Institute of Paper Science and Technology
Atlanta, Georgia

ANNUAL PROGRAM REVIEW

MECHANICAL PULPING AND BLEACHING PROJECT ADVISORY COMMITTEE

SLIDE MATERIAL

March 24, 1995

INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY
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- to sustain an international position of leadership in dynamic scientific research which is participated in by both students and faculty and which is focused on areas of significance to the pulp and paper industry; and

- to contribute to the economic and technical well-being of the nation through innovative educational, informational, and technical services.

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Fundamentals of Brightness Reversion
FO14
Annual Review

A.J. Ragauskas

Fundamentals of Brightness Reversion

Program Objective: Investigate the fundamental reactions which contribute to brightness reversion of mechanical pulps

Design new technologies which will halt or significantly hinder photoyellowing of high-yield pulps

Research Approach

Design new additive treatments for high-yield pulps

- UV absorbers
- Radical Scavengers

Photo-Stabilization Agents Available For Retarding Retarding Brightness Reversion of Mechanical Pulps

UV-absorbers: Benzophenone, Triazoles, Substituted Dienes

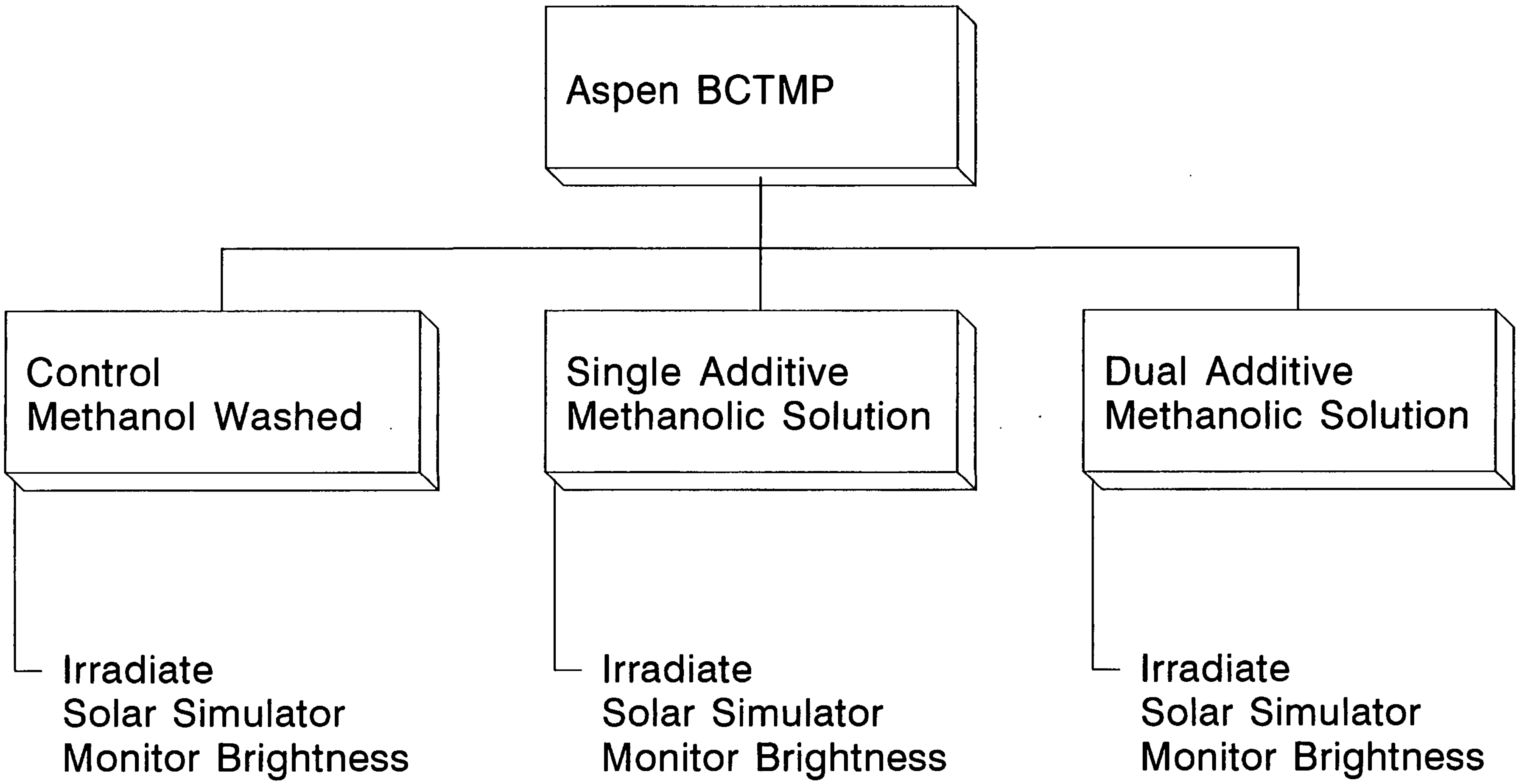
Radical Scavengers: Ascorbic Acid, Thiols, Disulfides

Polymers: Polyethylene glycol, Polyvinylpyrrolidone

Additives have demonstrated favorable photostabilization properties

Question: Do additives have any synergistic interactions?

Experimental Approach



Examination of Synergistic Interactions

Additives Examined To-date

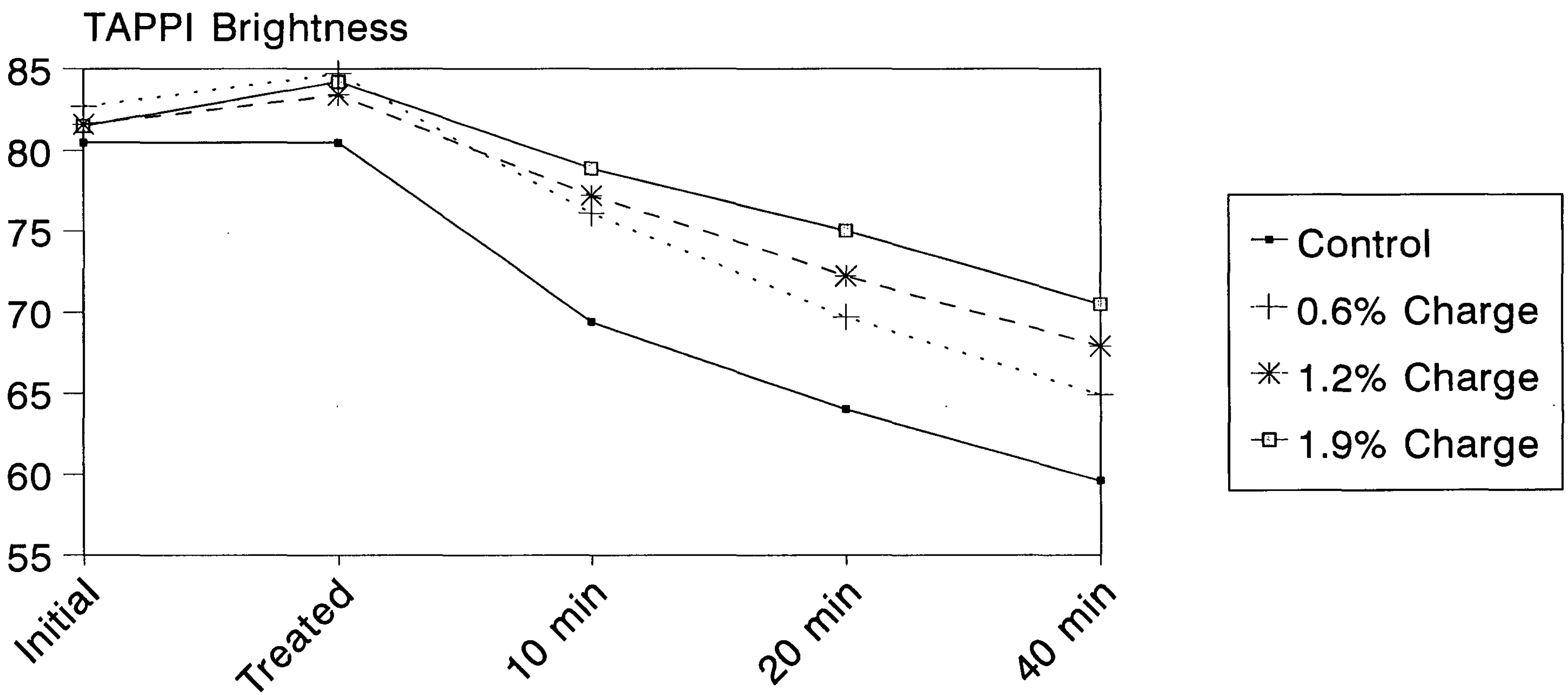
UV: 2,4-dihydroxybenzophenone, 5-phenylpenta-2,4-dienoic acid

Radical Scavengers: Ascorbic Acid, Ethylene glycolbisthioglycolate
3,3'-Dithiopropionic acid

Pulp: Hardwood & Softwood BCTMP

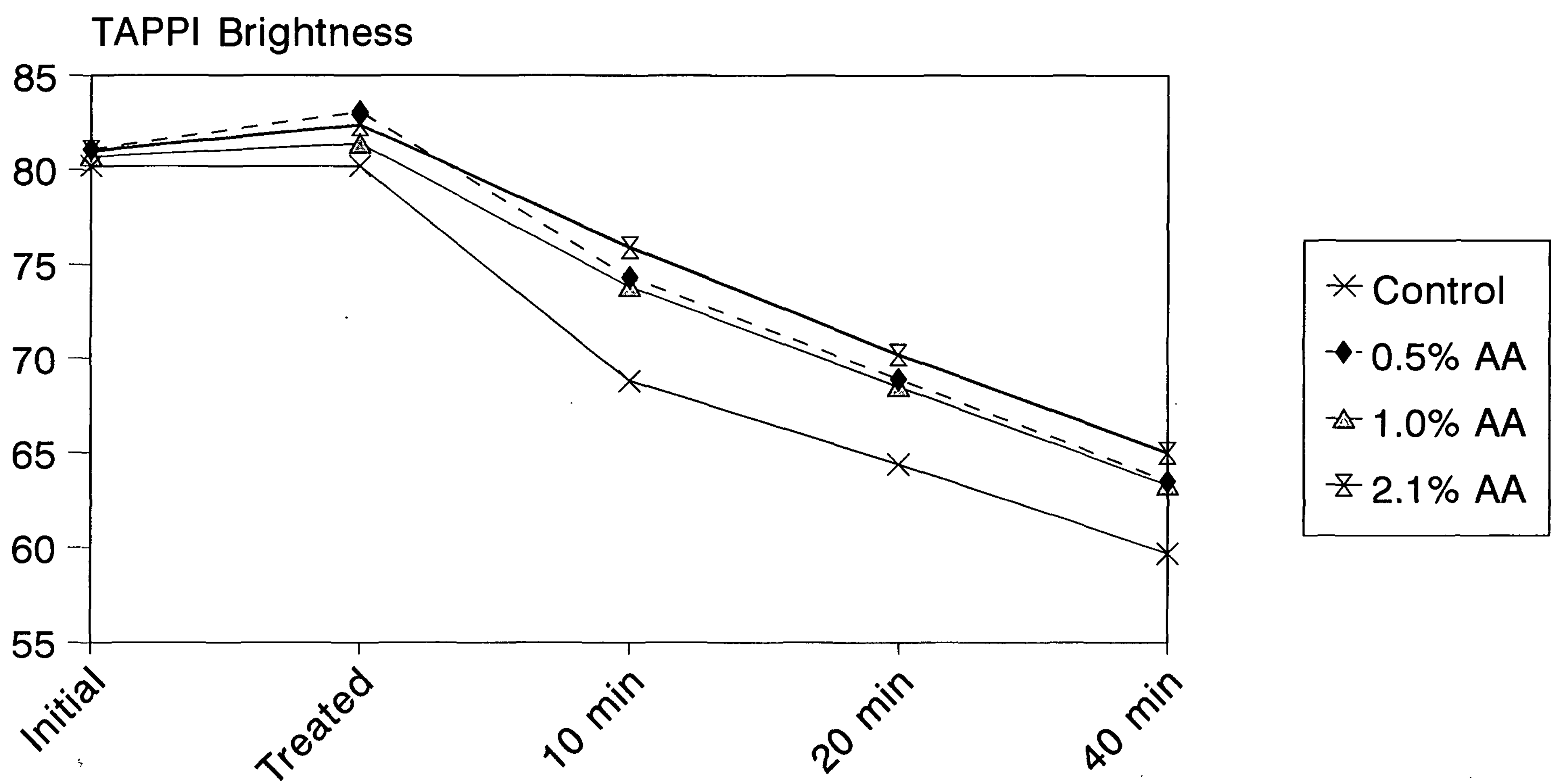
Hardwood BCTMP Reversion Studies

Reversion Properties BCTMP Impregnated With Ethylene Glycolbisthioglycolate

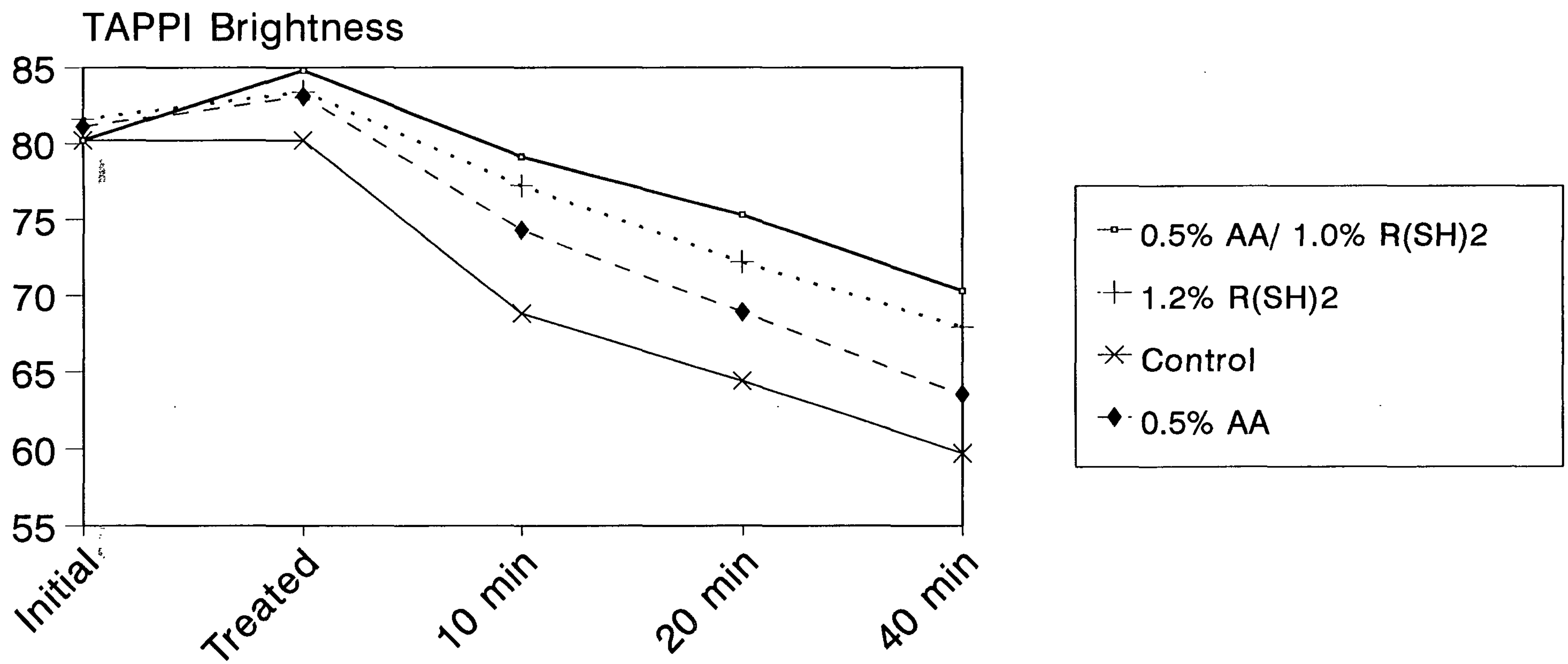


Note: Additive stabilization properties are not linearly related to the application level

Reversion Properties BCTMP Impregnated With Ascorbic Acid

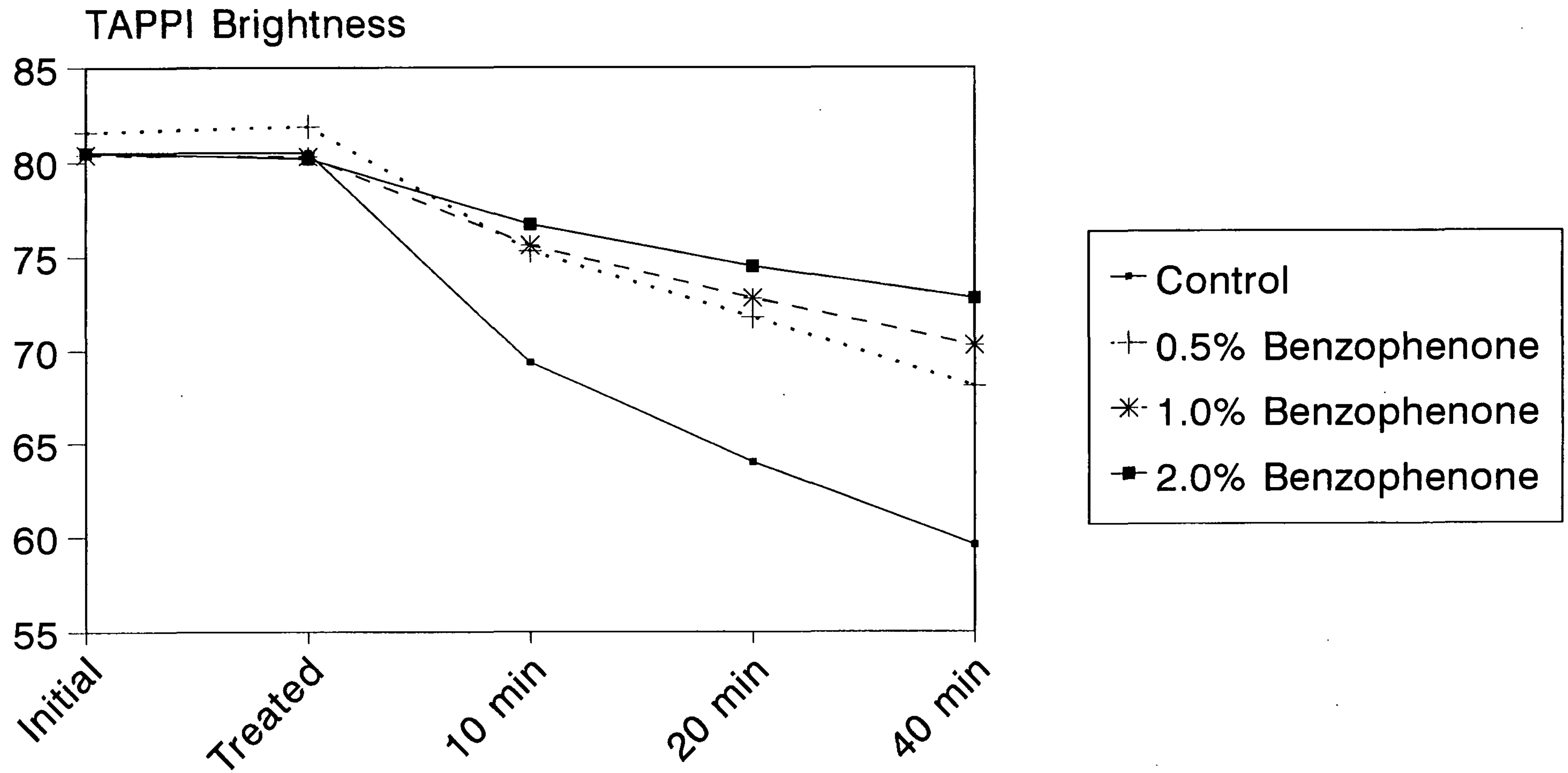


Reversion Properties BCTMP Impregnated With
Ascorbic Acid & Ethylene Glycolbisthioglycolate

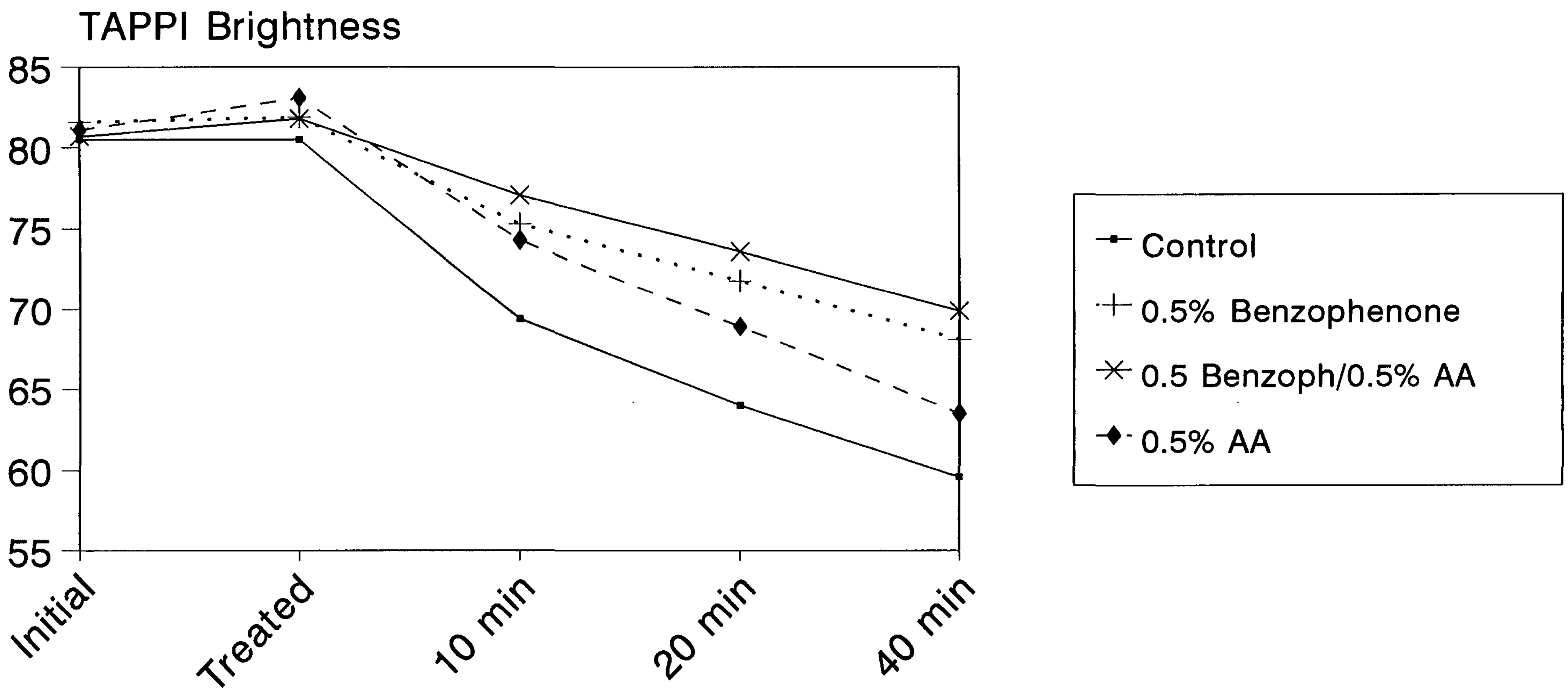


Need > 2% R(SH)2 or AA to get comparable reversion properties

Reversion Properties BCTMP Impregnated With 2,4-Dihydroxybenzophenone

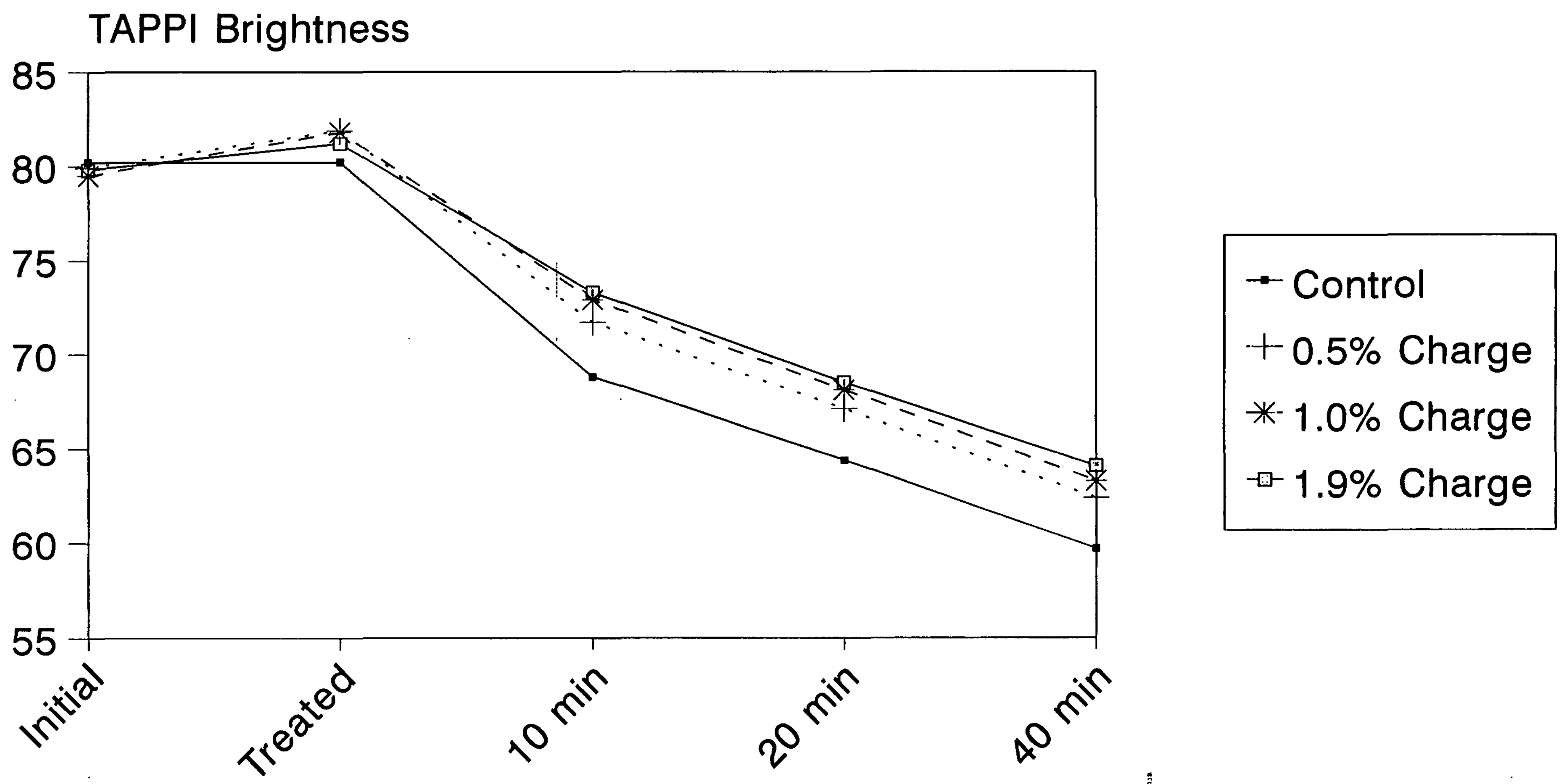


Reversion Properties BCTMP Impregnated With Benzophenone and Ascorbic Acid

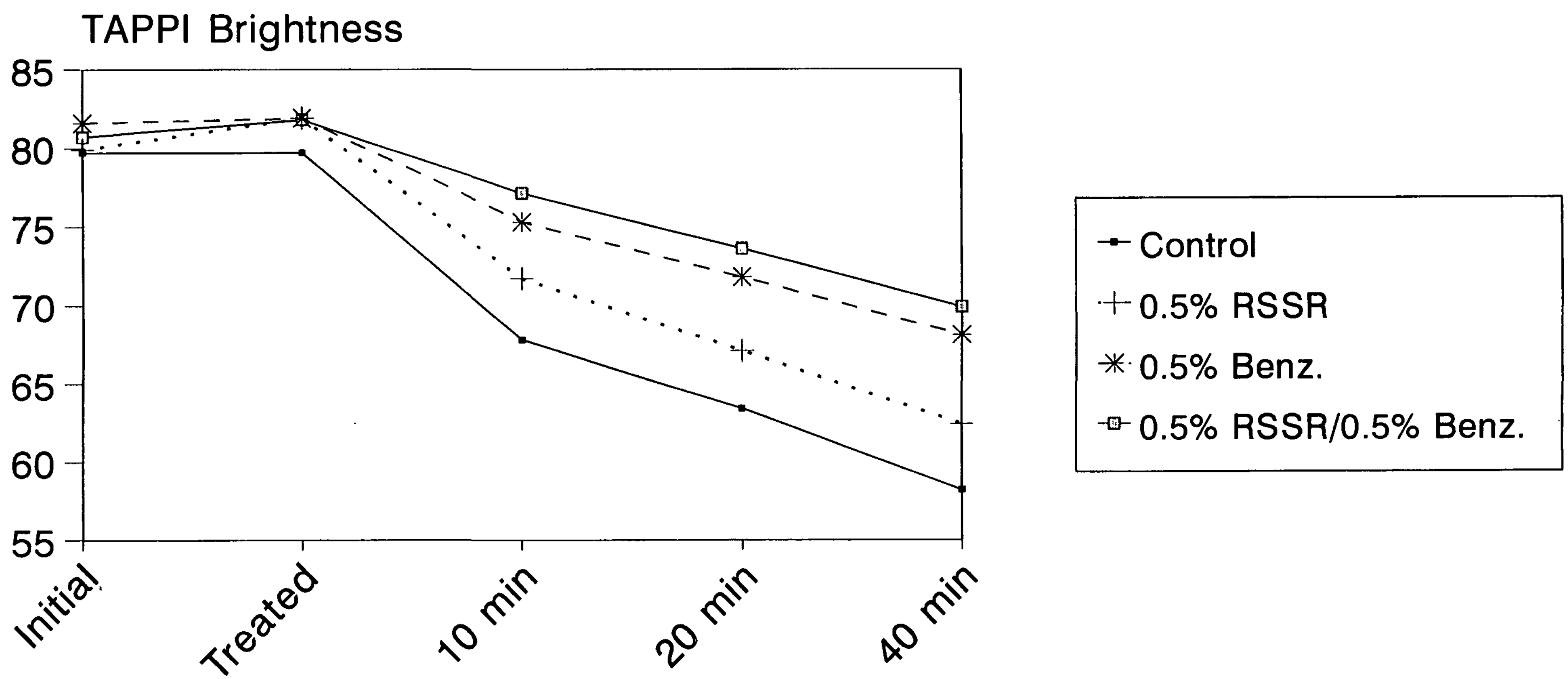


To get comparable levels of stabilization with AA need > 2%, with Benz. need > 1%

Reversion Properties BCTMP Impregnated With 3,3'-Dithiodipropionic Acid



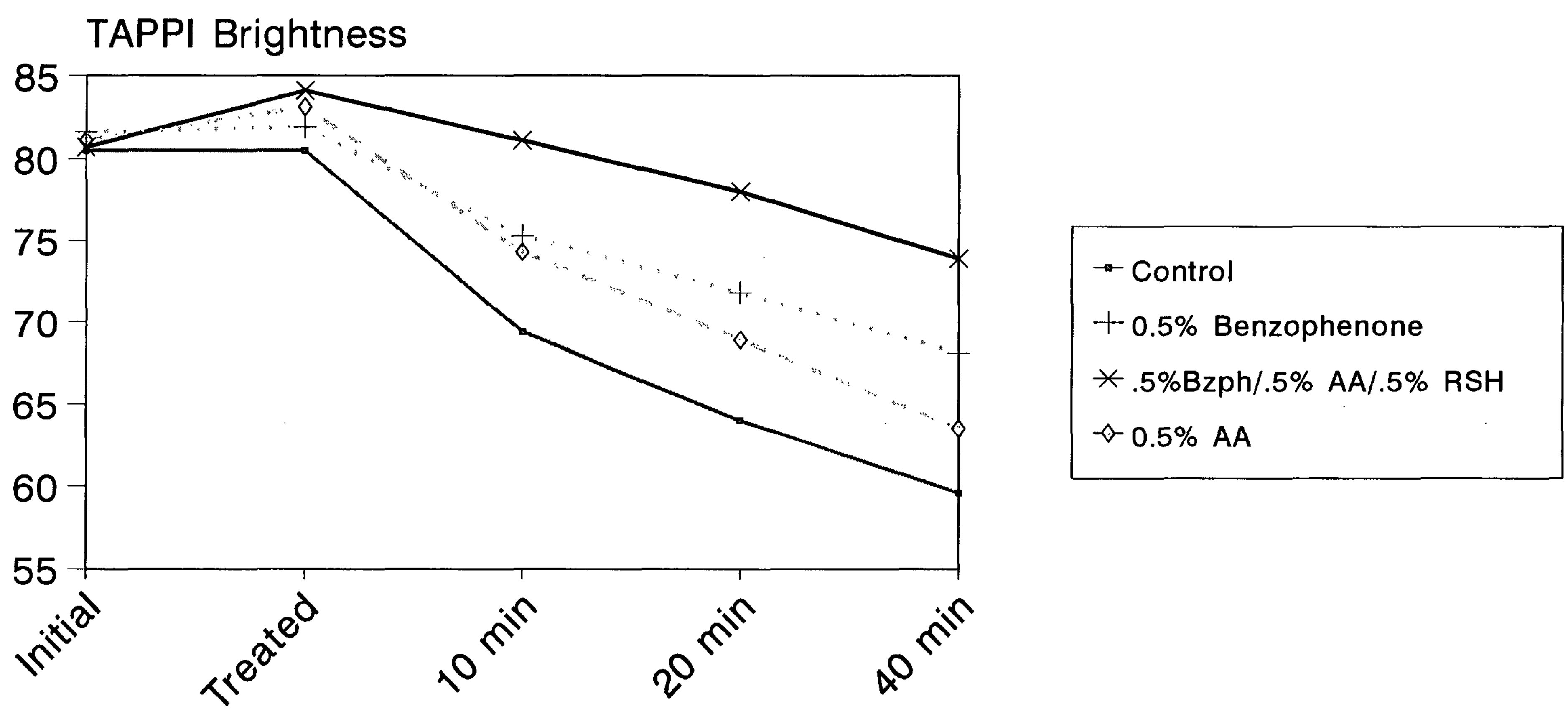
Reversion Properties BCTMP Impregnated With
2,4-Dihydroxybenzophenone & 3,3'Dithiodipropionic acid



Need > 2% RSSR or ca 1% Benz. to get an equivalent photostabilzation effect

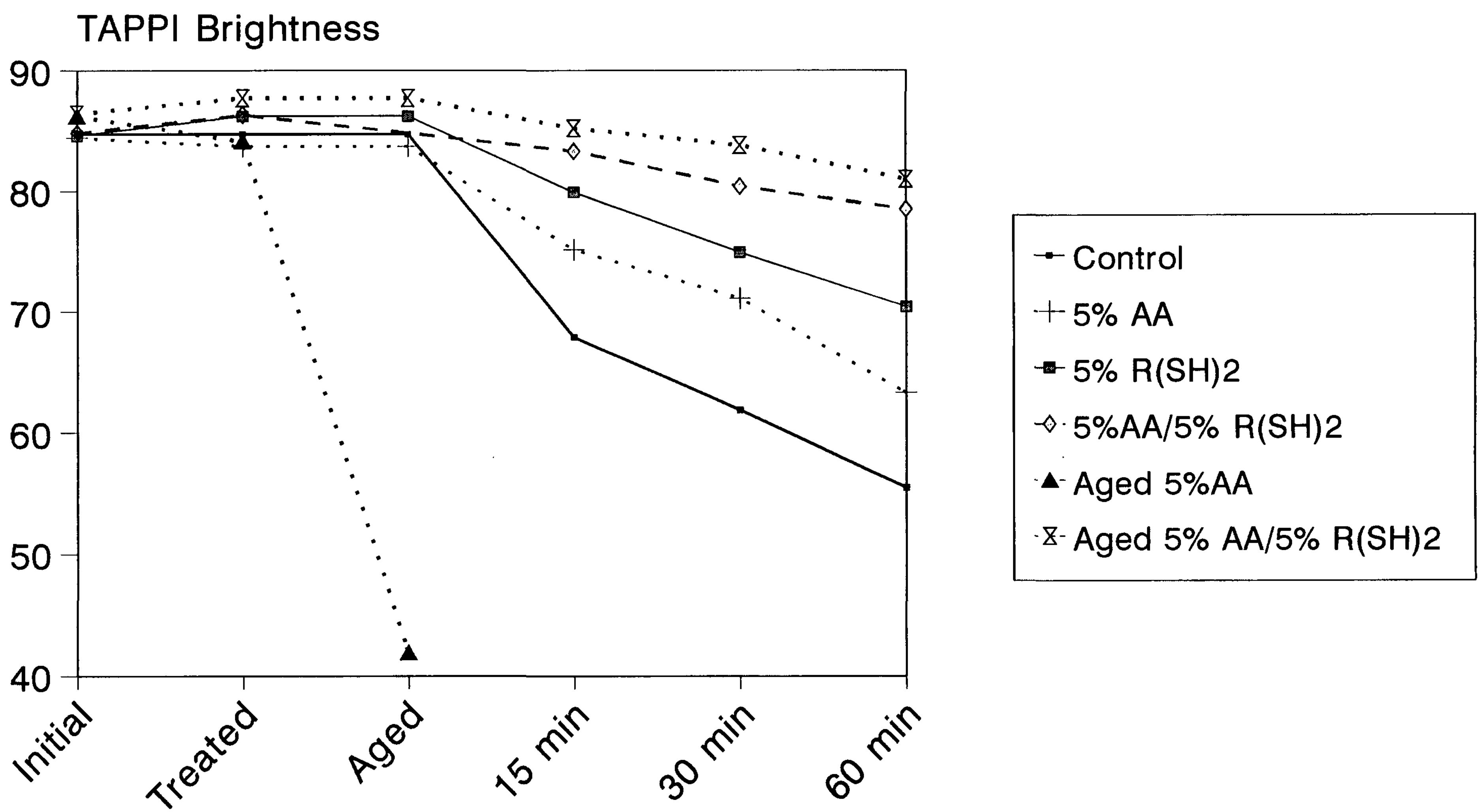
Reversion Properties BCTMP Impregnated with

Benzophenone/Ascorbic Acid/Ethylene Glycolbisthioglycolate



To get comparable levels of stabilization with AA, Benzoph., thiol need >> 2%

Reversion Properties For Impregnated BCTMP Handsheets



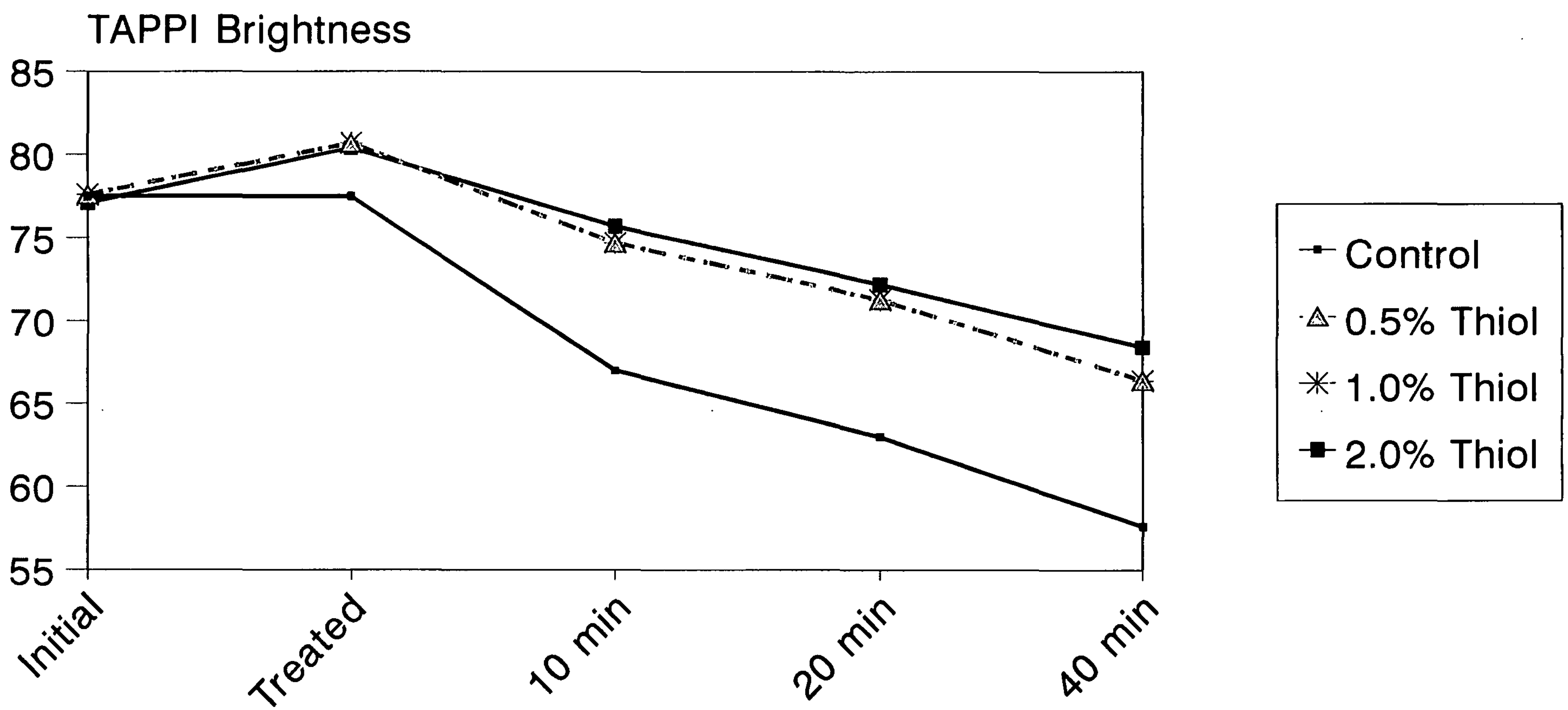
Absorption and Scattering Data for Selected Hardwood BCTMP Testsheets Impregnated with Ethylene Gylcol Bisthioglycolate or 2,4-dihydroxybenzophenone

	Application Level(%)	Absorption Coefficient 0	10 min	40min	Scattering Coefficient 0	10min	40 min
Control	--	0.20	0.45	0.86	34	33	33
Thiol	0.5	0.19	0.31	0.77	34	35	34
	1.0	0.16	0.25	0.59	35	35	34
	2.0	0.17	0.22	0.46	34	34	33
Benzophenone	0.5	0.21	0.36	0.69	34	34	33
	1.0	0.22	0.36	0.57	35	35	33
	2.0	0.22	0.32	0.53	35	35	33
Thiol/Benzophenone	0.5/0.5	0.16	0.24	0.62	34	35	34

NOTE: Scattering coefficient does not change during irradiation.

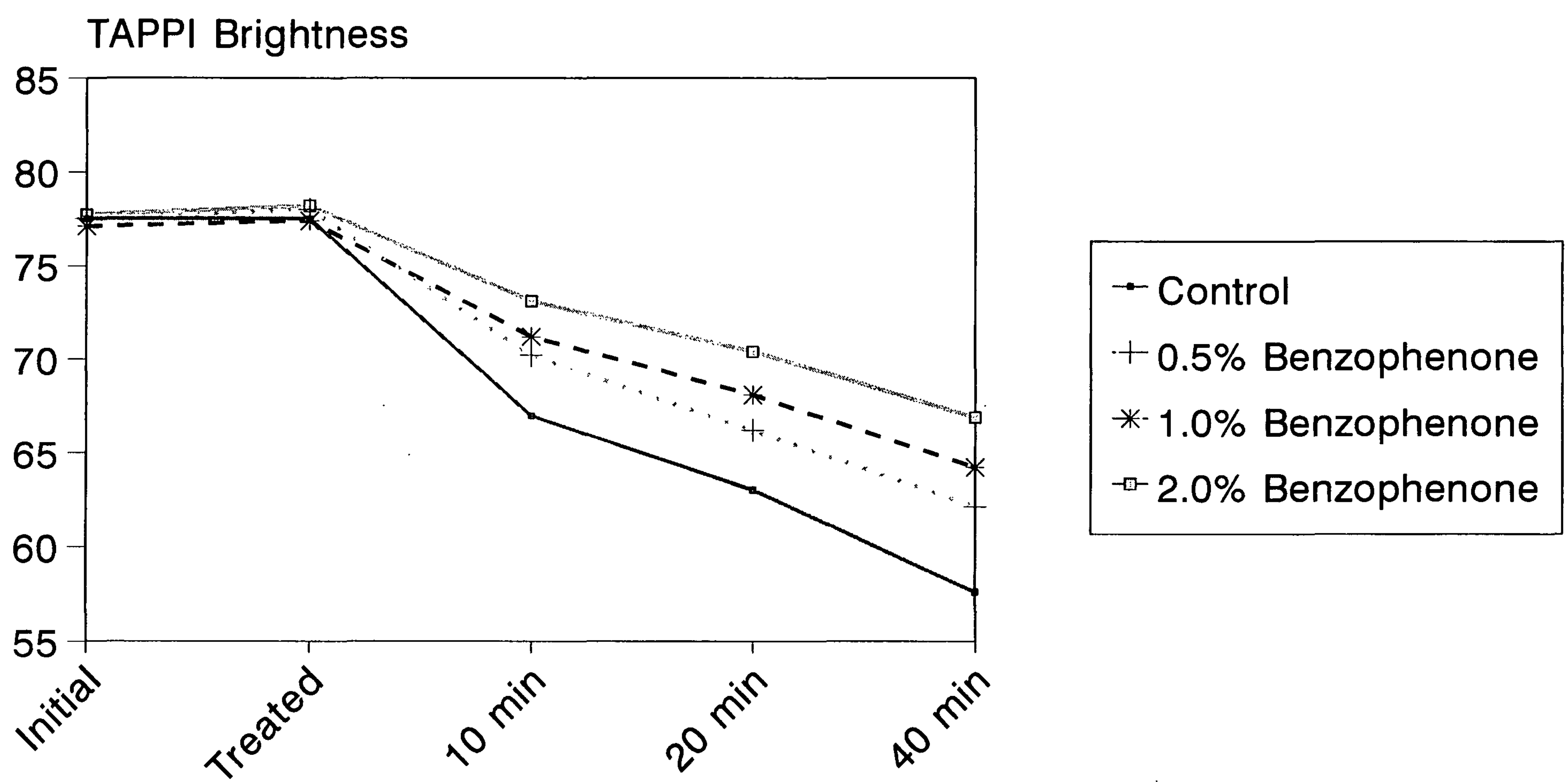
Softwood BCTMP Reversion Studies

Reversion Properties BCTMP Impregnated With Ethylene Glycolbisthioglycolate

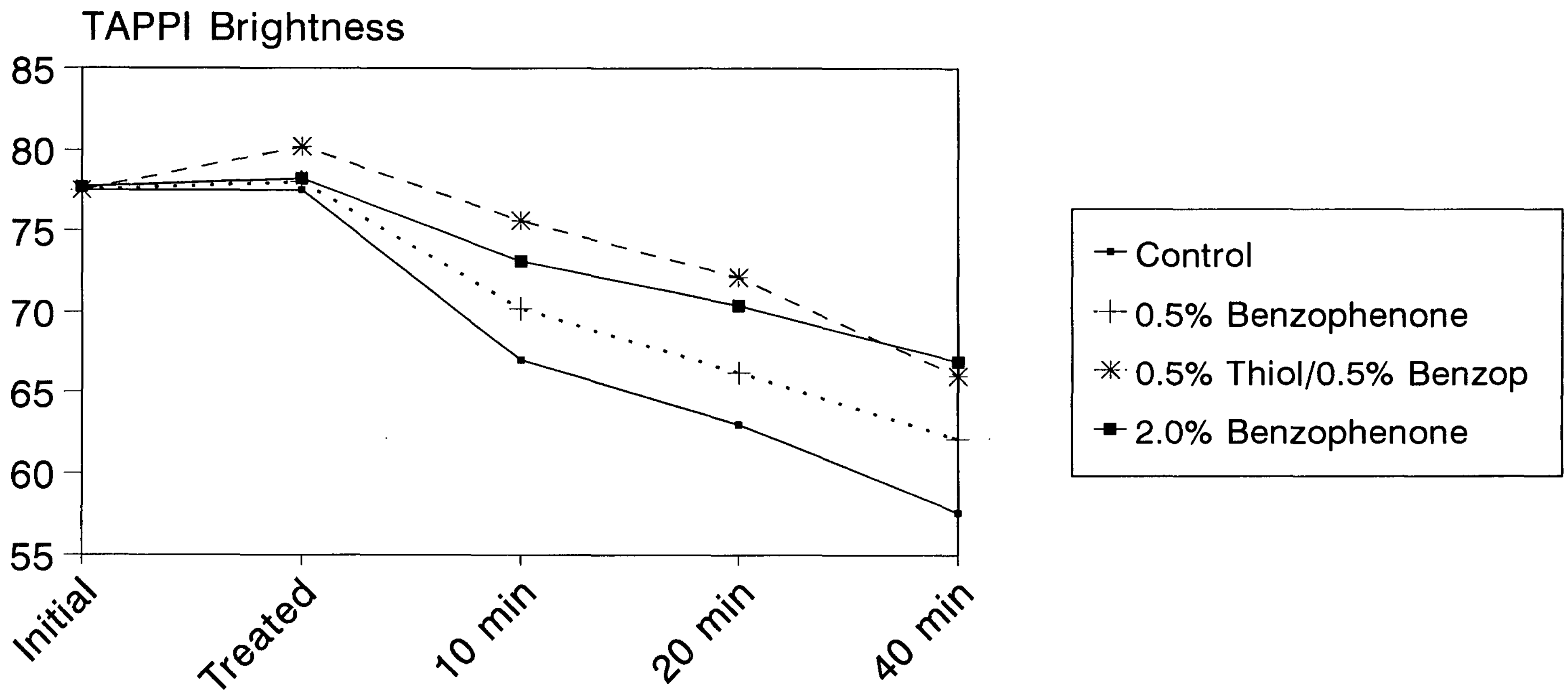


Softwood appears to be more responsive to thiol additive

Reversion Properties BCTMP Impregnated With 2,4-Dihydroxybenzophenone



Reversion Properties BCTMP Impregnated With
2,4-Dihydroxybenzophenone & Ethylene Glycolbisthioglycolate



Levels of photostabilization with 0.5% thiol & 0.5% benzophenone
> than 2% of single additive

Conclusions

- Photostabilization effects are not linear with additive application level
- Application of two or more additives can yield improved photostabilization properties for hardwood and softwood BCTMP
- For the additives examined improved reversion properties were noted for (thiol/benzophenone), (thiol/ascorbic acid) (ascorbic acid/benzophenone), & thiol/ascorbic acid/benzophenone
- Additive combinations can also enhance thermal reversion properties

Future Directions

- Improve the performance of the UV absorbers
- Develop new thiol additives
- Examine differing paper furnishes
- Optimize additive mixture effect

Acknowledgements

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L. Allison

Member Companies of IPST
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United States Department of Agriculture

Project F012

Improved Mechanical Pulping From Southern Pines

Alan W. Rudie
Blair Carter
Alex Shakhet
John Griffey (Bowater)

Objective:

Improve the performance of mechanical pulping processes.

Goal:

An improved understanding of the performance limitations in mechanical and chemimechanical pulping. The emphasis is on problem softwoods such as the dense pines.

AWR: PAC, 3/95

Prior work:
Development of a chlorite holopulping technique and test protocol for evaluating mechanical pulp fiber strength.

Current work:
Evaluation of seasonal changes in TMP strength.

Future Work:
Evaluation of wood breakdown patterns and earlywood and latewood fiber quality within the first stage of refining.

AWR: PAC, 3/95

Chlorite Holopulping

- Using a room temperature acetate/acetic acid buffered chlorite process. Each chlorite treatment takes 4 to 7 days.
- Each chlorite treatment is followed by a water wash (1 day) and an alkaline extraction (1 day).
- Four to five treatments are required to obtain usable pulp samples with minimal beating.

AWR: PAC, 3/95

MacMillan Bloedel
Impressafiner™ Evaluation

	Z span	Tensile	Tear	Fiber Length
Mill A				
Chips	148	103	8.4	1.76
Discharge	145	94	8.3	1.65
% Loss	2	9	1	6
Mill B				
Chips	148	104	8.2	1.81
Discharge	135	90	7.6	1.47
% Loss	9	14	7	19

AWR: PAC 3/95

Seasonal Strength Changes in TMP

- Southern TMP mills have noticed a seasonal variation in pulp strength.
- The project task is to use the chlorite holopulping process to help identify the source of the low strength in winter.

AWR: PAC, 3/95

Seasonal Strength Project

- The Bowater Inc, Calhoun mill agreed to participate in the project.
- Samples were to be collected over all four seasons and shipped to IPST.
- Samples were to be collected from the chip washer, primary refiner, secondary refiner and latency chest of one line.
- In each sample period, one group of samples was to be collected representing current mill production. A second set was collected under controlled conditions established with the first set of samples.

AWR: PAC, 3/95

What Question Will be Answered

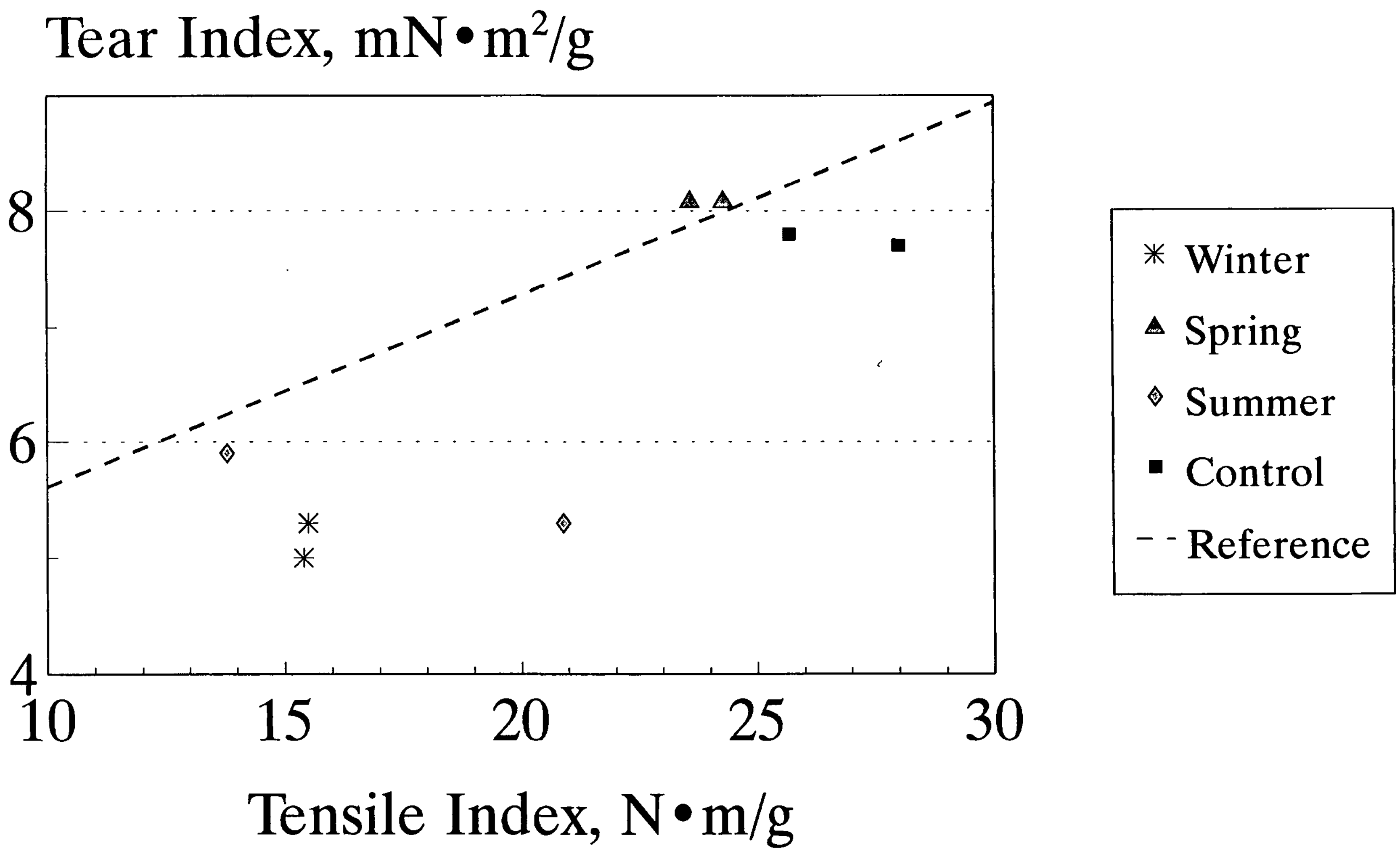
- Is the wood chip fiber strength low during periods of low pulp strength.
- Does fiber strength deteriorate more rapidly in refining during periods of low pulp strength.
- Where do we focus our attention to eliminate the seasonal strength problems, if fiber strength/damage is contributing to the problem.

What About Fiber Bond Strength?

- Fiber bond strength is the main strength parameter in TMP.
- **The project approach can only answer bond strength issues by eliminating fiber strength as a cause of low strength.**
- Bond strength development, and fiber strength may be correlated.

AWR: PAC,3/95

Pulp Tear/Tensile Relationship



AWR: PAC, 3/95

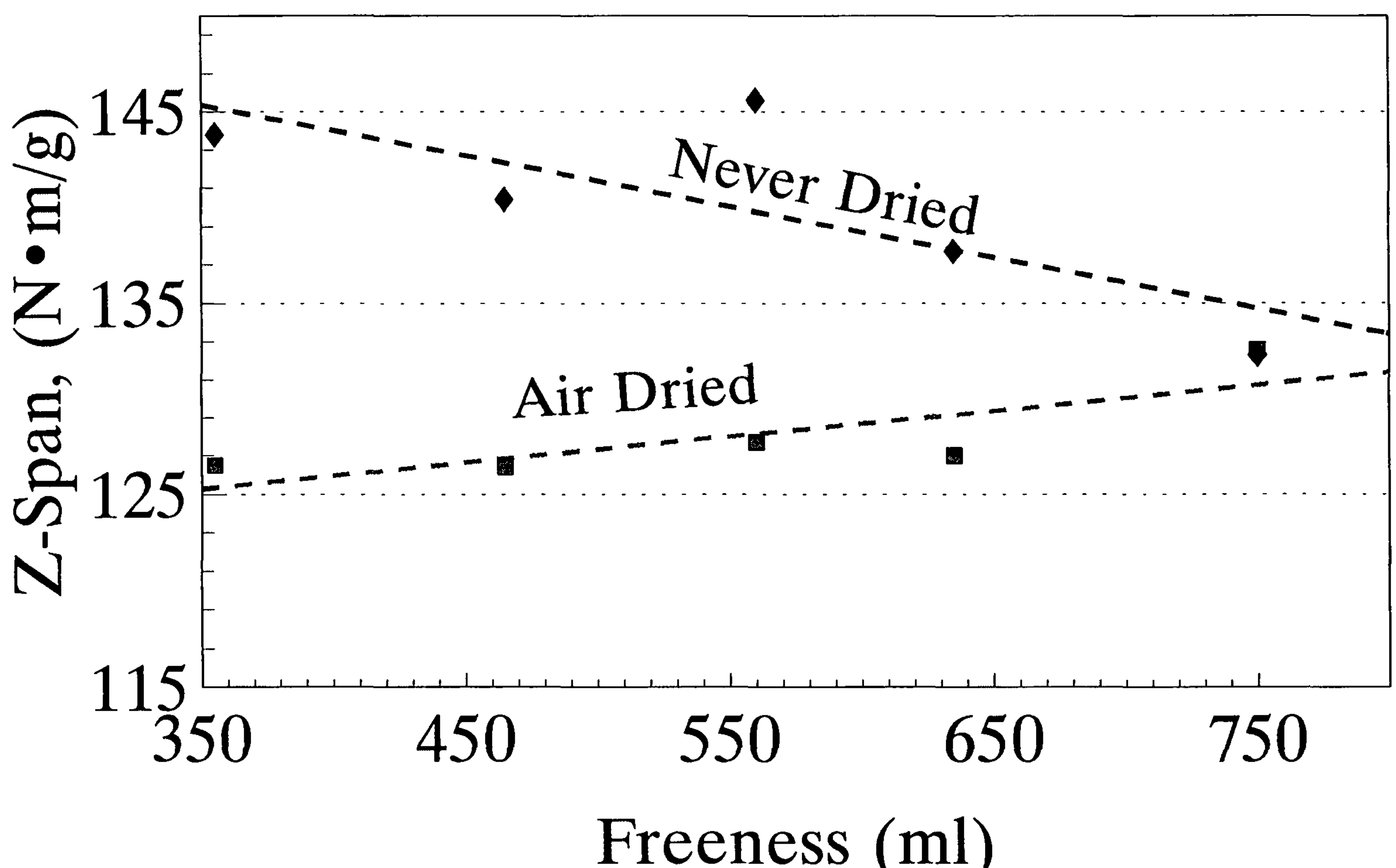
The Control

- The fall sample was delayed to collect a second low strength winter sample.
- The mill failed to experience a period of low strength in the winter of 93/94.
- A sample was finally collected in February 94 and used as a control wood sample.
- The "control" wood chips were used to prepare a large holopulp sample to evaluate the influence of number of treatment cycles, air drying, and pulp beating.

AWR: PAC, 3/95

Zero Span Tensile

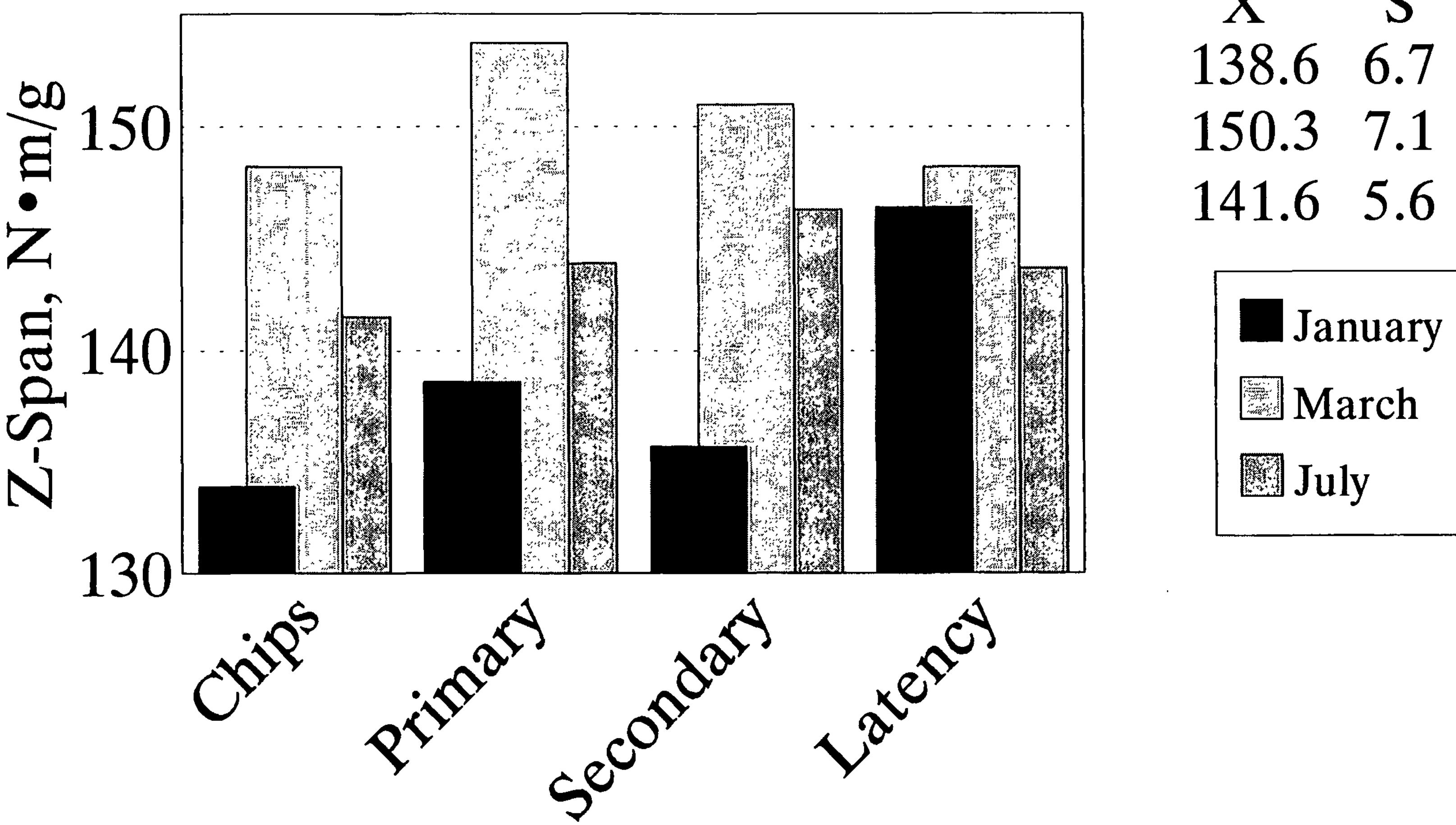
PFI Beater Data



AWR: PAC, 3/95

Zero Span Tensile

Normal Mill Production



$\overline{X}$	S
138.6	6.7
150.3	7.1
141.6	5.6

AWR: PAC, 3/95

Zero Span Tensile Tests

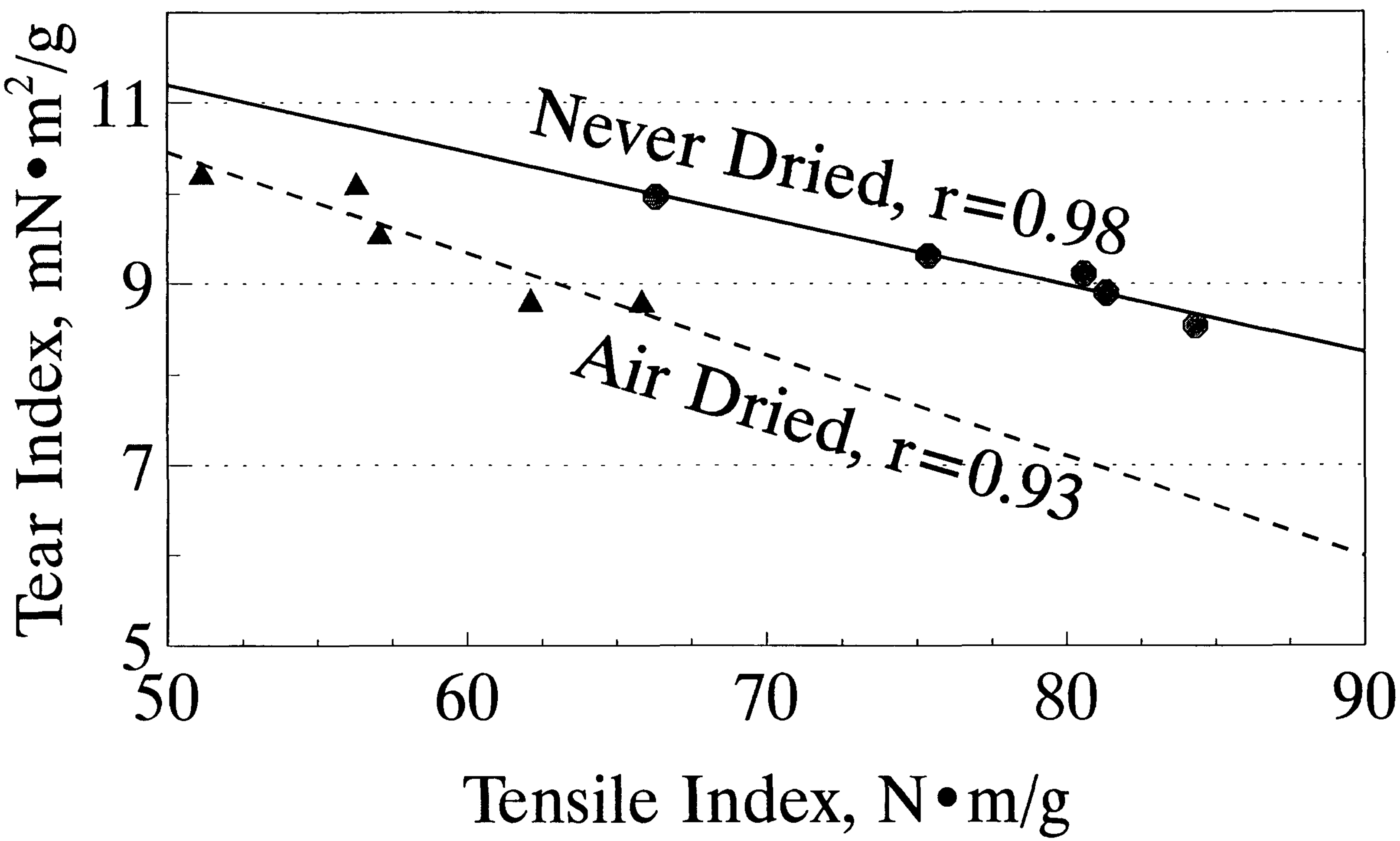
Paired *t*-tests

	WinN	WinC	SprN	SprC	SumN	SumC
Z-Span	139	139	150	152	142	144
WinN	0	-0.218	**3.230**	**3.668**	0.621	1.627
WinC	-0.218	0	**3.693**	**4.130**	0.912	**2.059**
SprN	**3.230**	**3.693**	0	0.526	**-2.706**	**-1.918**
SprC	**3.668**	**4.130**	0.526	0	**-3.169**	**-2.425**
SumN	0.621	0.912	**-2.716**	**-3.169**	0	0.994
SumC	1.627	**2.059**	**-1.918**	**-2.425**	0.994	0

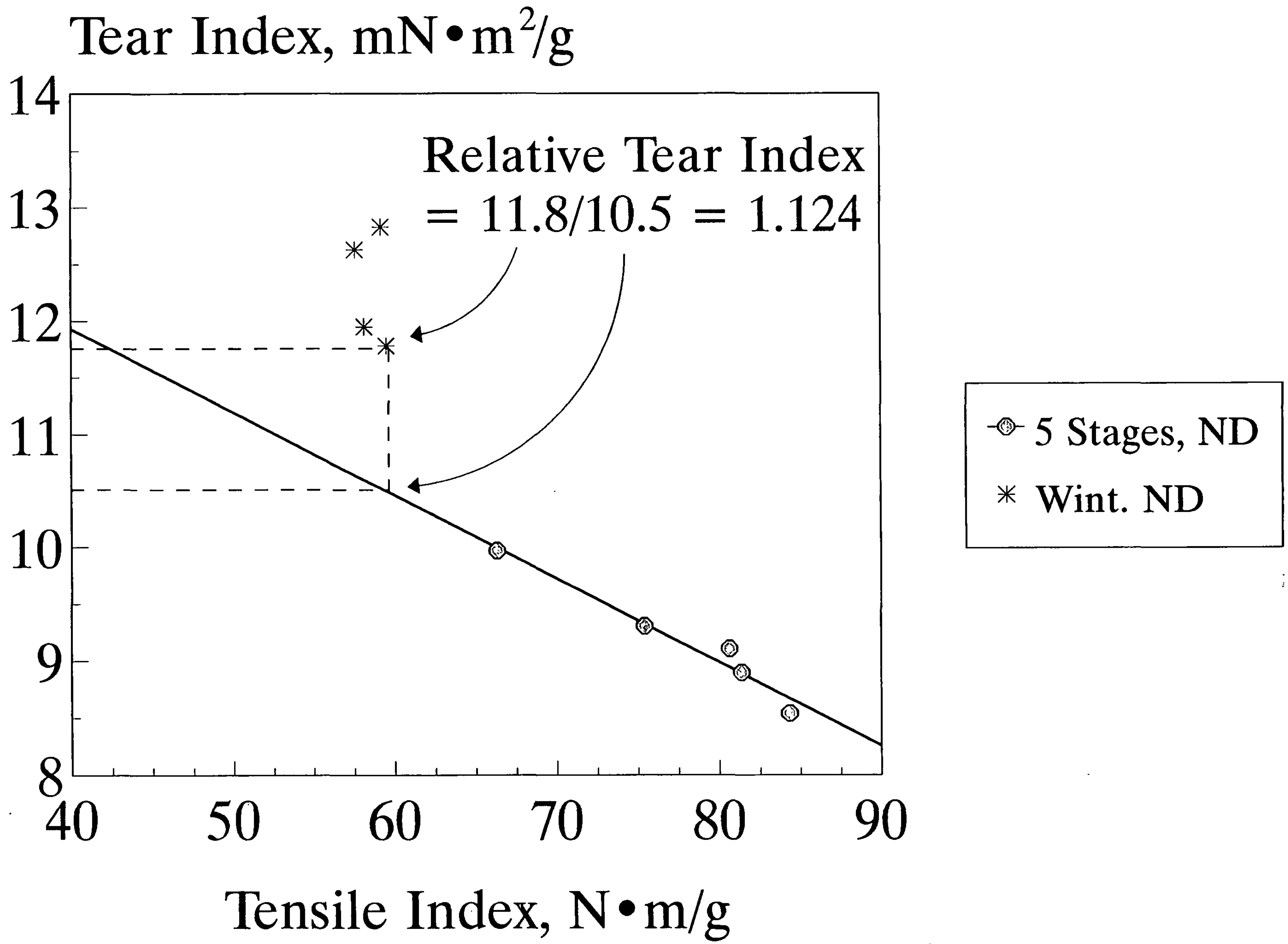
AWR: PAC, 3/95

Tear/Tensile Data

PFI Beater Data

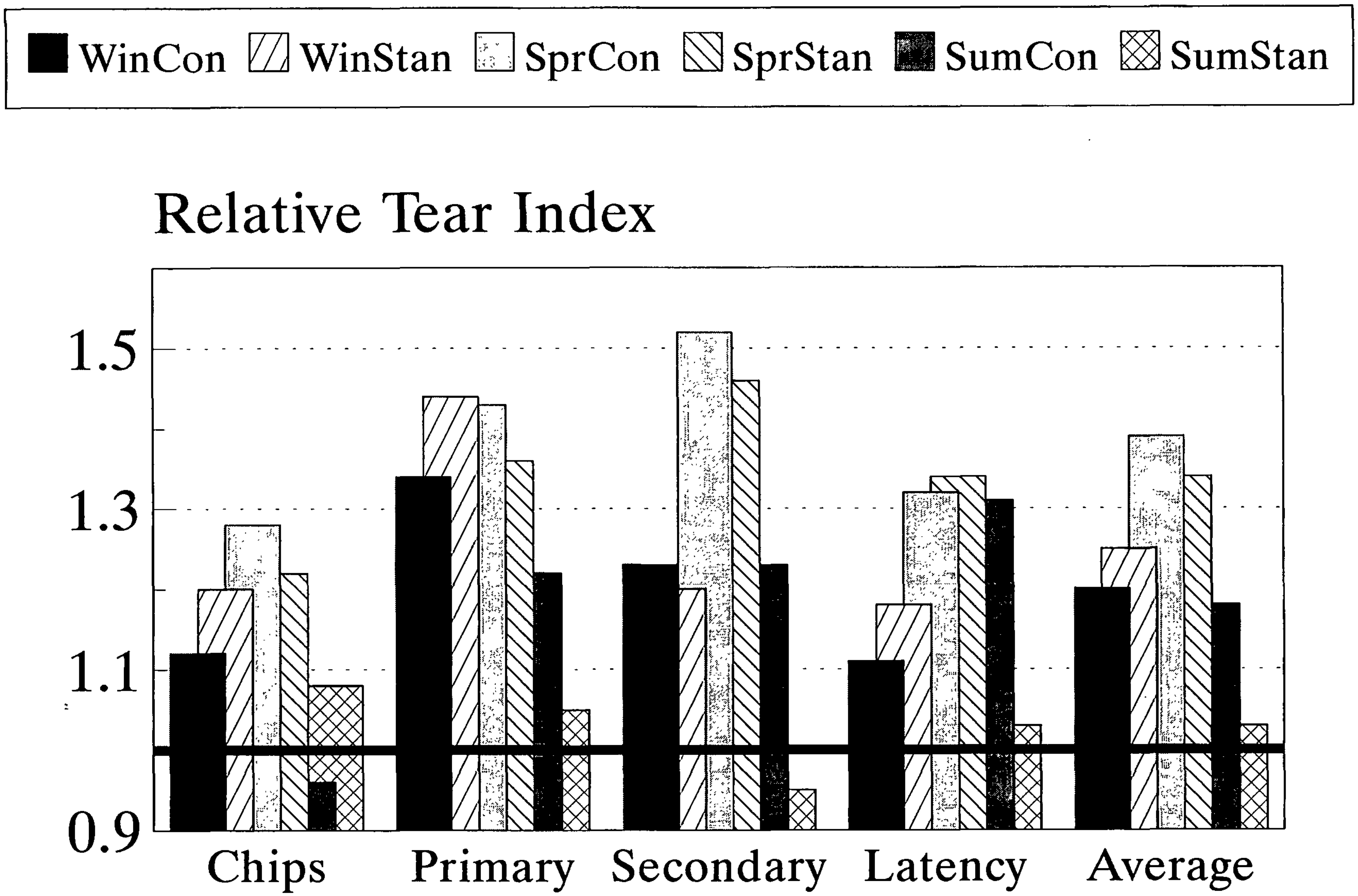


Freeness from 760 ml to 365 ml.
AWR: PAC, 3/95



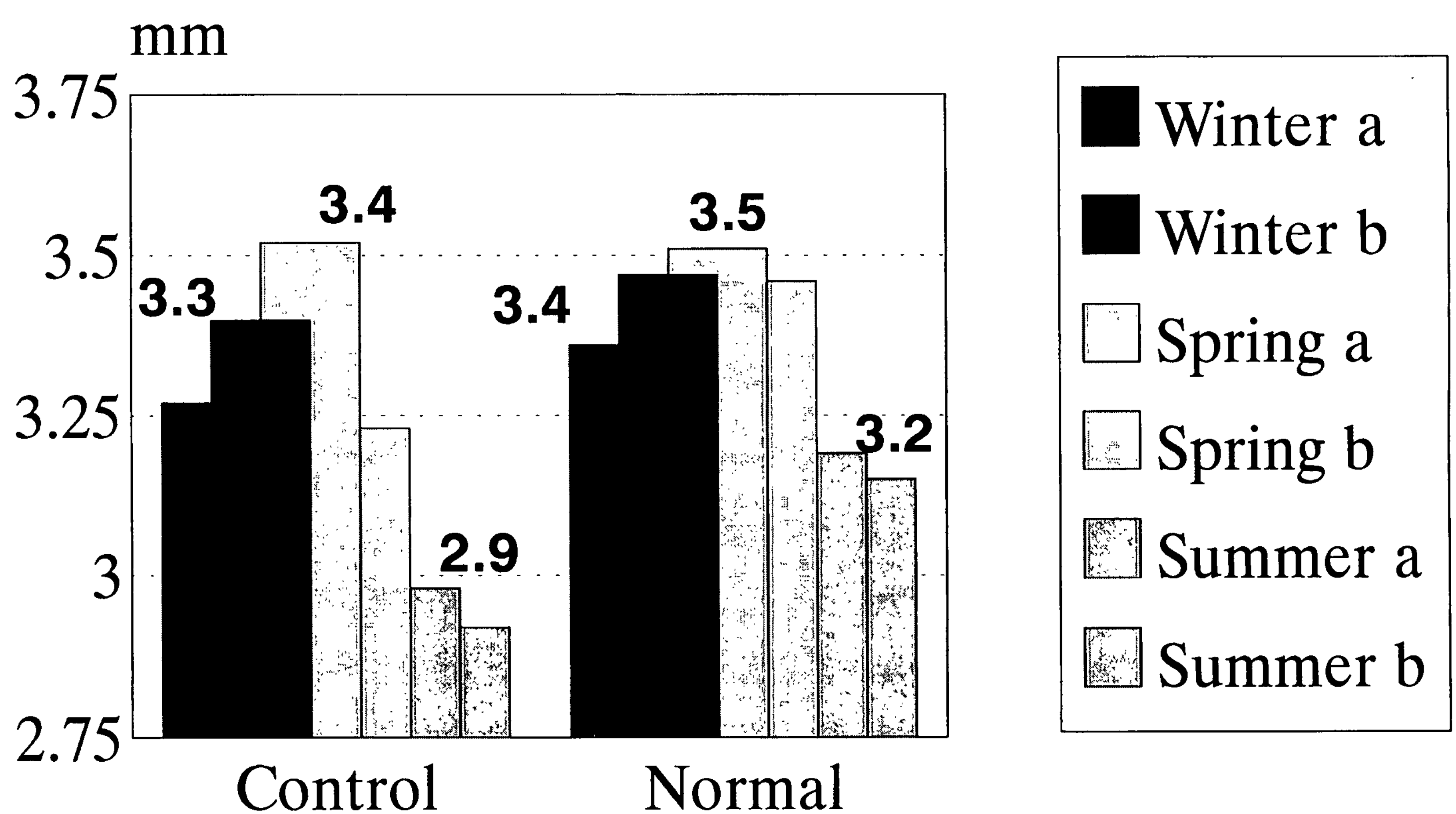
Tear Index, mN•m²/g
14
13
12
11
10
9
8
40
50
60
70
80
90
Tensile Index, N•m/g
Relative Tear Index
= 11.8/10.5 = 1.124
5 Stages, ND
Wint. ND

Tear @ Controlled Tensile



AWR: PAC,3/95

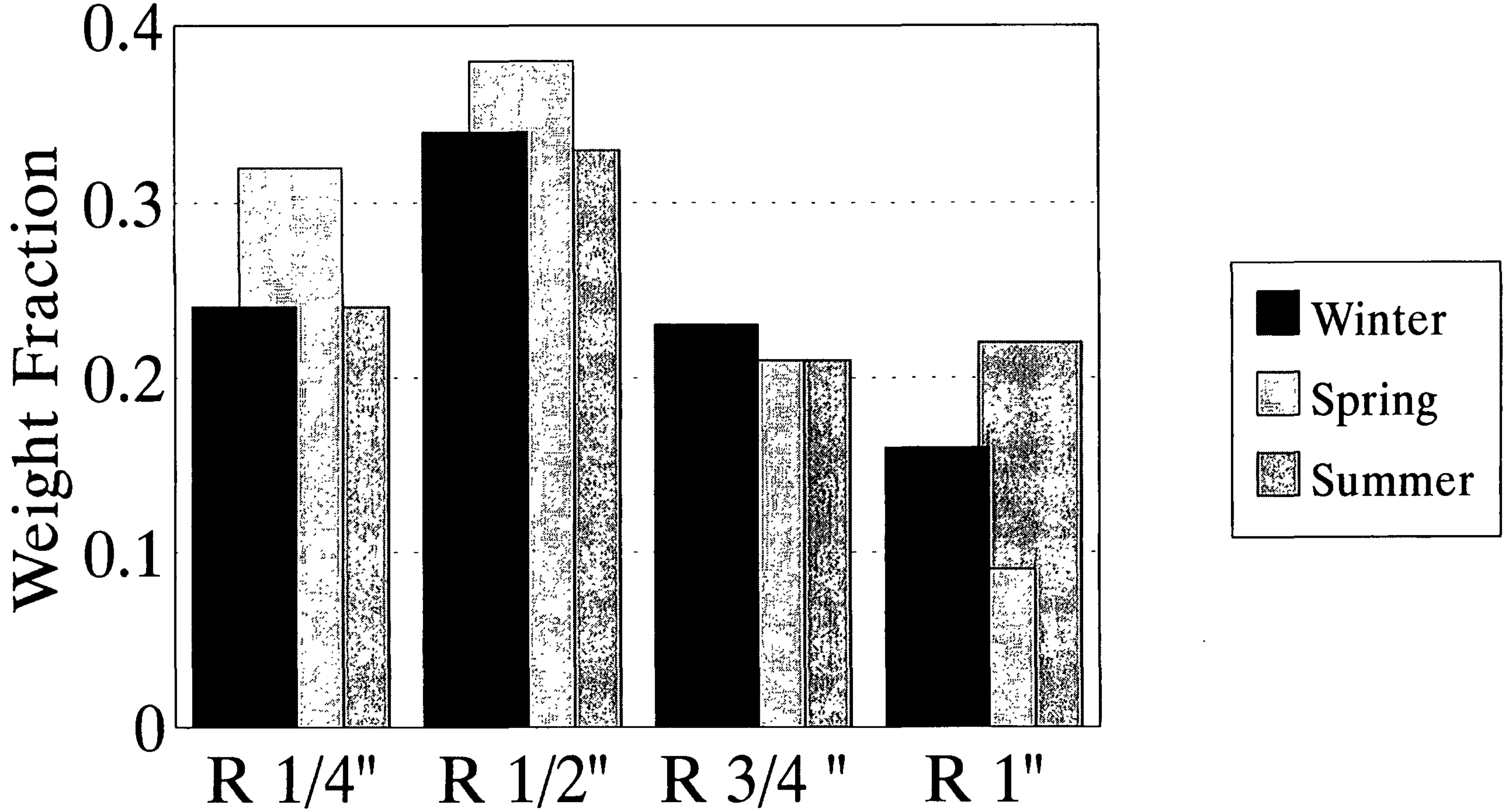
Wood Chip Fiber Length



AWR: PAC, 3/95

Chip Size Distribution

Bowater Seasonal Strength Data



AWR: PAC, 3/95

Influence of Chip Size

- An increase in chip size has been shown to decrease tensile and burst strength, but has little influence on tear.
- An increase in chip size should have no influence on native fiber strength.

Chip Quality

1" and 3/4" chips were inspected for knots bark and frayed cuts.

- There were no significant differences in bark or knot content for any of the three sample periods.

- There was a slight increase in ragged cuts in the spring samples.

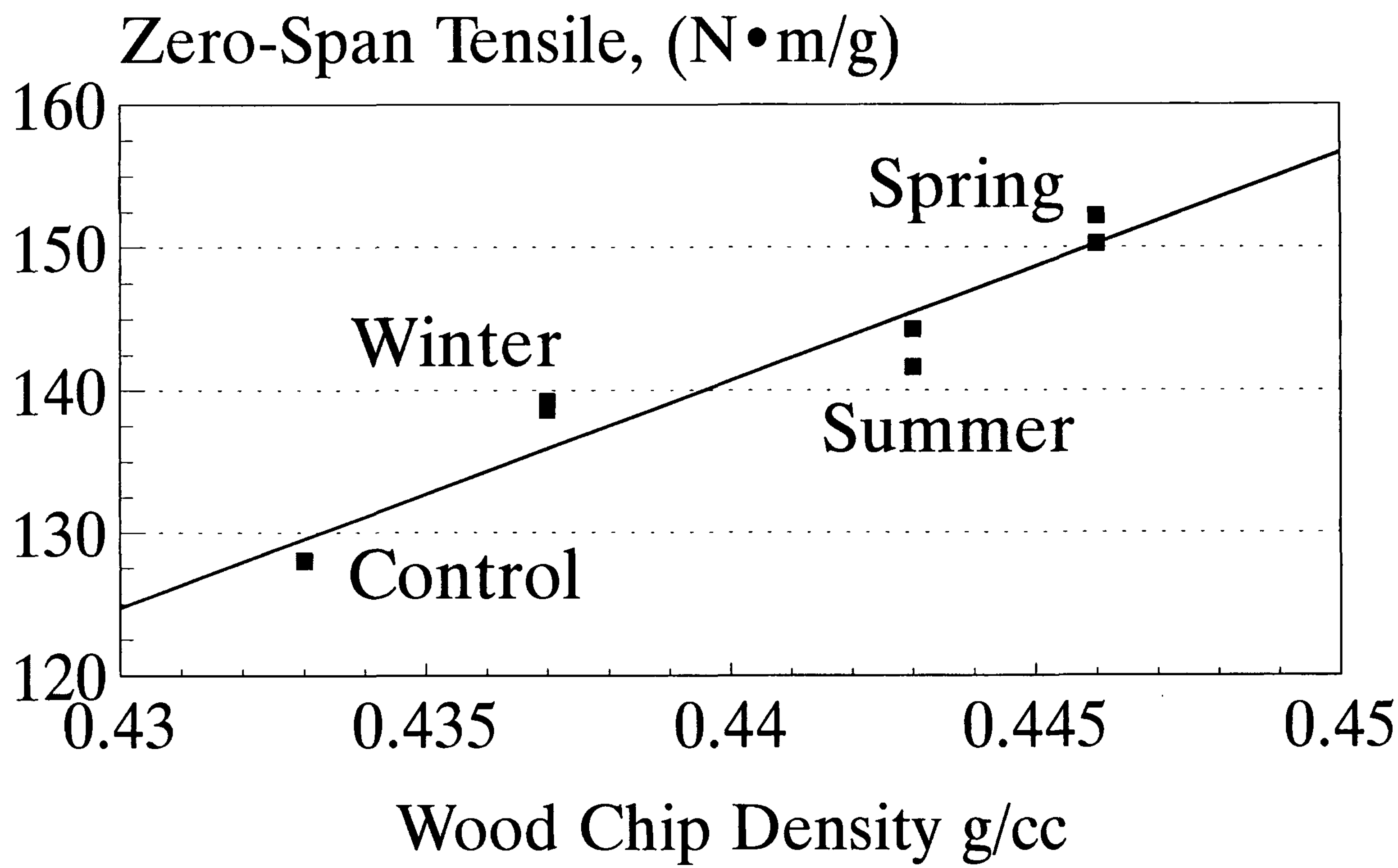
AWR: PAC, 3/95

Wood Chip Properties
Density and Increment Size

	Density g/cc	3/4" mm	1/2 " mm	% >6mm
Control	0.433	4.15	4.09	19
Winter	0.437	3.77	3.7	12
Spring	0.446	3.71	3.36	10
Summer	0.443	3.93	3.17	12

AWR: PAC,3/95

Influence of Wood Density



$r^2 = 0.93$

AWR: PAC, 3/95

Conclusions and Comments

- The most likely source of the seasonal change in pulp strength is a change in juvenile of plantation wood content.
- There is relatively little loss in pulp strength in TMP refining.
- The chlorite holopulping technique has successfully tracked seasonal fiber strength.

AWR: PAC, 3/95

Future Work

The project has initiated a task to evaluate how wood breaks down in the early stages of refining.

- The project will evaluate the influence of earlywood and latewood content and species (Spruce vs Pine) on the pattern of wood disintegration.

- A pilot refining trial using reduced diameter refiner plates is scheduled for June.

Acknowledgements:

Bob Harley

Melanie Gray

Shawn Wendell

Jim Turnbull

Peter Joyce

IPST Member Companies

Evaluation of Strain in Earlywood and Latewood of Loblolly Pine in Cyclic Compression

Cheryl B. Rueckert
Ph. D. Candidate

Advisory Committee:

- Alan Rudie (advisor)
- Earl Malcolm
- Pierre Brodeur

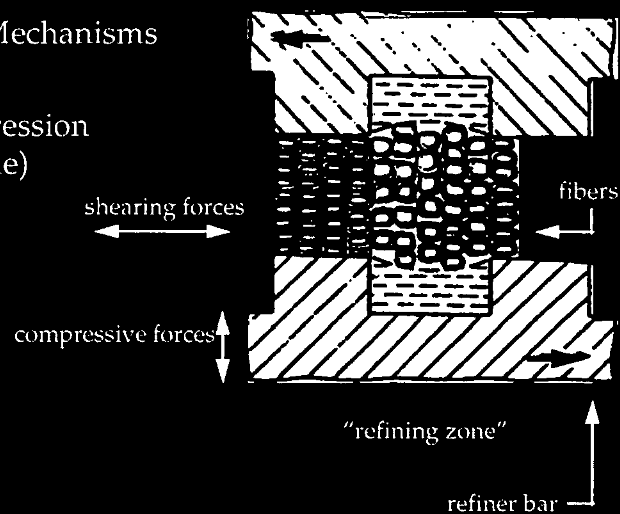
March 1995

Outline

- Introduction
  - Pulping Mechanisms
  - A190 Hickey
  - A190 St. Laurent
  - Shakhet
- A490 Rueckert
  - Thesis Objective
  - Experimental Plan
  - Experimental Finished
  - Experimental to be Completed

Introduction

- Pulping Mechanisms
  - Shear
  - Compression (Fatigue)



A190 Hickey Objectives

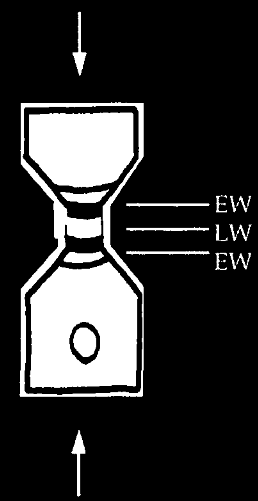
- Determine the strain distribution between the earlywood and latewood bands of Loblolly Pine during cyclic compression of model chips

- Evaluate the effects temperature and frequency have on this strain distribution

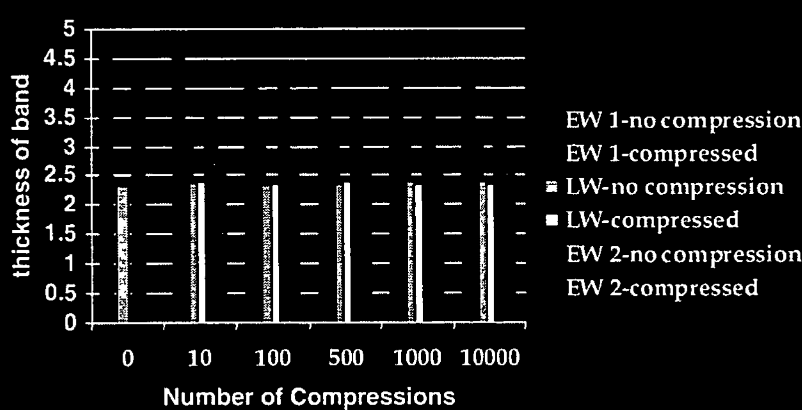
A190 Hickey Experimental Approach

- Cyclic compression at 1, 15, and 30 Hz
- Measure strain and temperature of model

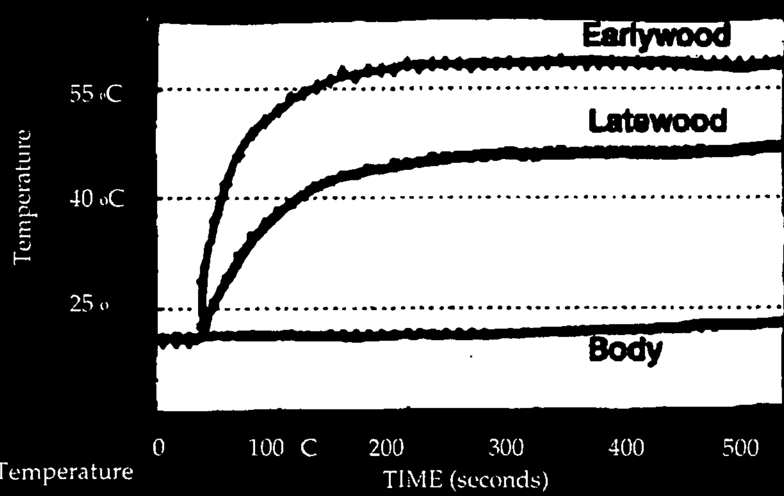
chips



A190 Hickey Results



A190 Hickey Results



Room Temperature
15 Hz

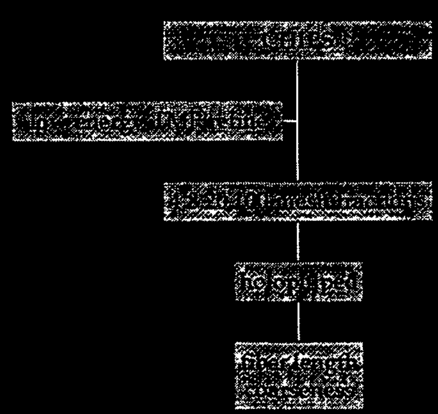
A190 Hickey Conclusion

- Loblolly Pine fatigue data shows that under compression, strain is concentrated in the earlywood band

A190 St. Laurent Objective (partial)

- Evaluate the distribution of earlywood and latewood in different particle size fractions obtained from early stage TMP pulping of Loblolly Pine

A190 St. Laurent Experimental Approach (partial)



A190 St. Laurent Results

Pure Earlywood or Latewood

Wood	Coarseness (mg/m)	Fiber length (mm)
Earlywood	0.14	3.08
Latewood	0.37	3.32

TMP Fractions

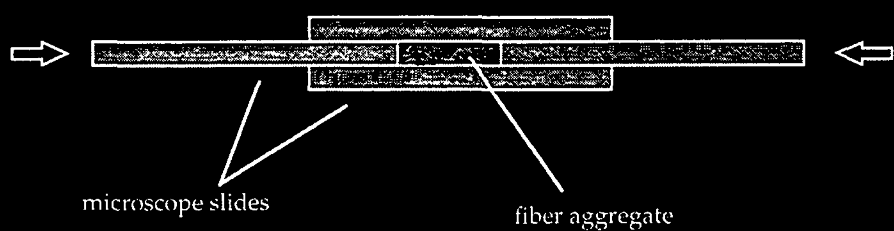
Mesh Size	Coarseness	Fiber length	% Earlywood	# Whole fibers EW	# Whole fibers LW
4	0.22	3.21	46	37	59
8	0.21	2.41	49	18	35
20	0.17	2.62	53	1	23
100	0.18	1.21	43	0	3

A190 St. Laurent Conclusion

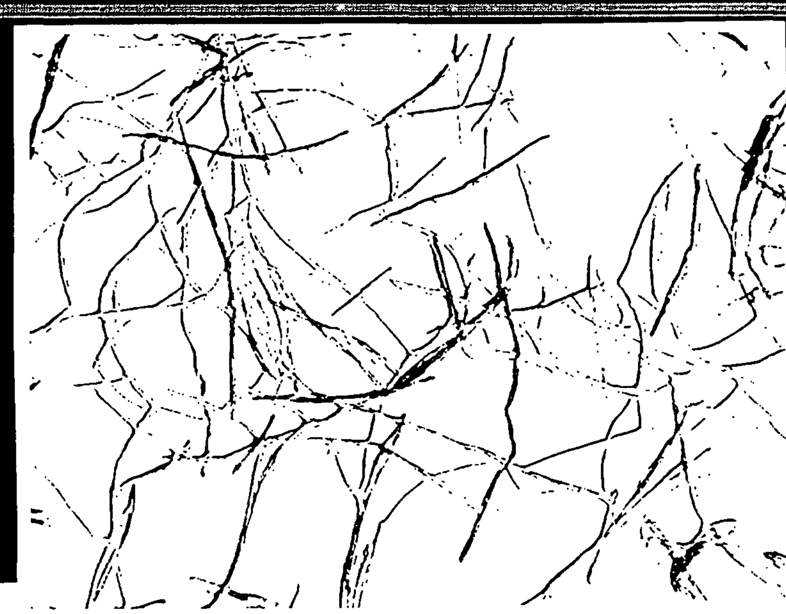
- There is evidence of earlywood enrichment in smaller particle size fractions from low energy refining

Shakhet

- Static compression of fiber aggregates



Shakhet Photograph - 1



Shakhet Photograph - 2

Data from Shakhet Photographs

EW (cm)		LW (cm)		t-Test: Two-Sample Assuming Unequal Variances	EW	LW
0.0	0.2	0.0	0.0		EW	LW
0.4	0.6	0.0	0.1	Mean	0.22	0.05
0.6	0.1	0.0	0.0	Variance	0.09	0.02
1.0	0.0	0.0	0.0	Observations	20	20
0.0	0.6	0.0	0.0	Pooled Variance	3.50	
0.5	0.0	0.0	0.1	df	26.99	
0.0	0.0	0.6	0.0	t	2.22	
0.0	0.0	0.0	0.0	P(T<=t) one-tail	0.02	
0.0	0.0	0.2	0.0	t Critical one-tail	1.71	
0.2	0.1	0.05	0.0	P(T<=t) two-tail	0.04	
				t Critical two-tail	2.06	

Data from Shakhet Photographs

t-Test: Two-Sample Assuming Unequal Variances (cm)

	EW	LW
Mean	0.26	0.14
Variance	0.14	0.03
Observations	52	45
Pooled Variance	3.50	
df	75.41	
t	1.96	
P(T<=t) one-tail	0.03	
t Critical one-tail	1.67	
P(T<=t) two-tail	0.05	
t Critical two-tail	1.99	

A490 Rueckert Thesis Objective

- Investigate the distribution of strain between earlywood and latewood in fiber aggregates subjected to cyclic loads

- Hypothesis: earlywood fibers will be preferentially strained and have a larger temperature increase than latewood fibers

A490 Rueckert Experimental Plan

- Development of an apparatus to induce and record high frequency cyclic compression
- Measure the strain distribution between earlywood and latewood fibers subjected to cyclic compression; change in fiber curl index will be used as a measure of strain

A490 Rueckert Experimental Plan

- Temperature differences between earlywood and latewood fibers will be measured using infrared imaging to confirm the distribution of strain
- Analysis of the strain distribution and temperature gradients will determine whether differences in energy absorption between the earlywood and latewood fibers exist and their frequency dependence

A490 Rueckert Experimental Finished

- Low frequency apparatus completed and experimentation begun

A490 Rueckert Experimental to be Completed

- Measure strain and temperature of fiber aggregates
- Purchase and assemble shaker system for higher frequency work

Frequency(Hz)	MTS	Shaker
10	X	
30	X	X
100		X
200		X
???		X

Effect of Fungal Treatment on Pulp Strength

Lois Forde Kohler

THESIS GOAL AND OBJECTIVES

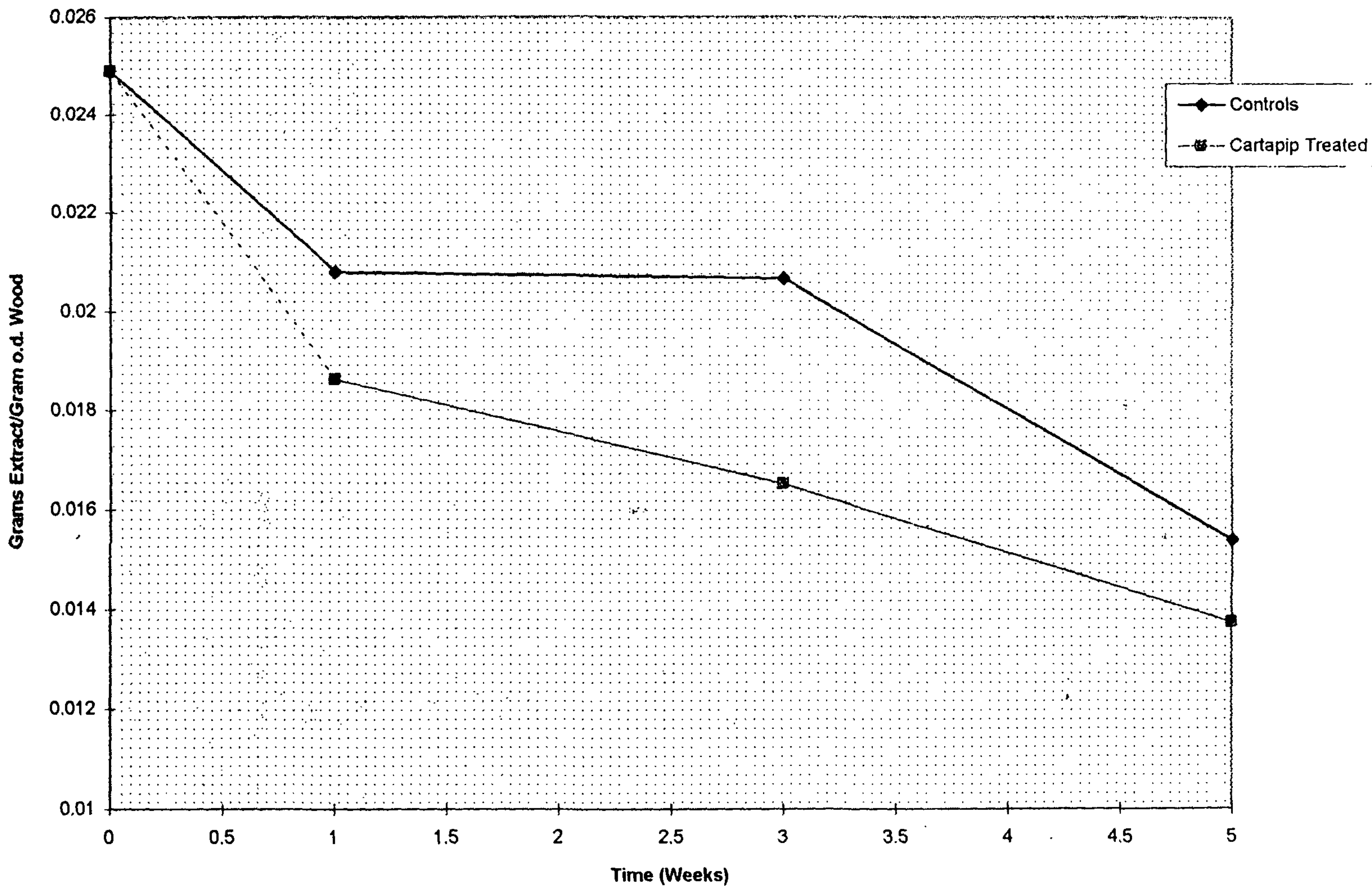
GOAL: Examine the effects of Ophiostoma piliferum on properties of wood pulp.

Objectives: Investigate the impact of Ophiostoma piliferum on the strength properties of handsheets.

SIGNIFICANCE OF EXTRACTIVE CONTENT AND FIBER MORPHOLOGY:

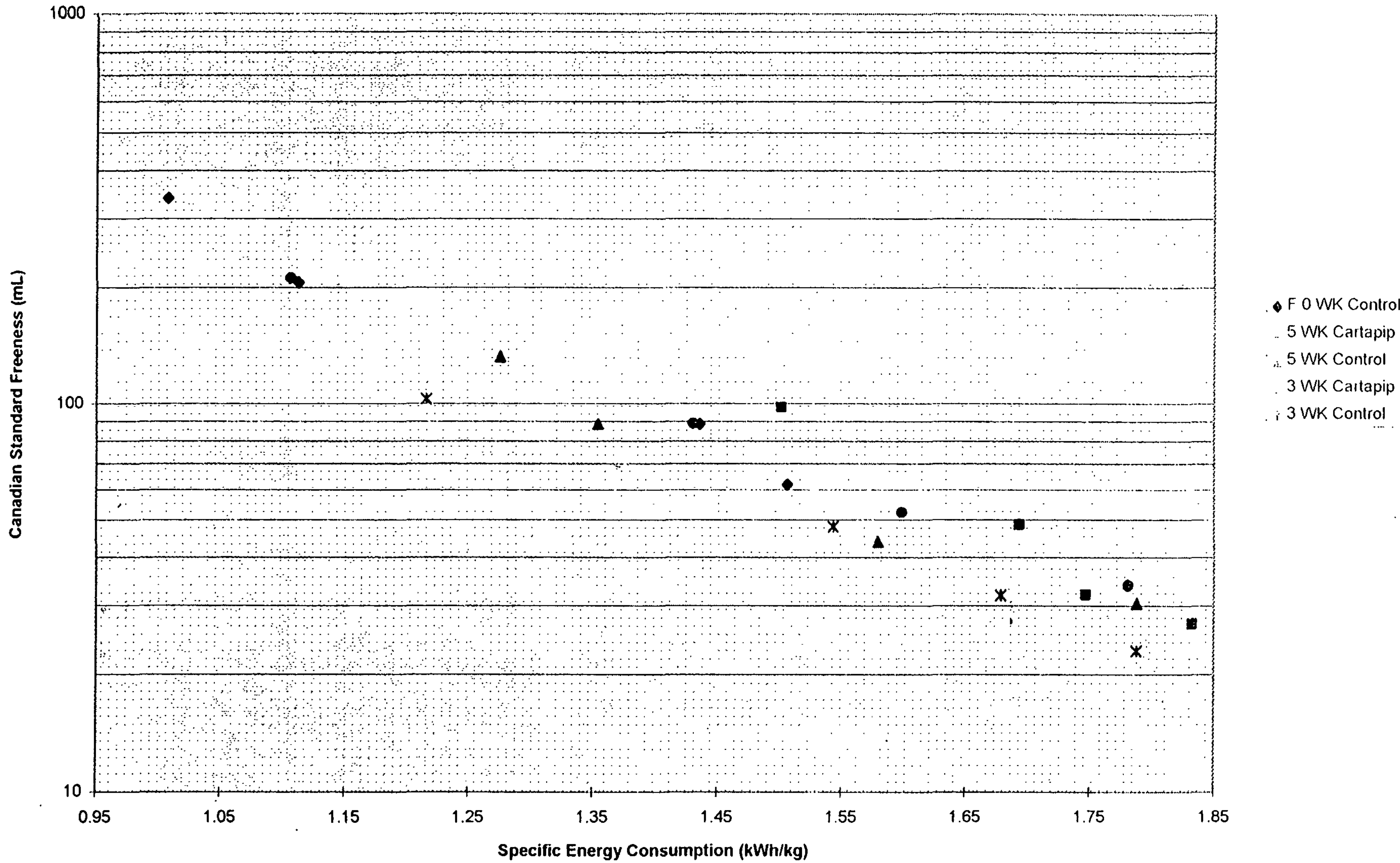
1. **Extractive Free Groundwood Pulp Yields Strong Handsheets**
2. **Extractive Content DECREASES dramatically within Treatment**
3. **Strength and Runnability Increase with Treatment**
4. **Ray Parenchyma Cells and Pit Membranes Contribute to Fines**
5. **Fungal Mycelia may be Contributing to Fines Content**

Figure 1. Relationship Between Extractive Content and Cartapip Residence Time



SCAT Chart 76

Figure 2. Freeness - Specific Energy Relationship



SCAT Chart 87

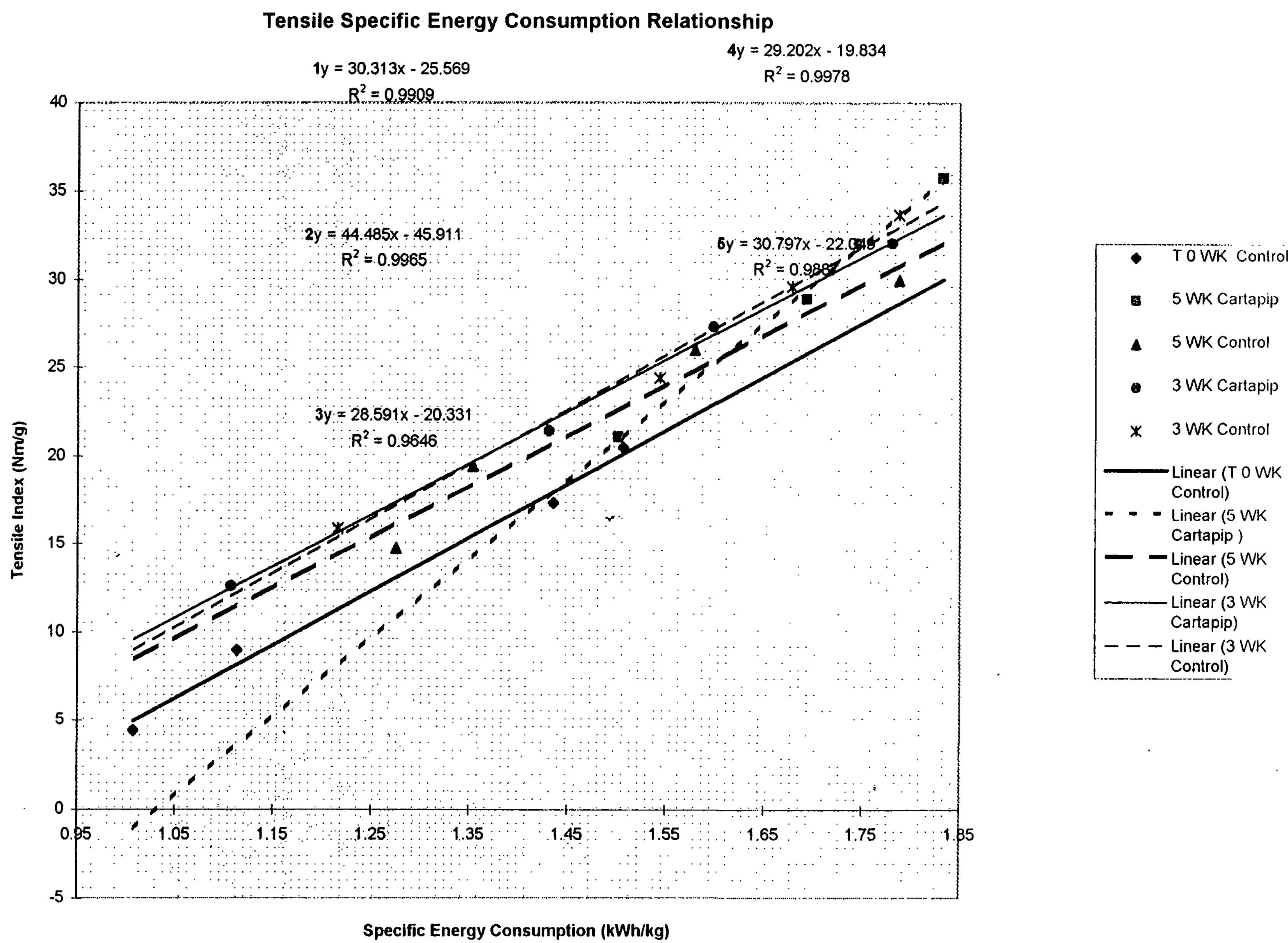


Figure 3. Fines Generation with Energy Consumption

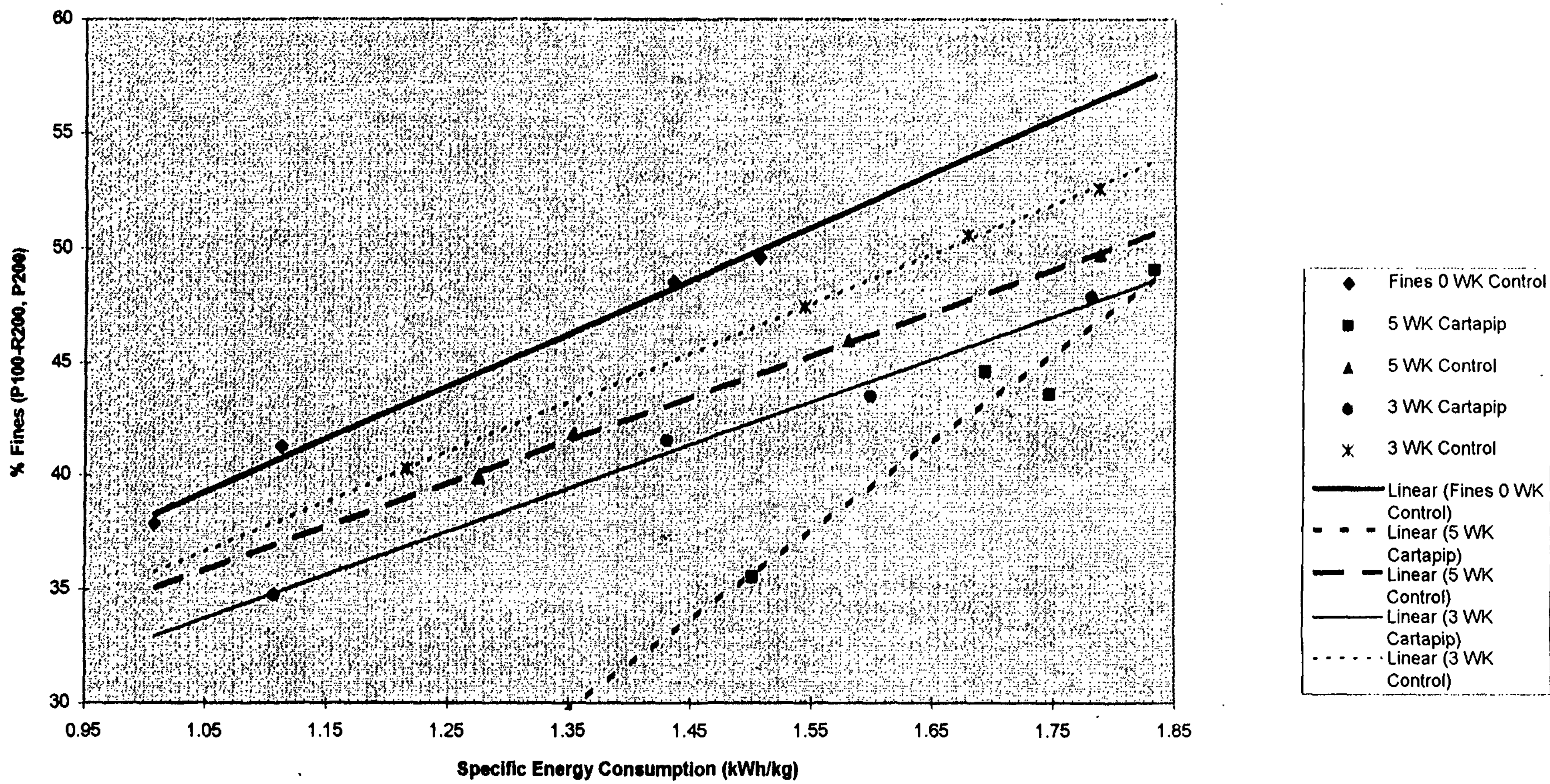
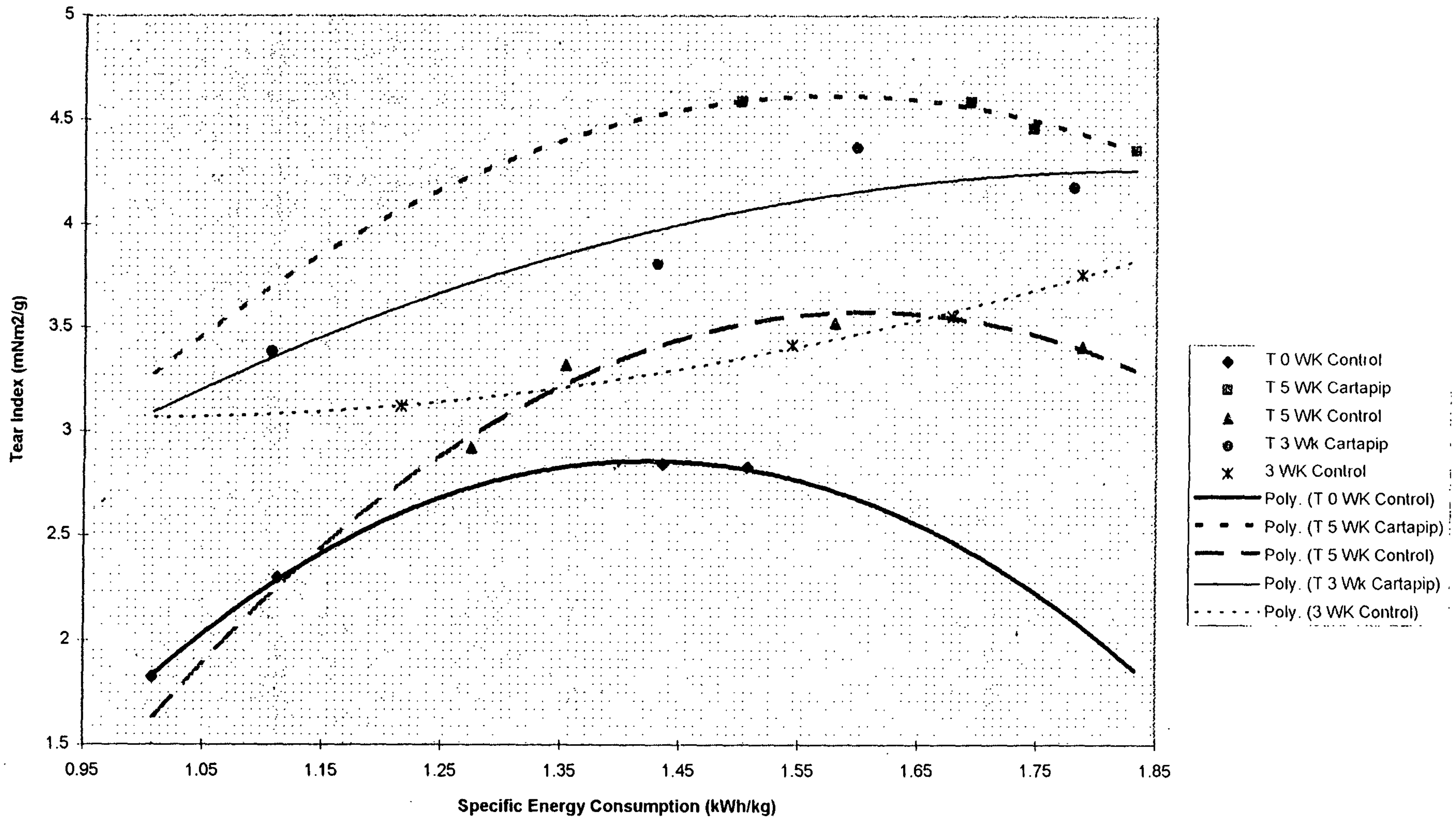


Figure 6. Tear Specific Energy Consumption Relationship



SCAT Chart 102

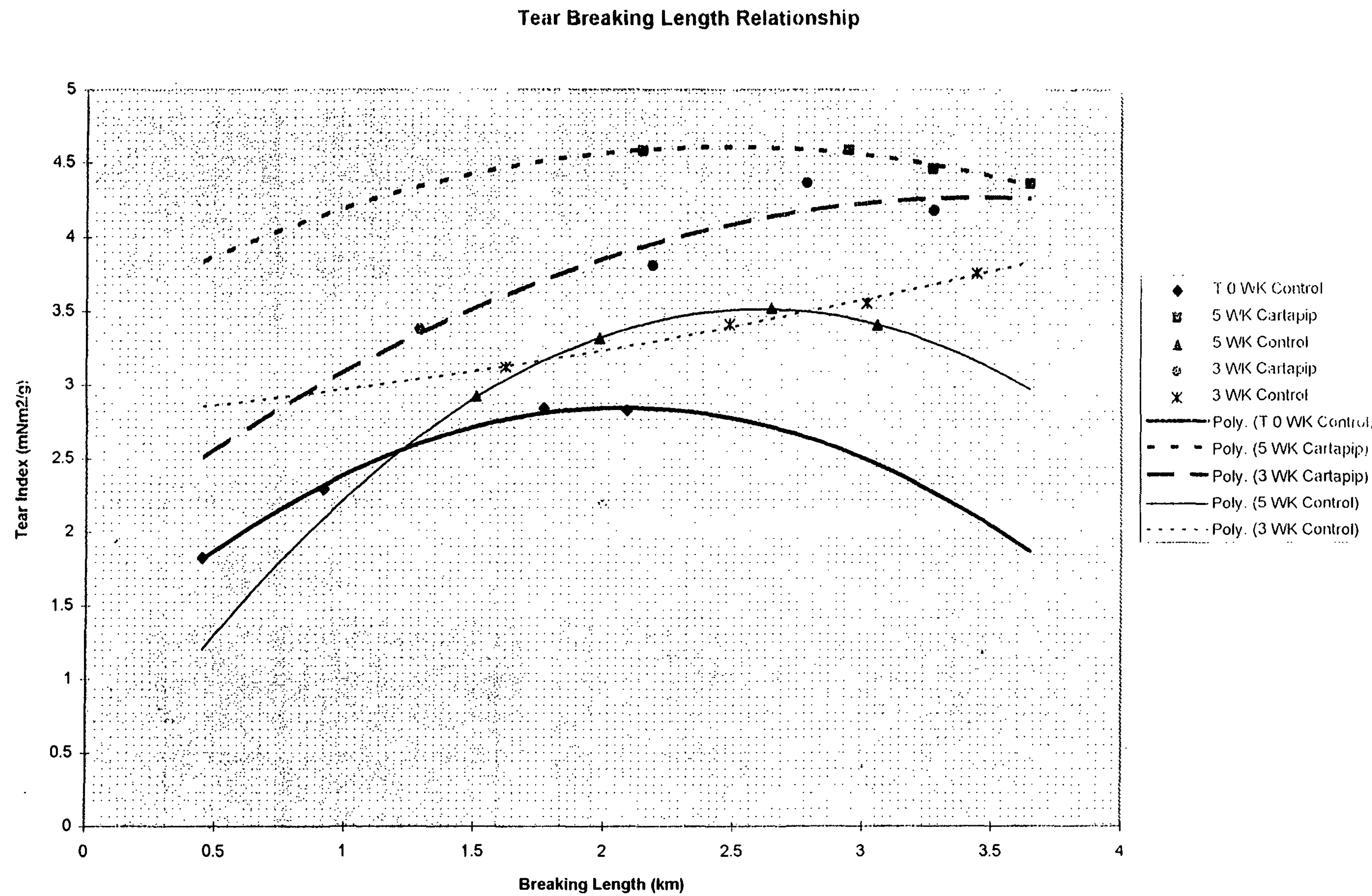
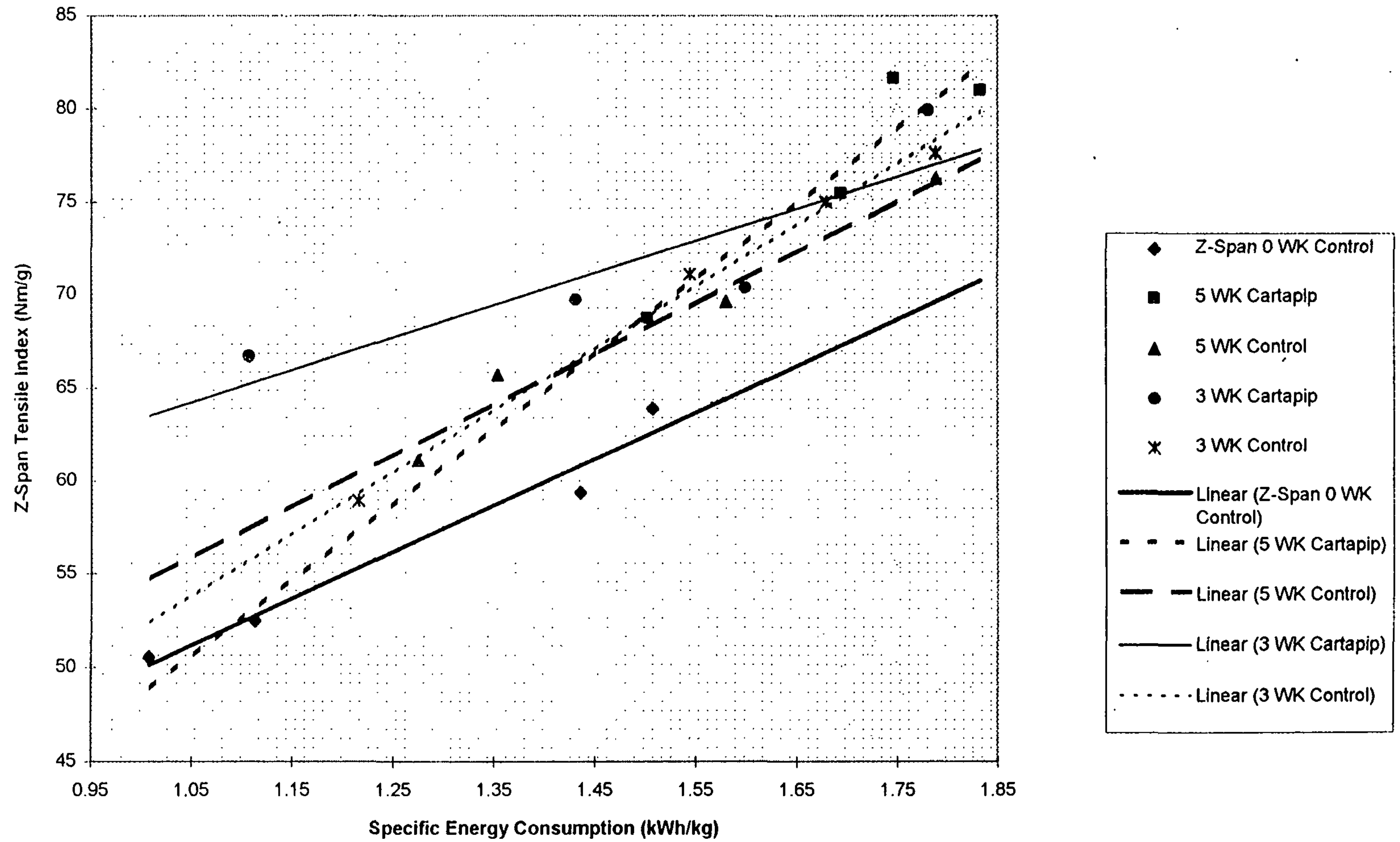
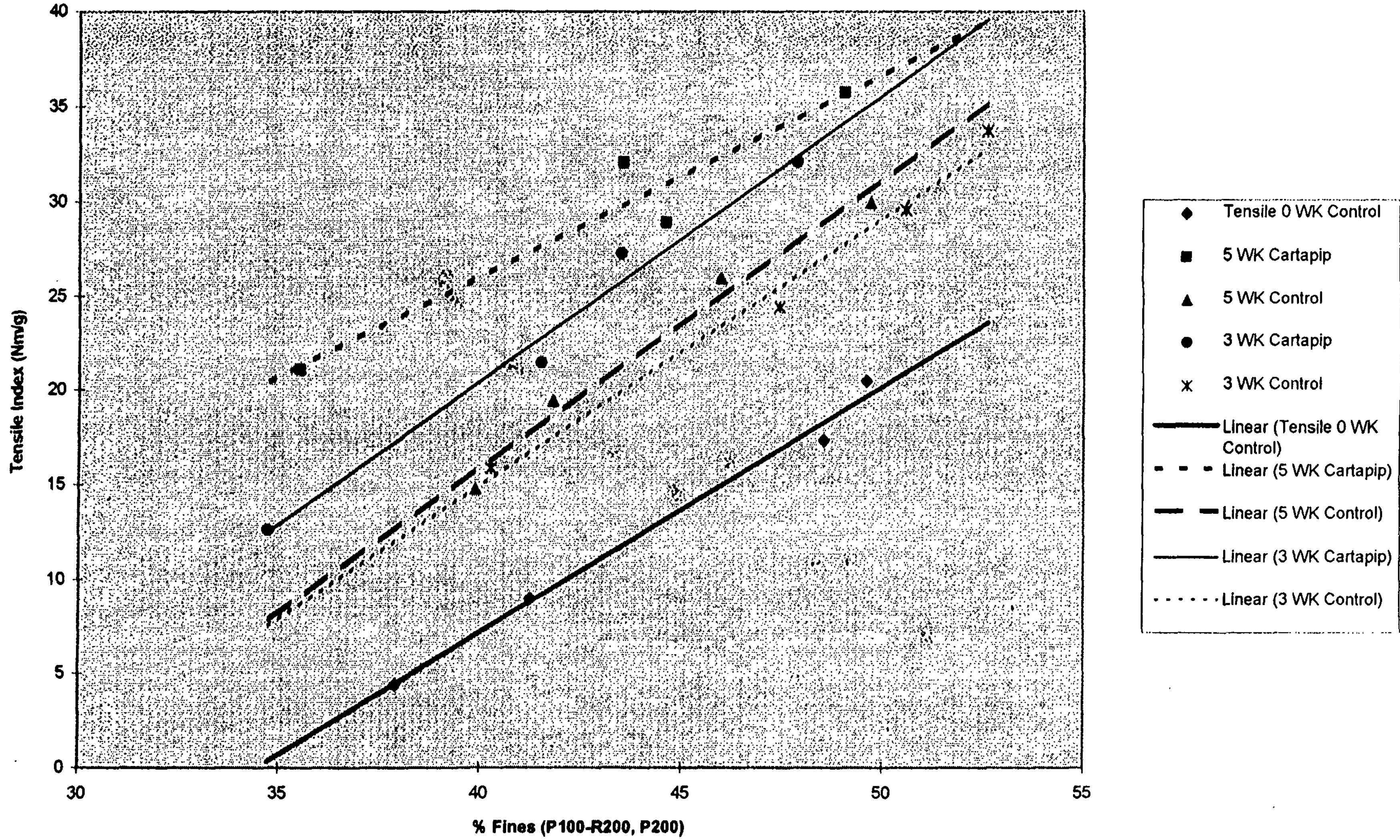


Figure 7. Z-Span Tensile Index vs. Specific Energy Consumption



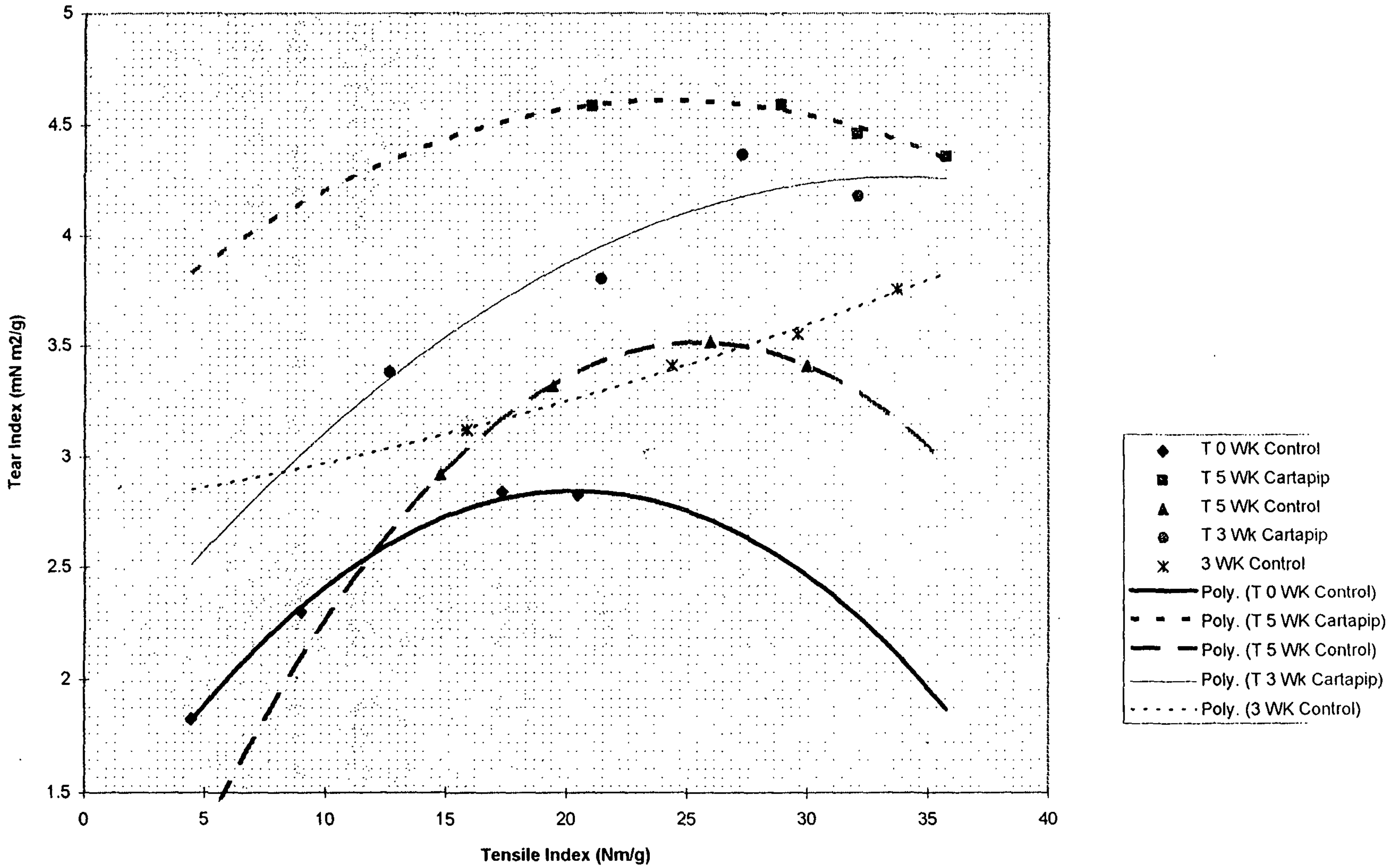
SCAT Chart 97

Figure 10. Fines Content Tensile Relationship

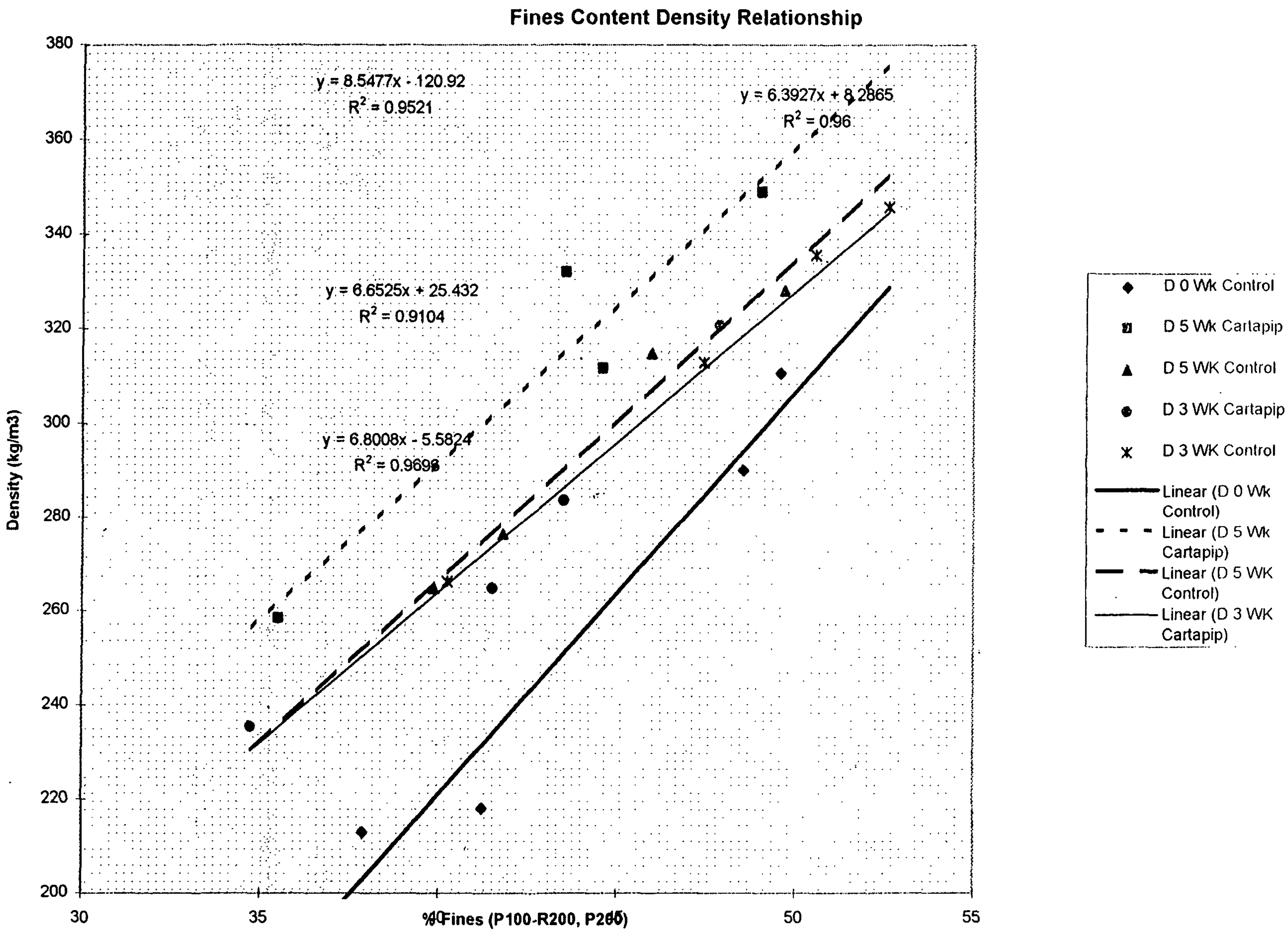


SCAT Chart 98

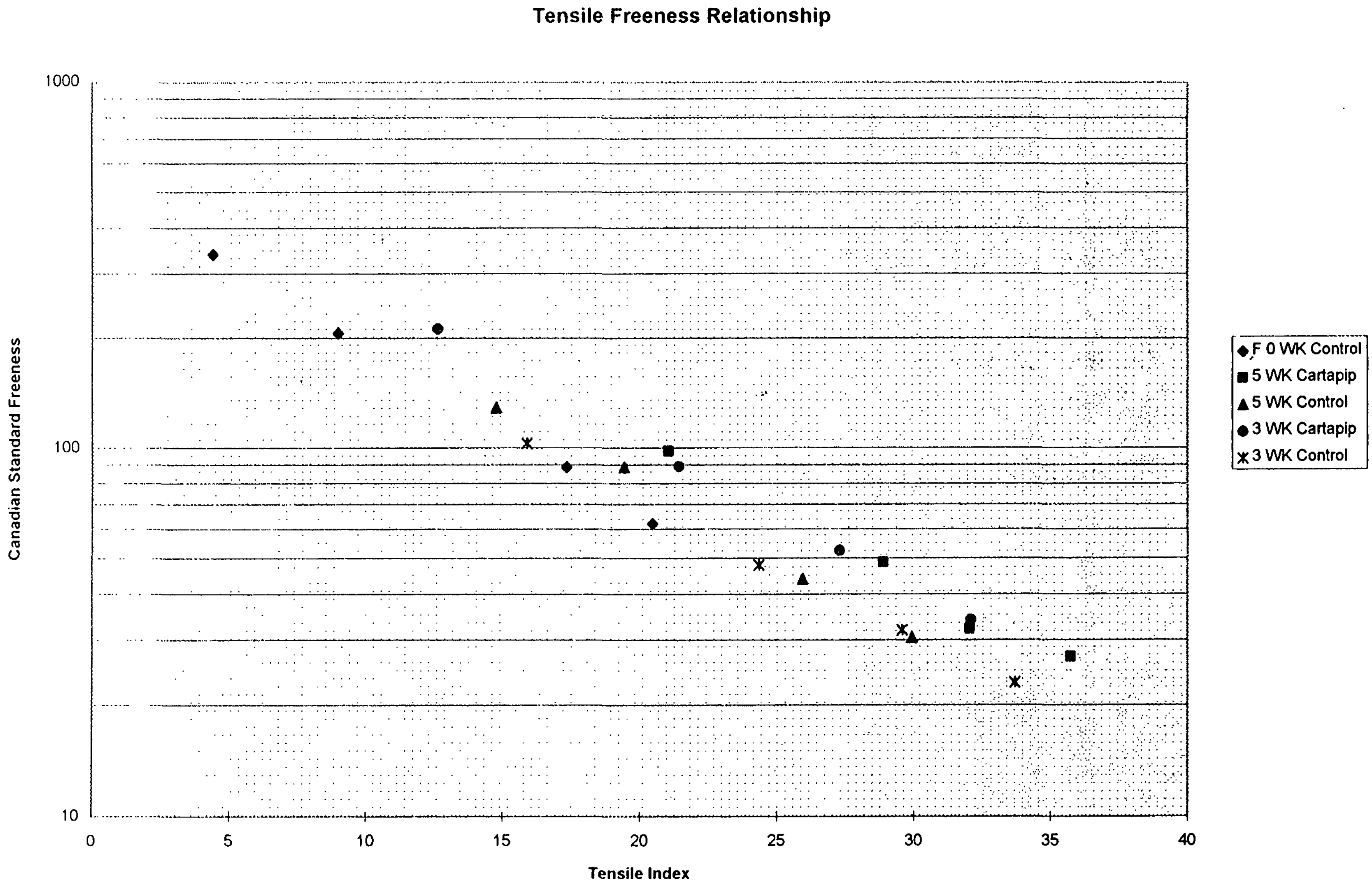
Figure 9. Tensile Tear Relationship



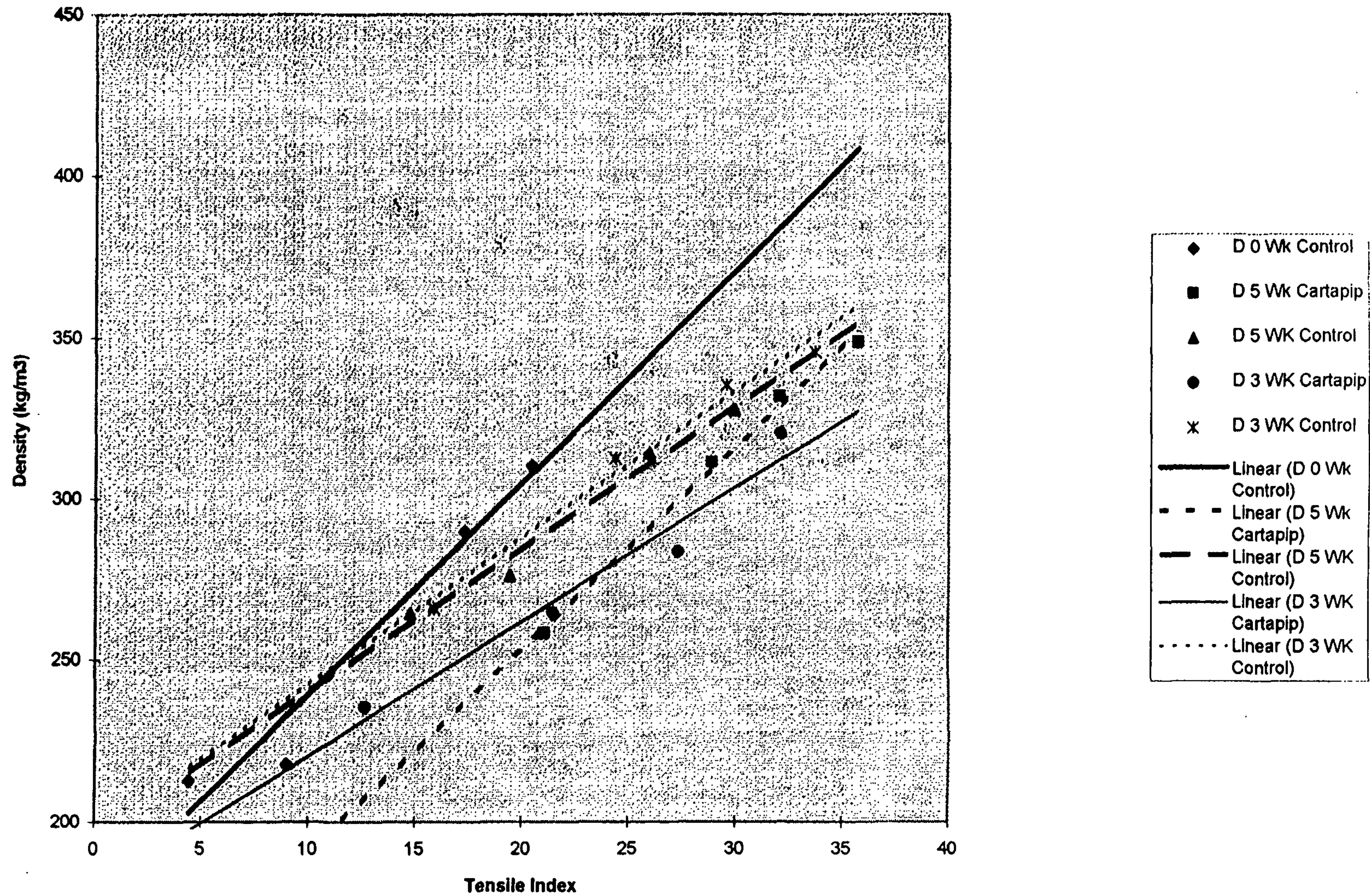
SCAT Chart 100



SCAT Chart 77



Density Tensile Relationship



GOALS FOR PAST QUARTER

1. Refine one week Cartapip treated sample and control.

2. Inoculate chips for 5 week residence for continued refining and handsheet studies.

3. Continue work with DCM extracts to determine proportions of extractive classes for handsheets produced from 5 week control and Cartapip treated samples.

GOALS FOR UPCOMING QUARTER

A. Complete Lab Work to Begin Thesis Writing with the Beginning of Spring Quarter.

1. Freeze dry, extract, and conductivity titrate pulp samples to compare with current conductivity titration results.

2. Refine and produce pulps and handsheets from one week control and Cartapip treated chips.

3. Repeat refining runs with T0 control, 5 week control, and 5 week Cartapip treated chips.

4. Use pulps from Goal 3 to mass produce handsheets for dichloromethane extraction and extractive class studies.