



Western Michigan University  
ScholarWorks at WMU

---

Master's Theses

Graduate College

---

4-2000

## Borax as Internal Stock Additive in Papermaking

Nejib S. Yousef

Follow this and additional works at: [https://scholarworks.wmich.edu/masters\\_theses](https://scholarworks.wmich.edu/masters_theses)



Part of the Wood Science and Pulp, Paper Technology Commons

---

### Recommended Citation

Yousef, Nejib S., "Borax as Internal Stock Additive in Papermaking" (2000). *Master's Theses*. 4942.  
[https://scholarworks.wmich.edu/masters\\_theses/4942](https://scholarworks.wmich.edu/masters_theses/4942)

This Masters Thesis-Open Access is brought to you for free and open access by the Graduate College at ScholarWorks at WMU. It has been accepted for inclusion in Master's Theses by an authorized administrator of ScholarWorks at WMU. For more information, please contact [wmu-scholarworks@wmich.edu](mailto:wmu-scholarworks@wmich.edu).



**BORAX AS INTERNAL STOCK ADDITIVE  
IN PAPERMAKING**

by

**Nejib S. Yousef**

**A Thesis  
Submitted to the  
Faculty of the Graduate College  
in partial fulfillment of the  
requirements for the  
Degree of Master of Arts  
Department of Paper and Printing  
Science and Engineering**

**Western Michigan University  
Kalamazoo, Michigan  
April 2000**

Copyright by  
Nejib S. Yousef  
2000

## ACKNOWLEDGMENTS

I want to express my deepest appreciation to my advisor Dr. Raja Aravamuthan for his guidance and support throughout the project. I extend my gratitude to the committee members, Dr. John Cameron and Dr. Peter Parker.

I also want to thank all members of the NCASI and the Western Michigan University pilot plant employees who constantly supported and encouraged me until the project came to completion.

Lastly, to my family members and friends who were helpful in every part of my life. I finally dedicate this thesis to my beloved parents who always loved me unconditionally.

Nejib S. Yousef

## BORAX AS INTERNAL STOCK ADDITIVE IN PAPERMAKING

Nejib S. Yousef, M.S.

Western Michigan University, 2000

The effect of borax on strength property of paper has been investigated in different ways. The use of borax either improved the physical properties or assisted in the reduction of refining energy to significant level.

The study of borax in the presence of precipitated and ground calcium carbonate was accomplished by using white water recycling. In the absence of retention aids, recycling of white was a necessary condition for the retention of ash content. As the ash content increased the strength property decreased. But the presence of borax slowed down the detrimental effect of fillers.

Sizing development progressively improved especially at high dosage of AKD addition and for all levels of borax addition. Noticeable change had not been observed in terms of strength properties. The inclusion of filler in the borax and AKD system showed detrimental effect on strength development. Neither borax nor AKD were able to counteract the negative role of the filler. The result of size reversion was inconclusive.

Borax in the surface application was insignificant to bring property changes.

## TABLE OF CONTENTS

ACKNOWLEDGMENTS .....	ii
LIST OF TABLES .....	vii
LIST OF FIGURES.....	x
CHAPTER	
I. INTRODUCTION.....	1
II. LITERATURE REVIEW.....	2
Nature of Paper Strength .....	2
Furnish Variables .....	3
Chemistry of Borax.....	5
Process Variables.....	6
III. FUNDAMENTAL ASPECTS OF SIZING .....	10
Chemical Structure .....	10
Hydrophobation With AKD .....	11
Reaction of AKD With water .....	12
Macro View of AKD Sizing Mechanism.....	12
Effect of Filler on Alkaline Papermaking .....	13
Effect of Filler on Strength Properties .....	14
Effect of AKD on Paper Strength.....	14
Size Reversion.....	15
IV. STATEMENT OF THE PROBLEM AND OBJECTIVES.....	17

## Table of Contents – Continued

### CHAPTER

V. EXPERIMENTAL DESIGN AND PROCEDURE .....	18
Phase 1: Optimizing of Borax Level .....	27
Phase 2: Effect of Borax in Presence of Filler .....	28
Phase 3: Effect of Borax on Alkaline Sizing .....	28
Phase 4: Effect of Borax in Surface Applications of Paper .....	29
VI. RESULTS AND DISCUSSION.....	30
Phase 1: Borax and Fiber System .....	30
Phase 1.1: Tensile Energy Absorption .....	31
Phase 1.2: Tensile Energy Absorption.....	34
Phase 1.1: Tear Index .....	36
Phase 1.2: Tear Index .....	38
Phase 1.1: Burst Index.....	39
Phase 1.2: Burst Index.....	41
Phase 1.1: Stiffness.....	43
Phase 1.2: Stiffness.....	45
Phase 1.1: Folding Endurance.....	46
Phase 1.2: Folding Endurance.....	48
Phase 1.1: Internal Bond Strength.....	49
Phase 1.2: Internal Bond Strength.....	51
Conclusion .....	53

## Table of Contents – Continued

### CHAPTER

Phase 2: Borax and Filler System .....	54
Ash Content .....	55
Tensile Energy Absorption for GCC and PCC Filled Papers .....	59
Tear Index for GCC and PCC Filled Papers .....	64
Burst Index for GCC and PCC Filled Papers .....	69
Stiffness for GCC and PCC Filled Papers .....	74
Folding Endurance for GCC and PCC Filled Papers .....	78
Conclusion .....	82
AKD Sizing System .....	82
Phase 3.0: Fiber, Borax and AKD System .....	83
Hercules Size Testing .....	84
Phase 3.1: AKD Sizing and Filler System .....	86
Hercules Size Testing .....	87
Stiffness.....	88
Phase 3.2: Size Reversion .....	90
Conclusion .....	91
Borax in Surface Application .....	92
Tear Index .....	92
Conclusion .....	94
VII. CONCLUSION .....	95



## Table of Contents – Continued

### CHAPTER

VIII. RECOMMENDATIONS FOR FURTHER STUDY .....	97
-----------------------------------------------	----

LITERATURE CITED.....	98
-----------------------	----

### APPENDICES

A. Analysis of Variance of the Strength Properties (Phase 1.1).....	100
---------------------------------------------------------------------	-----

B. Analysis of Variance for Strength Properties (Phase 1.2) .....	105
-------------------------------------------------------------------	-----

C. Raw Data of the Strength Properties in Relation to Filler Loading for GCC and PCC Filled Papers (Phase 2.0).....	110
------------------------------------------------------------------------------------------------------------------------	-----

D. Test Method to Measure Alkalinity .....	117
--------------------------------------------	-----

E. Effect of Borax as Surface Sizing on Strength Properties of Paper (Phase 4.0).....	119
------------------------------------------------------------------------------------------	-----

BIBLIOGRAPHY .....	122
--------------------	-----

## LIST OF TABLES

1. Fiber Characteristics Modified or Developed by Beating Action.....	7
2. Summary of the Experimental Levels for the Design Variables .....	24
3. TAPPI Standard Testing Methods.....	26
4. Phase 1.1: Effect of Borax and Refining on TEA ( $\text{kJ/m}^2$ ).....	33
5. Phase 1.2: Effect of Borax and Refining on TEA ( $\text{kJ/m}^2$ ).....	34
6. Phase 1.1: Effect of Borax and Refining on Tear Index ( $\text{mNm}^2/\text{g}$ ) .....	37
7. Phase 1.2: Effect of Borax and Refining on Tear Index ( $\text{mNm}^2/\text{g}$ ) .....	39
8. Phase 1.1: Effect of Borax and Refining on Burst Index ( $\text{kPam}^2/\text{g}$ ).....	41
9. Phase 1.2: Effect of Borax and Refining on Burst Index ( $\text{kPam}^2/\text{g}$ ).....	42
10. Phase 1.1: Effect of Borax and Refining on Stiffness (mN) .....	44
11. Phase 1.2: Effect of Borax and Refining on Stiffness (mN) .....	46
12. Phase 1.1: Effect of Borax and Refining on Folding Endurance .....	47
13. Phase 1.2: Effect of Borax and Refining on Folding Endurance .....	49
14. Phase 1.1: Effect of Borax and Refining on Internal Bond Strength (kPa).....	51
15. Phase 1.2: Effect of Borax and Refining on Internal Bond Strength (kPa).....	52
16. Effect of Borax Addition and Refining on Various Strength Properties.....	53
17. Phase 2.0: The Regression Analysis of Ash Content for GCC .....	57
18. Phase 2.0: The Regression Analysis of Ash Content for PCC.....	57
19. Ash Content of the GCC and PCC at 7500 Revolutions.....	58
20. Phase 2.0: The Regression Analysis of TEA for GCC.....	61

## List of Tables - Continued

21.	Phase 2.0: The Regression Analysis of TEA for PCC .....	62
22.	Effect of Borax and Filler Addition on TEA at 7500 Revolutions .....	63
23.	Phase 2.0: The Regression Analysis of Tear Index for GCC.....	66
24.	Phase 2.0: The Regression Analysis of Tear Index for PCC .....	67
25.	Effect of Borax and Filler Addition on Tear Index at 7500 Revolutions .....	68
26.	Phase 2.0: The Regression Analysis of Burst Index for GCC .....	71
27.	Phase 2.0: The Regression Analysis of Burst Index for PCC.....	72
28.	Effect of Borax and Filler Addition on Burst Index at 7500 Revolutions.....	73
29.	Phase 2.0: The Regression Analysis of Stiffness for GCC .....	75
30.	Phase 2.0: The Regression Analysis of Stiffness for PCC.....	76
31.	Effect of Borax and Filler Addition on Stiffness at 7500 Revolutions.....	77
32.	Phase 2.0: The Regression Analysis of Folding Endurance for GCC .....	80
33.	Phase 2.0: The Regression Analysis of Folding Endurance for PCC.....	80
34.	Effect of Borax and Filler Addition on Folding Endurance at 7500 Revolutions.....	81
35.	Phase 3.0: The Regression Analysis of HST .....	84
36.	Phase 3.0: Effect of Borax and AKD Addition on HST at 7500 Revolutions.....	85
37.	Phase 3.1: Effect of Borax, AKD and Filler Addition on HST at 7500 Revolutions.....	87
38.	Phase 3.1: Effect of Borax, AKD and Filler Addition on Stiffness at 7500 Revolutions.....	89
39.	Phase 3.2: Effect of Borax on Size Reversion.....	90
40.	Phase 4.0: The Regression Analysis of Tear Index.....	92

## List of Tables – Continued

41.	Statistical Output of the Strength Properties for Phase 1.1.....	101
42.	Statistical Output of the Strength Properties for Phase 1.2.....	106
43.	Effect of Ash Content and Borax Addition on Strength Properties of GCC Filled Papers at 7500 Revolutions.....	111
44.	Effect of Ash Content and Borax Addition on Strength Properties of PCC Filled Papers at 7500 Revolutions .....	114
45.	Effect of Borax as Surface Sizing Agent on Strength Properties .....	120

## LIST OF FIGURES

1. Borate Ester Cross-Linking.....	5
2. Alkyl Ketene Dimer (AKD).....	10
3. Reaction Between AKD and Cellulose .....	11
4. Hydrolysis of AKD .....	12
5. Steps in Sizing With AKD.....	13
6. Operation to Optimize Borax Addition (Phase 1.1).....	19
7. Operation at Optimal Level of Borax Addition (Phase 1.2).....	20
8 Borax Addition in the Presence of Filler or Alkaline Sizing Agent (Phase 2 & 3.1).....	21
9. Effect of Borax in Alkaline Sizing With Filler Inclusion (Phase 3.2).....	22
10. Borax as Surface Sizing Agent and Its Effect on Paper Properties (Phase 4).....	23
11. Effect of Borax Addition on Tensile Energy Absorption at Different PFI Mill Revolutions (Phase 1.1) .....	32
12. Effect of Borax Addition on Tensile Energy Absorption at Different PFI Mill Revolutions (Phase 1.2) .....	35
13. Effect of Borax Addition on Tear Index at Different PFI Mill Revolutions (Phase 1.1) .....	36
14. Effect of Borax Addition on Tear Index at Different PFI Mill Revolutions (Phase 1.2) .....	38
15. Effect of Borax Addition on Burst Index at Different PFI Mill Revolutions (Phase 1.1) .....	40
16. Effect of Borax Addition on Burst Index at Different PFI Mill Revolutions (Phase 1.2) .....	42

## List of Figures—continued

17.	Effect of Borax Addition on Stiffness at Different PFI Mill Revolutions Phase 1.1 .....	43
18.	Effect of Borax Addition on Stiffness at Different PFI Mill Revolutions (Phase 1.2).....	45
19.	Effect of Borax Addition on Folding Endurance at Different PFI Mill Revolutions (Phase 1.1) .....	47
20.	Effect of Borax Addition on Folding Endurance at Different PFI Mill Revolutions (Phase 1.2) .....	48
21.	Effect of Borax Addition on Internal Bond Strength at Different PFI Mill Revolutions (Phase 1.1) .....	50
22.	Effect of Borax Addition on Internal Bond Strength at Different PFI Mill Revolutions (Phase 1.2) .....	52
23.	Effect of Borax Addition in Filler Retention of GCC Filled Papers at 7500 PFI Mill Revolutions .....	55
24.	Effect of Borax Addition in Filler Retention of PCC Filled Papers at 7500 PFI Mill Revolutions .....	56
25.	Effect of Ash Content and Borax Addition on TEA of GCC Filled Papers at 7500 PFI Mill Revolutions .....	59
26.	Effect of Ash Content and Borax Addition on TEA of PCC Filled Papers at 7500 PFI Mill Revolutions .....	60
27.	Effect of Ash Content and Borax Addition on Tear Index of GCC Filled Papers at 7500 PFI Mill Revolutions .....	65
28.	Effect of Ash Content and Borax Addition on Tear Index of PCC Filled Papers at 7500 PFI Mill Revolutions .....	65
29.	Effect of Ash Content and Borax Addition on Burst Index of GCC Filled Papers at 7500 PFI Mill Revolutions.....	69
30.	Effect of Ash Content and Borax Addition on Burst Index of PCC Filled Papers at 7500 PFI Mill Revolutions .....	70

## List of Figures—continued

31. Effect of Ash Content and Borax Addition on Stiffness of PCC Filled Papers at 7500 PFI Mill Revolutions .....	74
32. Effect of Ash Content and Borax Addition on Stiffness of PCC Filled Papers at 7500 PFI Mill Revolutions .....	75
33. Effect of Ash Content and Borax Addition on Folding Endurance of GCC Filled Papers at 7500 PFI Mill Revolutions.....	78
34. Effect of Ash Content and Borax Addition on Folding Endurance of PCC Filled Papers at 7500 PFI Mill Revolutions.....	79
35. Effect of Borax and Sizing Addition on HST at 7500 PFI Mill Revolutions (Phase 3.0) .....	85
36. Effect of Borax and Sizing Addition on HST at 7500 PFI Mill Revolutions (Phase 3.1) .....	88
37. Effect of Borax, Sizing and Filler Addition on Stiffness at 7500 PFI Mill Revolutions (Phase 3.1) .....	89
38. Effect of Borax as Surface Sizing Agent on the Tear Index at Different PFI Mill Revolutions .....	93

## CHAPTER I

### INTRODUCTION

The increasing demand in the use of secondary fibers, wood-free fibers, more hardwood, high filler content and lower basis weight call for a means of maintaining or promoting bond strength of paper. Inexpensive chemical aids or chemical treatment of fibers reinforced by mechanical preparation are often introduced in order to meet the sheet specification. The current research of borax addition to kraft, bleached softwood is an investigation of improving the inter-fiber bonding with the intention to replace expensive or inefficient strength aids.

Paper strength is a complex function of several variables. The amount of fiber-to-fiber bonding is the most important factors of all the variables. Refining and chemical treatment of the fibers have a synergistic effect in the development of a high degree of inter-fiber bonding. Deficiency in bonding will lead to deficiency in the strength of paper. Sheet formation, wet pressing, and drying play a significant role in the sheet strength.

This research concentrates on the effect of borax as internal stock additive in papermaking and it tries to simulate industrial conditions by incorporating different types of fillers and alkaline sizing agents. It is also applied after wet pressing as a surface application agent.



## CHAPTER II

### LITERATURE REVIEW

#### Nature of Paper Strength

The fundamental factors that are related to paper strength were studied extensively by Swanson and Jones (1). According to their analysis, basic factors that contribute to paper strength are: fiber dimensions, fiber strength, bonded area, specific bond strength, distribution of bonds (sheet formation), fiber conformability (flexibility), and stress distribution.

Each of these factors is not an independent variable that can stand alone to govern the strength of paper. Most of the factors are influenced by other variables such as chemical and physical composition, mechanical preparation, degree of swelling, temperature, wet pressure, and presence of additives (adhesives, sizing agents, fillers, etc.) in the furnish.

In the papermaking process, the strength of the individual fibers cannot be improved because fiber strength is a function of wood species, and the pulping and bleaching process employed to obtain the fibers (2). Therefore, an already processed kraft bleached softwood have no chance of changing the intrinsic strength of fibers.

The analysis of these factors can be categorized into two major variables: the furnish variables and the process variables. The furnish variables consists of fibrous materials, filler loading, sizing agents etc. and the process variables comprise wet pressing, refining, surface treatment etc.

### Furnish Variables

Fiber dimensions have a significant effect on the strength of paper. It is known that longer fibers such as softwood produce greater strength compared to hardwood. Since softwoods have very thin cell walls, they show a marked effect on beating rate, swelling and fiber bonding, whereas hardwoods having shorter fibers, lead to papers with lower tear and tensile strength. Since hardwoods have thick cell walls with narrow lumen, they are stiff and collapse is not favored. Paper made from hardwoods is generally low in bonding (3).

Fiber bonding is a fundamental process that controls the mechanical properties of the paper. It is an essential parameter in the internal cohesion of paper. Nearly all-mechanical interaction between fibers, during papermaking, takes place through fiber bonding (4). In the mechanism of bonding of cellulose fibers, three conditions should be satisfied. They are: available area, proper surface contact, and hydrogen bonding (5). Bonded area is dependent on several factors. It is closely related to the surface area of the network which consists of the number of fibers in a given basis weight, and fiber dimension. External fibrillation and fines formation affect bonded area by expanding the total surface area and increasing Campbell's force. The extent,

intensity and nature of fiber bonds are determined by the degree of fiber collapse and fiber flexibility. The bonded area of fibers can be determined from relative bonded area (RBA) which decreases with an increasing cell-wall thickness. Direct estimation of bonded area in papers was made first by Parson (6) and Ratliff (7) using optical light scattering technique. The relative bonded area (RBA) is measured by a linear correlation between tensile strength and light scattering coefficient. The total surface area can be determined by extrapolating the tensile strength value to zero. The bonded surface area can be obtained by taking the difference between the total area at specific degree of beating and the unbonded area.

Fiber bonding is affected when adding non-fibrous material in the furnish. Hydrophobic additives such as rosin or paraffin show an adverse effect on bond strength. Hydrophilic materials such as clay, alumina and other fillers reduce the bonding ability of fibers. On the other hand, high molecular weight hydrophilic colloids such as starch and vegetable gums increase the strength of the sheet when they are used in the furnish. These materials act by being adsorbed on the fiber surface when they hold the sheet together through a fiber-to-binder-to-fiber bond (8). The compound that is going to be tried in this project is borax solution, which has the capacity to form complex molecule with cellulose and adsorb on the surface of fibers to enhance the bonding strength of papersheet. Borax has similar tendency of binding the fibers when compared to starch or vegetable gums. Borax is used in liner board with starch adhesive for the same purpose. Borax is considered as a stock additive because it is generally added prior to refining, though it can also be sprayed

after wet pressing as a surface application agent. Borax as a hydrophilic substance imparts special functional properties that cannot be achieved if the fibers are used alone.

Additives that increase the strength of paper might allow a higher percentage of filler without too much loss of strength. Some of the additives increase the retention of fillers in the process of papermaking. The adhesive quality of borax in the papermaking might be due to the anionic tetraborate molecule that would enter into some kind of interaction with the fibrous surface to form a fiber-borax-fiber bonding, which may be stronger than fiber-fiber bonding. If this is the case, the strength of the sheet will be definitely improved. The exact mechanism of the interaction of borax and cellulosic fiber is not understood at this point and it is an open question for further research.

### Chemistry of Borax

Structural formula of borax:  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$

Borax is essentially half titrated boric acid. It can generate tetraborate anion that can be used as a buffer. The tetraborate anion is a six membered ring with a triangular center of negatively charged boron as shown below in Figure 1. Most probably the

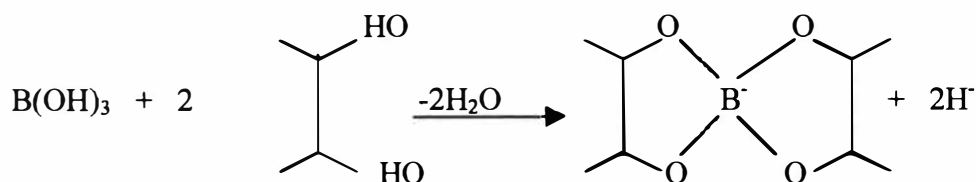


Figure 1. Borate Ester Cross-Linking.

borate anion attaches itself to opposite surfaces of cellulose through ester formation.

### Process Variables

Refining has been dealt within literature at great length; the discussion in this paper will be restricted to the relevant topic of fiber bonding and sheet strength. Paper formation, paper strength and other properties are strongly affected by fiber morphology, fiber surface and intra-wall characteristics. The properties that are strongly influenced by refining as described by Kibblewhite (10) are presented in Table 1.

It is impossible to obtain a strong sheet without the removal of primary wall. The secondary wall is the next layer to be exposed for further beating operation. The rubbing action of the refining bar causes bruises and the fibers develop fine fibrils that increase the exposed surface area of the fibers. The fibers become increasingly flexible and plastic. The surface tension increases due to the increased surface. The fibers are drawn together to form good adhesion at the time of sheet formation. During refining, the hydrogen bonds are broken and on drying, they subsequently reform between the fibers. This reformation provides the basic mechanism of paper strength (9). Refining affects paper properties through its effect on interfiber bonding. Fibers conform to one another at fiber crossings, leading to a denser, more highly bonded structure (2). Refining alone can improve properties to a limited extent and it has to be reinforced with the use of chemical additives.

Table 1

## Fiber Characteristics Modified or Developed by Beating Action

---

Dimensions	-	Length, diameter, wall thickness, Cross section, lumen shapes.
Stiffness	-	Flexibility, conformability
Surface Properties		
Structural	-	Wall layers, fibrillation, specific surface, specific volume, amorphous substances fibular substances, pits and other Discontinuities
Chemical	-	Composition, colloidal and electrical Phenomenon
Wall Structure	-	Wall layers, wall lamellae, discontinuities, delamination, fibrillation, microfibril angle.

---

The most important response to refining is fiber conformability and flexibility, which have a direct influence on fiber bonding. Beating has a minor influence on fiber collapsibility, but it increases wet fiber flexibility especially of thin-walled fibers. Refining thick-walled fibers results in extensive fibrillation and formation of more fines that are responsible in increasing the surface tension and bonded area. Wet fiber flexibility enhances bonded area and large bonded area is more important for network strength than bonding strength. Bonded area is a product of fiber conformability, total surface area, and the factors affecting surface tension forces (11). Flexible fibers respond better to surface tension forces and promote fiber-to-fiber bonds. Thick-walled fibers form an open network and fewer areas of fiber are in

contact with one another. An increase in basis weight might improve the bonded area provided fibers are flexible enough to form increased fiber-to-fiber contact. During papermaking most fibers collapse but this does not mean that a better fiber network is formed. Although fiber collapse increases surface area, flexibility plays a major role in determining the bonding ability of fiber-to-fiber contact. Fiber conformability characterizes the ability of wet fibers to plastic and elastic deformation. Fiber flexibility and collapsibility are dependent on cell wall thickness, fiber width, and the elasticity of the cell wall (12). In fact, fiber flexibility is an inverse function of cell wall thickness. Fiber collapsibility and flexibility are two of the most important factors for paper properties such as tensile strength, apparent density, light scattering coefficient, surface smoothness and sheet porosity. They also affect the flocculation tendency of fibers (formation) and network strength (13,14,15). Delamination and swelling have an adverse effect in cell wall elasticity and curtail the improvement of fiber flexibility and greater fiber collapse. Most softwood fibers collapse to some extent during consolidation and drying regardless of beating. During drying, fibers collapse and irreversible hydrogen bonds are formed. Fibers shrink and become rigid. The flexibility of never-dried pulp is half that of once-dried pulp, whereas wet fiber flexibility can be increased by a factor of three on beating (12). Lignin removal, fiber length and degree of beating improve flexibility. Since borax is added prior to beating, it gets sufficient contact time to adsorb on the fiber surface. It further exploits the flexibility of those fibers that can be bound through fiber-borax-fiber interaction.

Paavilainen (12) showed that the tensile strength of unbeaten softwood increases linearly as fiber flexibility increases. Moreover, It has been demonstrated beating improves the bonding ability of fibers, when the tensile strength of fibers of different cell wall thickness is compared with flexibility level. Generally, tensile strength is affected by bonded area, bonding strength, fiber length and intrinsic fiber strength.



## CHAPTER III

### FUNDAMENTAL ASPECTS OF SIZING

Alkyl Ketene Dimer (AKD) is an internal sizing agent that can be used at 0.05 – 0.2 % of addition levels on dry weight pulp which gives sufficient sizing to the sheets. Alkaline papermaking provides the benefit of low furnish costs and improved paper permanence. AKD has to be prepared in the form of emulsion prior to its addition into the papermaking system. Cationic starch or cationic polymer is often added to stabilize the AKD emulsion. AKD develops sizing by a direct reaction with the fiber in the neutral or near neutral pH zone.

#### Chemical Structure

Alkyl ketene Dimer (AKD) is the most commonly used of the synthetic sizes. It has the general chemical structure shown in Figure 2. The four membered rings

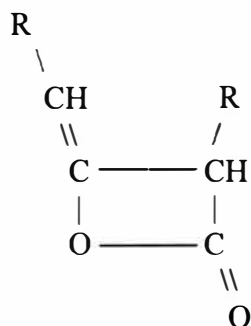


Figure 2. Alkyl Ketene Dimer (AKD).

provide a functional group that can react with cellulose hydroxyl groups. Synthetic sizing agents have a molecule having both hydrophilic and hydrophobic chemical groups.

### Hydrophobation With AKD

AKD can react with wood fibers during the drying of the paper with the formation of  $\beta$ -Ketoesters (13). Ring opening takes place at the acyl oxygen. The fiber surface gains hydrophobic property from the lengthy hydrocarbon chains. The reaction of AKD with cellulose is shown in Figure 3.

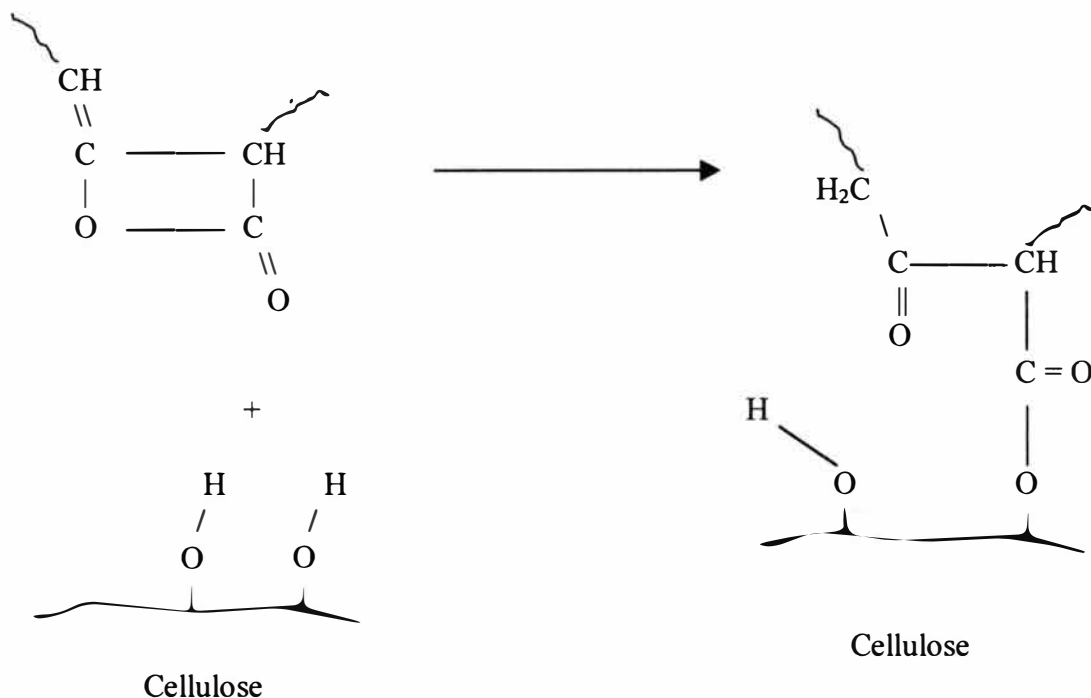


Figure 3. Reaction Between AKD and Cellulose.

### Reaction of AKD With Water

AKD can react with the hydroxyl group in water, which is the only competing reaction with AKD. The hydrolysis product is non-sizing and is referred to as hydrolyzate (14).

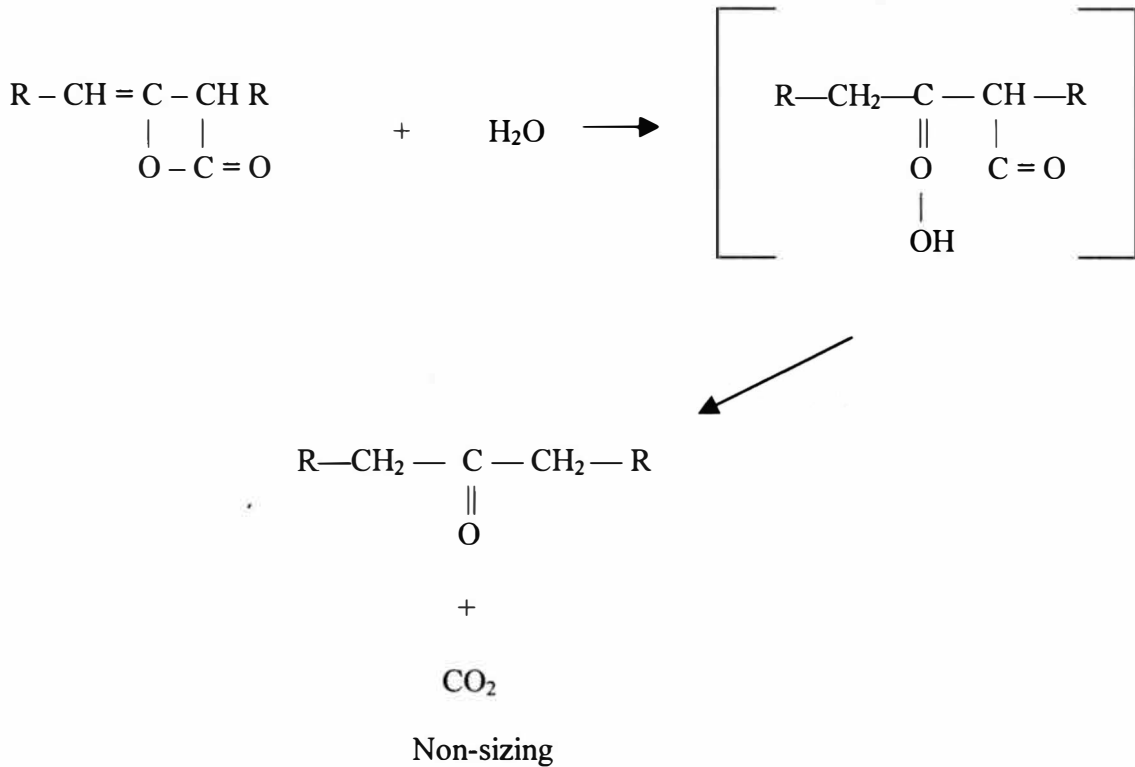


Figure 4. Hydrolysis of AKD.

### Macro View of AKD Sizing Mechanism

AKD sizing is often applied internally to the pulp slurry close to the paper machine to reduce hold time. AKD particles will adsorb onto the pulp and filler

surfaces, by electrostatic means. Since AKD has low surface energy its initial deposition will have few contact points between the cellulose and AKD molecules shown in Figure 5. In this stage no appreciable reaction can take place due to the surface tension of the surrounding water.

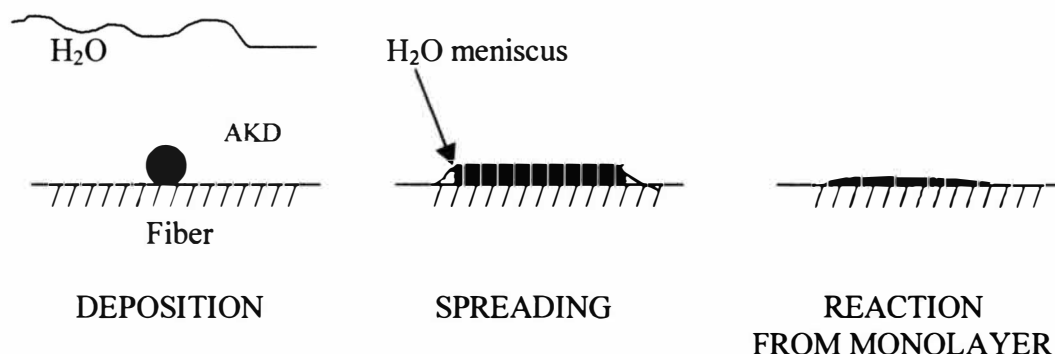


Figure 5. Steps in Sizing With AKD.

Extensive molecular dispersion takes place at elevated temperature during the drying of paper. An increase in temperature favors the formation of air-AKD surface, which is crucial condition for spreading. The continual spreading on the cellulose-air surface promotes a monomolecular layer formation. This layer reacts with the hydroxyl groups of the cellulose (13). In order for AKD to impart on alkaline papers, it has to be retained, distributed, and anchored on the fibers with proper molecular orientation.

### Effect of Filler on Alkaline Papermaking

The driving force for the conversion of acid papermaking to alkaline papermaking is the use of low-cost fillers, which can replace the fibers to certain

extent. An appropriate selection of filler particle size and morphology can allow high filler level to maintain strength, stiffness and sizing (17).

Normally fillers are added in the furnish to impact the required final properties in the paper sheet, such as brightness, opacity, smoothness, and printability. There is some penalty associated with the use of filler. The paper filled with filler has a low degree of bonding but higher density; it also has a wearing effect on the paper machine wire.

The interaction of fiber and filler is due to electrostatic and Van der Waals forces. Filler will be adsorbed on the fiber surfaces whenever opposite electrical charges exist. The electrostatic charge on the surface of filler particle plays an important role in the stability and retention of filler on the paper web.

#### Effect of Filler on Strength Properties

Filler has a negative effect on interfiber bonding. Particle size and particle shape determines the extent of this adverse effect on paper strength. In general, if the pigment is small, it affects fiber bonding severely. The decrease in mechanical properties is due to the intrafiber blocking of filler, which interferes with the fiber-to-fiber bonds. A 10% increase in the filler content reduces the strength by 20% or more (13).

#### Effect of AKD on Paper Strength

Papers made in acidic conditions run into a problem of aging at the

accelerated rate, lowering of fiber strength, and paper strength such as fold, tear, and tensile. Acid system encourages cellulose hydrolysis, which decreases the chain length of the cellulose molecule. Degradation of cellulose is high if the acidity level is high. The chain shortening process has a significant effect in weakening the fiber strength, which reduces the paper strength (15).

Alkaline papermaking using AKD sizing eliminates the papermaking process in acidic condition and use of alum. This allows AKD sizing to operate at a neutral or slightly alkaline pH range. Since AKD is less reactive compared to other synthetic sizes, it can tolerate a much higher pH before hydrolysis takes place. The ideal pH is between 7 and 8.5 and often  $\text{CaCO}_3$  is used to buffer in the 7.5 to 8.0 pH range (16).

A reactive group of AKD forms a covalent bond between the size and cellulose.

Under alkaline sizing, there is no loss in the strength of paper.

In the project, borax is included in the furnish with AKD and/or filler. It is safe to believe that borax competes with filler to adsorb on the surface of the fibers. Hence the bonding potential of fibers is somehow protected by the presence of borax and it would be less towards the sizing agents. The overall mechanism is not known at present but close investigation will bring some light to the problem.

### Size Reversion

Size reversion (fugitive) is the loss of water repellent ability of the sizing agent. The main cause for lack of sizing is attributed to anionic trash, wet end additives, AKD and filler interaction, and AKD hydrolysis products. It is generally

accepted that spreading of AKD wax can take place at higher temperature. At drying temperature, the fluidity of AKD wax increases. It will have the tendency to flow into the void structure of a porous pigment, and become unavailable for sizing. At high moisture content the 'non-reacted' AKD can be hydrolyzed at pH 7.5 and above. At this point, the AKD becomes non-sizing and a fugitive effect occurs (16). Borax being alkaline, may contribute to this size reversion. It remains to be seen whether this effect is significant.

## CHAPTER IV

### STATEMENT OF THE PROBLEM AND OBJECTIVES

Strength additives can bring an improvement in the strength of inter-fiber bonding and bonded area, which supplement the hydrogen bonding. Process variables, such as refining and the type of furnish, have an effect on the overall increase in strength. Papermakers use several approaches to increase sheet strength. They might modify the furnish by the inclusion of long fibers, reduction of filler or addition of strength additives. It may also be achieved by process manipulation, for example refining level or increased wet pressing (17).

In this study, borax is used as an internal stock additive in papermaking and is reinforced by the refining process. Borax treatment can enhance the bonding of the fiber network. The increase in the cohesiveness between the fibers was inferred from the preliminary trial conducted on a bleached kraft softwood pulp. Four objectives are set to further investigate the effect of borax on the paper properties. They are: (1) to confirm the effect of borax as an internal additive in increasing the strength of paper properties, (2) to study the effect of borax in the presence of filler, (3) to study the effect of borax on alkaline sizing, and (4) to study the effect of borax in surface applications of paper.



## CHAPTER V

### EXPERIMENTAL DESIGN AND PROCEDURE

The experimental design of this project was to evaluate the effect of borax on the strength properties of paper. The first phase was an attempt to determine the optimal level of borax. The second phase utilized two types of fillers i.e. PCC and GCC. The pulp was treated with borax solution prior to the addition of filler. A deliberate avoidance of retention aids caused a major problem of low and inconsistent retention of the filler. The slurry was recycled to increase the retention of the fillers to the required level. In the third phase, borax was used in the presence of alkaline sizing (AKD) agent and / or filler (PCC or GCC). This phase utilized both the concentrated and diluted AKD. Samples were stored for 2, 4 and 6 days before sizing tests were carried out. In the fourth phase, borax was sprayed on the surface of the paper after wet pressing.

The schematic of various phases is presented in Figures 6 to 10. The different factors involved in the study are summarized in Table 2. The summary includes the treatment combinations of each experiment in every phase. Standard handsheets with a nominal basis weight of 60 g/m<sup>2</sup> were prepared according to TAPPI standard. Appropriate TAPPI standard methods (Table 3) were implemented in the experimental procedures. Modified procedures, if any, are included in the appendix

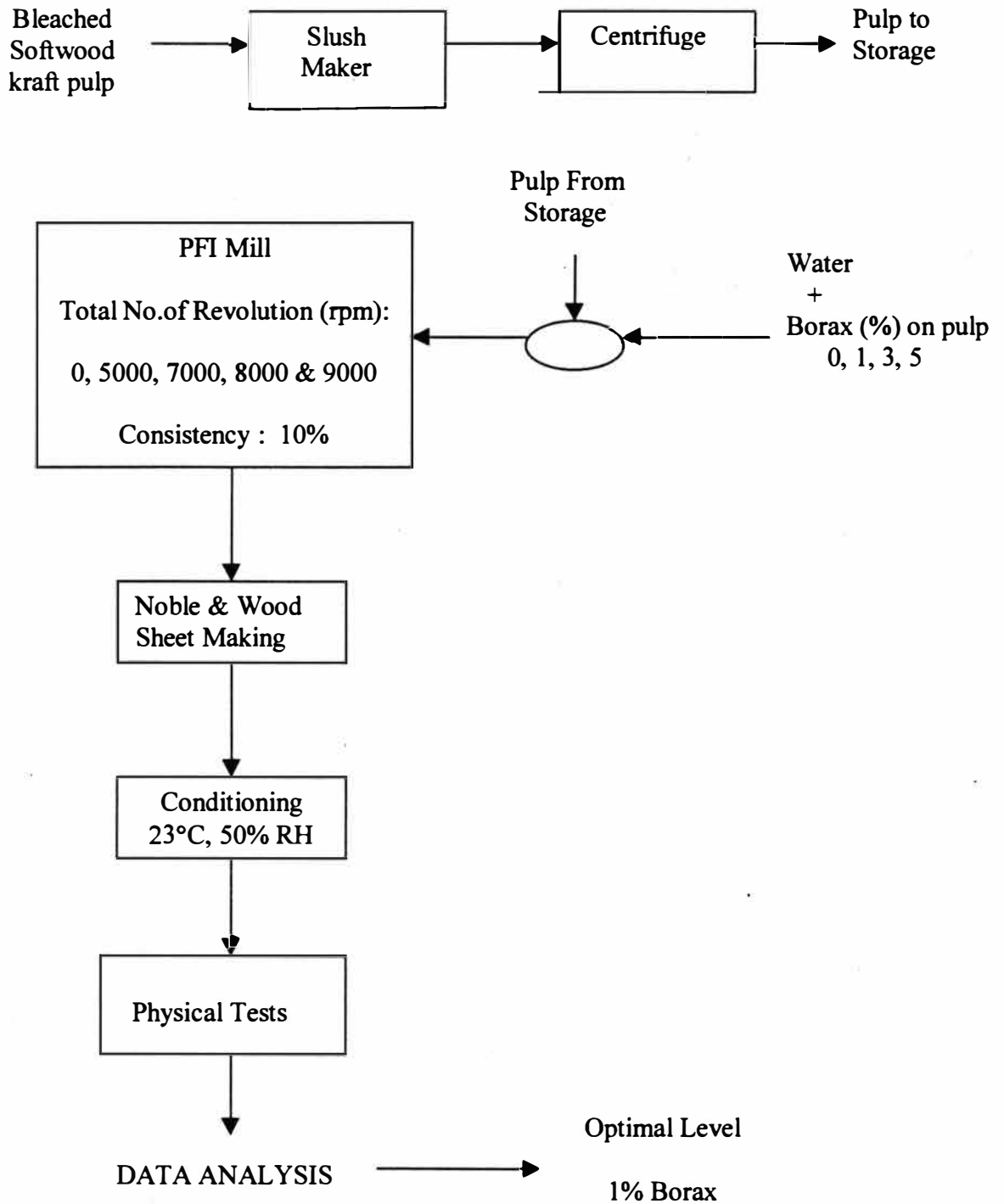


Figure 6. Operation to Optimize Borax Addition (Phase 1.1).

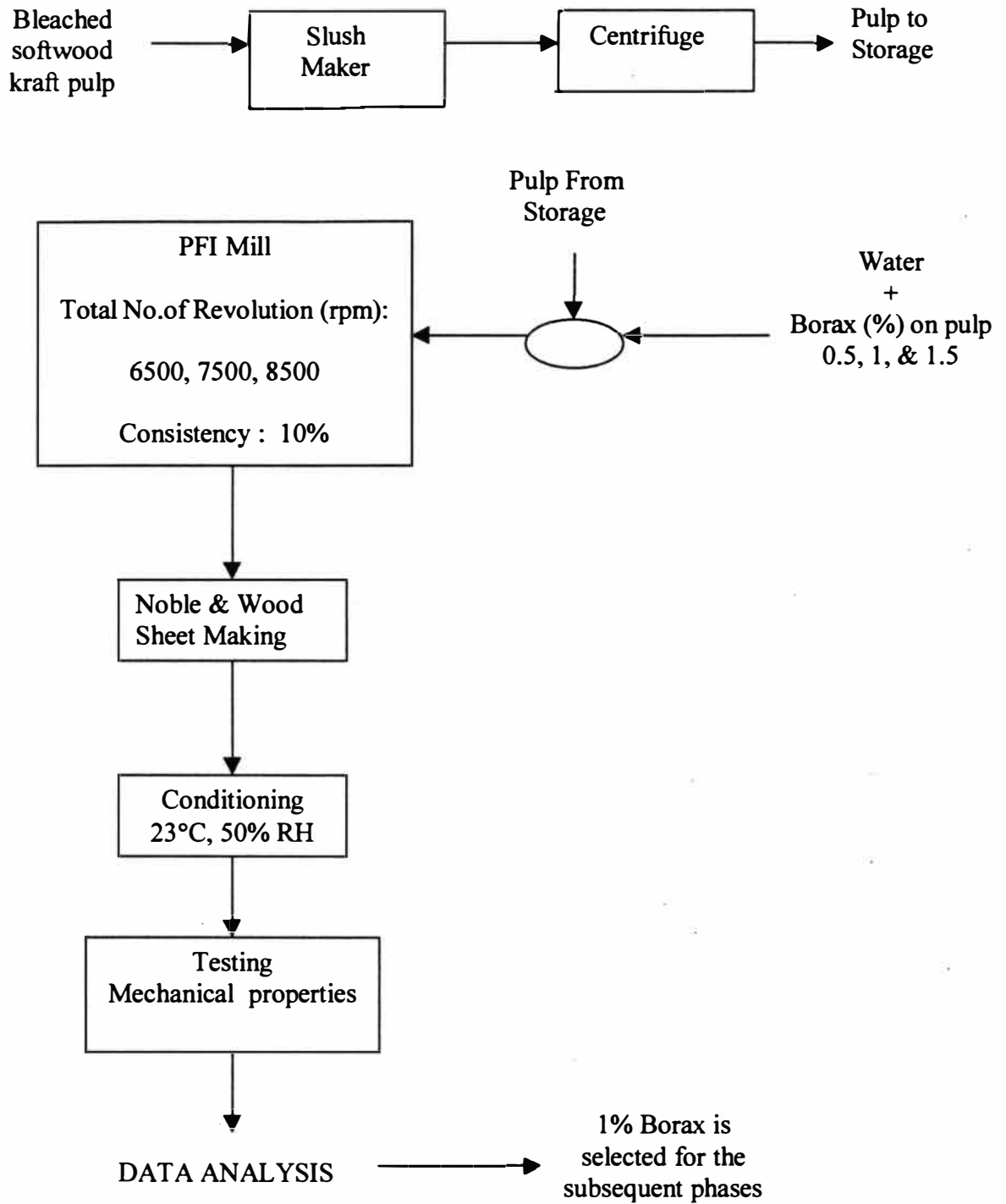


Figure 7. Operation at Optimal Level of Borax Addition (Phase 1.2).

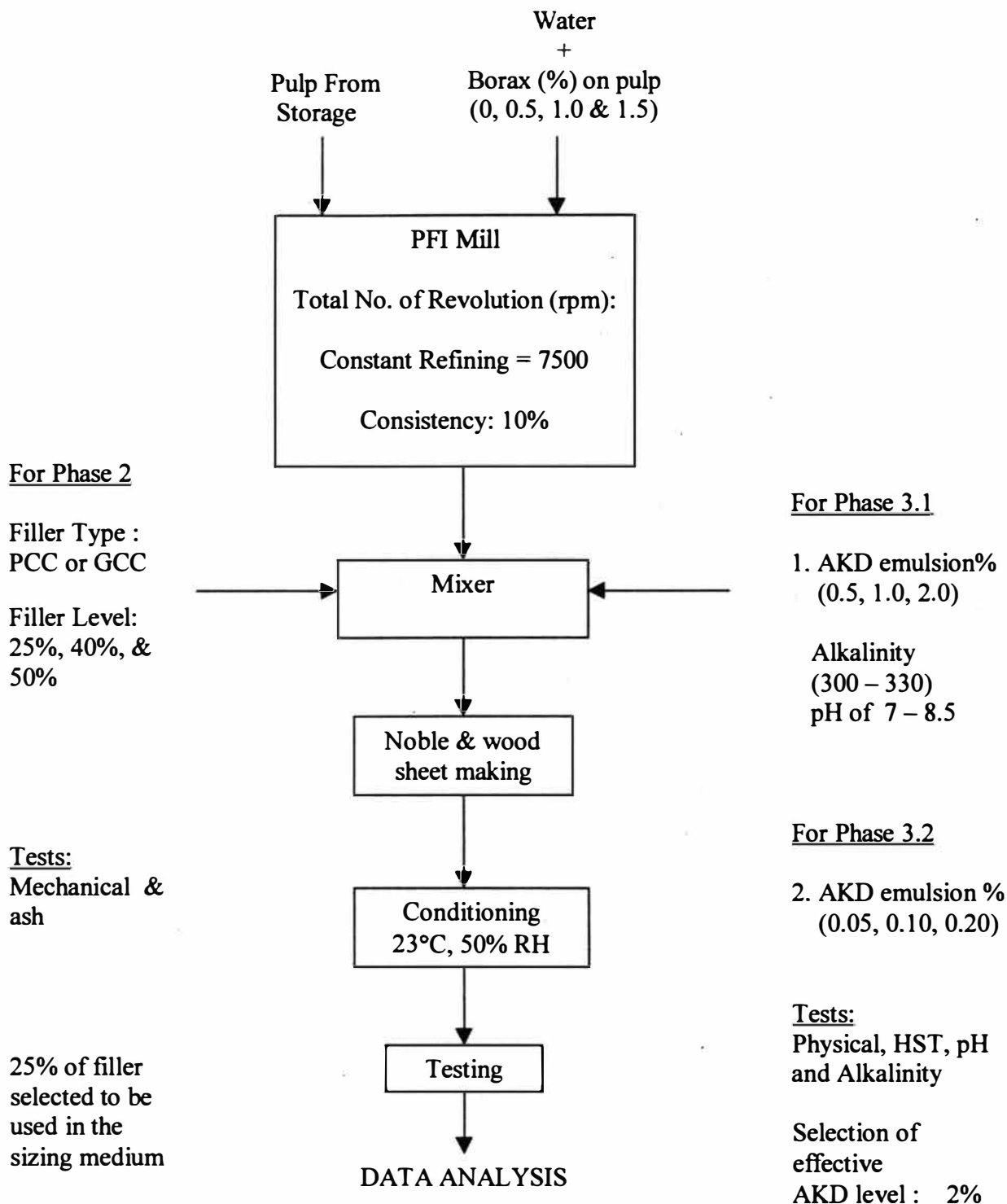


Figure 8. Borax Addition in the Presence of Filler or Alkaline Sizing Agent (Phase 2 & 3.1).

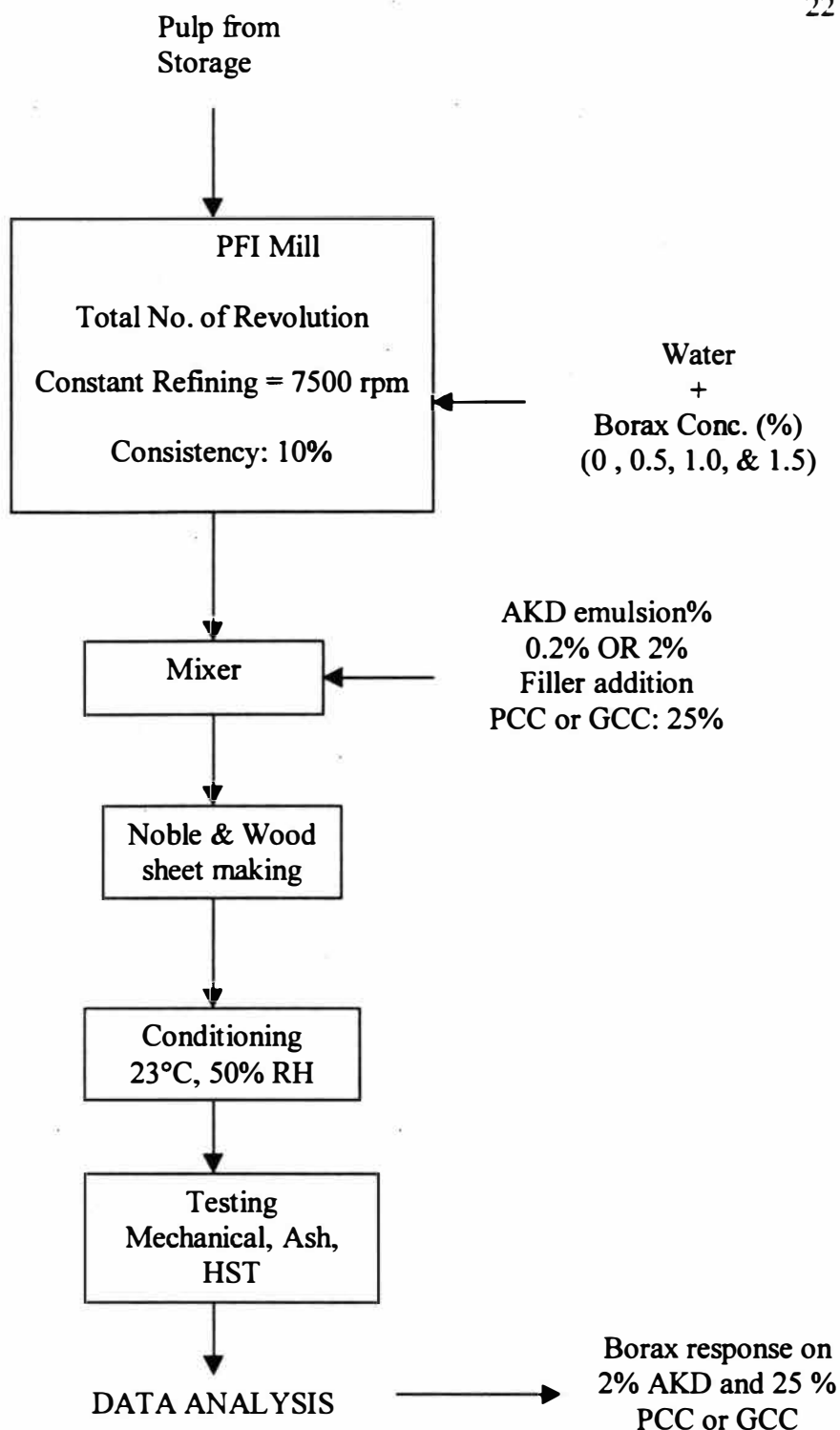


Figure 9. Effect of Borax in Alkaline Sizing With Filler Inclusion (Phase 3.2).

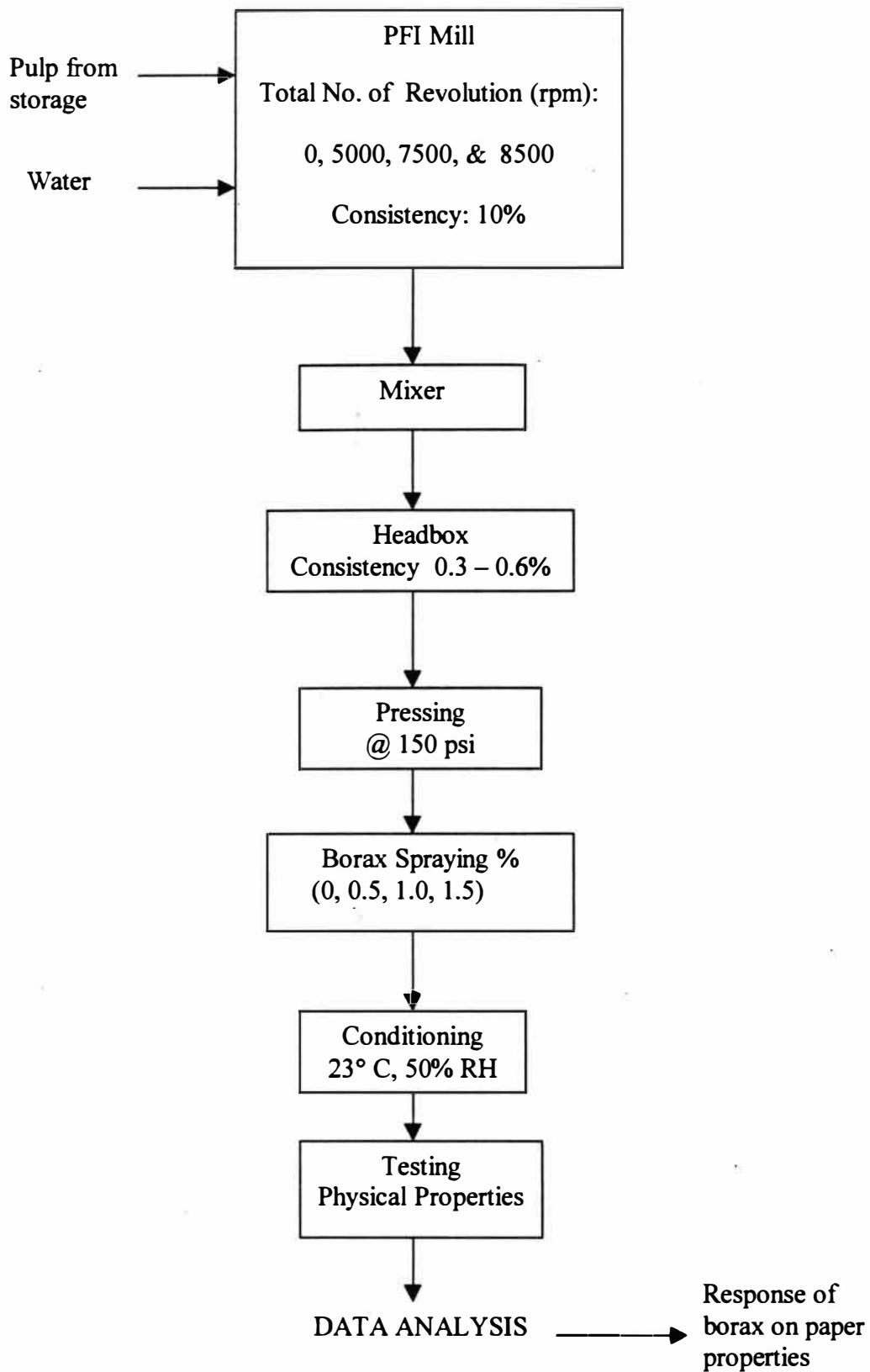


Figure 10. Borax as a Surface Additive and Its Effect on Paper Properties (Phase 4).

Table 2

## Summary of the Experimental Levels for the Design Variables

Phase No.	Description	Variables		
		Borax %	Filler or Sizing Levels %	Refining Level, PFI mill Revolutions
Phase 1	1.1 Preliminary Trial	0, 1, 3 & 5		0, 5000, 7000, 8000, & 9000
	1.2 Optimal level	0, 0.5, 1 & 1.5		0, 6500, 7500, & 8500
Phase 2	Filler loading	0, 0.5, 1 & 1.5	25, 40, 50 (PCC)	7500
		0, 0.5, 1 & 1.5	25, 40, 50 (GCC)	7500
Phase 3	3.0 Alkaline Sizing and Borax	0, 0.5, 1 & 1.5	0, 0.5, 1, 2 (AKD)	7500

Table 2- Continued

		Variables		
Phase No.	Description	Borax %	Filler or Sizing Levels %	Refining Level, PFI mill Revolutions
Phase 3				
	3.1 Borax, Alkaline sizing and Filler	0, 0.5, 1 & 1.5	0.2 or 2 (AKD) 25 (PCC)	7500
		0, 0.5, 1 & 1.5	0.2 or 2 (AKD) 25 (GCC)	7500
	3.2 Size Reversion	0, 0.5, 1, 1.5	0.05, 0.1, 0.2 (AKD)	7500
Phase 4				
	Borax as a surface additive	0, 0.5, 1 & 1.5		0, 5000, 7500, & 8500



Table 3

## TAPPI Standard Testing Methods

Method	Description
T 205 sp-95	Forming handsheets for physical tests of pulp
T 220 sp-96	Physical testing of pulp handsheets
T 227 om-94	Freeness of pulp (Canadian standard method)
T 248 cm-85	Laboratory beating of pulp (PFI mill method)
T 402 om-93	Standard conditioning and testing atmospheres for paper, board, pulp handsheets, and related products
T 494 om-88	Tensile breaking properties of paper and paperboard
T 530 om-94	Bending resistance of paper (Gurley type tester)
T 530 pm-89	Size test for paper by ink resistance (Hercules method)
T 541 om-89	Internal bond strength of paperboard (Z-directional tensile)
T 620 cm-83	Analysis of industrial process water (Alkalinity test)

section. Separate handsheets of high basis weight ( $200 \text{ g/m}^2$ ) were made for testing the internal bond strength.

### Phase 1: Optimizing of Borax Level

Bleached softwood kraft pulp was slushed and then refined in a PFI mill at different revolutions. Borax was charged at concentration levels of 0%, 1%, 3% and 5% prior to refining. The fiber was mixed with borax solution to a consistency of 10% and residence time of one hour. Each batch was refined at 0, 5000, 7000, 8000, and 9000 rpm and the Canadian Standard Freeness (CSF) were determined. For each level of refining five handsheets was produced after proper disintegration and dispersion were made using a Noble and Wood sheet making machine. Each formed web was pressed between felts and dried on a rotating steam (20 psi and 250 °F) heated dryer. The dried handsheets were stored in a conditioned room for 1 day at 23 °C and 50% RH. Physical properties were tested according to the TAPPI standards. After analyzing the results, the best level was selected for the next run. 1% borax level at 7500 revolution was selected from this phase (phase 1.1) as the optimal level (Figure 6).

Following the optimal level determination, the investigation continued in phase 1.2 as a further confirmation of the 1% borax level (Figure 7). Taking 1% borax as a base line, 0.5% borax above and below the base line were tried to observe the effect of borax on strength properties. The same procedures as in phase 1.1 were

implemented in phase 1.2 except for a change in the refining treatment (6500, 7500, and 8500 revolutions).

### Phase 2: Effect of Borax in Presence of Filler

Pulp mixed with the optimal borax was treated at a constant refining level, which is 7500 revolutions, in a PFI mill. PCC or GCC was added to the slurry in the disintegrator at a level of 25%, 40%, and 50% respectively (Figure 8). The slurry was transferred to a mixing tank for complete suspension of the fibers. Handsheets were made on the Noble and Wood machine. Initial handsheets tested at a very low ash level of 5%, were at the highest filler loading. Therefore, a shift towards recycling of the white water was introduced as the alternate method. The smallest negative impact of the filler content on the fiber bonding was observed at the 25% filler addition. This percentage of the filler content was used together with the sizing agent (AKD) in the following phase (Phase 3).

### Phase 3: Effect of Borax on Alkaline Sizing

Investigation in this phase consisted of two cases. The common procedures for the two cases are described below (Figures 8 and 9).

In phase 3.1, AKD emulsion with 15% solid was used without dilution. The three levels of AKD were 0.5%, 1%, and 2%. In phase 3.2, AKD emulsion was diluted ten times and applied to the furnish at a level of 0.05%, 0.10%, and 0.20%. In both cases, the mixing container was selected as the addition point for the AKD. The

operation was carried at a pH of 7.5 to 8.5 for efficient sizing and the pH level was monitored at regular intervals. Since the alkalinity level remained between 300 – 330 ppm, there was no need for the addition of  $\text{NaHCO}_3$  or  $\text{NaOH}$  to adjust the alkalinity level. Handsheets were made and stored (to equilibrate) in a conditioning room for 2, 4 and 6 days before testing. HST sizing was measured on each sample of paper using Hercules Test Ink # 2 at 80% reflectance.

1. Pulp with optimal borax addition, from phase 1, was treated at a constant refining level in a PFI mill (7500 revolutions) and AKD was added as shown in Figures 8 and 9.

2. The best level of filler from phase 2 (25% PCC or GCC) was mixed with the pulp treated with borax containing 2% AKD. The process schematic can be referred to in Figure 9.

#### Phase 4: Effect of Borax in Surface Applications of Paper

In this phase, pulp was refined without borax. Handsheets were made and the wet web was pressed in between felts. Four levels of borax additions (0, 0.5, 1, & 1.5%) were made by spraying borax solutions from a fixed distance on the pressed sheets (Figure 10). Each sample was then conditioned and tested for physical properties.

## CHAPTER VI

### RESULTS AND DISCUSSION

The data and analysis of each phase are presented in the respective sections. The best lines were fitted through the scatter data points using linear regression method. The  $R^2$  values estimate the percentage variations due to the change in control variables. The relationship between the factor level and the response variable are measured by the coefficient of determination or  $R^2$ . The two-way ANOVA procedures were used in performing the data analysis (23). Whenever there were missing data, the GLM (general linear model) has been used. Due to lack of replicates the interactive effect could not be obtained, as there was no degree of freedom left for the analysis. The confidence intervals shown in the appendix are all based on the pooled standard deviation of the response variables. These values can be obtained by calculating the square root of the MSE (means of square error) in the ANOVA table.

#### Phase 1: Borax and Fiber System

The strength properties of phase 1.1 and phase 1.2 are discussed one after the other. The graphs of phase 1.1 and phase 1.2 are presented in the succeeding pages following the interpretation of each properties. The figures presented in both phases address the multiple effect of borax addition and refining on the properties of paper.

Strength properties for borax and fiber system are plotted as a function of PFI mill revolutions, for each borax addition level. In every phase, the tensile energy absorption plot is presented to represent both the tensile index and the stretch properties.

In the first phase, the fibers treated with borax solution showed a slight increase in strength properties compared to that of the untreated fibers. The main cause for the property increase could be due to the formation of strong fiber bonding. The increase in the fiber bonding is attributed to the combined effect of refining and borax. Factors such as good formation, wet pressing, density and basis weight can also contribute to the strength properties in a positive way (24). These factors were maintained the same both in the control and in the trial experiments.

#### Phase 1.1: Tensile Energy Absorption

Tensile Energy Absorption measurement combines both the tensile strength and stretch of paper to rupture. This makes TEA an important strength property of paper. An increase in tensile strength automatically increases the tensile energy absorption. Increasing the elongation of a paper would also contribute to an increase in the value of TEA. Figure 11 shows the sample distribution of the TEA obtained by the controlled addition of borax and refining operation. The figure clearly indicates that the average TEA increases in a curvilinear manner. The TEA of the control (0% borax) falls below those of the 1%, 3%, and 5% borax addition levels. The values 3% and 5% of borax increase steadily until they level off. The 1%

borax addition results in an increase in the strength property rapidly to a maximum value at 6000 revolutions. The control reaches its maximum value at 7000

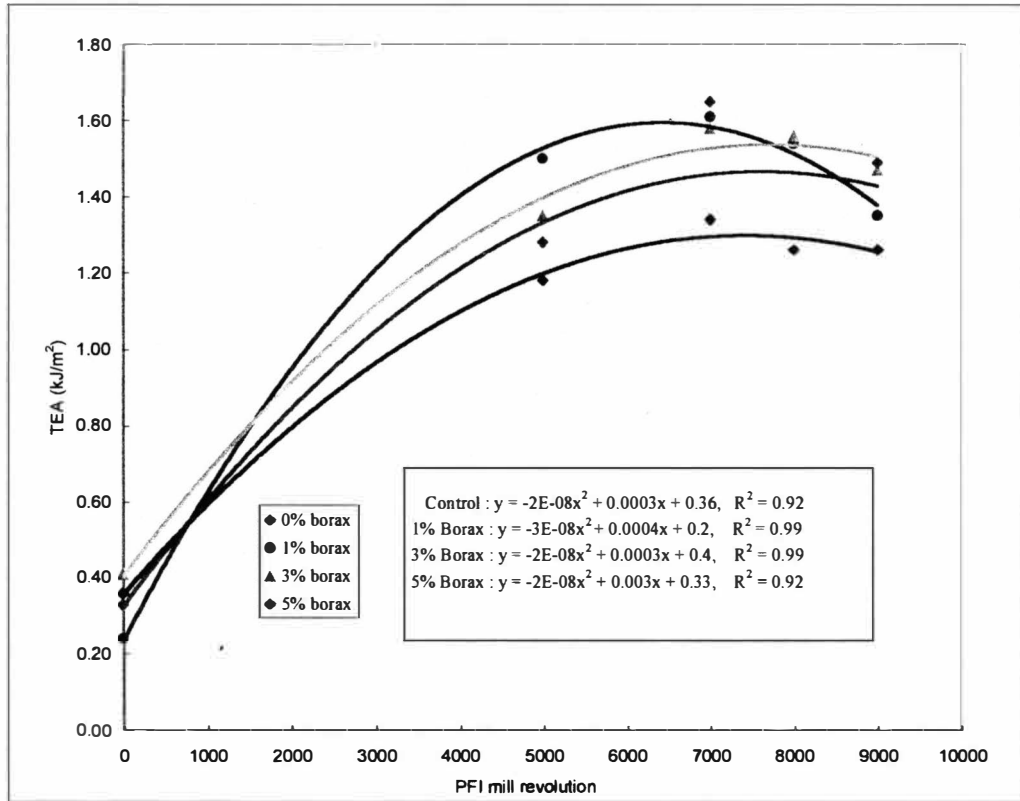


Figure 11. Effect of Borax Addition on Tensile Energy Absorption at Different PFI Mill Revolutions (Phase 1.1).

revolutions and the maximum value thus obtained was much less than those obtained with borax additions. The values of TEA at five refining levels and four levels of borax addition are presented in Table 4. The table indicates that strength development of the fiber is contributed to by a combination of refining and borax addition. The increase in strength property due to borax can easily be observed by choosing a constant refining level and comparing the TEA values obtained at various

borax addition levels. Even though refining normally favors the increase in the

Table 4

Phase 1.1: Effect of Borax and Refining on TEA (kJ/m<sup>2</sup>)

Revolution	Borax Concentration			
	0 %	1 %	3 %	5 %
0	0.36	0.24	0.41	0.33
5000	1.18	1.50	1.35	1.28
7000	1.34	1.61	1.58	1.65
8000	1.26	1.54	1.56	1.26
9000	1.26	1.35	1.47	1.49
Pooled Standard Deviation				0.12

degree of the interfiber bonding, borax clearly introduces a tangible difference in achieving a similar effect with less refining energy. The pooled standard deviation value also indicates that the observed increases in TEA values are statistically significant at 95% level.

Figure 11 and Table 4 suggests that small amount of borax addition can reduce the amount of energy required to produce the desired property. The two-way ANOVA (Appendix A) reveals that the effect due to borax level is close to be significant while the refining level is significant in contributing to the increase in the TEA values.



### Phase 1.2: Tensile Energy Absorption

From the results of experiments in phase 1.1, it was decided to continue the experiments around 1% borax addition and a refining level of 7500 revolutions. In this phase, there were three refining levels (6500, 7500 and 8500 revolutions) and three borax addition levels (0.5, 1.0 and 1.5%). These levels and the results obtained are shown in Table 5. The tensile energy absorption as a function of refining levels is plotted in Figure 12. This phase was designed to follow up on the results obtained in phase 1.1 so that a specific level of borax and refining level could be chosen for further experiment. As can be observed from the table, there is not much difference

Table 5

Phase 1.2: Effect of Borax and Refining on TEA (kJ/m<sup>2</sup>)

Revolution	Borax Concentration		
	0.5 %	1.0 %	1.5 %
6500	1.46	1.41	1.48
7500	1.33	1.23	1.80
8500	1.46	1.35	2.03
Pooled Standard Deviation			0.18

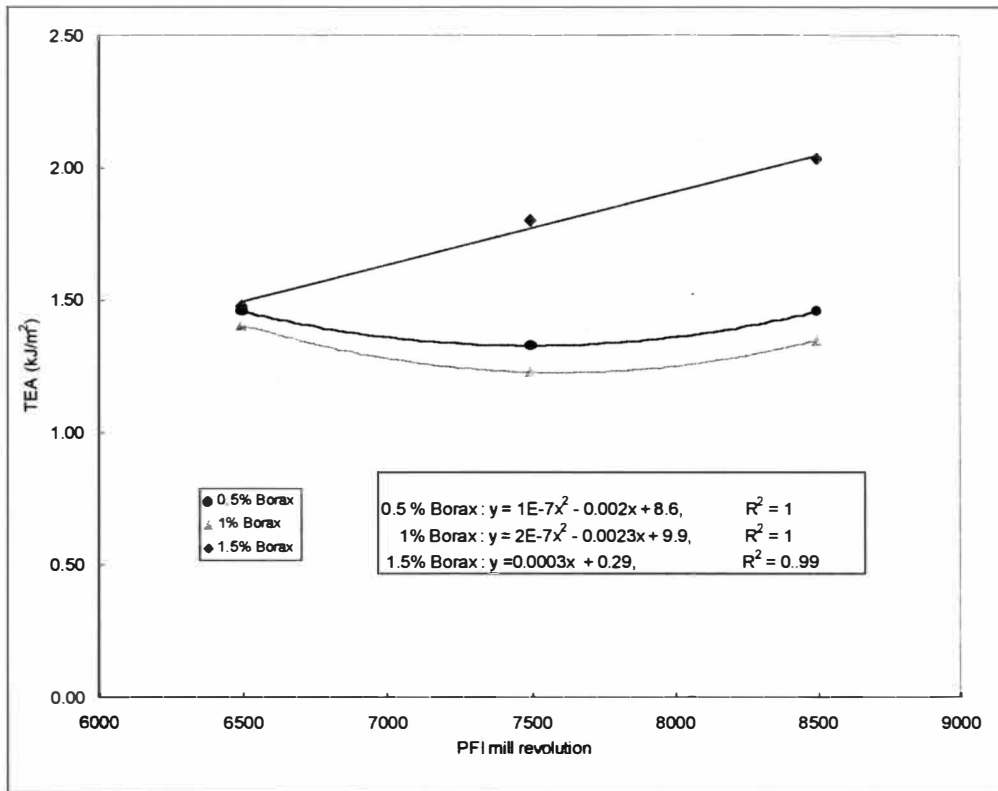


Figure 12. Effect of Borax Addition on Tensile Energy Absorption at Different PFI Mill (Phase 1.2).

between the three levels of borax addition. Also a refining level of around 7500 revolutions, seems to be adequate to develop the strength property; the obtained in this set of trials values are low when compared to phase 1.1 at 1% borax and 7000 revolutions. The 1.5% borax addition produced unexpected increase beyond what is observed in the previous phase. According to the two-way ANOVA, the pooled standard deviation was 0.18 and the F- test revealed that the results due to borax and refining were not significant. Hence a borax addition level of 1% and a refining level of 7500 revolutions can be chosen for further experiments.

### Phase 1.1: Tear Index

Internal tearing resistance was measured to determine the amount of work done in propagating the tear through a fixed distance. Tearing resistance is a significant property for the strength measurement. In this project the tear index showed a decreasing trend with refining for all treatment levels. The control values of the tear index were lower than those fibers treated with borax. There is an increase in the tear index at zero refining level for various addition of borax. The addition of borax must have allowed the fibers to develop a strong bond so that the fibers cannot be pulled out easily. Figure 13 shows the overall trend of the tear index as a function

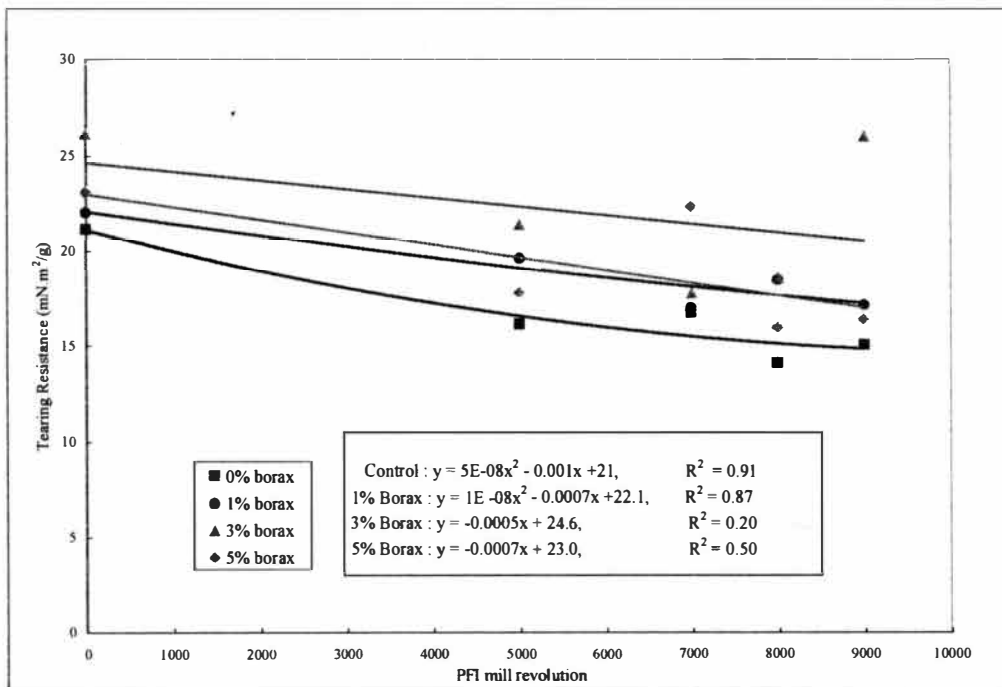


Figure 13. Effect of Borax Addition on Tear Index at Different PFI Mill Revolutions (Phase 1.1).

of the refining energy. At every refining level each value of the tear index was better than that obtained in control experiments (0% borax). Table 6 suggests that the values of the tear index at any refining level increased with borax addition. The

Table 6

Phase 1.1: Effect of Borax and Refining on Tear Index (mNm<sup>2</sup>/g)

Revolution	Borax Concentration			
	0 %	1 %	3 %	5 %
0	21.14	22.02	26.10	23.09
5000	16.13	19.62	21.40	17.83
7000	16.71	16.98	17.80	22.35
8000	14.09	18.50	18.60	15.97
9000	15.05	17.16	26.00	16.42
Pooled Standard Deviation				2.35

improvement in the tear index was due to the development of fiber-to-fiber bond formation. The strong bonds between the fibers were capable of absorbing the stress applied in the testing procedure. According to the P-values both factors refining level and borax addition were significant in affecting the tear index values (Appendix A).

## Phase 1.2: Tear Index

Narrow ranges of refining and borax addition were applied in this phase. A closer look of the behavior of borax showed that the addition of 1% borax progressively increased the tear index at each level of refining while the 0.5% and 1.5% borax led to constant tear index values. The tear index for 1% borax addition remained within the range of 17 – 24 mNm<sup>2</sup>/g and this value was consistent in both phases. In phase 1.1, the highest values of the tear index were obtained with the 3% borax and it had values of 18 – 26 mNm<sup>2</sup>/g. Figure 14 shows the tear index as a function of the refining level. In this figure, the linear fit showed that for 1% borax

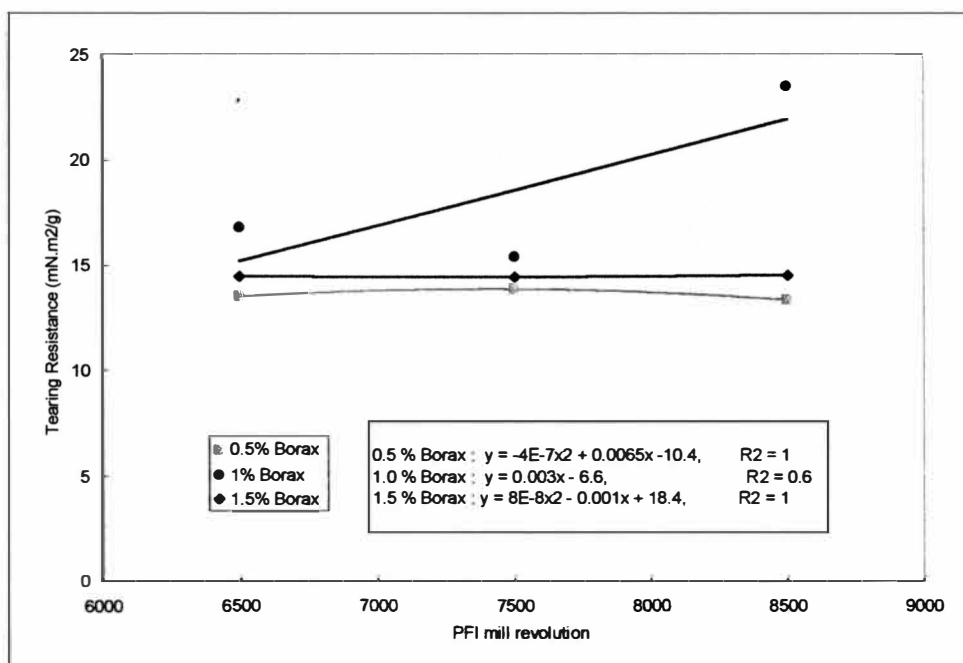


Figure 14. Effect of Borax Addition on Tear Index at Different PFI Mill Revolutions (phase 1.2).

addition, tear index had a positive correlation with the refining level also. The observed increase in the tear index values, for both phases, was due to the combined effect of the refining and the borax addition (Table 7). This means that the undamaged fibers formed a strong bond to adequately resist the applied stress. Thus, the tear index of phase 1.2 showed some improvement but not to a significant level.

Table 7

Phase 1.2: Effect of Borax and Refining on Tear Index ( $\text{mNm}^2/\text{g}$ )

Revolution	Borax Concentration		
	0.5 %	1.0 %	1.5 %
6500	13.48	16.80	14.48
7500	13.85	15.40	14.44
8500	13.34	23.50	14.55
Pooled Standard Deviation			2.50

#### Phase 1.1: Burst Index

Burst Index is a measure of the resistance to rupture when an increasing pressure is applied to one side of the test sample. In burst index the individual fiber length and the inter-fiber bonding play a major role. Normally, burst strength would increase with refining and level off after reaching its maximum value. Burst can be affected by other properties such as stretch and tensile strength. From Figure 15, it

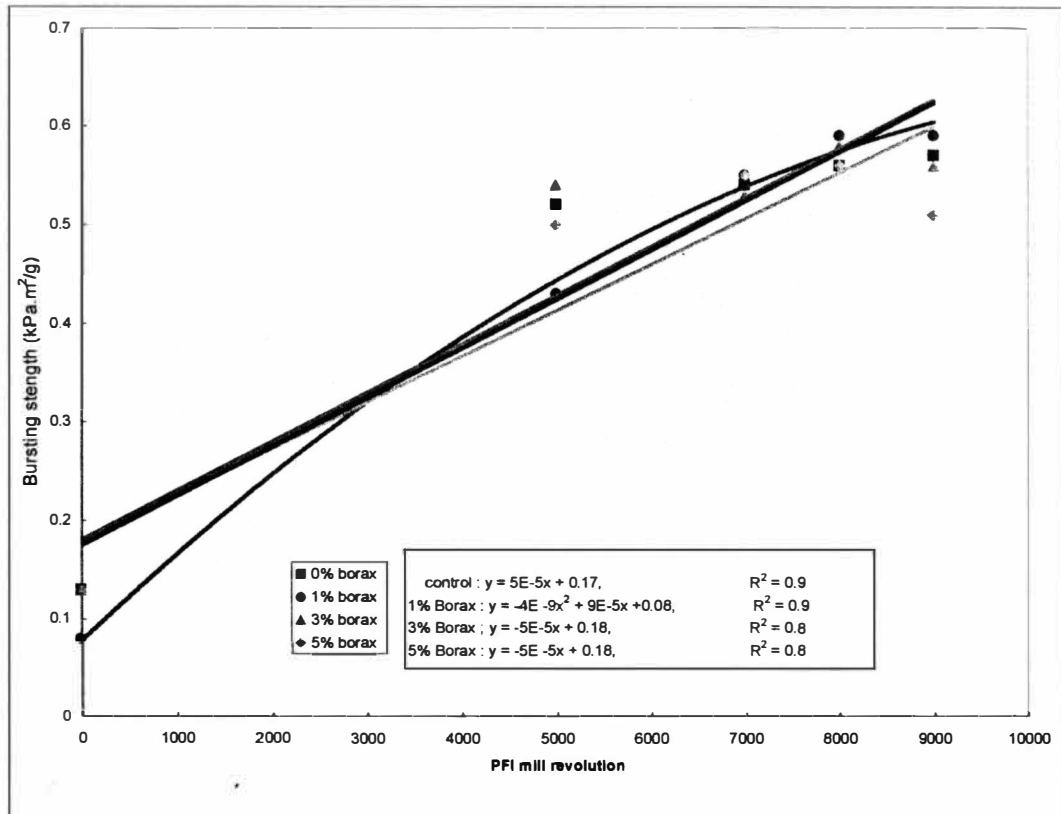


Figure 15. Effect of Borax Addition on Burst Strength at Different PFI Mill Revolutions (Phase 1.1).

can be seen that refining level and borax addition influenced the burst strength very slightly. The addition of 1% borax shows an increasing value of the burst index within the ranges of 5000 – 8000 revolutions (Table 8). The control and the 3% borax essentially overlap each other. The 5% borax falls below the control for higher values of refining. The reason for the trend of the 3% and 5% of borax could be the impact of refining on the fiber length. The statistical analysis presented in Appendix

A suggests that borax had no significant effect, while refining contributes significantly to the bursting strength values.

Table 8

Phase 1.1: Effect of Borax and Refining on Burst Index ( $\text{kPam}^2/\text{g}$ )

Revolution	Borax Concentration			
	0 %	1 %	3 %	5 %
0	0.13	0.08	0.13	0.13
5000	0.52	0.43	0.54	0.50
7000	0.54	0.55	0.53	0.55
8000	0.56	0.59	0.58	0.56
9000	0.56	0.59	0.56	0.51
Pooled Standard Deviation				0.03

### Phase 1.2: Burst Index

The burst index of phase 1.2 shows a clear distinction between the different results obtained due to the addition of borax and refining level. The plot of burst index as a function of refining is presented in Figure 16. The data of the burst index for the three levels of borax and three levels of refining are reported in Table 9.



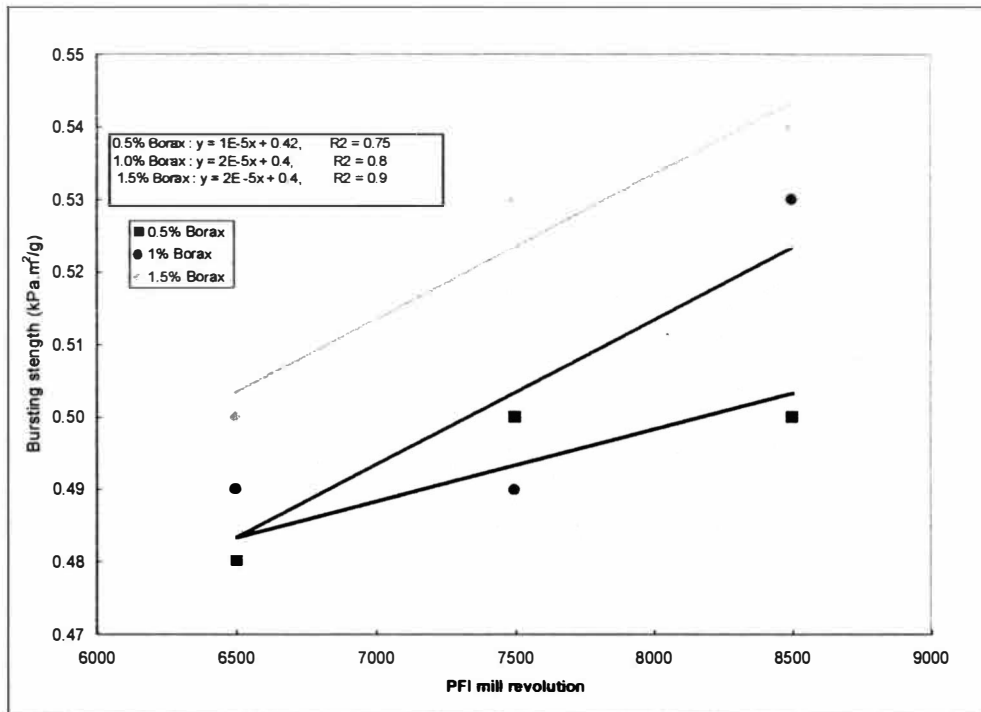


Figure 16. Effect of Borax Addition on Burst Strength at Different PFI Mill Revolutions (Phase 1.2).

Table 9

Phase 1.2: Effect of Borax and Refining on Burst Index (kPam<sup>2</sup>/g)

Revolution	Borax Concentration		
	0.5 %	1.0 %	1.5 %
6500	0.48	0.49	0.50
7500	0.50	0.49	0.53
8500	0.50	0.53	0.54
Pooled Standard Deviation			0.01

The sample distribution of the data for 1% and 1.5% borax addition showed a strong positive correlation with the refining level. Both levels of borax (1% and 1.5%) had a similar slope and their contribution towards the development of the burst index is very close to being significant not at 5% level but at 10% level.

### Phase 1.1: Stiffness

Stiffness is the resistance to bending or the ability of a paper to support its own weight. It is highly influenced by the basis weight and caliper. Stiffness is directly related to fiber bonding. Refining favors stiffness but too much refining has a detrimental effect on the property. Figure 17 shows that the results obtained with

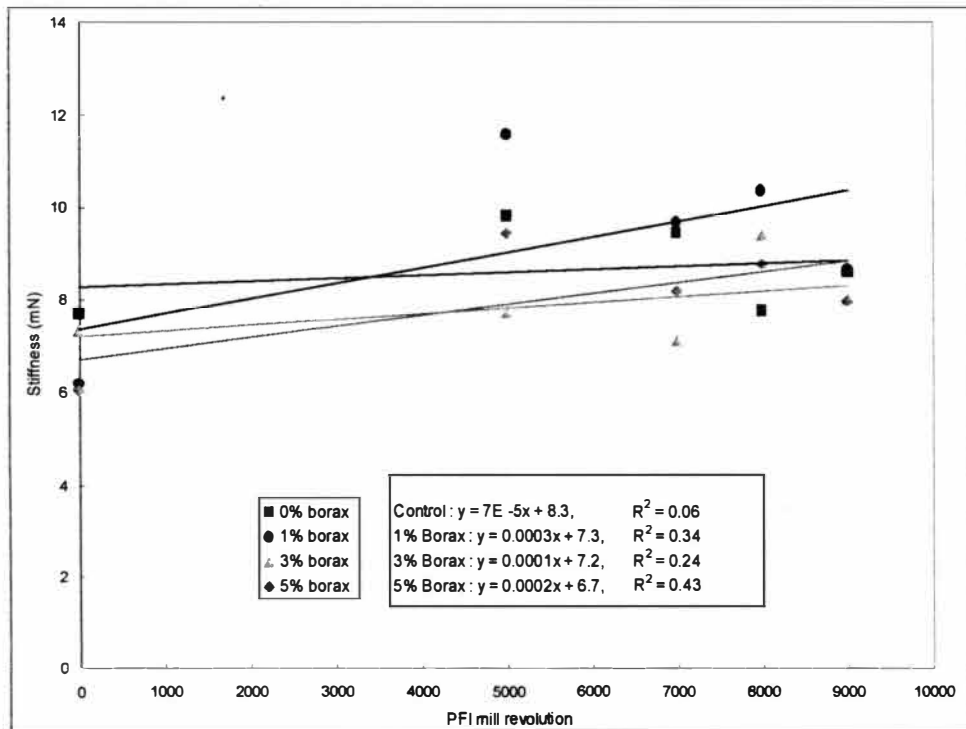


Figure 17. Effect of Borax Addition on Stiffness at Different PFI Mill Revolutions (Phase 1.1).

1% of borax addition are above the control values. All the borax additions seem to result initially in lower stiffness values but the stiffness values pick up with increased refining. Beyond 4000 revolutions, values obtained with 1% borax addition are better than those of control. However with 3% and 5% borax additions, stiffness values can become comparable with those obtained in experiments only at very high levels of refining (approx. 10,000 revolutions). The stiffness values for the different refining and borax levels are reported in Table 10. The statistical analysis (Appendix A) indicates that only refining is significant in increasing the stiffness values.

Table 10

## Phase 1.1: Effect of Borax and Refining on Stiffness (mN)

Revolution	Borax Concentration			
	0 %	1 %	3 %	5 %
0	7.68	6.17	7.30	6.05
5000	9.82	11.59	7.70	9.45
7000	9.44	9.70	7.10	8.18
8000	7.76	10.38	9.40	8.76
9000	8.56	8.66	8.00	7.96
Pooled Standard Deviation				1.00

### Phase 1.2: Stiffness

In this phase, an increase in stiffness is observed for 1% and 1.5% borax addition. The 1% borax addition reached its maximum stiffness value at 7500 revolution and drops thereafter. For various refining levels, the 1.5% borax addition had a stiffness value of 12 – 15.5 mN which is even higher than the values obtained in phase 1.1. These results can be seen in Figure 18. The values of stiffness of 1% and 1.5% were very close at 6500 - 7500 revolutions. This could be due to the effect of refining and borax in increasing the bonding ability of the fibers. The 1% borax

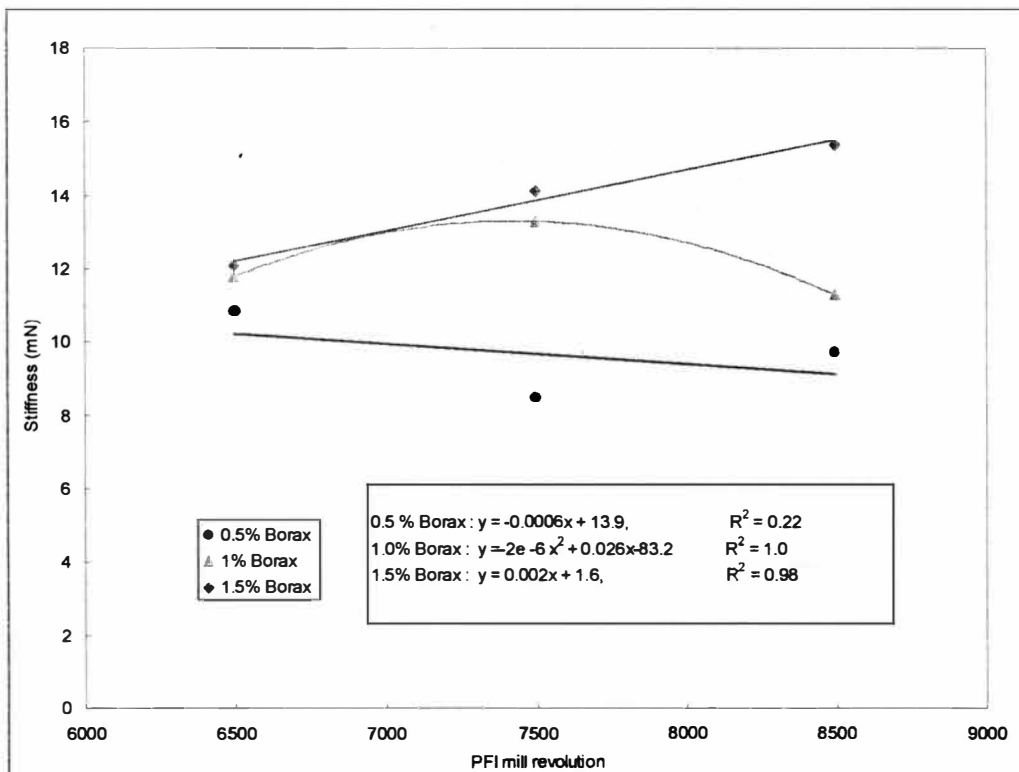


Figure 18. Effect of Borax Addition on Stiffness at Different PFI Mill (Phase 1.2).

produced similar result as in phase 1.1 except that the treatment of refining level was wider in the former case. These results can be seen in Table 10 and Table 11. From the statistical results of phase 1.2 (Appendix B), neither the refining nor the borax Revolutions addition level had any significant effect on the property.

Table 11  
Phase 1.2: Effect of Borax and Refining on Stiffness (mN)

Revolution	Borax Concentration		
	0.5 %	1.0 %	1.5 %
6500	10.83	11.80	12.09
7500	8.44	13.30	14.10
8500	9.70	11.30	15.36
Pooled Standard Deviation			1.60

#### Phase 1.1: Folding Endurance

Folding Endurance is a measure of the strength and flexibility of paper. It is normally sensitive to a slight change in refining. According to Figure 19, the values were clustered together for various levels of borax addition with the average folding values showing an increase with respect to the refining treatment. As the figure and the data in Table 12 indicate, there is very little difference between various levels of

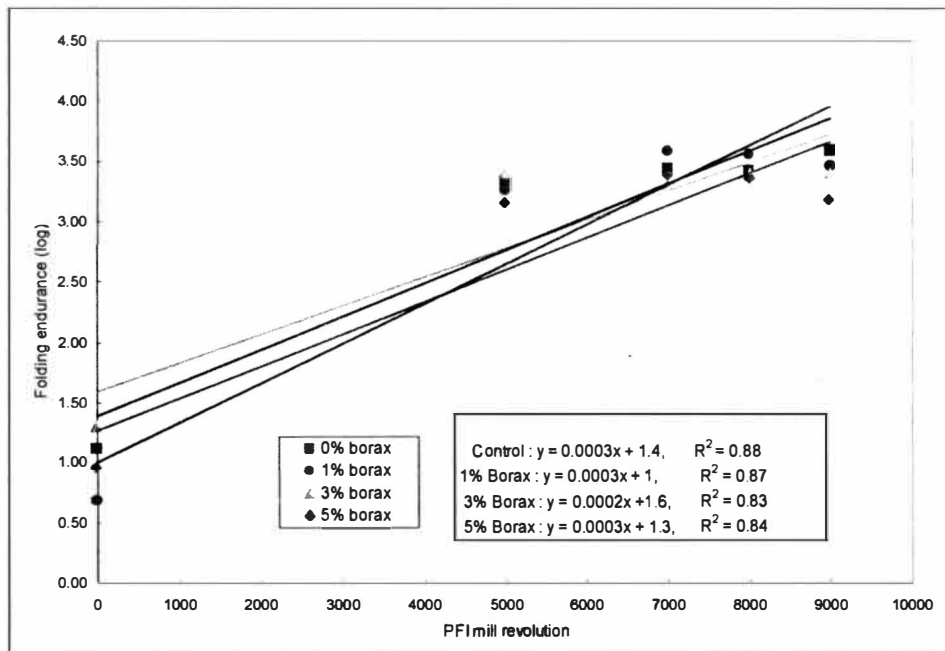


Figure 19. Effect of Borax Addition on Folding Endurance at Different PFI Mill Revolutions (Phase 1.1).

Table 12

Phase 1.1: Effect of Borax and Refining on Folding Endurance

Revolution	Borax Concentration			
	0 %	1 %	3 %	5 %
0	1.12	0.68	1.30	0.96
5000	3.33	3.26	3.40	3.16
7000	3.44	3.58	3.30	3.38
8000	3.42	3.56	3.40	3.36
9000	3.59	3.46	3.40	3.18
Pooled Standard Deviation				0.16

borax addition. The same borne out by the statistical analysis (Appendix A). The statistical analysis proved that the refining level had a significant effect while borax did not have any impact on the folding endurance.

### Phase 1.2: Folding Endurance

In this phase, the 0.5% and 1% borax addition show a decreasing trend through out the refining level. The values stayed within the 3.1 – 3.2 range. Figure 20 depicts a plot of folding endurance against the refining levels. The 0% borax essentially replicated the value observed in phase 1.1. In this phase of the trials, the

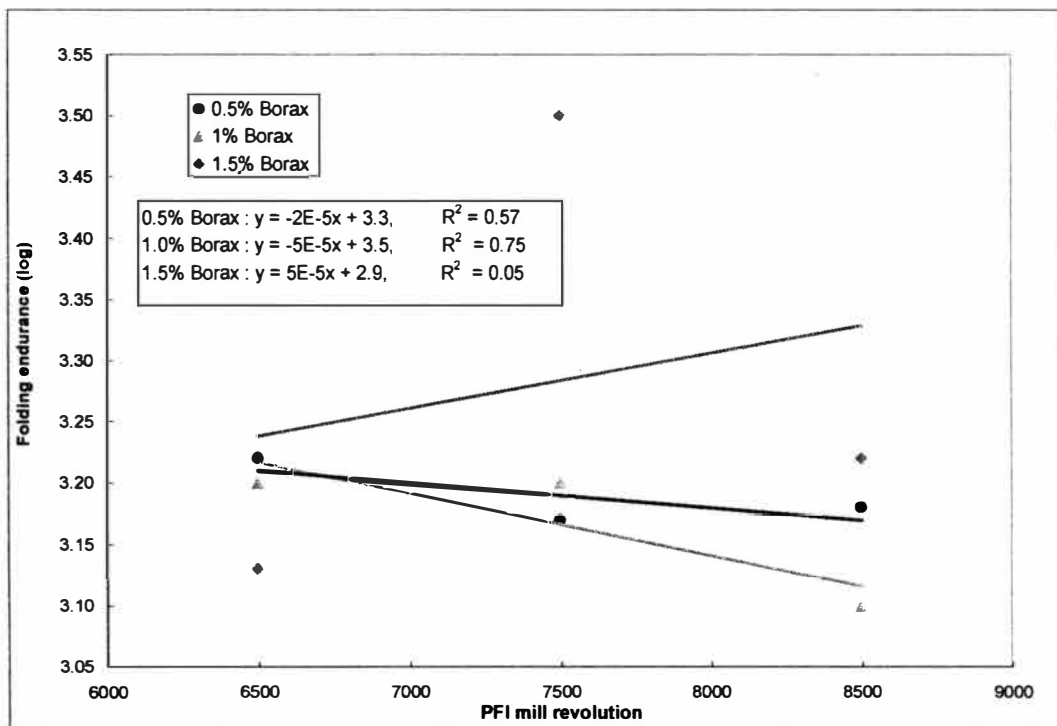


Figure 20. Effect of Borax Addition on Folding Endurance at Different PFI Mill Revolutions (Phase 1.2).

folding endurance did not show much change with respect to either refining or borax addition (Table 13). In fact, the statistical analysis proved that both refining and borax level played insignificant role in improving the folding properties.

Table 13

## Phase 1.2: Effect of Borax and Refining on Folding Endurance

Revolution	Borax Concentration		
	0.5 %	1 %	1.5 %
6500	3.22	3.20	3.13
7500	3.17	3.20	3.50
8500	3.18	3.10	3.22
Pooled Standard Deviation			0.12

Phase 1.1: Internal Bond Strength

The average values of Scott Bond are plotted in Figure 21. Since Scott Bond specifically measures the internal bonding strength, the strength relationship with respect to borax and refining can be predicted directly from the result. Figure 21 shows that as soon as refining and borax are introduced in the system, the internal bond strength started to increase. All percentage levels of borax showed.



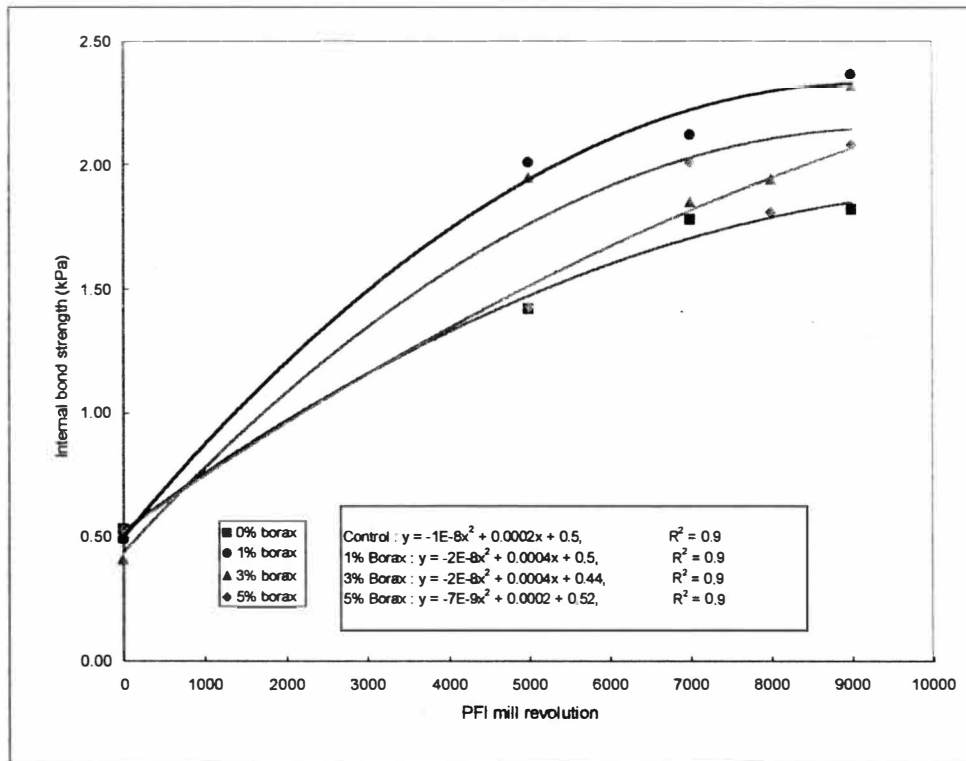


Figure 21. Effect of Borax Addition on Internal Bond Strength at Different PFI Mill Revolutions (Phase 1.1).

an increase in internal bonding compared to control. The 1% and 3% of borax addition showed a rapid increase in the internal bond strength at all levels of refining. At higher refining level 9000 revolution, the strength property reached its maximum value. There could be also a contribution from internal strength of the fibers since Scott Bond is loosely dependent on the fiber strength. It should be noted that the test was performed on a  $200 \text{ g/m}^2$  handsheet so as to evaluate the influence of refining and borax level on the internal strength of the paperboard. There are some missing data in Table 14 and the statistical analysis was based on the general linear model (GLM). The statistical results are presented in Appendix A.

Table 14

Phase 1.1: Effect of Borax and Refining on Internal Bond Strength (kPa)

Revolution	Borax Concentration			
	0 %	1 %	3 %	5 %
0	0.53	0.49	0.41	0.53
5000	1.42	2.01	1.95	1.43
7000	1.78	2.12	1.85	2.01
8000	-----	-----	1.94	1.81
9000	1.82	2.37	2.32	2.08
Pooled Standard Deviation				0.17

Phase 1.2: Internal Bond Strength

In this phase, a high value of internal strength was observed at 8500 revolutions and 1.5% borax addition. At lower level of refining, the 1.5% borax addition showed very low values when compared to 0.5% and 1% addition of borax. From Figure 22 and Table 15, it can be observed that the value of the internal bonding strength at 8500 revolution and 1.5% borax addition is an outlier. The statistical analysis showed that neither refining level nor borax addition had a significant effect on the internal bonding strength, in this phase of trials.

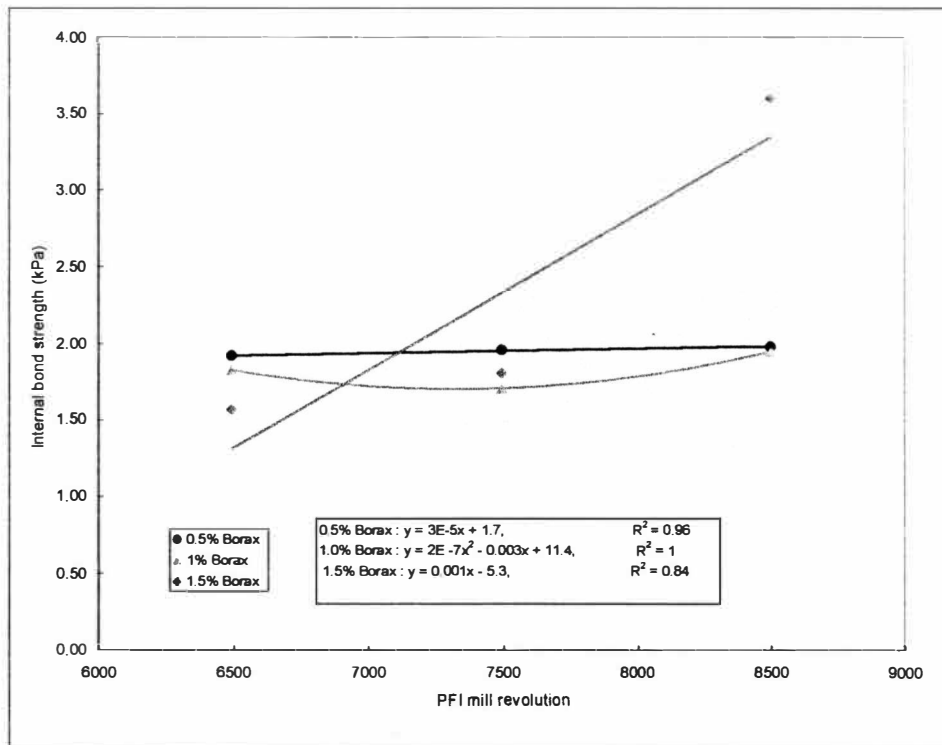


Figure 22. Effect of Borax Addition on Internal Bonding at Different PFI Mill Revolution (Phase 1.2).

Table 15

Phase 1.2: Effect of Borax and Refining on Internal Bond Strength (kPa)

Revolution	Borax Concentration		
	0.5 %	1 %	1.5 %
6500	1.92	1.83	1.57
7500	1.96	1.71	1.81
8500	1.98	1.95	3.60
Pooled Standard Deviation			0.60

## Conclusion

1. Addition of borax at 1% level had a significant effect on the increase of the tensile energy absorption (TEA) at a reduced refining energy.
2. The significant effects in the strength properties due to the factor levels of borax and refining are summarized below, in Table 16.

Table 16

Effect of Borax Addition and Refining on Various Strength Properties

Strength Properties	Level of Significance		Recommendation	
	for Borax	for Refining	Borax level	Revolution
TEA	6%	5%	1%	7500
Tear Index	5%	5%	3%	7500
Burst Index	None	5%	1%	7500
Stiffness	None	5%	1%	7500
Folding Endurance	None	5%	1%	7500
Internal Bonding Strength	5%	6%	1%	7500

3. The tear index was significantly affected by the treatment of borax and refining. The increased refining action shortens the fibers and the tear index values drops with the intensity of refining.

4. The internal bond strength is significantly affected by the combined effect of the addition of borax and refining levels. 1% borax addition showed the largest effect throughout the refining levels.

5. Other strength properties like burst index, stiffness and folding endurance are not affected by the addition of borax.

### Phase 2: Borax and Filler System

The objective of this phase is to observe the extent of the effect of borax on the strength properties of handsheets in the presence of fillers. The fibers were refined at a constant level of 7500 revolutions in the PFI mill. Two types of fillers were tried in the furnish. They were ground calcium carbonate (GCC) and precipitated calcium carbonate (PCC). Both types of fillers were added in various levels of 25%, 40% and 50% on dry fiber.

The first trial of this phase was repeated due to poor first pass retention of filler. In the first trial, the resulting ash content in paper was less than 5% and the results were not included in this report. The main reason for this failure was the filler particle size that made it escape into the drain before being entrapped within the fiber matrix. Since most of the filler were lost into the white water, it was logical to design another experiment that could increase the retention level to an acceptable percentage. This was achieved by the recycling of white water. The use of retention aids could have stabilized the fiber-filler system, but it was deliberately avoided in order to control the complexity of the investigation and to eliminate filler aggregation. The

analysis of this phase is based on the percentage of the ash content and not on the filler loading. Sixty observations were made for each property, which is included in Appendix C. The average values are displayed in various tables with succeeding pages and the regression analysis on the main and interactive effects are presented for each property at the appropriate locations.

### Ash Content

Since the analysis of this phase is based on the ash content, it is appropriate to discuss the effect of borax and filler loading on the ash content. Due to the recycling of the white water, the ash content was between 7 – 28 %. Figures 23 and 24 show

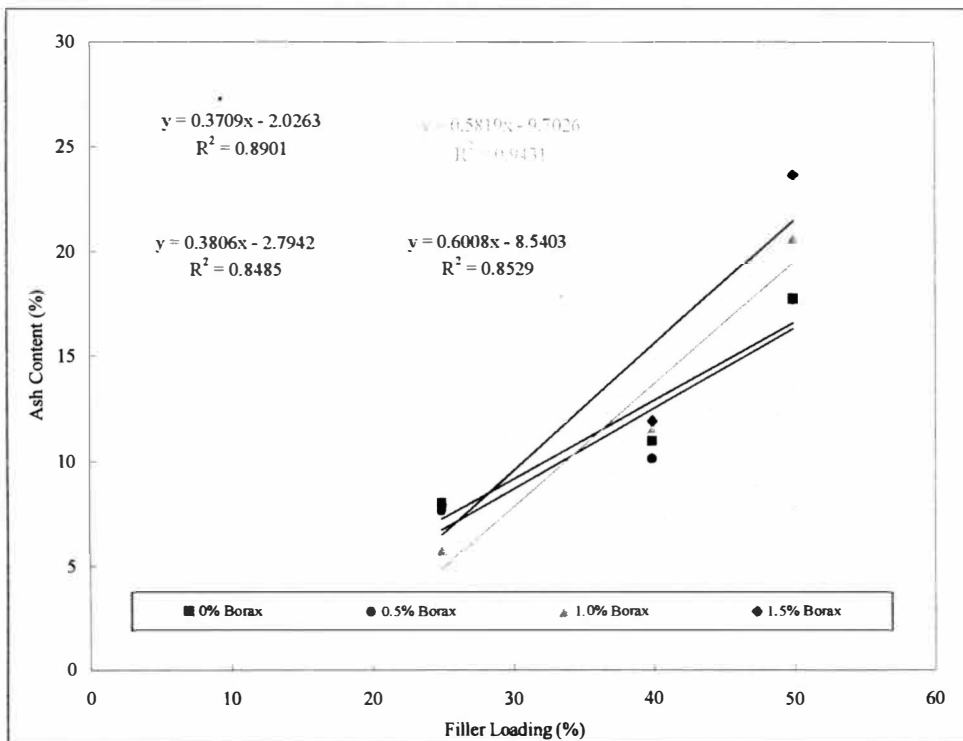


Figure 23. Effect of Borax Addition in Filler Retention of GCC Filled Papers at 7500 PFI Mill Revolutions.

the effect of borax addition and filler loading. The plot indicates that there is a positive correlation between the response and the main factors. But the regression equation presented in Tables 17 and 18 show a weak relationship between the ash content and the borax level / filler loading. The ash content of the PCC was higher than that of the GCC. This trend is clearly seen in the raw data presented in the Table 19. The main reason for the high concentration of ash in the PCC filled handsheet was due to the particle shape, its small size and narrower size distribution.

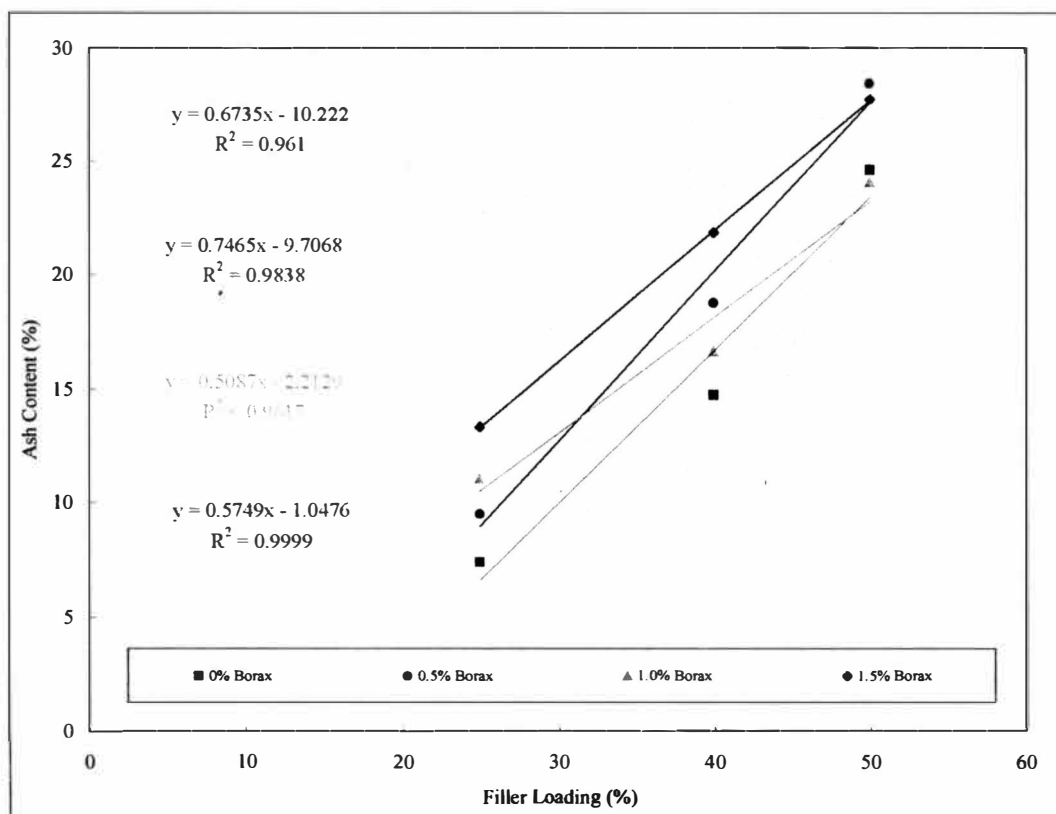


Figure 24. Effect of Borax in Filler Retention of PCC Filled Papers at 7500 PFI Mill Revolutions.

Table 17

## Phase 2.0: The Regression Analysis of Ash Content for GCC

---


$$\text{Ash Content} = 6.97 + 1.76 \text{ Borax} + 0.114 \text{ GCC}$$


---

Predictor	Coef	StDev	T	P
Constant	6.972	2.907	2.40	0.020
Borax	1.759	1.272	1.38	0.172
GCC	0.114	0.069	1.64	0.106

---

S = 5.5	R-Sq = 7.5 %	R-Sq (adj) = 4.2 %
---------	--------------	--------------------

---

Table 18

## Phase 2.0: The Regression Analysis of Ash Content for PCC

---


$$\text{Ash Content} = 10.0 + 2.92 \text{ Borax} + 0.156 \text{ PCC}$$


---

Predictor	Coef	StDev	T	P
Constant	10.01	3.605	2.78	0.007
Borax	2.920	1.577	1.85	0.070
PCC	0.156	0.086	1.82	0.074

---

S = 6.8	R-Sq = 10.6 %	R-Sq (adj) = 7.4 %
---------	---------------	--------------------

---



Table 19

Ash Content of the GCC and PCC at 7500 Revolutions

Filler (%) Loading	Borax Addition (%) - GCC				Borax Addition (%) - PCC			
	0.0	0.5	1.0	1.5	0.0	0.5	1.0	1.5
25	8.00	7.65	5.67	7.92	7.40	0.89	0.67	0.88
40	10.93	10.11	11.51	11.89	14.76	18.77	16.67	21.86
50	17.65	17.63	20.63	23.66	24.63	28.45	24.10	27.75
Pooled Standard Deviation	2.5				2.1			

### Tensile Energy Absorption for GCC and PCC Filled Papers

The tensile energy absorption of both types of fillers was evaluated with respect to ash content and borax addition. The operation was conducted at a constant refining level of 7500 revolutions. According to the plot shown in Figures 25 and 26, the strength property had a decreasing trend as the ash content increased from 7% to 28 %. This behavior was observed irrespective of the filler type. But the presence of

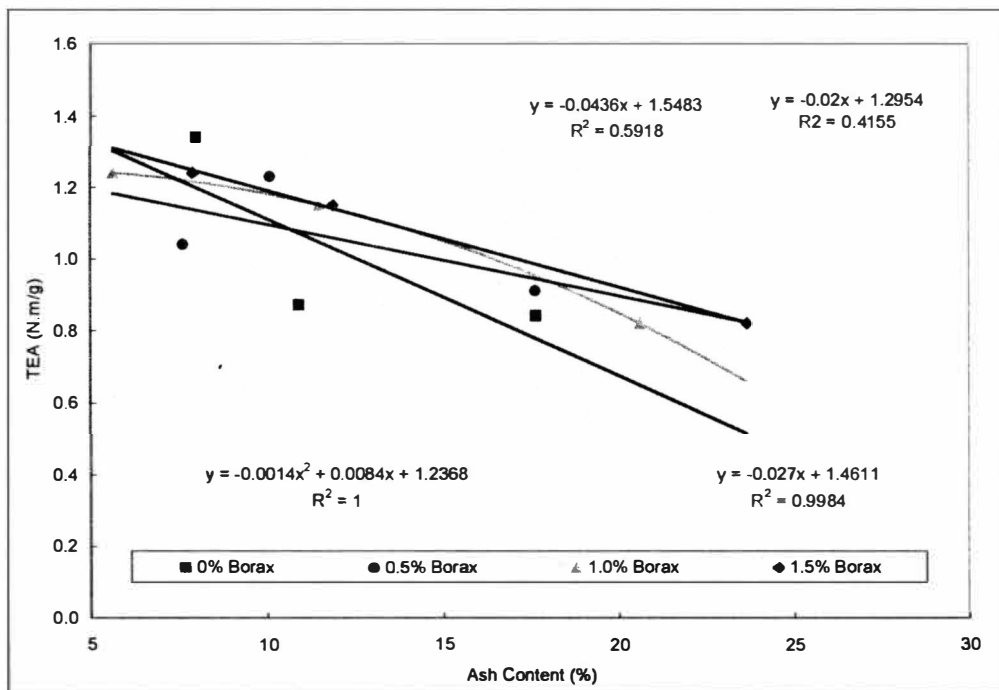


Figure 25. Effect of Ash Content and Borax Addition on TEA of GCC Filled Papers at 7500 PFI Mill Revolutions.

borax helped to retard the decreasing rate when compared to the test result that contained no borax treatment. For the PCC filled paper, the ash content at 23% was a cut-off point as far as the TEA is concerned and all levels of borax fell below the control at that point.

From examining the observed data, it is clear that the relationship between the average TEA and the factor variables is linear. A model that adequately represents this linear relationship is presented in Tables 20 and 21. The predicted average values of the TEA for GCC filled papers reveals that the average TEA drops by

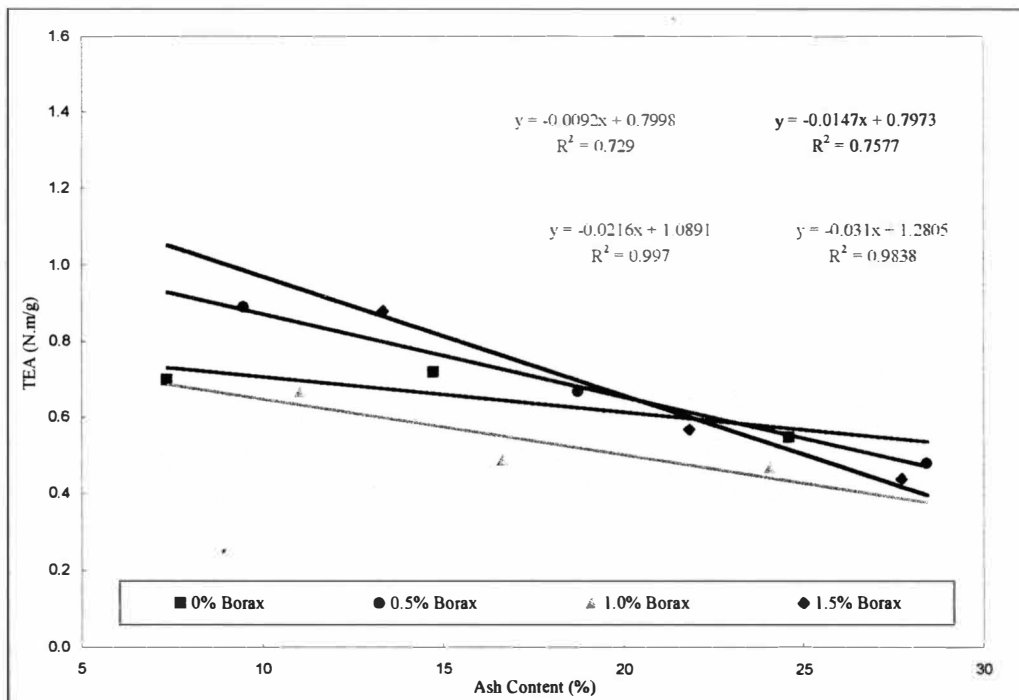


Figure 26. Effect of Ash Content and Borax Addition on TEA of PCC Filled Papers at 7500 PFI Mill Revolutions.

0.04% of ash content and 0.1% of borax level respectively. According to the t-ratio and p-value, the constant term and the ash content are significant. The estimated standard deviation around the regression line was 0.21 Nm/g. The coefficient of determination indicates that 31.4% of the variation in the TEA can be explained by its linear relationship to borax addition and ash level including the interactive factor. In the raw data, there were two observations whose standardized residual (2.47 and

2.48) was unusually high as opposed to the prediction made by the factors involved in the model.

In the case of PCC, the TEA had a strong negative relation with borax addition and a negative correlation with the ash content. As in GCC, the constant

Table 20

Phase 2.0: The Regression Analysis of TEA for GCC

$$\text{TEA} = 1.46 - 0.0357 \text{ Ash} - 0.097 \text{ Borax} + 0.0089 \text{ Ash-Borax}$$

Predictor	Coef	StDev	T	P
Constant	1.4634	0.1355	10.80	0.000
Ash	-0.0357	0.0107	-3.35	0.001
Borax	-0.0966	0.1317	-0.73	0.466
Ash-Borax	0.00893	0.0096	0.93	0.356
S = 0.2138				
R-Sq = 34.9%		R-Sq (adj) = 31.4%		
Unusual Observations				
Ash Content %	Borax Addition (%)		St. Residual	
8.3	1.5		2.47	
13.9	1.0		2.48	

coefficient and the ash content are significant. Borax addition and ash content can only attribute to 36% of the information representing the model of the average TEA. The standard deviation is 0.16 Nm/g. Three unusual observations were found in the

experimental unit whose ash content and borax level were shown in Table 21. Their standardized residual was 3.46, 3.12 and 0.63 respectively. The first two residuals indicate that the values are far above what could be predicted by using the model equation. The third residual has a large influence on the regression line. The average values of the TEA can be referred in Table 22. From the two-model equations, it can

Table 21  
Phase 2.0: The Regression Analysis of TEA for PCC

TEA = 0.844 – 0.0118 Ash – 0.149 Borax + 0.00773 Ash-Borax				
Predictor	Coef	StDev	T	P
Constant	0.8436	0.0830	10.16	0.000
Ash	-0.0118	0.0045	-2.59	0.012
Borax	-0.1488	0.1037	1.44	0.157
Ash-Borax	0.0077	0.0052	-1.48	0.145
S = 0.157		R-Sq = 39.3 %	R-Sq (adj) = 36.1%	
Unusual Observations				
Ash Content %	Borax Addition (%)		St. Residual	
10.2	0.5		3.46	
14.3	1.5		3.12	
22.7	1.5		0.63x	
x is an influential point				

Table 22

Effect of Borax and Filler Addition on TEA at 7500 Revolutions

		Borax Addition (%) - GCC				Borax Addition (%) - PCC					
Filler (%) Loading	Ash Content (%)	0.0	0.5	1.0	1.5		Ash Content (%)	0.0	0.5	1.0	1.5
25	7.3	1.34	1.04	1.24	1.17		10.3	0.70	0.89	0.67	0.88
40	11.0	0.87	1.23	1.15	0.95		18.0	0.72	0.67	0.49	0.57
50	20.0	0.84	0.91	0.82	0.78		26.2	0.55	0.48	0.47	0.44
Pooled Standard Deviation		0.12						0.09			

be concluded that the adverse effect of filler loading on the strength properties is more pronounced on the PCC than GCC.

The main reason was the difference in the particle size, shape and size distribution between the two types of fillers. GCC has a larger particle size and a broader size distribution than the precipitated material. The packing nature of the PCC has a great impact on the paper structure. The lower value of the TEA could be due to the high filler content disrupting the strength development.

#### Tear Index for GCC and PCC Filled Papers

The strength development of tear index is mainly dependent on the fiber length. Tear Index is less sensitive to an applied stress compared to TEA. Thus the tear index obtained with the addition of borax in the presence of filler produced a negative relationship for GCC filled sample and an opposite relation for the PCC filled paper. The plot of the tear index as a function of ash content at various borax levels is presented in Figures 27 and 28.

The regression equation of the GCC filled sample showed that all factors are significant in explaining the average values of the tear index (Table 23). The coefficient of the ash content and the borax level were both negatives (-0.86 and -3.85). The tear index of the PCC has a slight positive correlation with the ash content. The borax addition did not have any significant effect in altering the values of the strength property. All curves were clustered to produce tear index between the ranges of 12 and 14 mNm<sup>2</sup>/g. The ash content had a significant influence on the tear index,

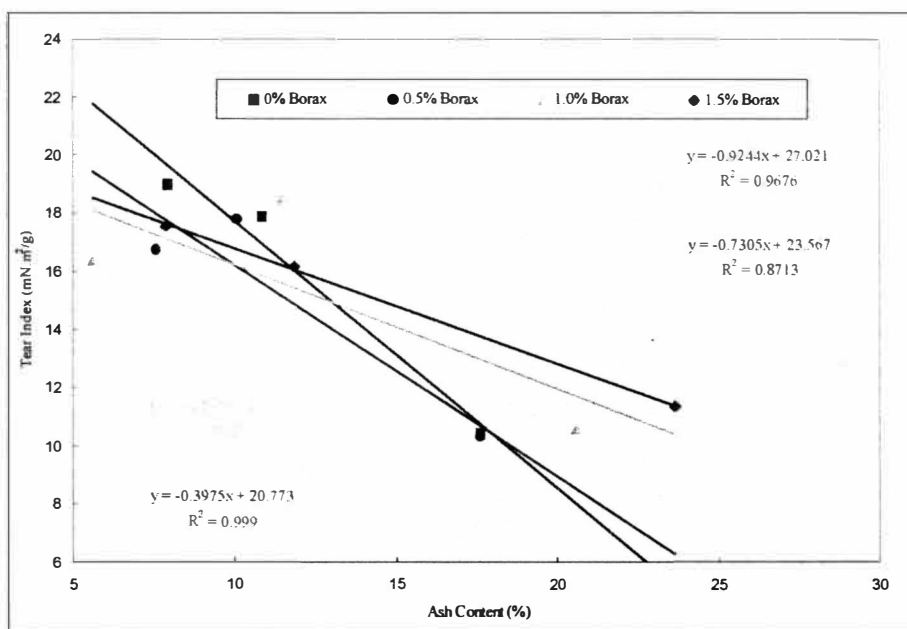


Figure 27. Effect of Ash Content and Borax Addition on Tear Index of GCC Filled Papers at 7500 PFI Mill Revolutions.

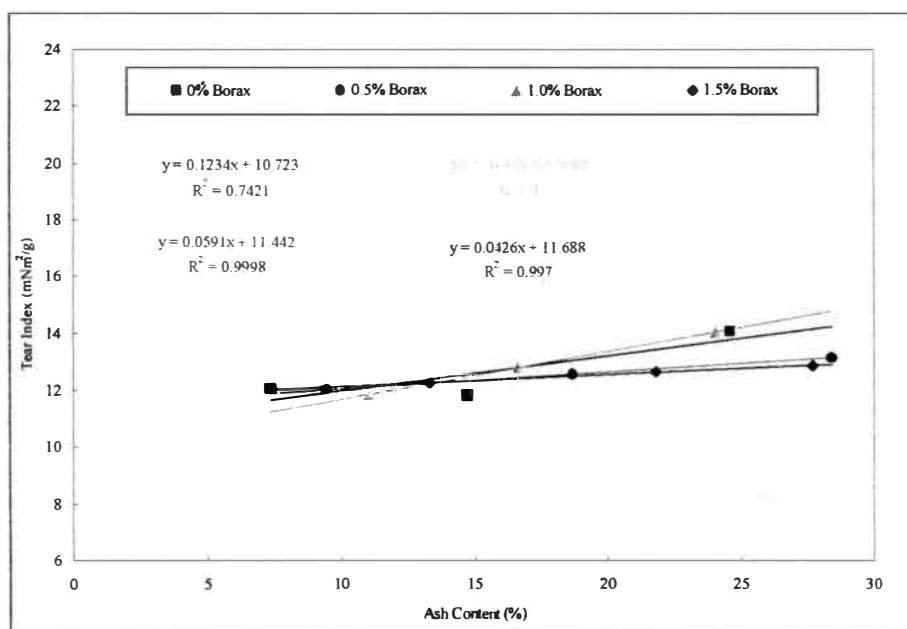


Figure 28. Effect of Ash Content and Borax Addition on Tear Index of PCC Filled Papers at 7500 PFI Mill Revolutions.



which was supported by the p-value of the statistical output as presented in Table 24. The adjusted  $R^2$  was only 28.4% meaning that the represented model equation was not adequate to explain the behavior of the tear index. There must be other unexplained factors that lead the tear index to drop 10 % of the ash content and 31% of the borax level. The corresponding values of the ash content and the borax

Table 23

Phase 2.0: The Regression Analysis of Tear Index for GCC

$$\text{Tear Index} = 25.4 - 0.862 \text{ Ash} - 3.85 \text{ Borax} + 0.349 \text{ Ash-Borax}$$

Predictor	Coef	StDev	T	P
Constant	25.44	1.148	22.15	0.000
Ash	-0.8619	0.0904	-9.53	0.000
Borax	-3.8540	1.1160	-3.45	0.001
Ash-Borax	0.3487	0.0814	4.28	0.000

S = 1.811

R-Sq = 75.0 %

R-Sq (adj) = 73.7 %

#### Unusual Observations

Ash Content %	Borax Addition (%)	St. Residual
7.8	0.5	-2.11
10.5	1.0	4.77

addition can be found in Table 25. Other factors that could appreciably lower the values of the tear strength are the reduction of the fiber length or the packing nature of the PCC particles. Since the surface area of PCC is high and occupies more space per unit weight, the interference of the filler could disrupt the fiber and borax

Table 24

## Phase 2.0: The Regression Analysis of Tear Index for PCC

$$\text{Tear Index} = 11.0 - 0.099 \text{ Ash} - 0.307 \text{ Borax} + 0.0281 \text{ Ash-Borax}$$

Predictor	Coef	StDev	T	P
Constant	11.04	0.4499	25.54	0.000
Ash	0.0995	0.0247	4.03	0.000
Borax	0.3072	0.5623	0.55	0.587
Ash-Borax	-0.0280	0.0284	-0.99	0.326
<hr/>				
S = 0.85	R-Sq = 32.1 %		R-Sq (adj) = 28.4 %	

## Unusual Observations

Ash Content %	Borax Addition (%)	St. Residual
14.8	0.0	-2.89
22.7	0.0	3.50
23.2	1.0	2.28
22.7	1.5	0.49x

x is an influential point

Table 25

Effect of Borax and Filler Addition on Tear Index at 7500 Revolutions

Filler (%) Loading	Ash Content (%)	Borax Addition (%) - GCC				Ash Content (%)	Borax Addition (%) - PCC			
		0.0	0.5	1.0	1.5		0.0	0.5	1.0	1.5
25	7.3	18.97	16.76	16.36	17.54	10.3	12.05	12.00	11.84	12.25
40	11.0	17.86	17.80	18.48	16.16	18.0	11.82	12.56	12.77	12.64
50	20.0	10.42	10.29	10.58	11.34	26.2	14.07	13.12	14.03	12.86
Pooled Standard Deviation		0.12				0.09				

interaction. The rigid nature of the filler could possibly enhance cracks, which could affect the tear index. From the statistical analysis one can conclude that PCC had a dominant role in reducing the strength property and borax did not show any improvement whatsoever.

### Burst Index for GCC and PCC Filled Papers

The burst strength of papers filled with both types of filler shows a marked reduction of values as more and more filler content was utilized. This trend can be observed in Figures 29 and 30. The burst index of both types showed a negative correlation with the ash content. The model equation presented in Tables 26 and 27

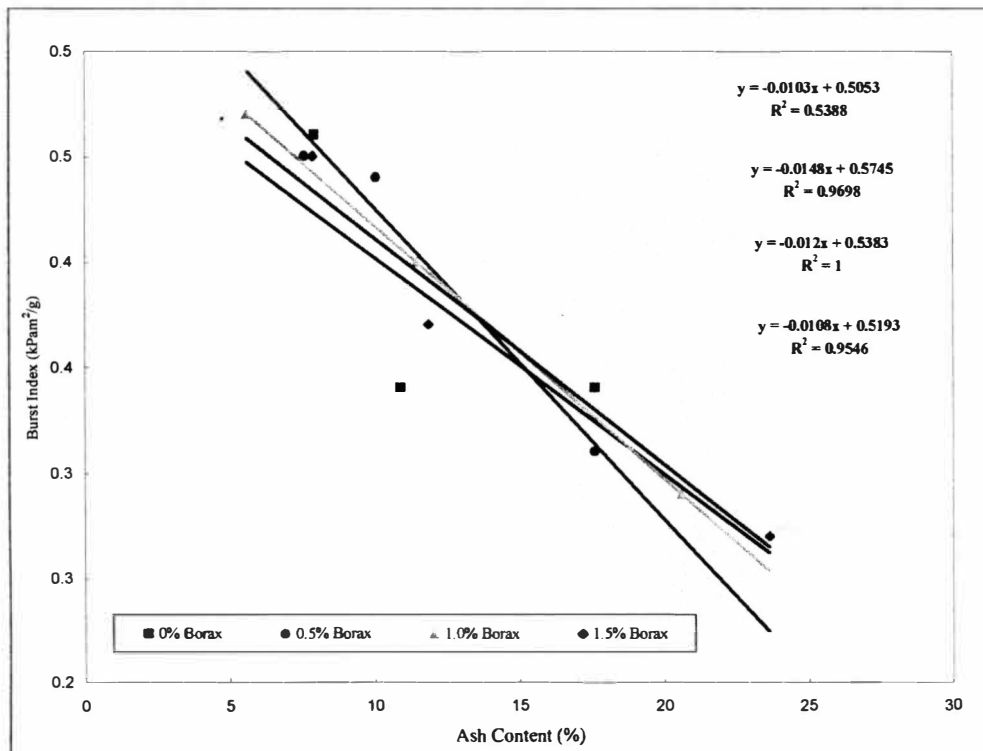


Figure 29. Effect of Ash Content and Borax Addition on Burst Index of GCC Filled Papers at 7500 PFI Mill Revolutions.

sufficiently quantifies the proportion of the total variation explained by the regression of burst index on the ash content. The value of  $R^2$ , for GCC and PCC filled papers, are 75% and 87% respectively. From both equations, it can be observed that only the

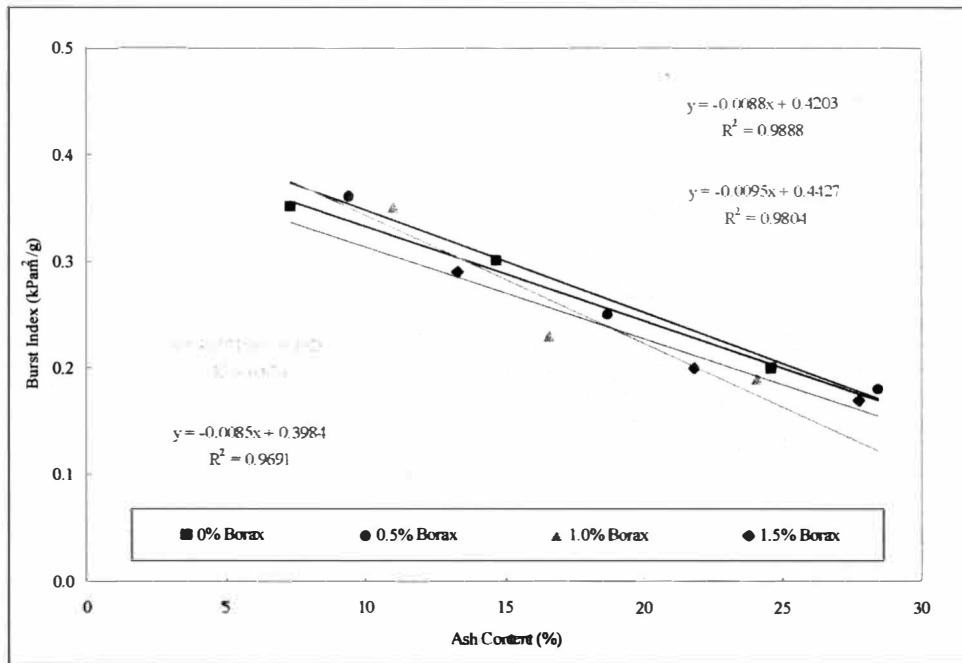


Figure 30. Effect of Ash Content and Borax Addition on Burst Index of PCC Filled Papers at 7500 PFI Mill Revolutions.

coefficient of the constant term and that of the ash are significant while the interactive effect and the borax addition remained insignificant. The estimated standard deviations of the samples are  $0.04 \text{ kPam}^2/\text{g}$  (for GCC) and  $0.03 \text{ kPam}^2/\text{g}$  (for PCC). There are a total of three unusual observations in each type of samples. These occurrences produce values higher than the predicted ones from the model equation. One of the standardized residual in the PCC filled sample was  $-0.05$  and it has a large

influence on the regression equation. The average data of the burst index is found in Table 28.

Other reasons for the overall relative loss in the burst index could be due to the dependency of burst property on the fiber length and fiber bonding. If the filler particles were embedded within the sheet as an expander, it could disrupt the fiber

Table 26

## Phase 2.0: The Regression Analysis of Burst Index for GCC

$$\text{Burst Index} = 25.4 - 0.862 \text{ Ash} - 3.85 \text{ Borax} + 0.349 \text{ Ash-Borax}$$

Predictor	Coef	StDev	T	P
Constant	0.5258	00230	22.88	0.000
Ash	-0.0117	0.0018	-6.45	0.000
Borax	-0.0040	0.0223	0.18	0.857
Ash-Borax	0.0003	0.0016	0.16	0.875
S = 0.04		R-Sq = 76.5 %		R-Sq (adj) = 75.2 %

## Unusual Observations

Ash Content %	Borax Addition (%)	St. Residual
9.4	0.0	-3.02
10.5	0.0	-2.65
10.8	0.0	-2.54

network leading to the drop in burst index. In this condition neither borax nor refining could save the fiber-to-fiber relationships. From the statistical analysis it could be concluded that the use of ash impaired the strength property. The more the percent of ash the lower the value of the strength property. The effect of borax in the filler filled papers produced no significant change in arresting the loss in the strength property.

Table 27

## Phase 2.0: The Regression Analysis of Burst Index for PCC

Burst Index = 0.421 – 0.009 Ash + 0.004 Borax - 0.0007 Ash-Borax				
Predictor	Coef	StDev	T	P
Constant	0.4205	0.0135	31.25	0.000
Ash	-0.0086	0.0007	-11.69	0.000
Borax	0.0036	0.0168	0.210	0.831
Ash-Borax	-0.0007	0.0008	-0.830	0.412
S = 0.03		R-Sq = 87.6 %	R-Sq (adj) = 87.0 %	
Unusual Observations				
Ash Content %	Borax Addition (%)		St. Residual	
11.6	1.0		3.40	
16.1	1.5		-2.13	
22.7	1.5		-0.05x	
x is an influential point				

Table 28

Effect of Borax and Filler Addition on Burst Index at 7500 Revolutions

		Borax Addition (%) - GCC				Borax Addition (%) - PCC				
Filler (%) Loading	Ash Content (%)	0.0	0.5	1.0	1.5	Ash Content (%)	0.0	0.5	1.0	1.5
25	7.3	0.46	0.45	0.47	0.45	10.3	0.35	0.36	0.35	0.29
40	11.0	0.34	0.44	0.40	0.37	18.0	0.30	0.25	0.23	0.20
50	20.0	0.34	0.31	0.29	0.27	26.2	0.20	0.18	0.19	0.17
Pooled Standard Deviation		0.03				0.02				



### Stiffness for GCC and PCC Filled Papers

Normally, the level of filler to some extent affects stiffness but stiffness is not as sensitive as tensile energy absorption. However, stiffness is related to tensile strength, and any impact on tensile strength will be reflected on stiffness as well. The effect of borax addition and ash content on stiffness is presented in Figures 31 and 32.

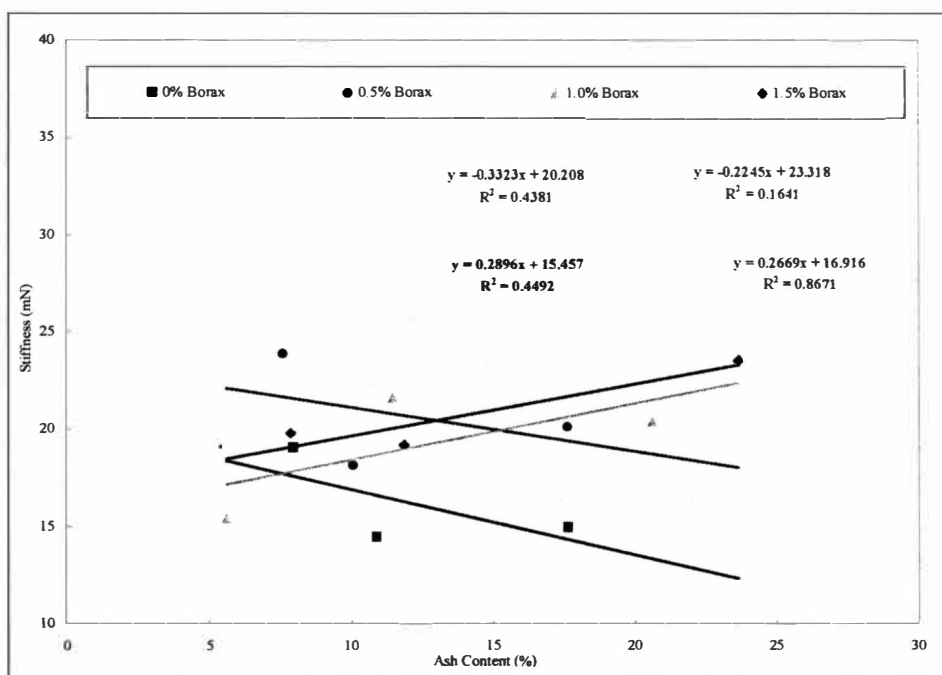


Figure 31. Effect of Ash Content and Borax Addition on Stiffness of GCC Filled Papers at 7500 PFI Mill Revolutions.

The GCC filled sample showed a mixed trend of positive and negative correlation with the response variable (Table 29). The PCC filled handsheet displayed a positive relationship between the ash content and the stiffness property. But the regression model presented can only explain about 7 – 14% of the total variation. More than 60% of the total variation is due to extraneous factors. In the GCC sample, all

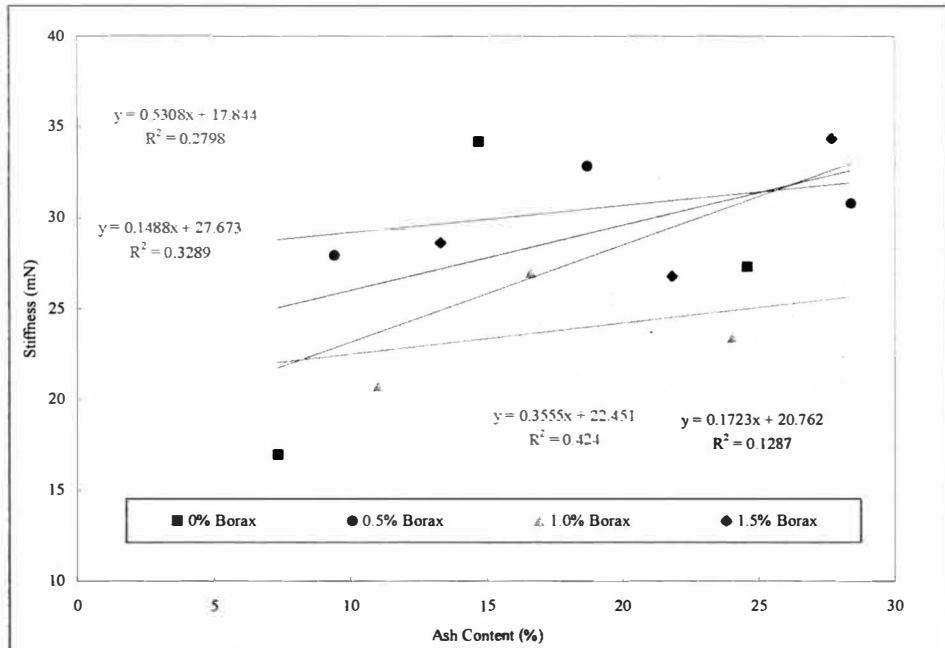


Figure 32. Effect of Ash Content and Borax Addition on Stiffness of PCC Filled Papers at 7500 PFI Mill Revolutions.

Table 29

Phase 2.0: The Regression Analysis of Stiffness for GCC

$$\text{Stiffness} = 20.3 - 0.248 \text{ Ash} - 2.43 \text{ Borax} + 0.376 \text{ Ash-Borax}$$

Predictor	Coef	StDev	T	P
Constant	20.330	2.6450	7.69	0.000
Ash	-0.2480	0.2082	-1.19	0.239
Borax	-2.4320	2.5700	-0.95	0.348
Ash-Borax	0.3762	0.1875	2.01	0.050
S = 4.2      R-Sq = 18.3 %      R-Sq (adj) = 13.9 %				

factors were insignificant except the constant term. In the case of PCC filled sample, the ash content and the constant terms remained insignificant. The average and statistical results are shown in Tables 30 and 31.

Table 30

## Phase 2.0: The Regression Analysis of Stiffness for PCC

$$\text{Stiffness} = 21.1 + 0.358 \text{ Ash} + 1.58 \text{ Borax} - 0.084 \text{ Ash-Borax}$$

Predictor	Coef	StDev	T	P
Constant	21.097	3.3110	6.37	0.000
Ash	0.3578	0.1815	1.97	0.054
Borax	1.5790	4.1380	0.38	0.704
Ash-Borax	-0.0838	0.2086	-0.40	0.689
<hr/>				
S = 6.3	R-Sq = 11.3 %		R-Sq (adj) = 6.6 %	

## Unusual Observations

Ash Content %	Borax Addition (%)	St. Residual
13.6	0.0	2.22
22.7	1.5	1.81x
<hr/>		
x is an influential point		

Table 31

Effect of Borax and Filler Addition on Stiffness at 7500 Revolutions

		Borax Addition (%) - GCC				Borax Addition (%) - PCC				
Filler (%) Loading	Ash Content (%)	0.0	0.5	1.0	1.5	Ash Content (%)	0.0	0.5	1.0	1.5
25	7.3	19.01	23.80	15.41	19.74	10.3	16.92	27.90	20.75	28.61
40	11.0	14.48	18.13	21.56	19.14	18.0	34.15	32.79	27.00	26.78
50	20.0	14.98	20.08	20.35	23.47	26.2	27.30	30.77	23.47	34.35
Pooled Standard Deviation		2.8				5.4				

### Folding Endurance for GCC and PCC Filled Paper

The folding endurance for both types of fillers showed a negative correlation with respect to the ash content. There is a slight difference between the two types of fillers as can be seen in Figures 33 and 34. According to the regression analysis, the coefficients of the ash content and the borax level are negative. In the model equation of both samples, the constant term and the ash content were particularly important.

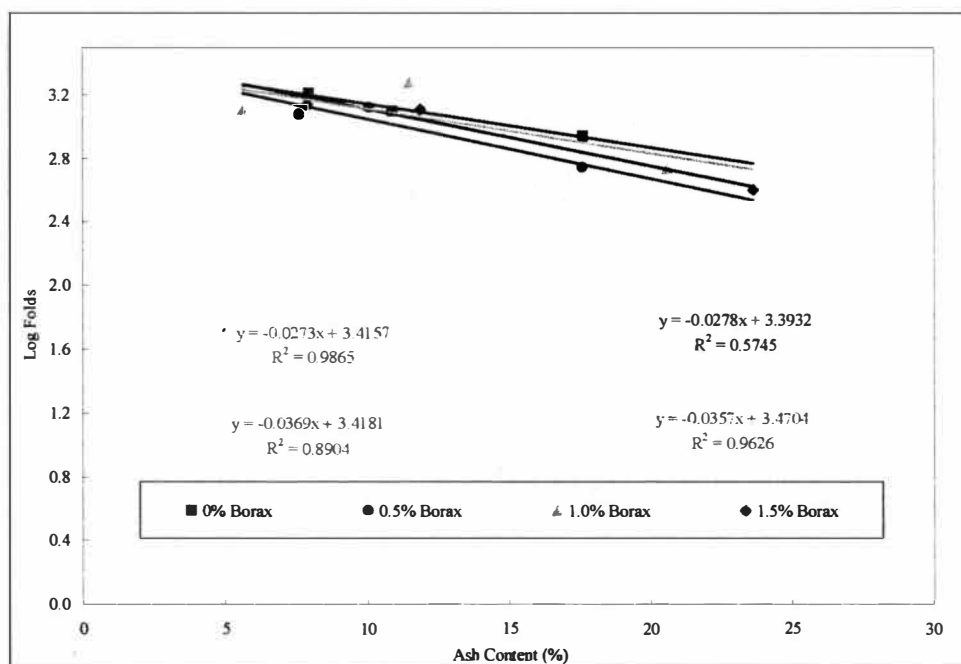


Figure 33. Effect of Ash Content and Borax Addition on Folding Endurance of GCC Filled Papers at 7500 PFI Mill Revolutions.

The  $R^2$  for the GCC and PCC filled samples are 54.4% and 81.7% respectively. From the coefficient of determination, one can realize that the main factors, especially the ash content had a strong influence on the strength property. From the statistical data

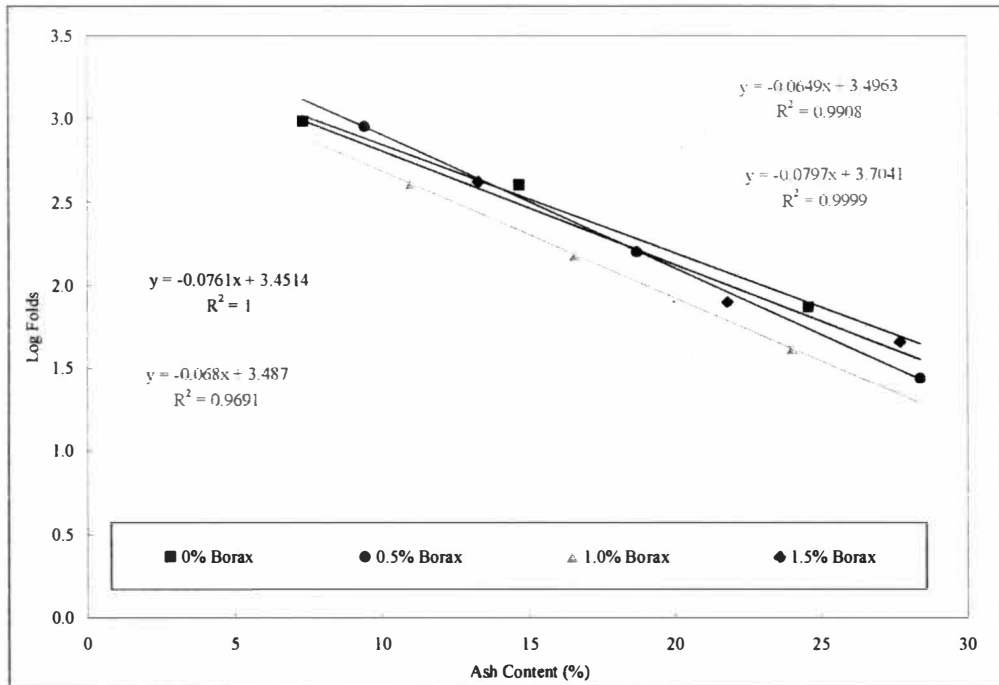


Figure 34. Effect of Ash Content and Borax Addition on Folding Endurance of PCC Filled Papers at 7500 PFI Mill Revolution.

as well as the plot, it can be concluded that borax did not contribute a significant effect on folding endurance. The plot showed that the control and borax treated sample had no difference in the presence of ash content. This leads one to believe that highly filled paper loses its strength as the filler takes the space of the fiber bonding weakening the sheet formation. This trend holds true for both types of fillers. In conclusion, ash content had a significant effect in reducing the folding endurance of the handsheets. The average and statistical values for both types of fillers are shown in Tables 32, 33 and 34.

Table 32

Phase 2.0: The Regression Analysis of Folding Endurance for GCC

Folding Endu. = 20.3 – 0.248 Ash – 2.43 Borax + 0.376 Ash-Borax				
Predictor	Coef	StDev	T	P
Constant	3.3993	0.1011	33.62	0.000
Ash	-0.2967	0.0080	-3.73	0.000
Borax	0.0115	0.0983	0.12	0.907
Ash-Borax	-0.0018	0.0072	-0.25	0.805
S = 0.16                      R-Sq = 56.8 %                      R-Sq (adj) = 54.4 %				
Unusual Observations				
Ash Content %	Borax Addition (%)		St. Residual	
17.8	0.5		-2.79	
20.4	1.0		2.05	

Table 33

Phase 2.0: The Regression Analysis of Folding Endurance for PCC

Folding Endu. = 3.58 – 0.0728 Ash – 0.181 Borax + 0.006 Ash-Borax				
Predictor	Coef	StDev	T	P
Constant	3.5841	0.1238	28.94	0.000
Ash	-0.0728	0.0068	-10.72	0.000
Borax	-0.1812	0.1548	-1.17	0.248
Ash-Borax	0.0064	0.0078	0.82	0.417
S = 0.23                      R-Sq = 82.6 %                      R-Sq (adj) = 81.7 %				

Table 34

Effect of Borax and Filler Addition on Folding Endurance at 7500 Revolutions

		Borax Addition (%) - GCC				Borax Addition (%) - PCC				
Filler (%) Loading	Ash Content (%)	0.0	0.5	1.0	1.5	Ash Content (%)	0.0	0.5	1.0	1.5
25	7.3	3.21	3.08	3.11	3.14	10.3	2.98	2.95	2.61	2.62
40	11.0	3.10	3.12	3.28	3.11	18.0	2.60	2.20	2.18	1.90
50	20.0	2.94	2.75	2.74	2.61	26.2	1.87	1.44	1.62	1.66
Pooled Standard Deviation		0.10				0.12				



## Conclusion

1. Tensile energy absorption is sensitive to filler addition. Both types of fillers had significant effect in reducing the TEA values. Borax had no effect in both cases.

2. The experimental result indicates that filler addition above the required level is detrimental in the strength development.

3. The ash content of the recycled furnish was above 5% and the values were high for the PCC than for GCC.

4. All various strength properties decreased in value with increasing ash content. Addition of borax was effective in arresting the decrease in strength. One of the two fillers, PCC led to a greater drop in strength properties than GCC. All the filler experiments have to be done with recycling of white water to help filler retention. The experimental data has various outlier points, which contributed to poor correlation and low coefficient of determination.

## AKD Sizing System

The experimental work on borax in the alkaline medium had three different phases. The first part is concerned with the treatment of the fiber with borax in the presence of alkyl ketene dimer. This work had provided information on the maximum sizing development (Phase 3.0). The system components of the second part of the project were fiber, borax, filler and AKD (phase 3.1). The last part of the investigation was to evaluate the behavior of borax addition with respect to size

reversion (phase 3.2). The refining level used in the stock preparation, for each phase, was 7500 revolutions. Each phase was operated in an alkaline medium of pH 8.1 to 8.5 and alkalinity level of 290 to 330 ppm. The HST results are reported on the corresponding sections of the analysis. The alkalinity test procedure is included in Appendix D.

### Phase 3.0: Fiber, Borax and AKD System

The investigation in this phase had been centered on the effect of borax in the presence of AKD. The system was designed to include fiber, borax and AKD. The pH and alkalinity are important parameters that affect sizing. During the experiment neither the pH nor the alkalinity level was adjusted. Borax might have acted as a buffer to maintain the pH level. Under alkaline environment fibers are prone to develop better strength than in acidic media. The main reason for this development was the swelling and flexibility of fibers in alkaline medium. Swelling of fibers has an impact on the consolidation of the web, which could lead to improved paper property. In short, pH has a role in establishing the strength of a formed sheet.

The main factors in the project were the borax level (0%, 0.5%, 1.0% & 1.5%), AKD sizing (0%, 0.5%, 1.0% & 2%) and a constant refining level of 7500 revolutions. The handsheets were aged for 48 hours before testing for HST values. The t-test and the p-value for phase 3.1 is organized in Table 35.

Table 35  
Phase 3.0: The Regression Analysis of HST

HST = 76.6 + 180 Borax + 900 AKD				
Predictor	Coef	StDev	T	P
Constant	76.63	82.03	0.93	0.354
Borax	179.85	59.29	3.03	0.004
AKD	899.70	52.43	17.16	0.000
S = 256.6	R-Sq = 84.4 %		R-Sq (adj) = 83.8 %	

### Hercules Size Testing

The results of internal sizing measured with the Hercules Sizing Tester (HST) at 80% reflectance are plotted as a function of borax concentration. Figure 35 shows the effect of borax addition on HST at various dosage of AKD. Increasing the content of AKD caused an increase in the internal sizing for all levels of borax. Table 36 shows the experimental results on the HST at various levels of Borax and AKD addition. The results suggested that borax promoted the efficiency of the sizing agent. From the plot it can be concluded that a minimum level of borax addition is sufficient to assist the retention and distribution of the sizing agent to generate an

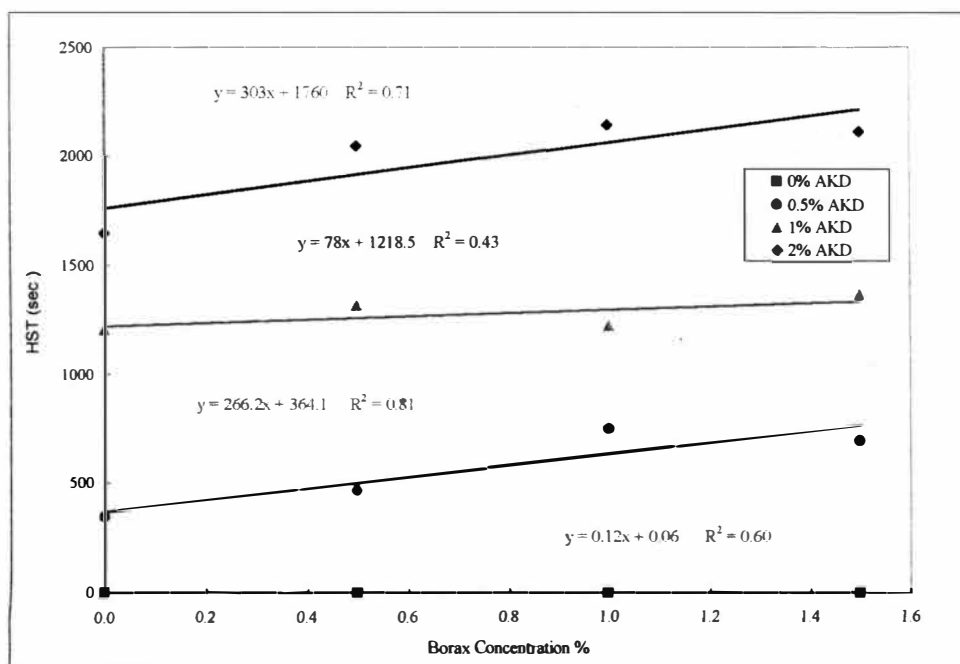


Figure 35. Effect of Borax and Sizing Addition on HST at 7500 PFI Mill Revolutions (Phase 3.0).

Table 36

Phase 3.0: Effect of Borax and AKD Addition on HST at 7500 Revolutions

BORAX (%)	AKD Addition			
	0.0 %	0.5%	1.0 %	2 %
0.0	0.0	346	1204	1645
0.5	0.2	465	1316	2043
1.0	0.2	749	1223	2145
1.5	0.2	695	1365	2116

acceptable result. From the statistical data, it is evident that there is a strong relationship between the response and the factor variables. Borax addition and the sizing agent can explain 84% of the variation in the average result of the HST. The regression analysis verifies that both variables significantly influence the resistance to penetration of the HST ink applied in the experimental procedure.

### Phase 3.1: AKD Sizing and Filler System

This phase is similar in terms of the operational conditions to that of phase 3.0 except for the inclusion of filler in the furnish. The amount of filler used in this trial was 25% of PCC and GCC which was determined in phase 2.0 of borax / filler process. Overuse of the filler materials can increase the negative effect on the fiber bonding, which was observed in phase 2.0. Controlling the amount of filler is important due to the filler's high surface area, which increases the consumption of the sizing system. Two levels of AKD were used with the 25% filler addition. A diluted amount of 0.2% AKD was mixed with the 25% PCC (on fiber) and the observed result was far low with respect to HST. Concentrated AKD at about 2% was then tried with 25% GCC (on fiber). The HST results are presented in Table 37.

As far as the strength properties are concerned only the stiffness showed a significant correlation with the HST values. Borax had no significant effect in the strength development of the TEA and tear index in the presence of AKD and filler addition.

Table 37

Phase 3.1: Effect of Borax, AKD and Filler Addition on HST  
at 7500 Revolutions

AKD (%)	PCC (%)	GCC (%)	BORAX (%)			
			0.0	0.5	1.0	1.5
0.0	0.0	0.0	0.0	0.2	0.2	0.2
0.2	25	0.0	6.0	5.0	2.0	1.0
2.0	0.0	25	436	445	502	423

#### Hercules Size Testing

A small amount of AKD of about 0.2% was used with 25% PCC and the sizing was not adequate to create interaction between the fiber and the sizing agent. Other reason could be the particle size of the PCC is so small and its surface area is large requiring too much of the AKD sizing. Figure 36 shows that the control (0% AKD) and 0.2% AKD addition overlapped on the horizontal axis and that the sizing agent could not impart any internal resistance against ink penetration. This could be due to the insufficient development of interaction between fibers and the sizing agent. On the other hand, when a 2% of the AKD level was incorporated with the 25% of GCC, a dramatic increase in HST values was observed. Even though there is a difference in the level of AKD addition, the handsheet filled with GCC showed a sign

to outperform the PCC filled handsheet. But it is unlikely to draw any conclusion due insufficient data.

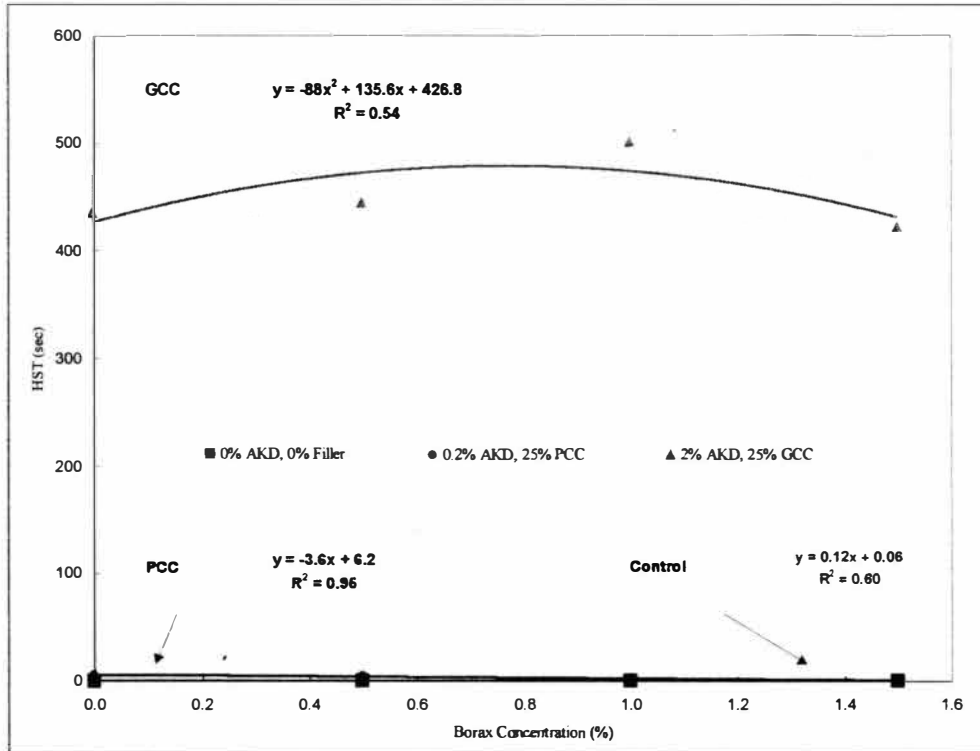


Figure 36. Effect of Borax, Sizing and Filler Addition on HST at 7500 PFI Mill Revolutions (Phase 3.1).

### Stiffness

PCC filled papers had a better stiffness than GCC filled papers as before. It is not clear whether the presence of AKD contributed to any of the results. The average result of the stiffness is presented in Figure 37 and Table 38 shows the stiffness with respect to borax addition and AKD level. The stiffness value of PCC filled papers (with 0.2% AKD) reached a maximum with 0.6 – 0.8 % borax addition.

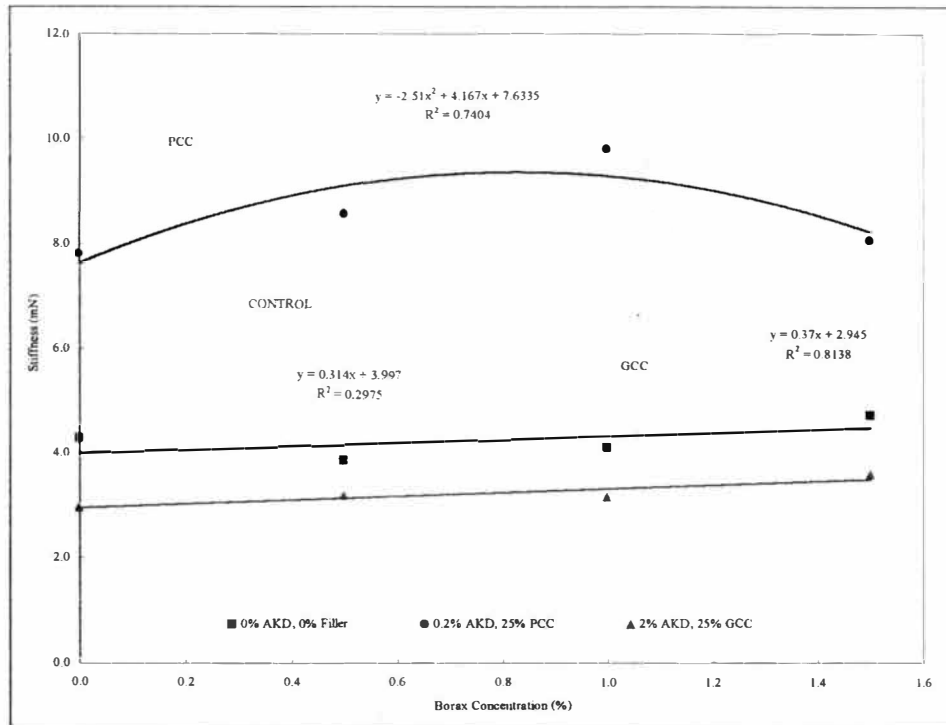


Figure 37. Effect of Borax, Sizing and Filler Addition on HST at 7500 PFI Mill Revolutions (Phase 3.1).

Table 38

Phase 3.1: Effect of Borax, AKD and Filler Addition on Stiffness at 7500 Revolutions

AKD (%)	PCC (%)	GCC (%)	BORAX (%)			
			0.0	0.5	1.0	1.5
0.0	0.0	0.0	4.28	3.84	4.09	4.72
0.2	25	0.0	7.81	8.56	9.82	8.06
2.0	0.0	25	2.96	3.19	3.15	3.59



### Phase 3.2: Size Reversion

In this trial, size reversion was examined without the inclusion of filler in the furnish. The experiment conducted in this phase tried to achieve sizing with a minimal concentration of AKD addition. Therefore, the amount of AKD selected for size reversion was smaller than the one tried in phase 3.1 and it was 0.05% to 0.2% at a pH of 7.1 to 8.4 with alkalinity level reaching a maximum of 320 ppm. The HST test was conducted at ambient temperature but at various storage times as indicated in Table 39. The main objective of the study was to observe the effect of borax on any sizing loss. It was found out that for the amount of AKD dosage used, there was no indication of sizing except at 1.5% of borax and 0.2% of AKD addition. The HST value at higher factor level showed some sizing with slight variation over time. These data were insufficient to conclude any relation between borax and size reversion and the question remains open.

Table 39

#### Phase 3.2: Effect of Borax in Size Reversion

AKD (%)	Borax (%)	pH	Alkalinity (ppm)	HST (sec)		
				day 2	day 4	day 6
0.05	0.0	7.8	320	0.4	0.4	0.3
	0.5	7.9	310	0.5	0.5	0.3
	1.0	8.0	310	0.4	0.5	0.5
	1.5	8.2	310	0.4	0.3	0.2

Tables 39 - Continued

0.1	0.0	7.8	250	0.9	1.0	0.8
	0.5	8.2	280	0.5	0.5	0.7
	1.0	7.1	290	0.4	0.3	0.3
	1.5	7.2	290	0.4	0.3	0.5
0.2	0.0	7.9	310	0.9	0.9	0.8
	0.5	8.1	310	1.2	1.1	1.0
	1.0	8.2	310	0.7	0.6	0.6
	1.5	8.4	310	25	31	27

### Conclusion

1. AKD and borax system showed that the addition of borax and AKD had significant effect on increasing the resistance to penetration, as measured by the HST values.

2. In the presence of AKD and filler, borax did not have any impact on strength properties such as TEA and tear index. The stiffness values of PCC filled paper were better result than GCC filled paper. But the results remained inconclusive as to the role of AKD in increasing the stiffness value.

3. No sizing was imparted at low level of AKD addition and 25% PCC loading. This could be due to a high surface area and small particle size of the PCC filled papers, which requires a high demand of sizing agent. It could also be due to the drainage of fines with the accompanying sizing chemical. The level of AKD used was insufficient for the development of sizing and hence size reversion studies are inconclusive.

## Borax in Surface Application

The objective of this phase was to treat the surface of the wet handsheet by spraying borax solution (0, 0.5, 1 and 1.5%) to improve the physical properties of paper. The spraying was done at the point between the wet press and drying process from a fixed distance. The refining levels were 0, 5000, 7500 and 8500 revolutions. After various strength measurements, the strength development was evaluated. Among all physical property tests only the tear index showed a slight improvement but not to any significant level. The summary result is displayed Appendix E.

### Tear Index

From the tear index curves, it can be seen that the tear index shows an improvement between the levels of 0.5 – 1.5 % borax addition. The regression analysis is shown in Table 40. As per the regression analysis refining level

Table 40

#### Phase 4.0: The Regression Analysis of Tear Index

---


$$\text{Tear Index} = 17.6 + 0.322 \text{ Borax} + 0.000276 \text{ Refining}$$


---

Predictor	Coef	StDev	T	P
Constant	17.6182	0.6736	26.16	0.000
Borax	0.3225	0.5210	0.62	0.547
Refining	0.0003	0.0001	3.12	0.008

---

S = 1.2	R-Sq = 43.8 %	R-Sq (adj) = 35.1 %
---------	---------------	---------------------

---

is more of a significant variable than the borax addition. However, the adjusted  $R^2$  value is too low (35%). One of the reasons for the failure of the surface treatment was the problem of penetration of borax into the sheet. The unavailability of retention measurement made it difficult to know how much of the borax was lost into the dryer felt which had reduced the effectiveness of the borax addition. Figure 38 shows the tear index as a function of refining energy when borax is used as surface sizing agent.

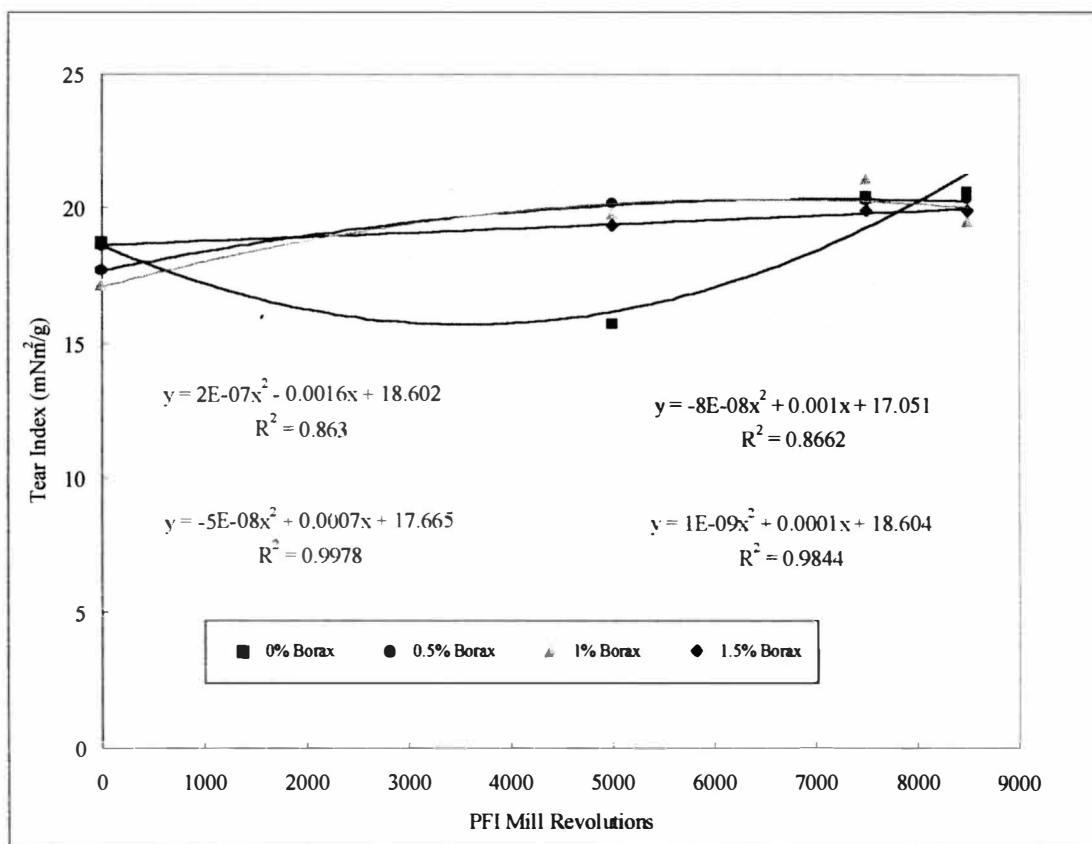


Figure 38. Effect of Borax as a Surface Sizing Agent on the Tear Index at Different PFI Mill Revolution.

## Conclusion

Borax in the surface application did not show any improvement in the strength properties. Tear index show some changes in the strength development but not to a significant level. Lack of improvement in the property could be associated either to the inability to spraying power of the borax penetrating the handsheet or the loss of borax due to the rubbing effect of the dryer felt with that of the wet handsheet.

## CHAPTER VII

### CONCLUSION

Borax addition had been tried with a desire to increasing strength properties of paper. At a reduced refining level a small quantity of borax (1%) had shown a significant effect on the tensile energy absorption (TEA), and internal bond strength. The combined effect of borax addition and refining level could not affect the strength properties such as tear index, burst index, stiffness, and folding endurance.

Even though loss of filler particles were encountered in the first trial with less than 5% retention of ash, the alternative method (recycling of white water) allowed a substantial gain in the filler retention. The recycling of white water made it possible for the evaluation of PCC and GCC filled papers. Although the addition of fillers, negatively affected the response towards the strength development, borax effectively reduced the level of deterioration in the strength properties. The experimental results suggested that there were various outlier points that contributed to poor correlation and low coefficient of determination.

Addition of borax in the presence of AKD promoted internal size development, which was evidenced by an increase in the values of HST. Inclusion of filler had the expected negative impact on the strength properties. Borax was not

arrest the influence of filler in reducing the strength properties. Due to the high demand on AKD, sizing could not be developed in the presence of PCC. At higher levels of AKD, sizing was effective in GCC filled paper. No size reversion was observed and the results remained inconclusive.

Borax, applied in the form of a surface spray, did not show any effect on the strength properties.

## CHAPTER VIII

### RECOMMENDATIONS FOR FURTHER STUDY

1. Addition of borax to the pulp prior to refining could bring a change in the strength properties development.
2. An investigation on a pilot plant scale could show the true effect of borax in the presence of filler and sizing additives.
3. Different levels of Addition of AKD in the absence of borax should be performed for comparison proposes.
4. The investigation of size reversion at higher level of AKD in the presence of fillers is worth trying over a longer storage time (20 – 60 days).



## LITERATURE CITED

1. Swanson, J.W. and Jones, E.J., "Factors Which Affect Strength Properties of Paper," Pulp and Paper Canada, 63(5): T251 - T258 (1962).
2. Scott, W.E., "Principles of Wet End Chemistry," 2<sup>nd</sup> Ed., Tappi Press, Atlanta, GA, p. 49 (1996).
3. Janes, R.L., "Fiber Characteristics," In, Stock Preparation Short Course, Tappi Press, Atlanta, GA, p. 3 (1992).
4. Retulainen, E. and Ebeling, K., "Fiber-Fiber Bonding and Ways to Characterize bond strength," Appita, 46(7): 282 (1993).
5. Campbell, W.B., "The Mechanism of Bonding," Tappi Journal, 42(12): 999 (1959).
6. Parsons, S. R., "Optical Characteristics of Paper as a Function of Fiber Classification," Tappi Journal (12), P 314 - 322 (1942).
7. Ratliff, F.T., "The Possible Correlation between Hemicelluloses and the Physical Properties of Bleached Kraft Pulps," Tappi Journal, 32(8): 357 (1949).
8. Casey, J. P., "Pulp and Paper Chemistry and Chemical Technology," 2<sup>nd</sup> Ed., Interscience, New York, (1960), p. 713
9. Bridge, N.K., and Hamer, R. J., "Beating and Basic Fiber Properties," Paper Technology, 18(2): 37 (1977).
10. Kibblewhite, R. P., "Effect of Beating on Fiber Morphology and Fiber Surface Structure," Appita, 26(3): 196 (1972).
11. Paavilainen, L., "Bonding Properties of Softwood Sulphate Pulp Fibers," Paperi Ja Puu, 76(1), (1994).
12. Paavilainen, L., "Wet Fiber Flexibility and Collapsibility of Softwood Sulphate Pulp Fibers," Paperi Ja Puu, 75, p. 9, (1993).

13. Lindström, T., and Eklund, D., "Paper Chemistry – An Introduction," Published by DT Paper Science Publications, Grankulla, Finland 1991, p. 215.
14. Alberts, R. M., "Alkyl Ketene Dimer Sizes," Tappi Sizing Short Course, 1987.
15. Eaton, C. K., and Janes, R. L., "The Effect of Internal Sizing on Paper Deterioration," Proceedings of 1992 Papermakers Conference Held at Nashville, TN., TAPPI Press, Atlanta, GA, p. 197.
16. Au, C.O., and Thorn, I., "Application of Wet-End Paper Chemistry," Published by Chapman and Hall, New York, 1995, p. 139.
17. Fairchild, G. H., "Increasing the Filler Content of PCC-Filled Alkaline Papers," Tappi Journal, 75(8): 85 (1992).
18. Jokinen, O., and Ebeling, K., "Flocculation Tendency of Papermaking Fibers", Paperi Ja Puu 67(5): 317 (1985).
19. Eissa, Y.Z., Naito, T., Usuda, M., and Kadoya, T., "Flow Resistance of Rotating Dilute Fiber Suspensions Role of Fundamental Research in Papermaking", Vol 1. British Paper and Board Maker's Association, p. 403 (1983).
20. Kerekes, R. J., "Pulp Flocculation in Decaying Turbulence: A literature Review", J. Pulp Paper Science 9 (7): TR86 (1983).
21. Binotto, A. P., and Nicholls, G. A., "Correlation of Fiber Morphological Variation and Wet Mat Compressibility of Loblolly Pine Bleached Kraft Pulp," Tappi Journal, 60(6): 91 (1977).
22. Marton, J., "Paper Chemistry", Roberts, J. C., 2<sup>nd</sup>, Ed., Chapman & Hall, New York p. 83 (1996).

## Appendix A

### Analysis of Variance of the Strength Properties (Phase 1.1)

Table 41

## Statistical Output of the Strength Properties for Phase 1.1

Response	Factors	F-ratio	P-value	Significant Level At $\alpha = 5\%$
TEA	Revolution	84.72	0.000	YES
	BORAX	3.25	0.060	YES (close)
Tear Index	Revolution	3.98	0.028	YES
	BORAX	4.37	0.027	YES
Burst Index	Revolution	151.7	0.000	YES
	BORAX	0.48	0.705	NO
Stiffness	Revolution	4.57	0.018	YES
	BORAX	2.00	0.168	NO
Folding Endurance	Revolution	179.24	0.000	YES
	BORAX	1.17	0.361	NO
Internal Bond Strength	Revolution	3.37	0.063	YES (close)
	BORAX	57.62	0.00	YES

TENSILE ENERGY ABSORPTION (Phase 1.1)

```
MTB> twoway 'TEA' 'BORAX' 'REFINING';
Subc> means 'REFINING' 'BORAX'
```

Analysis of Variance for TEA

Source	DF	SS	MS	F	P
REFINING	4	3.8503	0.9626	84.72	0.000
BORAX	3	0.1109	0.0370	3.25	0.060
Error	12	0.1363	0.0114		
Total	19	4.0976			

TEAR INDEX (Phase 1.1)

```
MTB > twoway 'TEAR' 'BORAX' 'REFINING';
SUBC> means 'REFINING' 'BORAX'.
```

Analysis of Variance for Tear Index

Source	DF	SS	MS	F	P
REFINING	4	87.82	21.96	3.98	0.028
BORAX	3	72.38	24.13	4.37	0.027
Error	12	66.27	5.52		
Total	19	226.48			

BURST INDEX (Phase 1.1)

MTB > twoway 'BURST' 'BORAX' 'REFINING';  
 SUBC> means 'REFINING' 'BORAX'.

Analysis of Variance for Burst Index

Source	DF	SS	MS	F	P
REFINING	4	0.588620	0.147155	151.79	0.000
BORAX	3	0.001380	0.000460	0.48	0.705
Error	12	0.011620	0.000968		
Total	19	0.601620			

FOLDING ENDURANCE (Phase 1.1)

MTB > twoway 'FOLDING' 'BORAX' 'REFINING';  
 SUBC> means 'REFINING' 'BORAX'.

Analysis of Variance for Folding Endurance

Source	DF	SS	MS	F	P
REFINING	4	18.0872	4.5218	179.24	0.000
BORAX	3	0.0887	0.0296	1.17	0.361
Error	12	0.3027	0.0252		
Total	19	18.4787			

STIFFNESS (Phase 1.1)

```
MTB > twoway 'STIFFNESS' 'BORAX' 'REFINING';
SUBC> means 'REFINING' 'BORAX'.
```

Analysis of Variance for Stiffness

Source	DF	SS	MS	F	P
REFINING	4	18.29	4.57	4.57	0.018
BORAX	3	5.99	2.00	2.00	0.168
Error	12	12.01	1.00		
Total	19	36.29			

INTERNAL BOND STRENGTH (Phase 1.1)

```
MTB > glm Internal Bond Strength = Borax,Refining
```

**General Linear Model**Analysis of Variance for Internal Bond Strength

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Borax	3	0.3155	0.2965	0.0988	3.37	0.063
Refining	4	6.7639	6.7639	1.6910	57.62	0.000
Error	10	0.2935	0.2935	0.0293		
Total	17	7.3728				

## Appendix B

### Analysis of Variance of the Strength Properties (Phase 1.2)



Table 42

## Statistical Output of the Strength Properties for Phase 1.2

Response	Factors	F-ratio	P-value	Significant Level At $\alpha = 5\%$
TEA	Revolution	0.81	0.505	NO
	BORAX	5.08	0.080	NO
Tear Index	Revolution	0.89	0.479	NO
	BORAX	3.27	0.144	NO
Burst Index	Revolution	6.25	0.059	YES
	BORAX	5.25	0.076	NO
Stiffness	Revolution	0.09	0.913	NO
	BORAX	5.33	0.074	NO
Folding Endurance	Revolution	0.97	0.455	NO
	BORAX	0.82	0.502	NO
Internal Bond Strength	Revolution	1.37	0.352	NO
	BORAX	0.54	0.619	NO

TENSILE ENERGY ABSORPTION (Phase 1.2)

```
MTB> twoway 'TEA' 'BORAX' 'REFINING';
Subc> means 'REFINING' 'BORAX'
```

Analysis of Variance for TEA

Source	DF	SS	MS	F	P
rpm	2	0.0523	0.0261	0.81	0.505
borax	2	0.3260	0.1630	5.08	0.080
Error	4	0.1284	0.0321		
Total	8	0.5066			

TEAR INDEX (Phase 1.2)

```
MTB > twoway 'TEAR' 'BORAX' 'REFINING';
SUBC> means 'REFINING' 'BORAX'.
```

Analysis of Variance for Tear Index

Source	DF	SS	MS	F	P
rpm	2	11.60	5.80	0.89	0.479
borax	2	42.59	21.30	3.27	0.144
Error	4	26.03	6.51		
Total	8	80.22			

BURST INDEX (Phase 1.2)

```
MTB > twoway 'BURST' 'BORAX' 'REFINING';
SUBC> means 'REFINING' 'BORAX'.
```

Analysis of Variance for Burst Index

Source	DF	SS	MS	F	P
rpm	2	0.001667	0.000833	6.25	0.059
borax	2	0.001400	0.000700	5.25	0.076
Error	4	0.000533	0.000133		
Total	8	0.003600			

FOLDING ENDURANCE (Phase 1.2)

```
MTB > twoway 'FOLDING' 'BORAX' 'REFINING';
SUBC> means 'REFINING' 'BORAX'.
```

Analysis of Variance for Folding Endurance

Source	DF	SS	MS	F	P
rpm	2	0.0269	0.0134	0.97	0.455
borax	2	0.0229	0.0114	0.82	0.502
Error	4	0.0557	0.0139		
Total	8	0.1054			

STIFFNESS (Phase 1.2)

```
MTB > twoway 'STIFFNESS' 'BORAX' 'REFINING';
SUBC> means 'REFINING' 'BORAX'.
```

Analysis of Variance for Stiffnes

Source	DF	SS	MS	F	P
rpm	2	0.47	0.23	0.09	0.913
borax	2	26.66	13.33	5.33	0.074
Error	4	10.00	2.50		
Total	8	37.13			

SCOTT BOND (Phase 1.2)

```
MTB > twoway 'scott-Bond' 'BORAX' 'REFINING';
SUBC> means 'REFINING' 'BORAX'.
```

Analysis of Variance for Scott Bond

Source	DF	SS	MS	F	P
rpm	2	1.012	0.506	1.37	0.352
borax	2	0.401	0.201	0.54	0.619
Error	4	1.479	0.370		
Total	8	2.893			

## Appendix C

### Raw Data of the Strength Properties in Relation to Filler Loading for GCC and PCC (Phase 2.0)

Table 43

Effect of Ash Content and Borax Addition on Strength Properties of GCC Filled Papers at 7500 Revolutions

Filler Load'g	Borax Add'n	TEA	Tear Index	Burst Index	Stiffness	Folding End.	Ash Content
(%)	(%)	kJ/m <sup>2</sup>	mNm <sup>2</sup> /g	kPam <sup>2</sup> /g	mN	(Log)	(%)
25	0.0	1.11	18.42	0.46	14.10	3.17	7.25
		1.47	21.44	0.44	23.67	3.26	7.00
		1.33	16.95	0.46	18.63	3.25	8.16
		1.40	18.53	0.50	15.11	3.21	8.00
		1.39	19.50	0.44	18.63	3.17	8.84
	0.5	1.12	14.37	0.42	24.68	3.20	7.85
		1.11	17.06	0.44	19.64	3.30	8.40
		0.75	17.00	0.40	35.26	2.87	9.09
		0.90	18.17	0.51	19.14	3.08	5.77
		1.34	17.22	0.47	21.15	2.96	7.32
	1.0	1.38	16.61	0.48	17.61	3.11	4.65
		1.20	16.68	0.42	13.60	3.23	5.27
		1.40	16.16	0.51	14.10	3.12	5.32
		1.14	16.49	0.47	14.61	2.91	6.54
		1.07	15.84	0.49	17.12	3.20	6.57
	1.5	0.97	16.75	0.43	12.59	3.13	7.23
		1.04	17.38	0.48	26.69	3.17	7.29
		1.31	18.07	0.43	20.15	3.04	7.98
		1.64	18.27	0.46	17.12	3.07	8.31
		0.88	17.25	0.44	22.16	3.28	8.15

Table 43 – Continued

Filler Load'g	Borax Add'n	TEA	Tear Index	Burst Index	Stiffness	Folding End.	Ash Content
(%)	(%)	kJ/m <sup>2</sup>	mNm <sup>2</sup> /g	kPam <sup>2</sup> /g	mN	(Log)	(%)
40	0.0	0.92	19.09	0.38	16.12	3.10	9.39
		0.78	19.11	0.31	12.59	3.10	9.41
		1.15	17.52	0.31	15.11	3.14	10.46
		0.70	16.40	0.31	16.12	3.06	10.79
		0.80	17.18	0.39	14.10	3.12	13.06
	0.5	1.23	16.00	0.42	19.64	3.23	8.12
		1.36	17.48	0.47	20.15	3.10	8.72
		1.45	17.93	0.44	14.10	3.16	9.51
		0.83	15.59	0.47	21.15	2.99	10.69
		1.30	17.80	0.42	17.12	3.12	11.53
	1.0	0.99	17.85	0.41	20.15	3.35	10.70
		1.25	24.75	0.40	24.68	3.37	10.47
		1.14	16.37	0.38	22.16	3.36	11.16
		0.83	17.37	0.39	20.65	3.04	11.32
		1.52	16.05	0.40	20.15	3.26	13.89
	1.5	0.85	16.10	0.35	20.65	3.18	12.35
		0.92	16.07	0.40	21.66	3.17	11.95
		0.72	16.42	0.34	17.63	2.99	13.13
		1.11	14.36	0.34	19.14	3.28	13.06
		1.13	17.84	0.42	16.62	2.95	8.94

Table 43 – Continued

Filler Load'g (%)	Borax Add'n (%)	TEA kJ/m <sup>2</sup>	Tear Index mNm <sup>2</sup> /g	Burst Index kPam <sup>2</sup> /g	Stiffness mN	Folding End. (Log)	Ash Content (%)
50	0.0	0.90	11.77	0.32	16.12	3.08	15.31
		0.90	10.05	0.37	13.60	3.02	17.14
		0.74	10.07	0.37	11.58	2.81	17.01
		0.75	9.66	0.31	16.62	2.83	18.70
		0.93	10.54	0.34	18.13	2.97	17.73
	0.5	0.96	10.21	0.32	17.63	2.93	16.86
		1.07	10.23	0.32	21.91	2.87	17.56
		0.90	10.51	0.29	26.44	2.87	17.09
		0.87	10.78	0.31	14.35	2.67	18.06
		0.70	9.74	0.33	17.63	2.43	17.80
	1.0	1.03	11.02	0.25	22.66	2.54	20.46
		1.04	10.62	0.29	17.63	3.09	20.37
		0.81	10.42	0.32	20.65	2.65	20.93
		0.63	10.64	0.30	19.14	2.67	20.37
		0.59	10.18	0.31	21.66	2.73	21.03
	1.5	1.10	11.31	0.26	30.72	2.57	24.46
		0.85	11.10	0.28	19.14	2.61	23.11
		0.67	10.74	0.28	24.18	2.42	23.01
		0.56	11.64	0.28	16.61	2.62	23.72
		0.73	11.89	0.27	26.69	2.83	24.02



Table 44

Effect of Ash Content and Borax Addition on Strength properties of PCC  
Filled Papers at 7500 Revolutions

Filler Load'g	Borax Add'n	TEA	Tear Index	Burst Index	Stiffness	Folding End.	Ash Content
(%)	(%)	kJ/m <sup>2</sup>	mNm <sup>2</sup> /g	kPam <sup>2</sup> /g	mN	(Log)	(%)
25	0.0	0.64	13.31	0.33	21.15	2.88	6.03
		0.60	11.37	0.39	14.10	2.68	6.81
		0.78	12.81	0.32	18.62	3.07	7.43
		0.84	10.00	0.37	17.12	3.11	7.71
		0.66	11.92	0.32	13.60	3.15	9.00
	0.5	0.70	11.60	0.36	33.74	3.06	9.27
		0.83	12.77	0.35	26.69	2.90	8.65
		0.86	11.75	0.37	28.71	2.93	9.41
		0.79	11.92	0.35	19.64	2.74	9.81
		1.29	11.96	0.37	30.72	3.12	10.16
	1.0	0.63	12.47	0.32	21.15	2.70	9.20
		0.66	10.85	0.36	26.69	2.39	11.51
		0.74	11.80	0.34	20.65	2.90	11.42
		0.74	12.09	0.34	14.10	2.86	11.68
		0.60	11.97	0.40	21.15	2.19	11.63
	1.5	0.90	12.26	0.29	31.73	2.75	13.37
		0.72	11.39	0.32	29.21	2.21	12.42
		0.96	12.37	0.29	28.20	2.67	14.05
		0.63	12.22	0.30	25.69	2.65	12.68
		1.20	13.03	0.27	28.20	2.80	14.30

Table 44 – Continued

Filler Load'g	Borax Add'n	TEA	Tear Index	Burst Index	Stiffness	Folding End.	Ash Content
(%)	(%)	$\text{kJ/m}^2$	$\text{mNm}^2/\text{g}$	$\text{kPam}^2/\text{g}$	mN	(Log)	(%)
40	0.0	0.75	10.11	0.31	38.28	2.66	14.70
		0.69	12.48	0.31	36.26	2.64	13.66
		0.75	12.14	0.29	39.79	2.82	13.60
		0.56	11.52	0.31	25.69	2.68	15.58
		0.86	12.83	0.26	30.72	2.22	16.21
	0.5	0.78	12.02	0.28	27.20	2.13	14.96
		0.59	12.72	0.24	29.72	2.16	19.43
		0.76	12.80	0.25	37.27	2.59	19.32
		0.78	12.01	0.25	37.02	2.04	20.87
		0.43	13.27	0.22	32.74	2.07	19.25
	1.0	0.40	12.56	0.25	24.18	1.96	15.18
		0.50	13.28	0.25	27.20	2.20	16.20
		0.46	12.57	0.22	30.22	2.26	16.05
		0.52	12.61	0.23	28.20	2.28	16.93
		0.55	12.85	0.21	25.18	2.21	18.97
	1.5	0.82	12.93	0.21	28.20	1.81	18.76
		0.52	12.37	0.24	24.18	2.47	22.18
		0.54	11.52	0.20	32.14	1.94	21.84
		0.52	12.80	0.19	25.69	1.77	22.24
		0.44	13.60	0.18	23.67	1.49	24.30

Table 44 – Continued

Filler Load'g (%)	Borax Add'n (%)	TEA kJ/m <sup>2</sup>	Tear Index mNm <sup>2</sup> /g	Burst Index kPam <sup>2</sup> /g	Stiffness mN	Folding End. (Log)	Ash Content (%)
50	0.0	0.85	16.10	0.22	33.74	2.10	22.71
		0.47	14.14	0.20	23.67	2.07	24.19
		0.45	13.39	0.19	30.22	1.68	23.53
		0.51	13.45	0.20	30.72	1.76	26.45
		0.47	13.29	0.21	18.13	1.73	26.27
	0.5	0.57	13.76	0.18	32.23	1.61	28.24
		0.51	13.66	0.17	33.74	1.20	28.05
		0.41	13.24	0.19	26.69	1.56	28.01
		0.37	12.23	0.17	27.95	1.32	29.44
		0.54	12.71	0.18	33.24	1.49	28.51
	1.0	0.69	14.94	0.18	27.20	1.85	23.17
		0.35	14.21	0.21	23.67	1.76	22.39
		0.47	13.24	0.20	24.68	1.64	22.40
		0.36	14.34	0.18	19.14	1.41	24.85
		0.48	13.41	0.19	22.66	1.45	27.70
	1.5	0.56	13.23	0.20	40.29	1.79	22.68
		0.35	12.19	0.18	40.29	1.64	27.38
		0.46	12.13	0.17	29.21	1.72	28.05
		0.40	13.12	0.15	36.26	1.69	29.66
		0.44	13.64	0.15	25.69	1.48	30.96

**Appendix D**  
**Test Method to Measure Alkalinity**

### Alkalinity Measurement

The alkalinity of pulp slurry with filler and AKD solution was measured by modifying the TAPPI test method T620 cm-83 section #6. Pulp slurry was filtered and 100 mL of the filtrate containing the filler or AKD was titrated with 0.2N  $\text{H}_2\text{SO}_4$ . Two drops of methyl orange were added to the sample. At the end point the solution turned from yellow to a faint pink. The amount of  $\text{H}_2\text{SO}_4$  required to produce a change in color was recorded and multiplied by a factor of 100. The result is reported in parts per million (ppm).

## Appendix E

### Effect of Borax as Surface Sizing on Strength Properties of Paper (Phase 4.0)

Table 45

Effect of Borax as Surface Sizing Agent on Strength Properties

Borax (%)	Refining (revolution)	TEA (kJ/m <sup>2</sup> )	Tear Index (mNm <sup>2</sup> /g)	Log Folds	Stiffness (mN)
0	0	0.25	18.67	0.87	2.08
	5000	1.58	15.67	3.09	6.77
	7500	1.86	20.42	2.98	4.72
	8500	1.44	20.57	3.10	4.03
0.5	0	0.20	17.66	0.57	4.41
	5000	1.32	20.14	3.30	6.34
	7500	1.60	20.26	3.09	5.76
	8500	1.14	20.31	3.08	4.11

Table 45 – Continued

Borax (%)	Refining (revolution)	TEA (kJ/m <sup>2</sup> )	Tear Index (mNm <sup>2</sup> /g)	Log Folds	Stiffness (mN)
0	0	0.19	17.10	0.50	3.90
	5000	1.24	19.80	2.90	4.40
	7500	1.31	21.10	3.10	5.50
	8500	1.02	19.50	3.10	5.20
0.5	0	1.26	18.61	3.07	4.60
	5000	1.19	19.34	2.93	4.80
	7500	1.34	19.91	3.15	4.92
	8500	1.26	19.91	3.10	3.83



## BIBLIOGRAPHY

- Alberts, R. M., "Alkyl Ketene Dimer Sizes," Tappi Sizing Short Course, 1987.
- Au, C.O., and Thorn, I., "Application of Wet-End Paper Chemistry," Published by Chapman and Hall, New York, 1995, p. 139.
- Bartz, W. J., Darroch, M.E., and Kurrle, F. L., "Alkyl Ketene Dimer Sizing Reversion and Efficiency in Papers Filled With Calcium Carbonate," Proceedings of 1994 Papermakers Conference Held at San Francisco, CA., Tappi Press, Atlanta, GA, p. 173.
- Binotto, A. P., and Nicholls, G. A., "Correlation of Fiber Morphological Variation and Wet Mat Compressibility of Loblolly Pine Bleached Kraft Pulp," Tappi Journal, 60(6): 91 (1977).
- Bridge, N.K., and Hamer, R. J., "Beating and Basic Fiber Properties," Paper Technology, 18(2): 37 (1977).
- Campbell, W.B., "The Mechanism of Bonding," Tappi Journal, 42(12): 999 (1959).
- Casey, J. P., "Pulp and Paper Chemistry and Chemical Technology," 2<sup>nd</sup> Ed., Interscience, New York, (1960), p. 713
- Eaton, C. K., and Janes, R. L., "The Effect of Internal Sizing on Paper Deterioration," Proceedings of 1992 Papermakers Conference Held at Nashville, TN., Tappi Press, Atlanta, GA, p. 197.
- Eissa, Y.Z., Naito, T., Usuda, M., and Kadoya, T., "Flow Resistance of Rotating Dilute Fiber Suspensions Role of Fundamental Research in Papermaking", Vol 1. British Paper and Board Maker's Association, p. 403 (1983).
- Fairchild, G. H., "Increasing the Filler Content of PCC-Filled Alkaline Papers," Tappi Journal, 75(8): 85 (1992).
- Hadgon, T.K., "Fiber-Liquid Interaction," Tappi Sizing Short Course 1997.
- Janes, R.L., "Fiber Characteristics," In, Stock Preparation Short Course, Tappi Press, Atlanta, GA, p. 3 (1992).

- Jokinen, O., and Ebeling, K., "Flocculation Tendency of Papermaking Fibers", *Paperi Ja Puu* 67(5): 317 (1985).
- Kerekes, R. J., "Pulp Flocculation in Decaying Turbulence: A literature Review", *J. Pulp Paper Science* 9 (7): TR86 (1983).
- Kibblewhite, R. P., "Effect of Beating on Fiber Morphology and Fiber Surface Structure," *Appita*, 26(3): 196 (1972).
- Lindström, T., and Eklund, D., "Paper Chemistry – An Introduction," Published by DT Paper Science Publications, Grankulla, Finland 1991, p. 215.
- Marton, J., "Paper Chemistry", Roberts, J. C., 2<sup>nd</sup>, Ed., Chapman & Hall, New York p. 83 (1996).
- Manson, D.W., "Alkaline Papermaking : A Tappi Press Anthology of Published Papers," Atlanta, GA, (1992).
- Montgomery, D. C., "Design and Analysis of Experiments" 3<sup>RD</sup> Ed., John Wiley and Sons, New York, (1991), p. 479.
- Neimo, L., "Paper Chemistry" Published in Cooperation With the Finnish Paper Engineers' Association and Tappi, (1999). p 151.
- Novak, R.W., and Rende, D.S., "Size Reversion in Alkaline Papermaking," *Tappi Journal* 76(8):117(1993).
- Paavilainen, L., "Bonding Properties of Softwood Sulphate Pulp Fibers," *Paperi Ja Puu*, 76(1), (1994).
- Paavilainen, L., "Wet Fiber Flexibility and Collapsibility of Softwood Sulphate Pulp Fibers," *Paperi Ja Puu*, 75, p. 9, (1993).
- Parsons, S. R., "Optical Characteristics of Paper as a Function of Fiber Classification," *Tappi Journal* (12), P 314 - 322 (1942).
- Penny A. P. "On the Mechanisms of AKD Sizing and Size Reversion," Proceedings of 1991 Tappi Papermakers Conference held at Seattle, WA, Tappi Press, Atlanta, GA, p. 415.
- Ratliff, F.T., "The Possible Correlation between Hemicelluloses and the Physical Properties of Bleached Kraft Pulps," *Tappi Journal*, 32(8): 357 (1949).

Retulainen, E. and Ebeling, K., "Fiber-Fiber Bonding and Ways to Characterize bond strength," *Appita*, 46(7): 282 (1993).

Roberts, J.C, and Garner, D. N., "The Mechanism of Alkyl Ketene Dimer Sizing of Paper, Part 1," *Tappi Journal* 68(4):118(1985).

Scott, W.E., "Principles of Wet End Chemistry," 2<sup>nd</sup> Ed., TAPPI Press, Atlanta, GA, p. 49 (1996).

Swanson, J.W. and Jones, E.J., "Factors Which Affect Strength Properties of Paper," *Pulp and Paper Canada*, 63(5): T251 - T258 (1962).