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STRENGTHENING OF CORRUGATED BOARDS BY CHEMICAL
IMPREGNATION

by

Rajesh PremNarayanSingh

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 2004

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Rajesh PremNarayanSingh

STRENGTHENING OF CORRUGATED BOARDS BY CHEMICAL IMPREGNATION

Rajesh PremNarayanSingh, M.S.

Western Michigan University, 2004

Linerboards are used for making corrugated boards. The paperboards can be strengthened by adding strength additives at the wet-end of the paper machine and also by sizing them. Impregnation is another effective approach of strengthening the paperboards, mostly offline. The aim of the thesis is to evaluate the performance of the modified Dixon coater and check the operating conditions with various chemicals used for impregnating 42 lb/1000 sq.ft grammage linerboard.

The major conclusions of the research were that the edge compression strength increases markedly validating the usage of impregnation for linerboards. Also, the stiffness of linerboard increases and the voids are effectively filled at higher levels of impregnation. The effect on water resistance depends on the nature of the impregnating chemicals; e.g., Kymene increases the water resistance while the hydrophilic starch or calcium lignosulfonate reduces it.

TABLE OF CONTENTS

ACKNOWLEDGMENTS	ii
LIST OF TABLES	vi
LIST OF FIGURES.....	vii
CHAPTER	
I. INTRODUCTION.....	1
II. LITERATURE REVIEW.....	3
Wet-End Addition on Paper Machine	4
Dry Strength Additives	4
Wet Strength Additives.....	6
Urea Formaldehyde Resin	7
Melamine Formaldehyde Resin (MF).....	8
Polyamide – Amine Epichlorohydrin Resin	9
Polyethylenimine Resins.....	11
Dialdehyde Starches	12
Coating of Paper	13
Impregnation of Paper	14
Emulsifiable Methylene Diisocyanate (EMDI).....	17
Method of Application	18
Starch	19
Method of Application	20
Urea Formaldehyde (UF) Resin	22

Table of Contents—continued

	Method of Application	22
	Ethylene Acrylic Polymer	23
	Method of Application	23
	Alkenyl Succinic Anhydride	24
	Method of Application	25
	Scripset740.....	25
	Method of Application	25
	Kymene	25
	Method of Application	26
	Calcium Lignosulfonate.....	26
	Method of Application	27
III.	PROBLEM STATEMENT	28
IV.	METHODOLOGY.....	29
	Trial Run on Dixon Coater	33
	Test Methods.....	34
	Burst Strength	34
	Porosity	35
	Stiffness.....	35
	Water resistance	36
	Crush resistance	36
	Experimental Run	37
	Trial Run I.....	37

Table of Contents—continued

	Trial Run II.....	37
	Trial Run III	38
	Trial Run IV	39
V.	RESULTS AND DISCUSSIONS.....	40
	Trial Run I	40
	Trial Run II.....	43
	Trial Run III.....	44
	Trial Run IV	49
	Influence of Operating Conditions on Properties of Impregnated Linerboard	50
VI.	CONCLUSIONS.....	58
VII.	FUTURE STUDIES.....	61
VIII.	REFERENCES	62

LIST OF TABLES

1. Effect of Pond and Shoe Pressure on Pickup and Surface Properties – 15% Solids Starch.....	41
2. Effect of Pond and Shoe Pressure on Strength Properties – 15% Solids Starch	42
3. Effect of Pond and Shoe Pressure on Pickup and Surface Properties – 25% Solids Starch.....	44
4. Effect of Pond and Shoe Pressure on Strength Properties – 25% Solids Starch	45
5. Effect of Pond and Shoe Pressure on Pickup and Surface Properties – Scripset740, ASA, Calcium Lignosulfonate and Kymene.....	46
6. Effect of Pond and Shoe Pressure on Strength Properties – Scripset740, ASA, Calcium Lignosulfonate and Kymene	48
7. Effect of Pond and Shoe Pressure on Pickup and Surface Properties – Calcium Lignosulfonate and Kymene	50
8. Effect of Pond and Shoe Pressure on Strength Properties – Calcium Lignosulfonate and Kymene	51
9. Influence of Operating Conditions at Equal Pickup – Kymene, 15% Starch and 25% Starch.....	54
10. Influence of Operating Conditions at Equal Pickup – 15% Starch and 25% Starch.....	55
11. Influence of Operating Conditions at Equal Pickup – 35% Calcium Lignosulfonate, ASA and Calcium Lignosulfonate with Kymene	55
12. Influence of Operating Conditions at Equal Pickup – Calcium Lignosulfonate and 15% Starch	56
13. Influence of Operating Conditions at Equal Pickup –ASA	56
14. Influence of Operating Conditions at Equal Pickup – Calcium Lignosulfonate with Kymene and Scripset740	57

LIST OF FIGURES

1. Formation of Methylol Amines.....	8
2. Cross Linking of Methylol Groups to Form Melamine Formaldehyde.....	10
3. Synthesis of Polyamide	11
4. Alkylation of Secondary Amine Groups in the Polyamide	11
5. Impregnator	32
6. Impregnator Shoe.....	33

CHAPTER I

INTRODUCTION

Corrugated board is made of recycled as well as virgin fibers and comprises of three components, the outer and inner liner and the corrugating medium. The liner is a flat surface, which is around 185 g/m² grammage and the corrugating medium is a fluted structure that is glued between the liners. The sandwich of the medium between liners provides excellent rigidity and cushioning effect to corrugating boards. The focus of the thesis was on finding a material that can profitably increase the strength properties like tensile, compression, burst, tear, and stiffness in the liner. The main problem was to determine a way to strengthen the liners, effectively. Generally, to improve the paper strength, additives are added at the wet end of paper machine. Barrier coatings on the paper can improve the surface properties alone. There is lesser retention of chemicals at the wet end and hence for effective usage of the chemicals, the liner can be impregnated with the chemicals offline. Impregnation, starch addition and coating are some of the commercially viable processes for enhancing surface and strength properties. Of these, impregnation can be more cost effective as all the material added is retained in the paper, as compared to wet end addition. This increased retention of the chemicals, tends to impart more strength to paper. Due to

increased strength, the basis weight of linerboard can be reduced. This would benefit the paper manufacturers as they can reduce fiber usage, thereby lowering production costs. The shipping costs for the boxes will be reduced in the process. The impregnation process could also enhance the performance of the corrugated containers, which encounter many mechanical actions during manufacturing and shipping and handling operations. The impregnating material should be cost-effective and also be easily recoverable during recycling of the paper. The optimum level of material to be impregnated for the best performance of corrugating medium is also to be determined. In addition, the process conditions that are conducive to achieve an optimum level of impregnation should also be determined.

CHAPTER II

LITERATURE REVIEW

Corrugated boxes are used for paperboard packaging. The corrugated boxes undergo a lot of wear and physical strain during shipping of products. Apart from physical stresses, moisture also tends to mar the performance of the boxes. In order to counter these deteriorating effects, the corrugation board should have wear resistance as well as water resistance. The strength of the liner can be increased either by increasing the fiber content or by replacing the fibers with other additives that impart more strength to the liner. The latter process sounds more effective as we can reduce the fiber content and try to make an economical replacement.

The replacement of the fibers can be done commercially by adding additives, coating the paper or by impregnation. We discuss below the various additives used and how they affect the strength properties when added at the wet end of the paper machine. Commonly used additives impart dry strength or wet strength or even both. Coating of paper enhances the barrier resistance properties but has no significant effect on physical strength properties. Impregnation has an effective response on both

physical strength and water resistance properties. The above-mentioned processes are discussed below in detail.

Wet-End Addition on Paper Machine

The additives added at the wet-end to impart strength to the paper are called wet-end additives. They are broadly classified as

1. Dry strength additives and
2. Wet strength additives.

Dry Strength Additives

Dry strength additives are water soluble, hydrogen bonded polymers. Some of the commonly used dry strength additives are starch, vegetable gum, and polyacrylamide.

Starch is the most widely used strength additive. Generally, cationic starches are used to have good starch retention on papermaking fibers. Usually, reaction with either tertiary or quaternary amine groups cationizes the starch.

The tertiary reagent cationized starch has a greater degree of dependence on pH, since the charge is lost in alkaline conditions. Quaternary reagent cationized starch retains the complete charge at all pH levels and hence are pH independent (1). Starch (about 2 to 2.5%) is usually applied at the inlet of pressure screens, which is a turbulent region and enough time is allowed for adsorption of starch on to fibers. Apart from increasing the strength properties, it also improves fiber retention and drainage.

Guar gum and locust bean gum are typical water-soluble vegetable gums used as dry strength additives. These gums then form hydrogen bond with the cellulose, providing strength to the paper. These additives are also added at the inlet of the pressure screen at around 0.1% - 0.35%. These gums have cations in them that hold the key to attraction between the gum and the fiber, thereby resulting in retention of the polymers (gums). Polyacrylamides are also dry strength resins used to increase the dry strength of paper. Polyacrylamides are either anionic or cationic. Anionic polyacrylamides are the result of co-polymerization of acrylamide with acrylic acid, resulting in the formation of ionizable carboxyl groups in the polymer chain. Cationic polyacrylamides are formed by co-polymerization with cationic monomers like methacryloxyethyl trimethyl ammonium methosulfate (METAMS), vinyl benzyl trimethyl ammonium chloride (VBTAC), 3-Acrylamido-3-methyl butyl trimethyl ammonium chloride (AMBTAC), etc (1).

Polyacrylamides are applied as aqueous solutions of 10 – 20% either to thick or thin stock where the mixing is good. Polyacrylamides are added at levels of 0.1 – 0.5% to the stock. Polyacrylamides increase the strength properties of paper without any adverse effects on bulk or appearance properties of paper.

Wet Strength Additives

A paper is said to have wet strength when it retains at least 15% of the dry strength after being saturated with water. Wet strength additives can be used depending on the end use. There are two types: temporary wet strength and permanent wet strength additives. The toilet tissues and paper tissues have temporary wet strength, as they need to be decomposed easily after use. Paper bags, carton box (flexible packing) for liquids, currency papers, etc., need permanent wet strength as they have to be coherent without losing integrity after being saturated with water. The time of saturation varies from paper to paper. Wet strength agents protect the existing hydrophilic hydrogen bonds; they also form new bonds insoluble in water and form cross-links between the fiber bonds to impart wet strength. In the case of dry strength additives, the retention of additives increases the strength; however, for wet strength additives, the curing of resin is more important than the retention to impart strength. Curing can be done by heating (thermosetting) or by chemically reacting with fiber bonds.

Some of the wet strength resins are urea-formaldehyde, melamine-formaldehyde, polyamide amine epichlorohydrin, polyethylenimine, and dialdehyde starches.

Urea Formaldehyde Resin

Urea formaldehyde resin (UF), resulting from urea reacting with formaldehyde, forms dimethylol urea that is a strengthening agent. But this has low solubility, which means it has limited use as a wet end additive. Polyfunctional amines such as diethylene triamine when polymerized with dimethylol urea give a cationic polymer, which is substantive to the pulp.

Urea formaldehyde resins are used with a small degree of polymerization at 25-35% solution. UF resins are acid cured; thus, the head box should have a pH of 4-4.5. UF resins are added from 0.5%-3% on dry basis, depending on the desired wet strength of paper. The alum-rosin reactions occur in acidic conditions and hence the acid curing UF resins are added at the fan pump. UF wet strength paper requires an additional accelerated curing of 5 – 15 min at 104°C as the curing is not completed on the paper machine (2).

Melamine Formaldehyde Resin (MF)

Melamine formaldehyde is the result of melamine reacting with formaldehyde, forming methylol melamines. The most effective MF resin is trimethylolmelamine treated with hydrochloric acid resulting in a colloidal suspension that is highly cationic and substantive to the pulp. Various combinations of melamine and formaldehyde can be used, but the key factor is the formation of ether and methylene cross-links with the fibers that impart the wet strength at low pH and high temperature (2).

Figures 1 and 2 explain the chemical reaction involved in the synthesis of melamine formaldehyde.

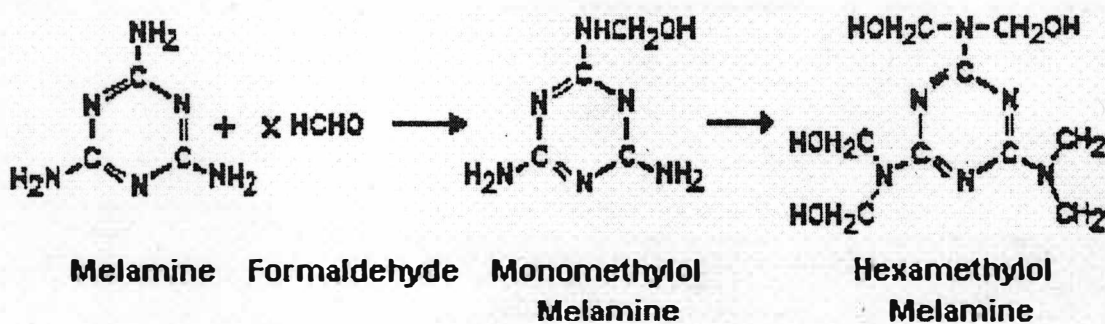


Figure 1. Formation of Methylol Amines (2).

MF resins are used in the acid colloid form (also available as a dry powder, but should be treated with dilute acid and aged under specific conditions before use).

MF resins are added depending on the desired wet strength of paper, say paper towels to paper bags. The addition levels vary from less than 1% to 5% based on dry fiber weight. The general norm is 1-3% for satisfactory results (2).

Polyamide – Amine Epichlorohydrin Resin (PAE)

PAE resins are the most common wet strength additives. First a polyamine like diethylenetriamine is reacted with a dibasic acid to form a polyamide. It is then treated with epichlorohydrin resulting in tertiary aminochlorohydrin groups and alkylating secondary amine groups.

PAE resins undergo two kinds of reaction for imparting wet strength. In the first type, the azetidinium group from one reacts with the secondary amine group of another molecule, producing a cross-link between the molecules. In the second type, two azetidinium groups of a molecule react with carbonyl groups on different fibers resulting in inter-fiber cross-linking (2).

PAE resins are thermosetting and generally are acidified in the pH range of 3.5 to 6 for stability reasons. PAE resins are used in the head box where pH can vary from 5 to 9, since in acidic conditions the resin is less reactive. PAE is added at 0.25 – 0.75% dilution near the fan pump. 25 – 35% solids is the concentration at which the resin is compatible with any kind of substance. It should be noted that since PAEs are

highly cationic they should be treated with mild anionic materials and should be kept away from other strongly anionic additives like rosin. At increased pH levels the resin will start to form a gel. For complete curing, the paper is cured at 80°C for 30 min.

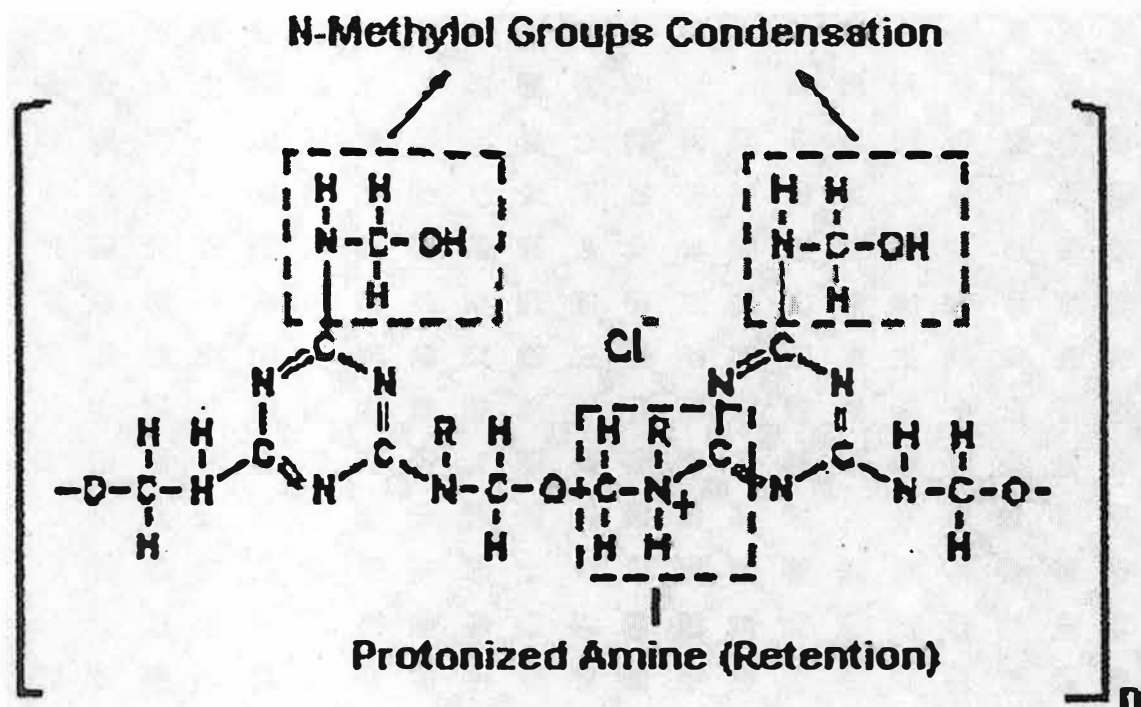


Figure 2. Cross Linking of Methylol Groups to Form Melamine Formaldehyde (2).

Figures 3 and 4 explain the chemical reaction involved in the synthesis of PAE.

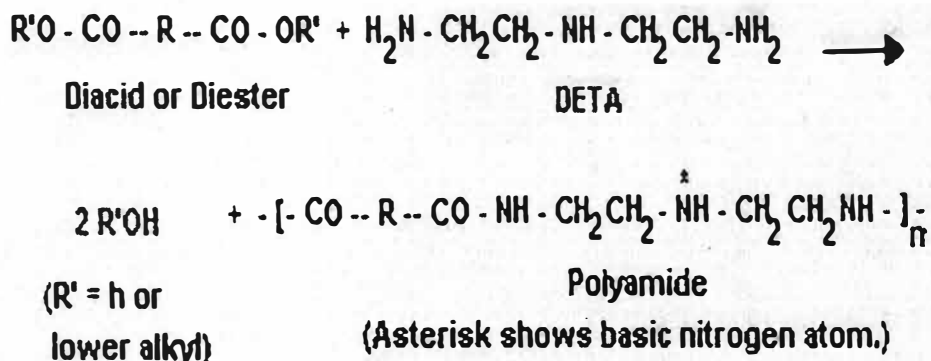


Figure 3. Synthesis of Polyamide (2).

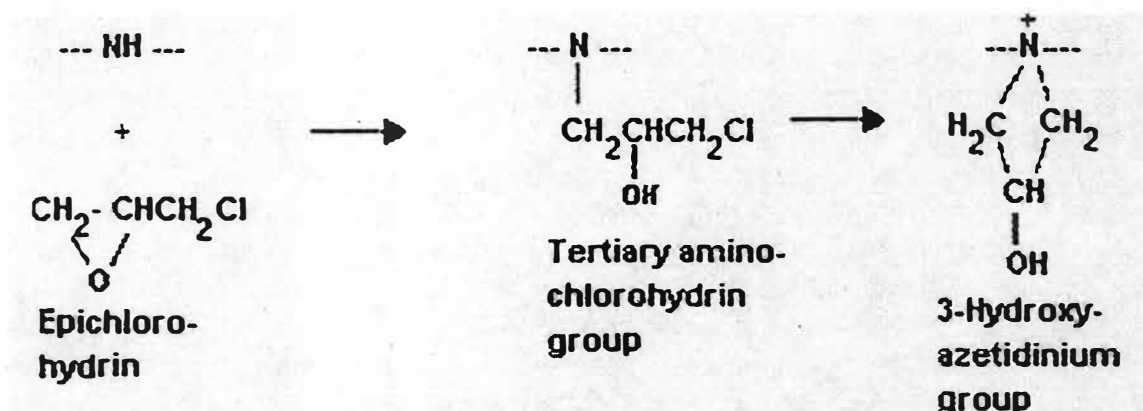


Figure 4. Alkylation of Secondary Amine Groups in the Polyamide (2).

Polyethylenimine Resins (PEI)

PEI is a polymer formed by polymerization of ethylenimine monomer in the presence of acid catalysts like mineral acids. PEI at low levels added to pulp increases the freeness of pulp, i.e., increases dewatering at the wet end of the paper machine and reduces the chances of a paper break when it is dried. PEI has a good flocculation

effect; the wet web along with various pigments and carbon black retains thereby more fines. Generally, PEI is added after the pulp is beaten as PEI retards the beating of pulps (2).

PEI cures by reaction of their amine groups with anionic groups in the pulp. PEI is added at about 2.5% of weight of pulp to have maximum wet strength, but the recommended level is 1- 1.5% of weight of pulp. Wet strength of PEI treated paper can be increased by treating with 2% formaldehyde solution, increasing the wet strength from about 16 to 22% of the dry strength. It can be further enhanced to 28% by curing at 105°C (2).

Dialdehyde Starches (DAS)

DAS is a highly modified starch, which has almost all anhydroglucose units oxidized by reaction with periodic acid. Initially, DAS dispersion is achieved with borax or sodium bisulfite; but this is anionic in nature and hence other cationic additives like alum and cationic starches are added prior to addition of DAS to make it substantive to pulp. But the varying levels of cationic agents vary the wet strength imparted to paper by DAS (3). Cationic starch is added at around the 2.5% level and DAS is added at around 0.25% - 0.75%. For these levels, the retention varies from 65% to 95%. Since cationic starch is also added, the dry strength of the paper also increases.

Coating of Paper

Barrier coatings applied onto the packaging board give protection against undesirable effects caused by such factors as light, oxygen, humidity, grease or aqueous liquids. The coating of paper is primarily done to increase barrier resistance and enhance printing properties of the paper. There are many coating formulations, which are used depending on the end-use of the paper. The desired attributes of a coated paper may be water resistance, abrasion resistance, etc. Kaolin clay, calcium carbonate, and titanium dioxide are the pigments that are mixed with binder and water to obtain the coating. The amount of solids is generally around 60% on the weight basis of solution.

Linerboard is made from recycled as well as virgin fibers and is coated to improve the aesthetic and printing properties. Clay based coatings are used for multilayered coating of the linerboards. The binder system also determines the barrier resistance and aesthetic properties. Binder system is selected depending upon the printing conditions of the corrugated board. Blade coater, air knife coater, and roll coaters are used depending upon the base sheet properties and thickness of the coating required. Mostly, combinations of the coating methods are used for multilayered coatings.

Impregnation of Paper

Linerboards are paperboards that are made from recycled or virgin fibers. They are used as a facing material on corrugated boxes and containers. Corrugated board is a versatile and inexpensive packaging medium. In a corrugated board, fluted sheets are glued to one or more liners. The resultant product of flutes and liners is much stronger than the paperboards from which it is made. Corrugated boards find their use in shipping and also are used for interior separators between products, and as corner blocks for blocking and bracing products that are used during shipping.

Folding cartons are boxes made from paperboards where the carton is printed, cut, and folded, and finally shaped. Perforations, scoring and cutting are done on the liner to facilitate the shaping operations.

Impregnation is done to strengthen the paper by forcing the impregnating materials into the paper. Due to a shortage in global production, calcium lignosulfonate is not widely used and hence an alternative must be found. Various materials can be used for impregnation, namely wax, starch and other organic and synthetic materials.

Wax coating/impregnation is used to treat the corrugating medium/liner board where water resistance and strength are to be enhanced under humid conditions. In

impregnation, wax is forced into the fibers rather than just coating. Impregnation helps to achieve cost-effective utilization of the fibers. Wax and polyethylene are the common impregnating materials. The corrugating medium is usually impregnated with wax and resin in a ratio from 70/30 to 50/50. Wax coating/impregnation imparts moisture resistance to the corrugated board. Wax coatings perpetuate the integrity and rigidity even when corrugated board is wet. Tests show that waxed corrugated board has three times the strength of non-waxed corrugated board.

Petroleum waxes are applied to corrugated board by the saturating or curtain coating methods. In the saturating method, a thick layer of wax is applied on the board at saturating conditions; the board picks up 40-50% of its weight in wax. Usually low melting point paraffin wax (130° F) is used along with polyethylene as an additive.

In curtain coating applications, the corrugated board passes horizontally through a curtain of falling wax. Around 6-7 lb of wax/ 1000 sq.feet of board is the weight applied. Paraffin wax (150°F), microcrystalline wax, and additives like ethylene vinyl acetate copolymer, tackifier resins, and antioxidants are combined to apply on the board. The waxes provide water-resistant properties. Ethylene vinyl acetate copolymers increase the viscosity of waxes so that they have a stable flow, and tackifier resins provide flexibility so as to avoid score line cracks on the boards. Antioxidants inhibit the oxidation of the melt.

Paraffin wax is the most widely used impregnating material. Paraffin wax is a by-product of crude petroleum refining. It is a compound of highly crystalline, structural hydrocarbons. It is obtained from paraffin base lubricating oil distillates. When completely refined, the wax has a melting point range from 120°F to 160°F; it is white, tasteless, odorless, hard and fairly brittle in nature. In the molten state, it is a clear, colorless, low viscosity liquid. The level of wax impregnation depends on the viscosity and melting point of wax, density of corrugated board, and the temperature at which it is done. The porosity, Cobb value, and the caliper uniformity of corrugated board also affect the impregnation.

The following properties characterize paraffin wax to be used as a waxing agent: melting point (100-160 °F) is the temperature at which most of the wax changes from solid to liquid. Hardness determines the penetrability of the wax; hardness is determined at 25°C. The higher the hardness value, the softer is the wax. The recommended values for paraffin wax are 9 – 20 at 25°C (ASTM D1321). The odor of the waxes should be 1 or less for paraffin (ASTM D1833). Oil content is the amount of oil in the wax; this measures the degree of refining. Fully refined wax has less than 0.5% oil, and semi-refined waxes have 0.5 – 1% oil. Viscosity is the resistance to flow of molten wax. 2.9 – 7.5 centistokes is the recommended value for paraffin wax. Color is measured by visual comparison of the molten wax color against glass color standards. The darkest color has the highest value; black has a value of 8 (ASTM D1500). The recommended value is 1.0.

40 – 60% wax pickup is the typical range for corrugated board. This imparts good stacking strength when exposed to humid conditions. Impregnated wax boards have increased dry strength, good appearance, reduced abrasion with the packaged products, and have high water resistance. These boards are rigid even in wet conditions. Compressive strength is reduced by 5%.

Wax cannot be used as an impregnating material because it poses problems at the time of recycling. Paraffin wax is not a recyclable material; hence resins or other organic compounds are used for impregnation. Hence a study was made to analyze the compounds that would impart good strength properties to liner after impregnation. The feasibility of the process of impregnation and the viability of the compound is to be taken into consideration. After the study, few compounds were short-listed and they are discussed below.

Emulsifiable Methylene Diisocyanate (EMDI)

Phenol-formaldehyde has to be cured at elevated temperatures for longer duration to prevent blocking. The major drawback is the loss of internal flexibility; this makes the paper brittle. Also, high costs limit its usage.

Emulsifiable methylene diisocyanate provides both strength and flexibility and also penetrates rapidly and completely into fibrous materials. It cures quickly at

normal temperature without blocking (no coagulation with in the paper layers). It also provides abrasion resistance and the cost is low when compared to phenol-formaldehyde. The EMDI described in US.Pat.No. 3,996,154 awarded to Johnson, et al comprises of aromatic diisocyanate and/or polyisocyanates of higher functionality having a methylene bridge. The impregnant is commercially available under the name Rubinate MF-178 by Rubicon Chemicals, Inc., of Wilmington, Del. It consists of approximately 50% diphenyl methane 4, 4' – diisocyanate, approximately 45% higher methylene bridged isocyanate polymers, and approximately 5% surfactant in the form of modified diphenyl methane diisocyanate. It is available as a liquid with approximately 95% solids (4).

Method of Application

Immersing in anhydrous EMDI saturates paper; an uncoated kraft paper had 88% take up during a 10 second saturation time (4). This high level of take up is uneconomical. Hence this impregnation is suitable only for non-impregnable materials; like 15-point kraft coated paper (it has only 18% take up, but excellent strength).

There is no theory governing the drying mechanism; but it is believed that EMDI reacts with the moisture in the paper to form a substituted urea, and with primary and secondary hydroxyl groups in the paper to form urethane cellulose. The

substituted urea reacts with paper layers to increase the bonding; the emulsifying agent can supplement the same (4). EMDI impregnated 42 lb linerboard cures to about 50% in 48 hours at 73°F and 50% RH (4), and completely in 12 – 14 days. As said earlier, water initiates the drying mechanism and then the paper comes into play. At an elevated temperature, 250°F, complete drying takes place in seconds. As EMDI is not an FDA approved compound, it restricts the usage of it for food packaging industry.

Effects of EMDI. 15-point kraft coated paper, immersed in Rubinate MF-178 for 10 seconds and dried completely was tested for strength properties. At 16% EMDI take up, the machine direction ring crush strength increased by 133%. Even after extended immersion in water, the impregnated paperboards retain around 36% of dry tensile strength. There is no significant change in flexibility, but there is reduced tear strength. The impregnated paper has an increase in basis weight of 22% without affecting the caliper. This increases water resistance and decreases the overall moisture absorbed by the paper (4).

Starch

Sodium hypochlorite modified, oxidized corn starch, was used (5). It is commercially available as Stayco M.TM from A. E. Staley Mfg. Company. The starch solution used at 26% solids and at 105°F and had a Brookfield viscosity of 118 cp at

100 rpm using a #2 spindle. Pearl cornstarch modified by ammonium per sulfate oxidation could also be used.

The starch solution is prepared in a 30-gallon batch starch cooker with 25 gallons of water and 100 pounds of the desired starch. The dispersed starch solution is heated to about 190-220°F for about 25 minutes and cooked for about 30 minutes. 30 mL of defoamer is added and applied at 26% solids at about 130 – 150°F.

Method of Application

When starch is applied with pressure, it penetrates into the interior of the liner; the resulting corrugated board made from the linerboard has enhanced crush resistance.

As per the author (5), the paper is assumed to be divided into 3 layers, namely, a first surface and a second surface of 0.0025" thick, and sandwiched between them lies the intermediate zone where at least 30 – 40 % of starch is to be absorbed for the best results. 16% starch on a dry weight basis will be applied, but significant effects are produced when around 3 – 6 % on dry weight basis is applied where at least 30 – 40 % of starch is absorbed in the intermediate region of paper. At least 0.008" thick paper should be used (5).

The paper used should be greater than about 20lbs/1000 ft² (98 g/m²). For liners, it is about 30 – 45lbs/1000 ft² (171- 220 g/m²), and for medium, it is about 15-30 lbs/1000 ft² (73 - 147 g/m²). The caliper should be at least 0.008", but can range from 0.01" to 0.04". On the size press of speed 800 – 2500 ft/min, a nip pressure of 300 to 550 pli is to be applied for effective penetration. If there is no effective penetration of starch, it will lead to brittleness and reduced adhesion of liner and medium. Taper grinding the paper surface prior to performing the iodine test can determine the distribution of starch. The impregnated liner is ground at a taper angle so that the base to the upper surface is exposed and then starch is applied and the indication of blue color tells the amount of distance, starch has been impregnated (5).

Effects of Starch. The ring crush strength increases in both the cross machine direction and the machine direction by about 5 – 7 % for every 1 % increase in starch when applied at levels of 4 – 10 % by weight. Tensile index value also increases by about 2 – 3 % for every 1 % increase in starch content when starch was applied at levels varying between 4 – 16 % by weight. The bending stiffness increased by about 11 – 13 % for every 1 % of starch was applied at levels of 3 – 6%.

Significant strength can be obtained at lower basis weight, which increases the paper production rate, reduces the energy consumption for driers, and decreases freight costs when liners have to be shipped.

Urea Formaldehyde (UF) Resin

UF resin is cheaper than maleic resin and phenol formaldehyde resin. The breaking load of paper doubles when maleic resin is used for impregnation, but UF is preferred because of its cost.

UF resin is prepared by heating urea and formaldehyde in a molar ratio of 1:4 in an alkaline medium (pH 8.5) for 2 hrs with constant stirring on a heating mantle, which is used as heat source (6).

Method of Application

A 50% UF resin solution in xylene is prepared. The board is vertically dipped in the solution for various time intervals, say 5 min, 10 min, 15 min, 20 min and 60 min, and the excess resin is removed by pressing. The board is then cured in an oven. 20 min immersion is the optimum level as there is no increase in breaking load on prolonged immersion (beyond 20 min).

Effects of Urea Formaldehyde resin. UF resin treated board reduces swelling and water absorption when the board is exposed to water.

Ethylene Acrylic Polymer

Ethylene acrylic copolymer, with a glass transition temperature of 38°C, when impregnated, provides better short-span compression strength at 95% RH.

Lignin and acrylic polymer (in ratio 2:1) when impregnated provide good short-span compression strength at 95% RH. The lignin is first dissolved in acetone and then reprecipitated in ammonia before adding to an ammonia-stabilized styrene-acrylic emulsion to prepare lignin dispersion. Care should be taken, as acetone is a toxic and flammable solvent (7).

Method of Application

When applied without pressure, the resin penetrates into the Z-direction of paper (due to only macro scale distribution), but, with impregnation, interaction with fiber takes place, leading to better strength properties even at 95% RH. The interaction with fibers takes place due to micro scale distribution. The pressure applied during impregnation helps in penetrating the fiber walls and crossover points that lead to effective distribution of the impregnant.

Effects of Ethylene Acrylic polymer. Due to impregnation, there is an internal reinforcing network whereby the compressive tensile loading is evenly distributed in the sheet.

Alkenyl Succinic Anhydride

Alkenyl succinic anhydride is a major synthetic sizing agent that is used in neutral and alkaline paper making (in pH range of 6 to 9). Alkenyl succinic anhydride is easily hydrolyzed by water and hence it is emulsified at the point of application. The alkenyl succinic anhydride is emulsified using cationic starch to be retained by the anionic furnish. Pure alkenyl succinic anhydride is made by polymerizing ethylene to form linear alkenes that are isomerized and reacted with maleic anhydride (8).

The alkenyl succinic anhydride emulsion is very unstable and has to be used immediately at the point of application. The alkenyl succinic anhydride emulsion is prepared by beating 150g alkenyl succinic anhydride with 2850g of 4% acidified starch in a blender for 90 seconds. The prepared emulsion is then added to 8250g of additional starch. The final emulsion contains 1.3% alkenyl succinic anhydride and 3.95% starch.

Method of Application

The alkenyl succinic anhydride can be applied using the impregnator.

Scripset740

Scripset740 alkaline resin solution is a commercially available sizing agent that is an aqueous solution of mixed methyl and isobutyl partial ester of styrene or maleic anhydride copolymer, ammonium salt. Chemically it is 2-butenedioic acid (2)-monomethyl ester that has been polymerized with ethenyl benzene, 2,5-furandione and (Z)-2-methylpropyl hydrogen 2-butenedioate and ammonium hydroxide. It is available at 10% solids concentration.

Method of Application

The Scripset740 can be applied using the impregnator.

Kymene

Kymene is a polyamide – amine epichlorohydrin resin (PAE). First a polyamine like diethylenetriamine is reacted with a dibasic acid to form a polyamide. It is then treated with epichlorohydrin resulting in tertiary aminochlorohydrin groups and alkylating secondary amine groups. PAE resins undergo two kinds of reaction for

imparting wet strength. In the first type, the azetidinium group from one reacts with the secondary amine group of another molecule, producing a cross-link between the molecules. In the second type, two azetidinium groups of a molecule react with carbonyl groups on different fibers resulting in inter-fiber cross-linking (2). PAE resins are thermosetting and generally are acidified in the pH range of 3.5 to 6 for stability reasons. PAE resins are used in the head box where pH can vary from 5 to 9, since in acidic conditions the resin is less reactive. PAE is added at 0.25 – 0.75% dilution near the fan pump. 25 – 35% solids is the concentration at which the resin has the highest compatibility. It should be noted that since PAEs are highly cationic they should be treated with mild anionic materials and should be kept away from other strongly anionic additives like rosin. At increased pH levels the resin will start to form a gel. For complete curing, the paper is cured at 80°C for 30 min.

Method of Application

Kymene (even though it is a surface treatment) can be applied using impregnator on the web that has been pre-impregnated with calcium lignosulfonate.

Calcium Lignosulfonate

Paper boards can be strengthened by applying aqueous solutions of lignosulfonates. Lignosulfonates are metal salts of lignosulfonic acids. The

lignosulfonates are highly hydrophilic in nature. The hydrophilic nature of the lignosulfonates mars the appearance of the paperboard when exposed to moisture. The lignosulfonates lose their integrity when they come in contact with water. Barrier coatings can be a possible remedy but it would be expensive. The cheaper solution would be to experiment with the chemistry of the chemical, like creating a cross linked structure that has higher water resistance. The cross linking can be done by reacting other compounds with the reactive agents like the sulfonic or hydroxyl groups.

Method of Application

The calcium lignosulfonate also can be applied using the impregnator.

CHAPTER III

PROBLEM STATEMENT

Impregnation is done as an offline process on the paperboard. The initial tasks were to evaluate the performance of the impregnator. The impregnator is designed exclusively for the research work and trial runs were done to evaluate the operating conditions of the impregnator. After the successful operation of the impregnator, various chemicals were used for impregnation. The effect of impregnation of these impregnating agents on the 42 lb/1000 sq.ft grammage linerboard was evaluated. The research work concentrates on studying the effect of impregnation of the linerboard, using various chemicals, on the paper properties like burst, porosity, water resistance, crush resistance, and, stiffness.

CHAPTER IV

METHODOLOGY

Impregnator is to be used in the impregnation experiments. Dixon coater is an off-machine coater. Blade or air-knife coating is done on Dixon coater up to a speed of 150 m/min. It is used commercially for barrier coating process. It has manual, semi or automatic winding and rewinding systems to ensure the continuous coating process. It is extensively used for barrier coatings apart from photographic film coatings.

In the impregnator a pressurized impregnation unit replaces the coating unit. The guide rolls have been changed from 14" to 16" in length to facilitate the usage of 15" wide web roll. The treated width of the web can be up to a maximum of 11". The impregnation unit consists of a knurled anilox roll and an impregnating shoe. It has an unwinding unit from where the web is taken through guide rolls to the impregnation unit. Tension of 120 pounds (8 pli for 15" wide web) is maintained on the roll to ensure that the paper does not sag when the machine is operated. The tension also accounts for the pull of paper from the web roll. Also a manually controlled brake system is present on the infeed system to ensure that the web is fed in to the coater

under tension. The paper is then passed through the impregnation unit. At the impregnation unit, the chemical is sprayed at pressure on one side of the paper. There is a spraying/impregnating shoe that has four nozzles in it. The pressurized chemical is brought to these nozzles and sprayed on the paper from a high-pressure cylinder of 1.3 gallons capacity. These four nozzles cover the entire width of the paper to be impregnated. After the impregnation, the paper is taken to a dryer and then wound on a roll on the other side of the machine.

The impregnator has a maximum speed of 225 feet/min. There is a drain to recover the overflowing chemicals during impregnation. The pressure is applied on the impregnating chemical and the impregnating shoe, separately. The pressure applied on both of these can be varied from 0 – 160 psi. There are three drives on the machine namely, lead, follower#1, and follower#2. The lead drive is geared in at 87.9 HZ for 225 FPM. The speed of operation can be entered manually on the keypad in the panel board. The followers 1 and 2 operate at 99.4% and 99.3% of the lead drive speed, respectively to match the roll surface speeds. This is done as the web may stretch or shrink when wetted or dried. The dryer has hot air blowing on the web. Electric coils heat the air and the temperature is maintained around 250°F.

The performance of the impregnator was evaluated with starch as impregnating chemical. Many modifications had to be done to the impregnation unit for the successful operation of the impregnator. Initially there was uneven application

of the chemicals across the width of the paper. The impregnation unit was modified to obtain an even application across the web prior to the experimental usage. The impregnator shoe was abraded and the level of the shoe had to be fixed for even application of the impregnating chemical. The dryer temperature was maintained at 250°F and the impregnator was operated at 15 fpm considering the drying constraints of the impregnated stock.

The following figures (5&6) present the schematic diagram of the Dixon coater and the cross section of the impregnator shoe.

As discussed earlier; the pressure is applied separately on the impregnating chemical and on the web at the nip (impregnation unit). The impregnating chemical stored in a vessel is applied on the web under pressure called as pond pressure and the web during impregnation is also under pressure of the knurled roll called as shoe pressure. Pond pressure and shoe pressure were the initial variables of concern that influence the amount of impregnation taking place. An interesting observation was made during the initial evaluation of the impregnator. Pond pressure's upper limit is 50 percent of the shoe pressure for the given shoe pressure used for impregnation. When the pond pressure exceeds the 50% critical value there was no successful impregnation. Observations indicate that when pond pressure is more than 50 percent of the shoe pressure it exceeds the counteracting shoe pressure leading to formation of

a skidding layer of the chemical; there is more accumulation of the chemical on the surface rather than impregnation.

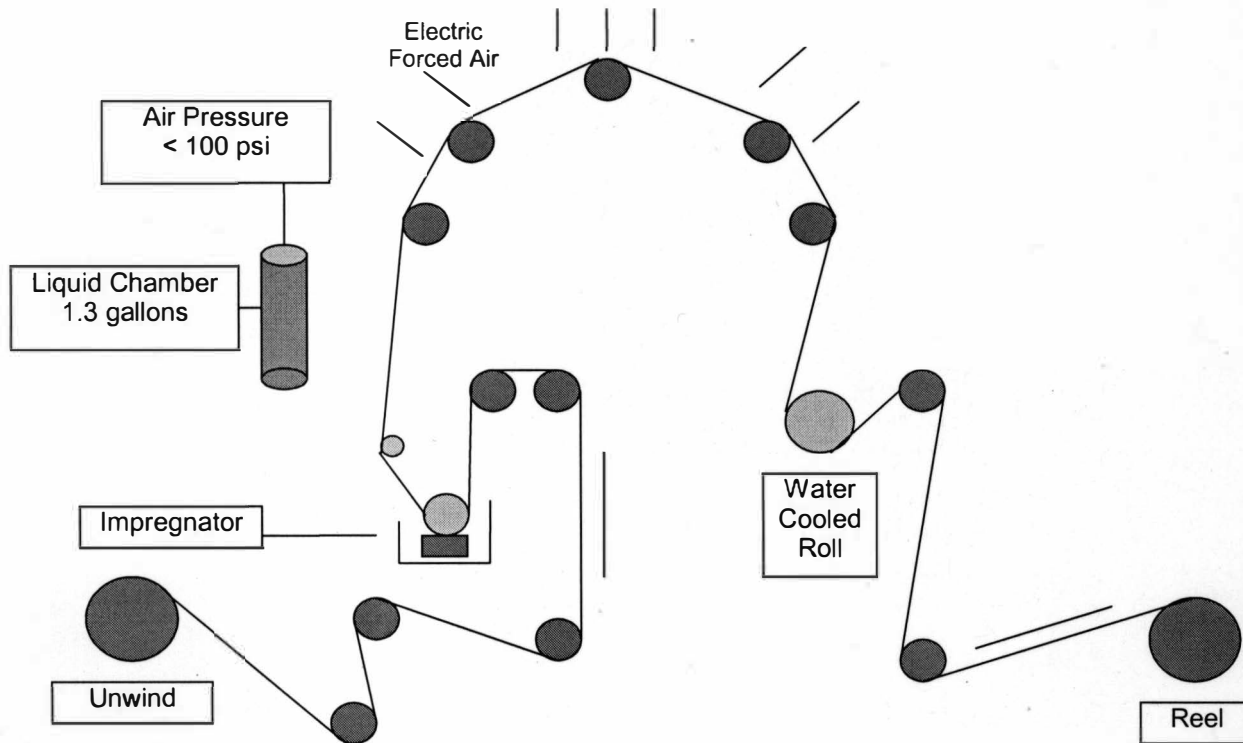


Figure 5. Impregnator.

The next step was to evaluate the optimum shoe pressure. After a couple of trials with starch as impregnating chemical, it was found that 40-60 psi was the optimum range of shoe pressure for effective impregnation. The level of penetration and the pickup of the impregnating chemical have a direct relation to pond and shoe pressure.

Trial Run on Dixon Coater

42 lb/1000squarefeet grammage liner roll from Green Bay Papers Inc. of 15" width was mounted on the infeed. Pneumatic brakes were applied at 25 psi pressure. The modified Dixon coater was run at 15 fpm for various combinations of pond pressure and shoe pressure. The dried web was wound on the rewind roll. The impregnated sheet was then conditioned and tested for various properties like bursting strength, stiffness, porosity, water resistance, and, crushing resistance. The treated side is called the top side and the untreated side is called the bottom side.

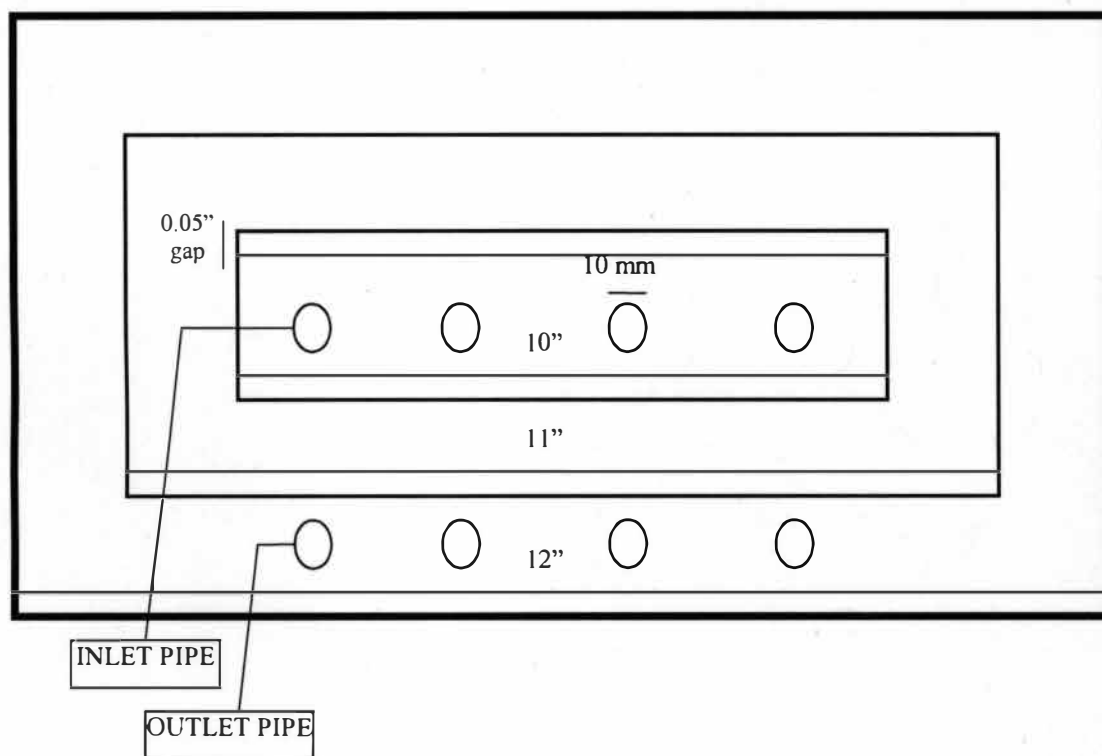


Figure 6. Impregnator Shoe.

Emulsifiable methylene diisocyanate could not be used as an impregnating chemical as it is not a FDA approved chemical. Urea formaldehyde along with xylene couldn't be used as there is the possibility of xylene catching fire in the dryer. The synthesis of ethylene acrylic polymer in lignin dispersion cannot be done on lab scale. Hence calcium lignosulfonate and Kymene were used as impregnating agents. They also conform to the Procter and Gamble research work (U.S. patent No. 4,588,616) that was based on these chemicals. The performance of the chemicals on the impregnator can thus be compared to the norms of the patent. Starch was used at 15 percent and 25 percent solids level. Alkenyl succinic anhydride being a cheap chemical could be a replacement for water resistance properties. Scripset 740, another commercially available sizing agent was also used.

Test Methods

Burst Strength

Burst strength is measured using Mullen tester following the TAPPI test method T403 om-97. The sample to be tested is held between annular clamps without any slippage and a rubber diaphragm exerts an increasing pressure. The diaphragm expands due to the hydraulic pressure that is applied until the sample ruptures. The pressure reading at the point of rupture is recorded as the bursting strength. The burst

index is calculated based on the formulae, $\text{Burst index} = \text{Burst strength (kPa)} / \text{Grammage (g/sq.m)} (9)$.

Porosity

The porosity was measured using Parker-Print Surfness tester. Ten readings were taken at random on the impregnated sheet and the mean was calculated. The clamping pressure was at 1000. The mean value is the porosity value given in mL/min.

Stiffness

The stiffness is measured to find the flexibility of the paperboard using TAPPI test method T489 om-92 by calculating the bending moment in gram centimeters to deflect the free end of a 38 mm wide vertically clamped sample 15 degrees from center when the load is applied 50mm from the clamp (9). The sample is fastened on one end and the other end is free. The clamp rotates for 15 degrees in both left and right direction and the readings are noted down for both directions. The average of the reading is given as the stiffness value in Taber units.

Water Resistance

Water resistance is inverse function of water absorption. The Cobb test is used to measure the amount of water absorbed by one square meter of paper for a given time. The lower the cobb values better the water resistance. The cobb test is done using TAPPI test method T441 om-98. The sample is mounted on the water absorption apparatus so that one side of the paper is wetted uniformly during the test. The apparatus covers area of 100 square centimeter of the paper and 100 mL of water is used to wet the sample for 120 seconds. The sample is then removed and pressed between blotting papers using a 20 cm wide roller weighing approximately 22 pounds. The sample is weighed before and after the test. The difference in the weights before and after the test multiplied by 100 gives the cobb value in grams per square meter (9).

Crush Resistance

The ring crush test is done to find the edge compression test of the paperboard. The sample is loaded in a ring that is held in a kind of jig between two platens and the upper platen exerts a compressive on the lower platen when in contact. The compressive force required to rupture the sample is calculated. The test is performed using TAPPI test method T818 om-97 (9).

Experimental Run

Trial Run I

After the initial evaluation of the impregnator, the experimentation started with 270 Penford starch. The starch was cooked in a digester at 15% solids concentration. A 30 gallon batch was prepared in the digester at a temperature of 190°F. The starch was at 55 centipoise viscosity on a Brookfield viscometer with spindle number 1 at 60 rpm speed. The high pressure vessel was loaded with the 15% solids starch and the impregnation was done for various levels of pond pressure and shoe pressure. At higher levels of pond pressure for given shoe pressure, there was over dosage of the starch into the web leading to web breakage. Also there was a wet layer of starch over the treated side of the web.

Trial Run II

The next trial was a step forward towards evaluating the performance of the impregnator with higher concentration of impregnating liquor. 25% solids starch (270 Penford starch) was used as the impregnating chemical. A 30 gallon batch was prepared in the digester at temperature of 190°F. The starch was at 8 centipoise viscosity on Brookfield viscometer on spindle number 3 at 60 rpm. The trial was successful as the impregnator could push in the 25 percent solids starch. Similar to 15

percent solids starch there was oozing out of the impregnating chemical on the web at higher pond pressures for a given shoe pressure.

Trial Run III

After successful trials with two different solids level of starch, calcium lignosulfonate and Kymene along with other sizing agents were used as impregnating chemical. Calcium lignosulfonate and Kymene, chemicals used in the Procter and Gamble patent, were used to find their performance on the impregnator. Borresperse CA-50% liquid supplied by Lignotech USA, Inc. was the commercially available calcium lignosulfonate. It is a brown liquid of 50 percent solids with slight odor and specific gravity of 1.248 at 25 degree Celsius. It has 100 percent solubility in water. Kymene 557H supplied by Hercules Incorporated was the commercial one used for impregnation. It is a pale amber liquid of 12.5% solids with viscosity around 40 - 60 centipoise. Scripset740 alkaline resin solution supplied by Monsanto was also used as an impregnating chemical. Alkenyl succinic anhydride emulsion with starch was also used as another impregnating chemical. Scripset740 at 5 percent solids was first impregnated into the web for various levels of pond pressure for given shoe pressure. Then alkenyl succinic anhydride emulsion at 5.25 percent solids was impregnated for various levels of pond pressure for a given shoe pressure. Calcium lignosulfonate was then impregnated for various levels of pond pressure for given shoe pressure. Then the web impregnated with calcium lignosulfonate at 20 psi pond pressure and 50 psi

shoe pressure was re-impregnated with Kymene for various levels of pond pressure for given shoe pressure.

Trial Run IV

In this trial, impregnation was done with various concentrations of calcium lignosulfonate (25%, 35%, and 50% solids) to study the effect of concentration. The order of impregnation was also changed during the trial by applying Kymene first. Calcium lignosulfonate was then applied in a second run on the Kymene impregnated linerboard. Kymene was impregnated separately for various levels of pond pressure for given shoe pressure. Then calcium lignosulfonate (35% solids concentration) was impregnated on web that had already been impregnated with Kymene at 15 psi pond pressure and 60 psi shoe pressure.

For all the trials, the impregnated samples were conditioned at 23°C and 50% relative humidity. The conditioned samples were then tested for burst strength, stiffness, porosity, water resistance and crush resistance.

CHAPTER V

RESULTS AND DISCUSSIONS

The trial runs were conducted successfully and the measured values for various tests were tabulated. The burst index, porosity, stiffness, edge compression resistance, water resistance, and pickup percentage of the impregnated chemical were calculated for the 42 lb/1000 sq.ft linerboard.

Trial Run I

Table 1 shows the values of pickup percentage, water resistance, and porosity for various levels of pond pressure for each level of shoe pressure for 15% solids starch impregnation on the impregnator.

The pickup percentage increases with an increase for pond pressure and shoe pressure. The increase in pressure makes the impregnating shoe to force more chemical into the linerboard.

The water resistance is found to be lower than the untreated linerboard, due to the hydrophilic nature of starch. As the percentage of starch pickup increases with increase in shoe and pond pressure levels, it leads to more water absorption during the cobb test. The porosity of the linerboard reduces with increased level of shoe and pond pressure levels because starch fills the voids in the linerboard.

Table 1

Effect of Pond and Shoe Pressure on Pickup and Surface Properties – 15% Solids Starch

Pond Pressure psi	Shoe Pressure psi	%Pickup	Cobb Value g/m ²	Cobb Value g/m ²	Porosity mL/min	Porosity mL/min
			Bottom	Top	Bottom	Top
Untreated Sample	Untreated Sample		47.30	58.36	509.2	506.3
5.0	20	1.19	50.00	67.00	103.9	90.80
10	20	2.27	55.30	43.60	80.83	62.95
15	20	3.32	52.20	40.70	75.39	49.91
10	30	2.48	65.00	42.90	100.5	71.29
15	30	2.94	79.70	84.20	107.7	75.90
20	40	6.60	105.2	82.50	158.7	152.0
20	60	2.91	99.90	47.60	151.3	140.8
25	60	2.65	107.0	53.60	166.3	175.1
30	60	4.42	112.1	66.90	143.5	138.6
35	60	4.95	61.00	52.70	114.8	116.4
30	80	10.2	147.0	94.30	193.5	189.2
Standard Deviation			5.240	6.370	6.320	6.530

Table 2 shows the values of burst index edge compression resistance, and stiffness for various levels of pond pressure for each level of shoe pressure for 15% solids starch impregnation on the impregnator.

The burst index increases with the increase in shoe and pond pressure levels. At higher levels of impregnation, the impregnating chemical enters more into the fiber matrix giving good strength properties. The edge compression increases with increased level of shoe and pond pressure levels. The compression strength is found to increase significantly in cross machine direction and that is a positive aspect for consideration as linerboards should possess good compression strength, to find applications in corrugated boards. The stiffness also increases with increase in level of impregnation. When starch fills the voids in the linerboard, the flexibility of the substrate is lost leading to higher stiffness values.

Table 2

Effect of Pond and Shoe Pressure on Strength Properties – 15% Solids Starch

Pressure psi		%Pickup	Burst Index kPam ² /g	Burst Index kPam ² /g	Edge Compression lbs	Edge Compression lbs
Pond	Shoe		Bottom	Top	MD	CD
Untreated Sample	US		3.57	3.34	67.00	80.50
5.0	20	1.19	3.87	4.13	71.50	85.50
10	20	2.27	3.74	3.96	79.50	101.5
15	20	3.32	3.60	4.37	74.00	119.5
10	30	2.48	3.87	4.37	89.00	108.0
15	30	2.94	3.91	4.37	96.00	111.0
20	40	6.60	4.91	5.00	81.50	109.5
20	60	2.91	4.19	4.23	84.50	120.5
25	60	2.65	4.12	4.33	98.50	118.0
30	60	4.42	4.55	4.62	98.00	138.5
35	60	4.95	4.61	4.68	94.00	146.5
30	80	10.2	5.10	5.33	100.0	152.0
Standard Deviation			7.75	7.80	2.870	3.190

The first trial run was used to evaluate the optimum shoe pressure and pond pressure. The results indicated that the pond pressure could have its upper limit at half

the value of shoe pressure for effective impregnation. Also the effective shoe pressure levels were between 40 and 60 psi. The next trial run was conducted with 25% solids starch at shoe pressure levels of 40 psi and 60 psi.

Trial Run II

Table 3 shows the values of pickup percentage, water resistance and porosity for various levels of pond pressure for each level of shoe pressure for 25% solids starch impregnation. The pickup percentage is found to be higher for impregnation with 25% solids starch rather 15% solids starch, at the same level of pond and shoe pressures. The water resistance increases for impregnation with 25% solids starch (25 psi pond pressure) than with 15% solids starch (30 psi pond pressure) at same pickup level for impregnation at 60 psi shoe pressure. The porosity reduces for impregnation with 25% starch than 15% starch. Also, the stiffness of the linerboard increases for impregnation with 25% solids starch compared to that with 15% solids.

Table 4 shows the values of burst index and stiffness for various levels of pond pressure for each level of shoe pressure for 25% solids starch impregnation. The burst index of the impregnated board doesn't increase when impregnated with 25% solids when compared to 15% solids starch.

Table 3

Effect of Pond and Shoe Pressure on Pickup and Surface Properties – 25% Solids Starch

Pond Pressure psi	Shoe Pressure psi	% Pickup	Cobb Value g/m ²		Porosity mL/min	
			Bottom	Top	Bottom	Top
Untreated Sample	Untreated Sample		47.30	58.36	509.2	506.3
5.0	40	2.39	47.96	39.92	55.68	35.29
10	40	2.77	47.50	38.88	57.57	27.72
15	40	3.29	50.92	40.30	55.19	32.94
20	40	4.10	56.78	38.62	45.34	30.38
5.0	60	3.65	52.30	36.98	70.88	50.50
10	60	3.44	55.00	38.20	69.19	46.25
15	60	3.60	50.80	45.80	66.12	48.67
20	60	4.02	58.84	38.54	77.46	49.09
25	60	4.37	58.66	35.26	81.20	62.43
30	60	5.28	62.64	34.58	77.59	61.66
Standard Deviation			4.130	3.980	5.120	7.210

After successful impregnation with starch at 25% solids, various other chemicals were used for impregnation.

Trial Run III

Table 5 shows the values of pickup percentage, water resistance, and porosity for various levels of pond pressure for each level of shoe pressure for impregnation on the impregnator using Scripset740, ASA, Calcium lignosulfonate and Kymene.

Table 4

Effect of Pond and Shoe Pressure on Strength Properties – 25% Solids Starch

Pressure psi		% Pickup	Burst Index kPam ² /g		Stiffness Taber Units
Pond	Shoe		Bottom	Top	
US	US		3.57	3.34	34.90
5.0	40	2.39	4.05	4.10	53.80
10	40	2.77	4.06	4.30	50.42
15	40	3.29	3.91	4.18	50.35
20	40	4.10	4.22	4.26	52.19
5.0	60	3.65	4.27	4.25	53.17
10	60	3.44	3.89	4.25	48.35
15	60	3.60	4.06	4.26	51.86
20	60	4.02	4.02	4.32	53.32
25	60	4.37	4.04	4.37	51.06
30	60	5.28	4.20	4.55	55.20
Standard Deviation			7.21	7.38	2.830

The pickup percentage is low for Scripset740 and ASA as they were impregnated at 5% solids. The pickup level of calcium lignosulfonate was low at 50 psi shoe pressure due to excessive loss of calcium lignosulfonate through the outlet pipe. In general, if the pressure levels were increased beyond the optimum, pickup values dropped in all cases.

Table 5

Effect of Pond and Shoe Pressure on Pickup and Surface Properties – Scripset740, ASA, Calcium Lignosulfonate, and Kymene

	Pond Pressure psi	Shoe Pressure psi	% Pickup	Cobb Value g/m ²		Porosity mL/min	
				Bottom	Top	Bottom	Top
Untreated Sample				47.30	58.36	509.2	506.3
Scripset740	10	20	1.89	34.00	35.90	88.27	84.29
(5% solids)	5.0	40	1.32	40.67	37.23	92.60	89.25
	10	40	1.04	33.33	33.50	80.10	51.15
ASA	10	20	0.71	38.93	43.47	209.7	184.5
(5% solids)	5.0	30	0.49	40.87	42.20	322.4	320.0
	10	30	0.29	42.73	44.73	255.4	224.7
	10	40	0.15	40.07	41.63	205.8	192.6
	20	40	0.35	38.13	39.17	192.2	168.8
	20	60	0.36	38.23	38.73	249.9	233.0
	30	60	0.85	43.87	47.60	171.0	171.4
CaLS	10	30	3.80	54.67	70.17	39.19	23.45
(50% solids)	10	40	4.18	62.63	77.67	38.56	25.53
	15	40	4.81	102.6	111.1	38.47	26.41
	20	50	1.45	92.93	92.10	35.01	29.84
CaLS+Kymene	10	50	0.77	76.87	59.50	7.110	7.110
(12.5% solids Kymene)	20	50	1.38	56.10	83.63	6.060	6.060
	20	60	1.30	76.30	55.20	17.91	7.250
Standard Deviation				6.970	7.830	6.380	6.740

The water resistance values were significantly high for impregnation with the commercial sizing agents - Scripset740 and ASA emulsion. Calcium lignosulfonate is however hydrophilic in nature leading to low water resistance value but impregnation of Kymene (12.5% solids) over calcium lignosulfonate (50% solids applied at 20 psi pond pressure and 50 psi shoe pressure) increases the water resistance as Kymene cross links with calcium lignosulfonate and hence makes it impervious to water.

Table 6 shows the values of burst index, edge compression resistance, and stiffness for various levels of pond pressure for each level of shoe pressure for impregnation on the impregnator using Scripset740, ASA, Calcium lignosulfonate, and Kymene.

The burst index is lower for impregnation with the above chemicals when compared to starch. The edge compression strength and stiffness is significantly high for calcium lignosulfonate and Kymene when compared to other impregnating agents. The edge compression strength and stiffness values are in the same range for starch, scripset740 and ASA.

The edge compression has no significant increase for impregnation with ASA. The compression strength for impregnation with calcium lignosulfonate is twice the untreated sample in both machine and cross machine direction. The calcium lignosulfonate and Kymene impregnated samples showed greater increase in compression strength than those impregnated with calcium lignosulfonate alone. These significant increases in compression strength along machine and cross machine direction of liner board makes calcium lignosulfonate and Kymene a better choice for usage in corrugated boards manufacture.

Table 6

Effect of Pond and Shoe Pressure on Strength Properties – Scripset740, ASA, Calcium Lignosulfonate, and Kymene

	Pressure psi		% Pickup	Burst Index kPa ^m /g		Edge Compression lbs	
	Pond	Shoe		Bottom	Top	MD	CD
Untreated Sample	US	US		3.57	3.34	67.00	80.50
Scripset	10	20	1.89	4.07	4.23	78.50	102.0
(5% solids)	5.0	40	1.32	4.13	4.11	71.50	98.50
	10	40	1.04	4.14	4.56	83.00	104.5
ASA	10	20	0.71	3.79	3.73	68.50	89.00
(5% solids)	5.0	30	0.49	3.60	3.78	68.00	84.50
	10	30	0.29	3.73	3.82	66.50	87.00
	10	40	0.15	3.49	3.68	65.50	76.50
	20	40	0.35	3.61	3.91	69.00	91.50
	20	60	0.36	3.74	3.95	65.50	95.00
	30	60	0.85	3.81	4.15	68.00	87.50
CaLS	10	30	3.80	3.90	3.79	105.5	134.5
(50% solids)	10	40	4.18	3.80	3.69	104.5	134.0
	15	40	4.81	3.90	3.75	120.0	156.5
	20	50	1.45	4.09	3.82	132.5	172.0
CaLS+Kymene	10	50	0.77	3.36	3.78	122.0	179.0
(12.5% solids Kymene)	20	50	1.38	3.52	3.77	155.0	183.0
	20	60	1.30	3.52	3.61	145.0	173.0
Standard Deviation				6.94	7.37	2.170	2.780

To hone the findings from the trial run 3, various solid levels of calcium lignosulfonate as well as Kymene alone were tried in the fourth set of impregnation trials.

Trial Run IV

Table 7 shows the values of pickup percentage, water resistance, and porosity for impregnation of various levels of calcium lignosulfonate and Kymene. The pickup increases as the solids level of calcium lignosulfonate decreases.

The pickup level is around the same range for chemicals that are in the same range of solids. For example, 15% solids starch and Kymene (12.5% solids) have pickup around 2-4%. The water resistance is the least when calcium lignosulfonate is impregnated due to its hydrophilic nature. The water resistance is better than starch and equivalent to the commercial sizing agents when Kymene alone is impregnated but porosity is not improved markedly. Calcium lignosulfonate fills the pores effectively at all solids level but porosity reduces as we impregnate with higher solids of calcium lignosulfonate.

Table 8 shows the values of burst Index, edge compression resistance, and stiffness for impregnation of various levels of calcium lignosulfonate and Kymene.

Impregnation with Kymene alone has better burst index than calcium lignosulfonate. The burst index values and the edge compression strength are in the same range for impregnation with starch and Kymene. The stiffness values are slightly better for the starch impregnation than Kymene. The starch impregnation is

better than calcium lignosulfonate as far as burst index is concerned. The stiffness values for impregnation with calcium lignosulfonate are significantly high even at 25% solids. The edge compression strength is also on the higher side for calcium lignosulfonate impregnated linerboards when compared to the other impregnating chemicals.

Table 7

Effect of Pond and Shoe Pressure on Pickup and Surface Properties –Calcium Lignosulfonate and Kymene

	Pond Pressure psi	Shoe Pressure psi	%Pickup	Cobb Value g/m ²		Porosity mL/min	
				Bottom	Top	Bottom	Top
Untreated Sample				47.30	58.36	509.2	506.3
Cals 50% solids	10	60	2.31	161.6	165.3	96.30	87.70
Cals 35% solids	10	60	5.02	78.30	89.93	61.67	38.56
	20	60	1.10	146.3	139.2	117.6	101.1
	30	60	0.80	163.2	165.3	207.4	108.0
Cals 25% solids	10	60	8.70	146.4	167.5	191.7	185.8
Kymene 12.5% solids	10	60	2.61	60.00	112.3	506.5	490.4
	15	60	4.03	41.93	70.03	443.3	439.1
	20	60	4.09	41.37	64.67	480.8	449.1
	30	60	2.05	43.67	67.97	473.5	463.3
Kymene+ Cals 35% solids	10	60	11.4	66.03	54.30	138.4	130.1
Standard Deviation				6.790	7.420	5.970	6.630

Influence of Operating Conditions on Properties of Impregnated Linerboard

The results were also analyzed to determine whether the difference in properties arose due to operating pressure or due to the innate chemistry of the chemical used. Various properties were hence compared at the same pickup

percentage at different operating conditions for different chemicals. These results are presented in Tables 9 to 14. In all these tables pair wise comparison of impregnating chemicals are presented.

Table 8

Effect of Pond and Shoe Pressure on Strength Properties – Calcium Lignosulfonate and Kymene

	Pressure psi		%Pickup	Burst Index kPam ² /g		Edge Compression lbs	
	Pond	Shoe		BFU	TFU	MD	CD
Untreated Sample	US	US		3.57	3.34	67.00	80.50
Cals 50% solids	10	60	2.31	3.41	3.55		
Cals 35% solids	10	60	5.02	3.80	3.52	107.5	141.0
	20	60	1.10	3.77	3.91	118.0	150.0
	30	60	0.80	3.59	3.32	141.5	150.0
Cals 25% solids	10	60	8.70	3.86	3.92	107.0	116.0
Kymene 12.5% solids	10	60	2.61	3.91	3.64	74.00	81.00
	15	60	4.03	4.25	4.45	70.50	104.0
	20	60	4.09	4.17	4.43	74.00	104.5
	30	60	2.05	4.39	4.66	83.00	107.0
Kymene+Cals 35% solids	10	60	11.4	3.71	3.73	131.5	132.5
Standard Deviation				7.14	7.53	3.160	2.970

From Table 9, we find that with 60 psi shoe pressure; Kymene (12.5% solids) at 10 psi pond pressure has nearly the same amount of pickup as 15% starch at 25 psi pond pressure. However, the starch reduces the porosity better than Kymene. Burst index and edge compression are also higher for 15% starch than Kymene (Table 9). The better performance of 15% starch can be attributed to the chemistry of starch and to some extent to the increased pond pressure used for impregnation. Similarly at

same shoe pressure of 60 psi, Kymene (15 psi pond pressure) has the same pickup level as 25% starch (20 psi pond pressure). But, 25% starch reduces porosity significantly than Kymene. At 20 psi pond pressure, Kymene (60 psi shoe pressure) has the same amount of pickup as 25% starch (40 psi shoe pressure). The difference in solids level could be a possible explanation for similar pickup levels at higher shoe pressure for Kymene. Porosity is reduced by starch impregnation at same pond pressure.

It can be seen from Table 10 that at equal pickup, porosity decreases with 25% starch as compared to 15% starch. Equal pickup occurs at lower pond to shoe pressure ratio with 25% starch. The cobb values don't exhibit a pattern. At equal pickup there is no difference in strength properties.

Table 11 indicates that water resistance is due to the chemicals and not operating conditions. At equal pickup and identical operating conditions, calcium lignosulfonate has cobb values in the range of 160 g/m² while ASA produces value around 47 g/m². Calcium lignosulfonate and Kymene with the same pickup level of Kymene as in the above two cases has a cobb value of about 60-75 g/m² that falls between the values obtained by calcium lignosulfonate and ASA. It is also seen that when Kymene is impregnated on calcium lignosulfonate porosity value decreases enormously due to cross linking. Calcium lignosulfonate increases all the strength properties much better than ASA.

Table 12 shows that water resistance is better for 15% starch than calcium lignosulfonate at equal pickup. Calcium lignosulfonate has lower pond to shoe pressure ratio than starch. Porosity is also reduced for 15% starch impregnation.

Table 13 indicates that ASA at equal pickup and different operating conditions has similar water resistance and strength properties. However porosity decreases with higher shoe pressure.

Table 14 indicates that water resistance is better for impregnation with Scripset740 than calcium lignosulfonate with Kymene due to the chemical used rather than the operating conditions. Calcium lignosulfonate and Kymene impregnated board has lower porosity value than Scripset740 impregnated board. Calcium lignosulfonate with Kymene also increases the crush strength properties much better than Scripset740.

Overall, the difference in physical and surface properties at equal absorption values is largely due to the nature of the chemicals rather than due to the operating pressures. The operating pressures have to be maintained at different levels for different chemicals to ensure same pickup.

Table 9

Influence of Operating Conditions at Equal Pickup – Kymene, 15% Starch and 25% starch

	Pressure psi		%Pick up	Cobb Value g/m ²	Cobb Value g/m ²	Porosity mL/min	Porosity mL/min	Burst Index kPam ² /g	Burst Index kPam ² /g	Edge Compr ession lbs	Egde Compr ession lbs	Stiffness Taber units
	Pond	Shoe		Bottom	Top	Bottom	Top	Bottom	Top	MD	CD	
15% Starch	25	60	2.65	107.0	53.60	166.3	175.1	4.12	4.33	98.50	118.0	53.20
Kymene	10	60	2.61	60.00	112.3	506.5	490.4	3.91	3.64	74.00	81.00	44.22
Standard Deviaition				3.100	4.670	5.890	6.340	8.52	8.98	4.650	3.740	1.770
25% Starch	20	40	4.10	56.78	38.62	45.34	30.38	4.22	4.26			52.19
Kymene	20	60	4.09	41.37	64.67	480.8	449.1	4.17	4.43	74.00	104.5	48.74
Standard Deviaition				3.120	3.270	4.360	4.920	4.92	6.87			2.530
25% Starch	20	60	4.02	58.84	38.54	77.46	49.09	4.02	4.32			53.32
Kymene	15	60	4.03	41.93	70.03	443.3	439.1	4.25	4.45	70.50	104.0	46.53
Standard Deviaition				3.330	3.960	5.280	5.860	6.57	5.90			2.850

Table 10

Influence of Operating Conditions at Equal Pickup – 15% Starch and 25% starch

	Pressure psi		%Pick up	Cobb Value g/m ²	Cobb Value g/m ²	Porosity mL/min	Porosity mL/min	Burst Index kPam ² /g	Burst Index kPam ² /g	Edge Compr ession lbs	Egde Compr ession lbs	Stiffness Taber units
	Pond	Shoe		Bottom	Top	Bottom	Top	Bottom	Top	MD	CD	
15% Starch	15	20	3.32	52.20	40.70	75.39	49.91	3.60	4.37	74.00	119.5	54.70
25% Starch	15	40	3.29	50.92	40.30	55.19	32.94	3.91	4.18			50.35
Standard Deviaition				3.580	2.560	6.470	5.780	6.48	7.32			2.710
15% Starch	30	60	4.42	112.1	66.90	143.5	138.6	4.55	4.62	98.00	138.5	51.00
25% Starch	25	60	4.37	58.66	35.26	81.20	62.43	4.04	4.37			51.06
Standard Deviaition				3.460	2.490	5.450	6.290	7.21	6.98			1.930

Table 11

Influence of Operating Conditions at Equal Pickup – 35% Calcium Lignosulfonate, ASA and Calcium Lignosulfonate with Kymene

	Pressure psi		%Pick up	Cobb Value g/m ²	Cobb Value g/m ²	Porosity mL/min	Porosity mL/min	Burst Index kPam ² /g	Burst Index kPam ² /g	Edge Compr ession lbs	Egde Compr ession lbs	Stiffness Taber units
	Pond	Shoe		Bottom	Top	Bottom	Top	Bottom	Top	MD	CD	
Cals 35	30	60	0.80	163.2	165.3	207.4	108.0	3.59	3.32	141.5	150.0	89.05
ASA	30	60	0.85	43.87	47.60	171.0	171.4	3.81	4.15	68.00	87.50	50.44
Cals Kymene	10	50	0.77	76.87	59.50	7.110	7.110	3.36	3.78	122.0	179.0	77.63
Standard Deviaition				2.940	8.540	7.490	6.820	4.38	5.79	2.830	2.510	2.270

Table 12

Influence of Operating Conditions at Equal Pickup – Calcium Lignosulfonate and 15% Starch

	Pressure psi		%Pick up	Cobb Value g/m ²	Cobb Value g/m ²	Porosity mL/min	Porosity mL/min	Burst Index kPam ² /g	Burst Index kPam ² /g	Edge Compr ession lbs	Egde Compr ession lbs	Stiffness Taber units
	Pond	Shoe		Bottom	Top	Bottom	Top	Bottom	Top	MD	CD	
Cals 50	10	60	2.31	161.6	165.3	96.30	87.70	3.41	3.55			78.53
15% Starch	10	20	2.27	55.30	43.60	80.83	62.95	3.74	3.96	79.50	101.5	49.00
Standard Deviaition				3.580	2.940	4.870	6.170	6.18	7.24			2.280

Table 13

Influence of Operating Conditions at Equal Pickup – ASA

	Pressure psi		%Pick up	Cobb Value g/m ²	Cobb Value g/m ²	Porosity mL/min	Porosity mL/min	Burst Index kPam ² /g	Burst Index kPam ² /g	Edge Compr ession lbs	Egde Compr ession lbs	Stiffness Taber units
	Pond	Shoe		Bottom	Top	Bottom	Top	Bottom	Top	MD	CD	
ASA	20	40	0.35	38.13	39.17	192.2	168.8	3.61	3.91	69.00	91.50	44.76
ASA	20	60	0.36	38.23	38.73	249.9	233.0	3.74	3.95	65.50	95.00	43.36
Standard Deviaition				1.260	1.110	6.370	5.810	4.62	4.39	1.870	1.740	2.170

Table 14

Influence of Operating Conditions at Equal Pickup – Calcium Lignosulfonate with Kymene and Scripset740

	Pressure psi		%Pick up	Cobb Value g/m ²	Cobb Value g/m ²	Porosity mL/min	Porosity mL/min	Burst Index kPam ² / g	Burst Index kPam ² /g	Edge Compr ession lbs	Edge Compr ession lbs	Stiffness Taber units
	Pond	Shoe		Bottom	Top	Bottom	Top	Bottom	Top	MD	CD	
Cals Kymene	20	60	1.30	76.30	55.20	17.91	7.250	3.52	3.61	145.0	173.0	63.61
Scripset740	5.0	40	1.32	40.67	37.23	92.60	89.25	4.13	4.11	71.50	98.50	37.47
Standard Deviaition				1.430	1.240	5.930	6.420	6.51	5.99	2.950	2.370	2.490

CHAPTER VI

CONCLUSIONS

After four successful trials with the impregnator, the following conclusions can be drawn:

1. For effective impregnation, the pond pressure should have its upper limit as half the shoe pressure value used for impregnation. The shoe pressure itself has an optimum range between 40-60 psi that depends on the chemical and its concentration.
2. Pickup percentage remains relatively low around 3-5% for all impregnating chemicals except for Scripset740 and ASA. Also for the same pond pressure, an increase in shoe pressure causes an increase in the pickup percentage.
3. Edge compression strength increases nearly twice that of the untreated sample when impregnated with calcium lingsulfonate and also for impregnation with Kymene on a linerboard pre-impregnated with calcium lingsulfonate. Kymene when impregnated upon calcium lingsulfonate enhances the edge compression strength of the linerboard but doesn't increase the edge compression strength significantly when

impregnated alone. Compression strength increase is mainly due to interactions of calcium lignosulfonate with the fibers. Kymene enhances the compression strength by cross-linking with the calcium lignosulfonate. Starch tends to increase the edge compression strength by nearly 50% of the original value. Scripset740 and ASA don't have significant influence on the edge compression strength.

4. Porosity value falls drastically to single digit when Kymene is impregnated on a calcium lignosulfonate impregnated linerboard because cross linking takes place between them. Even the other chemicals reduce the porosity values significantly.

5. Stiffness values increases markedly for higher levels of impregnation irrespective of the impregnating chemical used.

6. High water absorption value of calcium lignosulfonate impregnated linerboards is due to its hydrophilic nature. 15% solids starch also has higher cobb values due to its hydrophilic nature. However, impregnation with 25% starch shows better water resistance than 15% starch due to lower porosity values. Scripset740 and ASA tend to increase the water resistance properties of the linerboard, by virtue of their sizing characteristics.

7. The difference in physical and surface properties at equal absorption values is largely due to the nature of the chemicals rather than due to the operating pressures. The operating pressures have to be maintained at different levels for different chemicals to ensure same pickup.

CHAPTER VII

FUTURE STUDIES

The following additional studies need to be conducted:

1. Impregnation of calcium lignosulfonate and Kymene on linerboard of different grammage and initial sizing to study (a) the optimum sizing level and (b) the extent of reduction in basis weight that could be achieved without losing strength properties.
2. Creating boxes to study the effective crushing resistance of the linerboard.
3. Measuring the extent of penetration by pore distribution test using BET analysis.
4. Study the printability characteristics as well as gluability of the impregnated paper.
5. Study the correlation between the printability of the linerboard and the distance of penetration after impregnation.

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