2 2	43 50 4.47   39 43 47.20   108 194   77 64   134 85
ASH (%)	1.71 0.76 1.71 7.34 7.66	154 162 30.89 117 107 132.80	ASH 7.85 (%) 7.69	04 133 196 47.65 25 178 82 127.10
FOLD (log 10) (# double folds)	7.50 0.000 0.301 0.301 0.477	2.079 2.274 2.158 2.164	FOLD (log 10) 0.000 (# double folds) 0.301 0.301	77
BULK (cm³/g)	0.000 0.19 0.20 2.62	1.970 1.970 0.10   2.188 2.210 0.10   2.068 2.029 2.11	0.477 0.477 0.4 0.602 0.000 0.3 BULK 2.0 (cm <sup>3</sup> /g)	2:127 1:329 0.16   2:124 2:292 0.16   31 2:250 1:914 2:08   84 1.64

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# Handsheet Testing Raw Data 10% PCC Filler

	GF	RADE - 10% PCC FILI	LER	
	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE
EIGHT Tams)	4.3 4.28 4.24 4.33 4.35	0.04 4.30	5.64 5.72 5.58 5.55 5.54	0.07 5.61
ICK-UP %)			28.18 30.00 29.77 28.77 28.54	0.79 29.05
ALIPER nils)	6.38 6.25 6.42 6.54 6.29	0.11 6.38	5.92 5.85 5.86 5.71 5.59	0.13 5.79
100THNESS heffi <b>e</b> ld Units)	395 396 394 399 399	2.30 395.40	377 361 369 372 353	9.48 366.40
OROSITY Surley Units)	2.75 2.69 2.62 2.58	0.07	6.18 6.43 8.14 6.66	0.27
ENSILE (ilogram)	2.65 2.701 3.0 2.845 2.7 2.77 2 2.709 2.1	2.66 014 764 .81 754 0.15	5.96 9.003 8.77 9.543 8.34 9.145 8.62 9.179 8.00	6.27 74 01 23 06 0.60
	3.118 2.0	2.81	8.439 7.5	73 8.66

### Table 18-Continued

	UNSATURATED	STDEV AVERAGE	SATURATED	STD AVE
ELONGATION				
(%)	1.321 1.5	51	7.56 6.7	55
	1.347 1.1	18	7.102 7.2	01
	1.549 1.7	73	6.98 6.2	93
	1.65 1.3	68 0.24	6.299 7.3	87
	1.822 1.	79 1.53	7.56 7.8	31
TEAR	60		65	
(grams force)	54		63	
	51		70	
	58	3.97	65	
	60	30.00	69	
BURST	ND ND		44	46
(psig)	ND ND	_	46	50
	ND ND	_	47	45
	ND ND		40	**
			4/	43
FOLD	ND ND		77 1	56
(# double folds)	ND ND	-	115	84
	ND ND	_	107 1	14
	ND ND		133 1	19
			108	82 1
ASH	9.65		•	
(%)	9.83		· · ·	
			· · · ·	
			· · · ·	
		9.74	<u> </u>	
				-
FULD (10g 10)			1.886 2.1	83
(# double folds)			2.061 1.9	
			2.029 2.0	78
		ND	2.033 1.0	14
			2.000 1.8	
BULK		2.65		
(cm³/g)				

		GR	ADE - 2.5% GCC FILLER				REPEAT GRAD	DE - 2.5% GCC FILLER		
	WEIGHT	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE	WEIGHT	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE
	(grams)	4.43 4.43 4.43 4.33	0.04	5.6 5.7 5.66 5.77	0.06	(grams)	4.33 4.37 4.33 4.36	0.02	5.49 5.49 5.5 5.63	0.06
	РІСК-UP (%)	4.41	4.41	27.85 27.52 28.05	5.68	PICK-UP (%)	4.37	4.35	26.79 27.38 26.73	5.54
65	CALIPER (mils)	6.56		27.65 26.68	0.53 27.55	CALIPER (mils)	6.34		28.25 26.82	0.64 27.19
		6.48 6.84 6.26 6.42	0.21 6.51	5.96 5.94 6.1 5.96	0.09 5.96		6.32 6.48 6.3 6.38	0.07 6.36	5.62 5.7 5.96 5.76	0.14 5.73
	SMOOTHNESS (Sheffield Units)	400 400 400		385 391 383		SMOOTHNESS (Sheffield Units)	400 399 400	iù	386 371 378	
	POROSITY (Gurley Units)	400 400	0.00 400.00	388 380 5.48	4.28 385.40	POROSITY (Gurley Units)	400 400	0.45 399.80	392 383 5.55	7.97 382.00
		2.09 2.24 2.26 2.11	0.09 2.20	5.59 5.8 5.43 5.52	0.07 5.52		2.5 2.51 2.4 2.58	0.08 2.48	6.06 6.19 5.92 5.72	0.26 5.89
	(kilogram)	3.607 3.44 3.545 3.26 2.977 1.75 3.335 3.45	6 11 11	10.758 9.72 9.98 9.77 10.252 11.42 9.14 10.151	1	(kilogram)	3.249 3.73 2.94 3.211 3.73 3.261 3.113 2.90	3] ]] ].	9.597 10 10.227 9.807 9 10.178 10	482 9.4 943
		2.554 3.17	4 3.09	9.74	10.11		3.223 3.014	3.24	10.079 9	.807 9.96

# Handsheet Testing Raw Data 2.5% GCC Filler

Table 19

### Table 19-Continued

	GRADE	- 2.5% GCC FILLER				REPEAT GRA	DE - 2.5% GCC FILLER		
ELONGATION (%)	UNSATURATED ST AV 1.825 1.842 1.913 1.824	DEV ERAGE	SATURATED 7.387 8 8.118	STDEV AVERAGE	ELONGATION (%)	UNSATURATED	STDEV AVERAGE 8]	SATURATED	STDEV AVERAGE
TEAR	1.368 1.004 1.368 1.732 1.363 1.822	0.30 1.59	7.378 7 7.204 8 7.114	.114 .025 0.42 7.56	TEAD	1.459 1.84 1.458 1.45 1.368 1.27	2 9 0.20 8 1.52	7.294 7.1 7.387 7. 7.387 7.	934 478 0.32 023 7.34
(grams force)	84 83 82 82	2.28 81.80	78 75 78 72 69	3.91 74.40	(grams force)	90 82 94 95	7.33 92.60	70 89 75 77 77	3.49 73.20
BURST (psig)	5 8 5 5 5 4 5 4 4 3	0.84 4.60	55 46 55 51 45	50 51 45 48 4.04 45 48.90	BURST (psig)	5 6 5 5 5	4 5 4 5 0.57 5 4.90	49 45 50 52 45	50 44 50 44 3.75 55 48.40
FOLD (# double folds)	8 8 4 8 3 5 3 3 4 3	1.72 4.50	231 215 108 69 138	97 134 113 184 49.31 154 148.30	FOLD (# double folds)	11 4 8 4 - 4 -	4 7 3 2.62 5.38	194 206 197 138 203	334 253 118 139 63.18 173 195.30
ASH (%)	2.5 2.68	2.59			ASH (%)	2.77	2.73		
FOLD (log 10) (# double folds)	0.776 0.903 0.602 0.776 0.477 0.699 0.477 0.477 0.602 0.477	0.18 0.63	2.364 1 2.332 2 2.033 2 1.949 2 2.140 2	.987 .127 .053 .265 0.14 .188 2.14	FOLD (log 10) (# double folds)	1.041 0.60 0.602 0.84 0.778 0.47 0.602 0.602	2 5 7 0.18 0.89	2.288 2.5   2.314 2.6   2.294 2.0   2.134 2.   2.307 2.5	524 103 172 143 0.13 238 2.27
BULK (cm <sup>3</sup> /g)		2.65		1.88	BULK (cm <sup>3</sup> /g)		2.82		1. <b>85</b>

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### Table 20

# Handsheet Testing Raw Data 5% GCC Filler

	UNSATURATED	STDEV	SATURATED	STDEV
		AVERAGE		AVERAGE
(grams)	4.4 4.35 4.32 4.42	0.04	5.57 5.54 5.65 5.47	0.08
	4.41	4.38	5.46	5.54
(%)			26.02 25.34 26.12 25.75 26.39	0.40 25.92
CALIPER	··			
(mils)	6.1 6.46 5.98 6.3	0.19	5.8 5.62 5.74 5.5 5.48	0.14
SMOOTHNESS	0.12	0.15	<u> </u>	5.05
(Sheffield Units)	398 400 395 397	1.82 397.40	390 387 383 378	6.50 382.40
POROSITY	397	337.40		502.40
(Gurley Units)	2.75 2.57 2.78		6.08 6.22 6.13	
	2.5	0.12	6.26	0.12
	2.65	2.65	6.39	6.22
kilogram)	3.15 3.347 3.174 3.619		9.857 10. 10.907 10.	203 005
	3.792 3.42	0.27	10.561 10	375 0.27
	2 458 3 743	3.52	10 289 10	10.27

### Table 20-Continued

	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE
(%)	1.642 1.276   1.642 1.44   1.824 1.56   1.82 1.456   1.82 1.456   1.549 2.262	3 3 3 2 1.65	6.84 7.7   7.478 7.33   8.745 8.56   7.023 7.21   7.297 7.66	19 37 38 37 0.40 31 7.20
TEAR (grams force)	85 91 94 86 85	4.09 88.20	78 69 69 67 67	4.76 69.60
BURST (psig)	2 3 3 3 5 5	2] 5] 5 1.06 1] 3.30	55 5 54 4 45 4 46 5	51 14 19 50 3.74 16 49.00
FOLD (# double folds		)                 	106 22 53 21 125 9 86 5	19 12 17 19 62.94
ASH (%)	5.05	5.12		
FOLD (log 10) (# double folds	) 0.477 0.477 0.778 0.699 0.699 0.477 0.602 0.477	0.12	2.025 2.36 1.724 2.33 2.097 1.96 1.934 1.77	0 8 77 1 0.23
BULK (cm³/g)	L0.602[0.477	2.53	2.158 1.70	1.82

GRADE - 5% PCC FILLER

		GRAI	DE - 7.5% GCC FILLER				REPEAT GRAI	DE - 7.5% GCC FILLER		
	WEIGHT	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE	WEIGHT	UNSATURATED	STDEV AVERAGE	SATURATED	STDEV AVERAGE
	(grams)	4.38 4.27 4.3 4.33 4.33	0.04	5.49 5.45 5.53 5.61	0.07	(grams)	4.33 4.41 4.28 4.34	0.06	5.43 5.32 5.39 5.43 5.58	0.10
	PICK-UP (%)			28.27 27.34 26.83	1.04	PICK-UP (%)			25.40 26.97 25.06 25.6	0.70
69	CALIPER (mils)	8.24 6.08		23.20 27.04	27.75	CALIPER (mils)	5.86		20.37 25.96 5.76 5.58	25.99
	SMOOTHNESS (Sheffield Units)	5.84 6.22 6.04	0.16 6.08	5.84 5.64 5.48	0.17 5.60	SMOOTHNESS (Sheffield Units)	6.16 6.36 6.02 391	0.19 6.09	5.52 5.7 5.54	0.10 5.62
	POROSITY	400 400 400 398	3.03 398.20	370 372 383 389	7.91 378.00	POROSITY	389 395 396 396	3.21 393.40	380 377 375 357	8.98 372.20
	(Gurley Units)	2.81 2.55 2.8 2.72 2.57	0.12 2.69	5.91 5.77 6.17 6.27 6.2	0.21 6.06	(Gurley Units)	3.05 2.62 2.91 2.86 2.78	0.16 2.84	7.17 5.83 6.28 5.84 6.78	0.59 6.38
	(kilogram)	3.249 3.236 3.088 3.347 2.927 2.334 3.856 3.57 3.249 2.68	0.40 3.13	9.239 9.536 9.481 8.387 10.067 9.499 9.227 10.325 9.894 8.819	0.58 9.45	(kilogram)	2.186 3.224 2.47 2.456 3.236 1.482 2.767 2.68 3.36	0.60 2.85	8.646 8.251   8.819 9.289   9.807 9.758   9.684 9.511   10.511 10.375	0.73 9.47

## Handsheet Testing Raw Data 7.5% GCC Filler

### Table 21-Continued

	-	GRAI	DE - 7.5% GCC FILLER				REPEAT GRAD	- 7.5% GCC FILLER		
	ELONGATION (%)	UNSATURATED 1.459 1.825 1.549 1.459 1.275 1.185 1.823 1.825 1.551 1.368	STDEV AVERAGE 0.23 1.53	7.568 7.57   6.293 6.02   7.295 7.751   6.75 7.661   7.751 8.75	STDEV AVERAGE 0.64 7.14	ELONGATION (%)	UNSATURATED 1.642 1.732 1.368 1.641 1.915 1.386 1.641 2.006 1.915	STDEV AVERAGE 0.23 1.69	SATURATED 5.745 6.292 6.75 7.113 6.566 7.023 7.367 6.75 6.366 7.203	STDEV AVERAGE
	TEAR (grams force)	85 86 81 84 63	1.92 63.80	74 69 70 60 69	2.17 70.20	TEAR (grams force)	86 82 80 84 66	2.61 83.60	82 72 72 69 70	5.20 73.00
70	BURST (p <del>s</del> ig)	4 2 3 5 4 4 3 6 2 5	1.32 3.60	45 56   45 49   49 51   51 45   47 40	4.42 47.80	BURST (psig)	2 2 3 3 4 4 2 3 3 3	0.74 2.90	51 46   52 53   41 46   42 46   49 51	4.16 47.70
	FOLD (# double folds)	3 3 3 4 1 4 4 5 3 3 3	1.06 3.30	105 61 65 95 93 124 202 120 52 97	39.56 105.40	FOLD (# double folds)	2 4 4 3 2 3 5 4 4 5	1.07 3.60	218 82 59 190 106 129 76 95 104 105	52.28 114.40
	ASH (%)	7.31	7.28			ASH (%)	7.69	7.57		
	FOLD (log 10) (# double folds)	0.477 0.477 0.477 0.602 0.000 0.602 0.602 0.699 0.477 0.477	0.19 0.49	2.021 1.908 1.929 1.978 1.968 2.093 2.305 2.079 1.718 1.987	0.15 2.00	FOLD (log 10) (# double folds)	0.301 0.602 0.602 0.477 0.301 0.477 0.699 0.602 0.602 0.699	0.14 0.54	2.338 1.792 1.771 2.279 2.025 2.111 1.881 1.978 2.017 2.021	0.19 2.02
	BULK (cm²/g)		2.53		1.82	BULK (cm³/g)		2.52		1.85

### Table 22

# Handsheet Testing Raw Data 10% GCC Filler

	GR	ADE - 10% GCC FIL	LER		
	UNSATURATED	STDEV	SATURATED	STDEV	
WEIGHT					
(grams)	4.38 4.42 4.31 4.44	0.05	5.55 5.59 5.62 5.46	0.07	
	4.39	4.39	5.49	5.54	
PICK-UP (%)			25.57 26.47 25.45		
			25.52	0.73	
			27.08	26.02	
CALIPER					
(miis)	6.25 5.93 5.98 6.25 6.18	0.15	5.74 5.56 5.6 5.82 5.39	0.17	
SMOOTHNESS	0.10	0.12	5.58	0.02	
(Sheffield Units)	399 396 400 395 396	2.17 397.20	388 385 385 392 378	5.13 385.60	
POROSITY					
(Gurley Units)	2.81 2.9 2.86		6.41 6.49 6.39		
	2.69	0.08	6.52	0.06	
	2.79	2.81	6.39	6.44	
TENSILE (kilogram)	2.835 2.80 2.542 2.55	2] 6]	9.305 9.5 9.308 9.3	58 04	
	2.0/2 2.44	0 16	9.362 9.2	35 0.11	
	2.004 2.00	3 2.65	9.442 9.2	05 0.11	

### Table 22-Continued

		_			
	UNSATURATED	STD AVE	EV RAGE	SATURATED	STDEV AVERAGE
ELONGATION (%)	1.585 1.30 1.709 1.40 1.669 1.50 1.711 1.45 1.549 1.90	5 5 6 3 6	0.18 1.58	7.23 8.995 7.308 8.753 6.575 6.566 6.86 6.501 6.575 6.487	0.30
TEAD					
(grams force)	64 66 90			72 69 65	
	63 64		2.79 85.40	63	3.54 67.00
BURST		_			-
(psig)	ND ND ND ND ND ND ND ND			47 44 43 44 45 46 46 46	2.08
	ND ND	JND		48 50	45.90
(# double folds)	ND ND ND ND ND ND ND ND ND ND			107 126 99 59 196 44 190 94 75 52	53.38 104.20
ASH					
(	9.69		9.83		
FOLD (log 10) (# double folds)				2.029 2.100 1.996 1.771 2.292 1.643 2.279 1.973	
BULK		ND		1.875 1.716	1.97
(cm³/g)			2.50		1.82

GRADE - 10% GCC FILLER

APPENDIX B

### GRAPHICAL REPRESENTATION OF TEST RESULTS



Figure 7. Caliper Comparison of Unimpregnated & Impregnated Grades with 0% Filler



Figure 8. Smoothness Comparison of Unimpregnated & Impregnated Grades with 0% Filler



Figure 9. Permeability Comparison of Unimpregnated & Impregnated Grades with 0% Filler



Figure 10. Tensile Comparison of Unimpregnated & Impregnated Grades with 0% Filler



Figure 11. Elongation Comparison of Unimpregnated & Impregnated Grades with 0% Filler



Figure 12. Tear Comparison of Unimpregnated & Impregnated Grades with 0% Filler



Figure 13. Burst Comparison of Unimpregnated & Impregnated Grades with 0% Filler



Figure 14. Fold Comparison of Unimpregnated & Impregnated Grades with 0% Filler



Figure 15. Saturant Pick-up Variation with Increased Ground Calcium Carbonate (GCC) Addition



Figure 16. Caliper Variation of Impregnated Handsheets with Increased Ground Calcium Carbonate (GCC) Addition



Figure 17. Bulk Variation of Impregnated Handsheets with Increased Ground Calcium Carbonate (GCC) Addition



Figure 18. Smoothness Variation of Impregnated Handsheets with Increased Ground Calcium Carbonate (GCC) Addition



Figure 19. Air Resistance Variation of Impregnated Handsheets with Increased Ground Calcium Carbonate (GCC) Addition



Figure 20. Tensile Strength Variation of Impregnated Handsheets with Increased Ground Calcium Carbonate (GCC) Addition



Figure 21. Elongation Variation of Impregnated Handsheets with Increased Ground Calcium Carbonate (GCC) Addition



Figure 22. Tear Variation of Impregnated Handsheets with Increased Ground Calcium Carbonate (GCC) Addition



Figure 23. Burst Variation of Impregnated Handsheets with Increased Ground Calcium Carbonate (GCC) Addition



Figure 24. Fold Variation of Impregnated Handsheets with Increased Ground Calcium Carbonate (GCC) Addition



Figure 25. Saturant Pick-up Variation for All Filler Types and Addition Levels



Figure 26. Bulk Variation of All Unimpregnated Handsheets



Figure 27. Bulk Variation of All Impregnated Handsheets



Figure 28. Caliper Variation of All Unimpregnated Handsheets



Figure 29. Caliper Variation of All Impregnated Handsheets



Figure 30. Smoothness Variation of All Unimpregnated Handsheets



Figure 31. Smoothness Variation of All Impregnated Handsheets



Figure 32. Air Resistance Variation of All Unimpregnated Handsheets



Figure 33. Air Resistance of All Impregnated Handsheets



Figure 34. Tensile Strength Variation of All Unimpregnated Handsheets



Figure 35. Tensile Strength Variation of All Impregnated Handsheets



Figure 36. Tensile Elongation Variation of All Unimpregnated Handsheets



Figure 37. Tensile Elongation Variation of All Impregnated Handsheets



Figure 38. Tear Variation of All Unimpregnated Handsheets



Figure 39. Tear Variation of All Impregnated Handsheets



Figure 40. Burst Variation of All Unimpregnated Handsheets



Figure 41. Burst Variation of All Impregnated Handsheets



Figure 42. Fold Variation of All Unimpregnated Handsheets



Figure 43. Fold Variation of All Impregnated Handsheets

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