

Simplified Pulping & Bleaching of Corn Stalks

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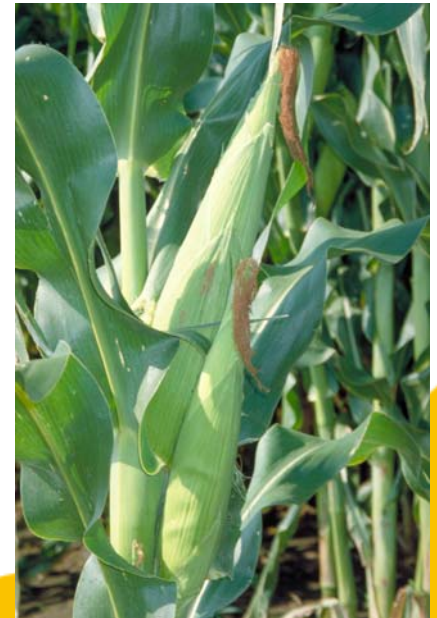
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OUTLINE

- Why Cornstalks?
- Why *NOT* Cornstalks?
- Our experiences with cornstalk pulping
- A new approach
- Experimental
- Process Results
- PFI Refining and Handsheet Testing
- Additional Work



WHY CORNSTALKS?



1. Why Nonwoods?

- Trees....available for the future?
- Trees...more valuable for other uses?
- Regional shortages of hardwoods
- Consumer demand for “tree-free” paper

2. It's an Agricultural Residue

- Grain pays for growing costs
- Source of extra income for farmer

3. Lots of It Available

Residue Type	U.S. Availability, OD tons/year
Corn stalks	300,800,000
Wheat straw	78,900,000
Barley straw	12,000,000
Sorghum straw	12,000,000
Rice straw	7,500,000

Rowell and Cook, 1998 N. American Nonwood Fiber Symposium

4. Physical/Chemical Composition

	Cornstalks	Hardwoods
Fiber length, mm	1-1.5	0.7-1.6
Fiber diameter, microns	20	20-40
Lignin content, %	15-18	23-30
Cellulose content, %	44-47	38-49

>Atchison, Pulp and Paper Manufacture, Vol. 3

>Eroglu, 1992 TAPPI Pulping Conference, Book 1

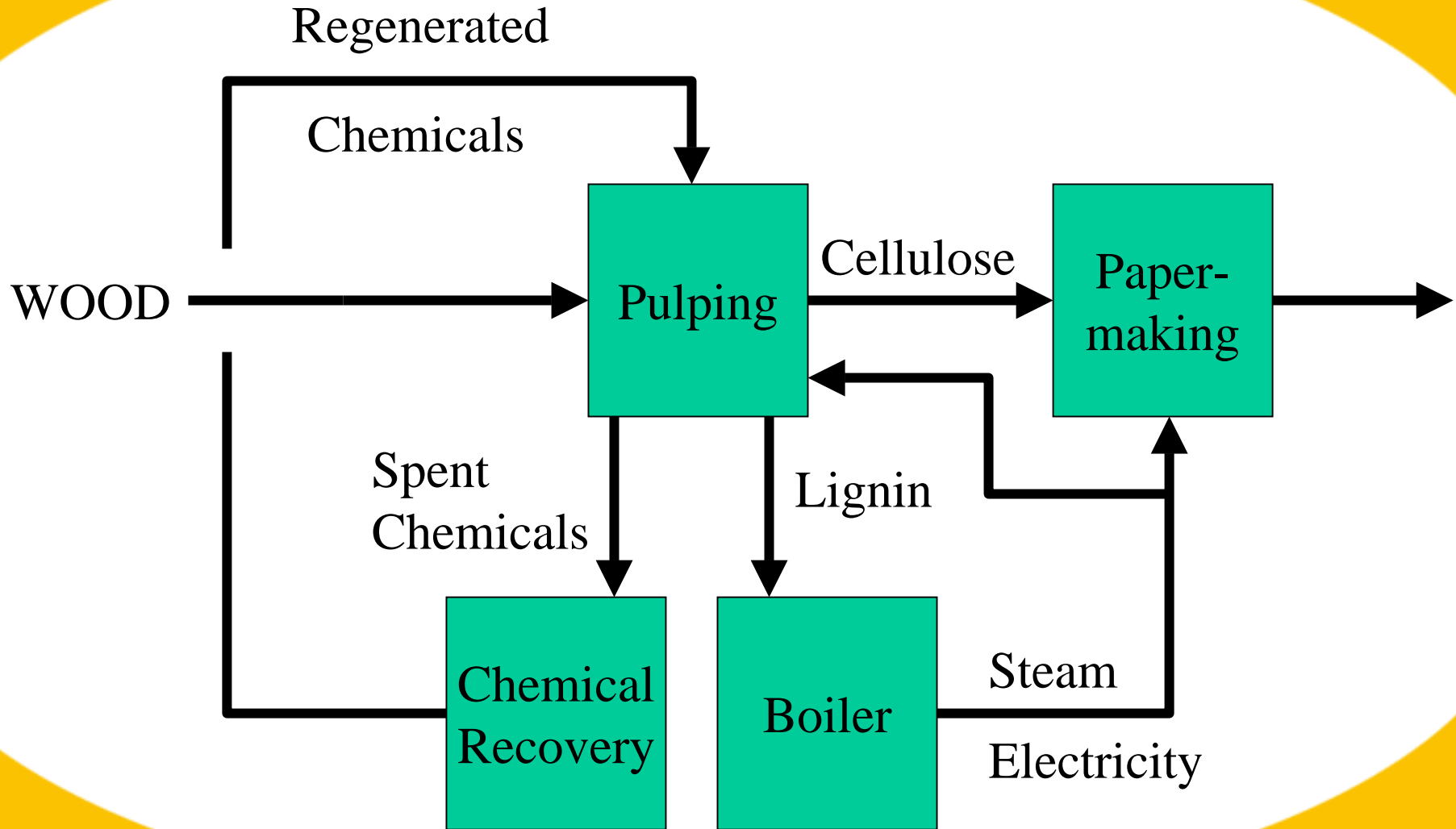
Why *NOT* Cornstalks?



1. Trees and Wood

- Wood is a *good* fiber supply for papermaking
- Forest growth in the US exceeds harvests by 37 %
- Wood has a clean, uniform composition --with few contaminants, and fibers well-suited for papermaking
- The wood-based pulping and bleaching process is very, very well-balanced – at least at large scale

A DAMN GOOD SYSTEM



2. The Challenges of Annual Crops

- Must harvest at one time and store all year
- Susceptibility to pests, fire, disasters
- Cost volatility?

3. The Challenges of Corn Stalks

- Low bulk density – storage, transport costs
- Can't process in wood-based equipment
- Modest fiber quality
- Silica (?)

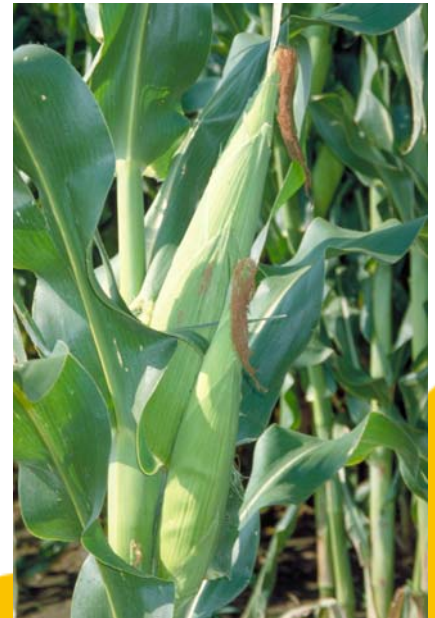
4. Extraneous Materials

- High content of pith – parenchyma and fines
- Poor drainage, high chemical consumption
- Requires de-pithing prior to pulping

What is Needed to Make Cornstalk Pulping Work

- A simplified process that lends itself to “mini-mills” located in the supply area
- A process that deals with pith without expensive mechanical de-pithing
- A process that preserves drainage rate

Our Experiences in Pulping Non-Depithed Cornstalks, With TCF Bleaching



1. Soda Pulping with TCF Bleaching

	Brightness, % ISO	Freeness, CSF
Pulping, 20 % NaOH, 140 C, 90 minutes	25-30	400-450
Bleaching, Q-P-P	68-75	350-400
Bleaching, Q-Pp-P	82-84	280-360
Bleaching, Q-Pp-Q-P	84-88	270-300

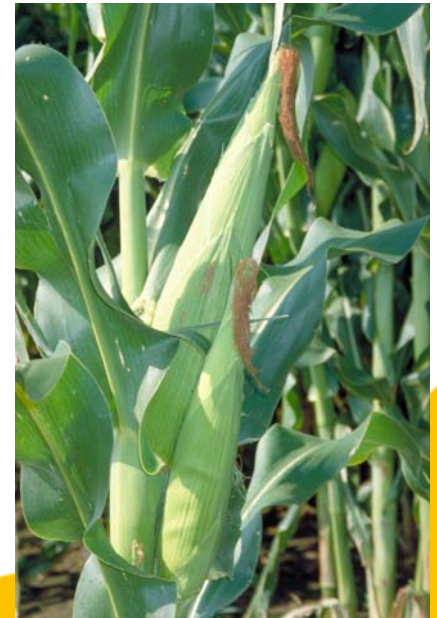
2. Soda-AQ Pulping with TCF Bleaching

	Brightness, % ISO	Freeness, CSF
Pulping, 11 % NaOH, 0.1 % AQ, 140 C, 60 minutes	14-25	360-423
Bleaching, Q-P-P	78-82	280-332

The Deficiencies

- “Traditional” pulping and bleaching approaches do not remove or passivate pith
- The pith breaks up in the bleaching sequence (especially acid stages), reducing freeness significantly

A New Approach --
The E-A-Z-P Process...
US Patent Number 6,302,997



Attributes

- A simple, 2- or 3-stage process (3- or 4-stage including screening)
- Requires no raw material depithing
- Produces bright, free-draining pulp with good papermaking properties

Two Key Concepts

- *Lowered pulping intensity* – many processes tend to “overcook” cereal straws, actually reducing lignin removal
- *In-process treatment of pith* – deals with pith and parenchyma chemically in the process, rather than using mechanical depithing of the raw material

E – Alkaline Extraction

- Milder than a typical soda or soda-AQ cook
- Typical conditions
 - NaOH charge = 12-14 % on OD
 - Temperature = 115-118 C
 - Time = 60 minutes
 - Liquor:Fiber = 8:1
- Kappa = 18-20

A - Acid Chelation

- Can use nitric, sulfuric, or acetic acids
- Typical conditions
 - Acid charge = 5 % on OD
 - DTPA (chelant) charge = 0.5 % on OD
 - Consistency = 10-15 %
 - Temperature = 60 C
 - Time = 30-60 minutes

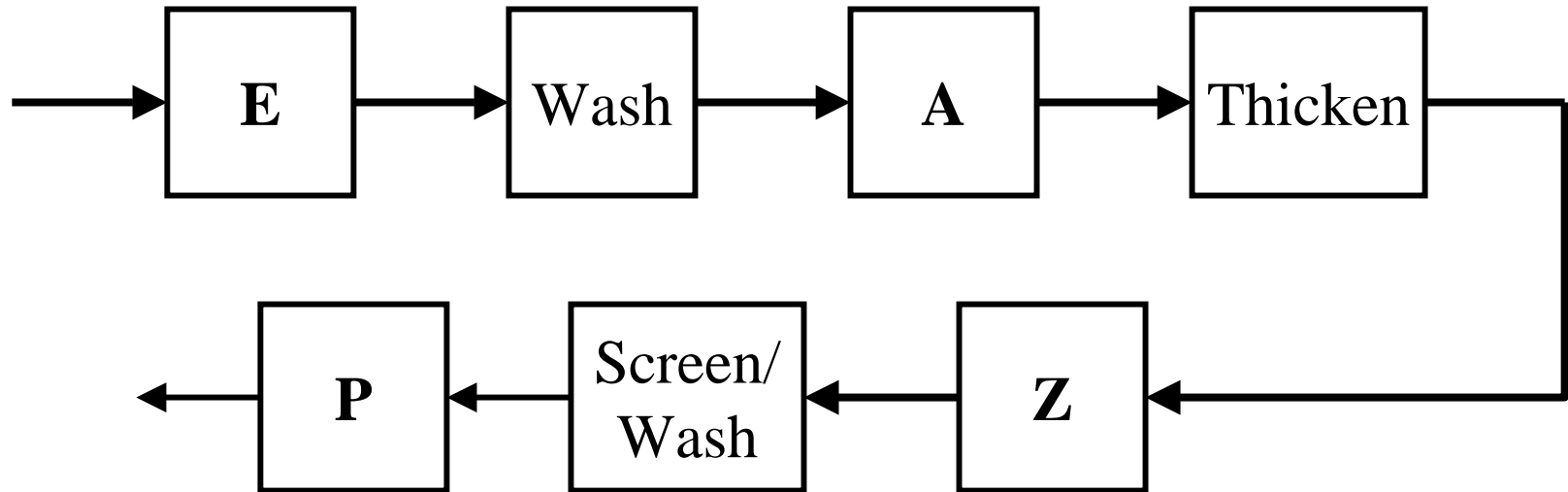
Z – Ozone Treatment

- Typical conditions
 - Ozone consumption = 0.7 – 1 % on OD
 - Consistency = 3 %
 - pH = 1.5
 - Temperature = 30 C
 - Time = 10 minutes

P = Pressurized Peroxide Bleaching

- Typical conditions
 - Peroxide charge = 4 % on OD
 - NaOH charge = 5 % on OD
 - DTMPA (chelant) charge = 0.2 % on OD
 - MgSO₄ and Silicate charge = 0.5 % on OD
 - Consistency = 10-12 %
 - Temperature = 105 C
 - Time = 90 minutes

Basic Process Flowsheet



Experimental Methods



Raw Material

- Corn stover from Iowa, aged 1 year
- Composition = 70 % stalk, 30 % leaves and husks
- Prior to pulping, soaked in hot water (130 F) for 30 minutes, then drained

E Stage

- Carried out in Paprican-designed “finger reactor”
- Good for emulating screw-type digester
- Cooked fiber passed through disk refiner, 0.035-inch gap
- Washed



A Stage

- Carried out in sealed plastic bags placed into a heated water bath
- Nitric acid used
- Kneaded periodically

Dewatering Stage

- Acid-treated fiber centrifuged in fine-mesh poly bag for 5 minutes
- Discharge consistency = 35 %
- Z stage followed immediately

Z Stage

- Centrifuged pulp diluted to 3 % consistency with distilled water
- Acid added
- Put into modified blender with non-cutting rotor and gas sparger into mixing zone
- Ozone gas of known flow rate and concentration injected into blender; excess taken off top and bubbled into kill solution
- Reacted for 10 minutes

Screening/Washing Stage

- Z stage pulp diluted with distilled water to approximately 0.5 % consistency
- Screened through vibrating flat screen with 0.010-inch slot
- Accepts dewatered to 35 % consistency

P Stage

- Carried out in 3-liter bombs placed into heated oven on rotating rack
- Chemicals mixed in using industrial-style kitchen mixer

Process Results

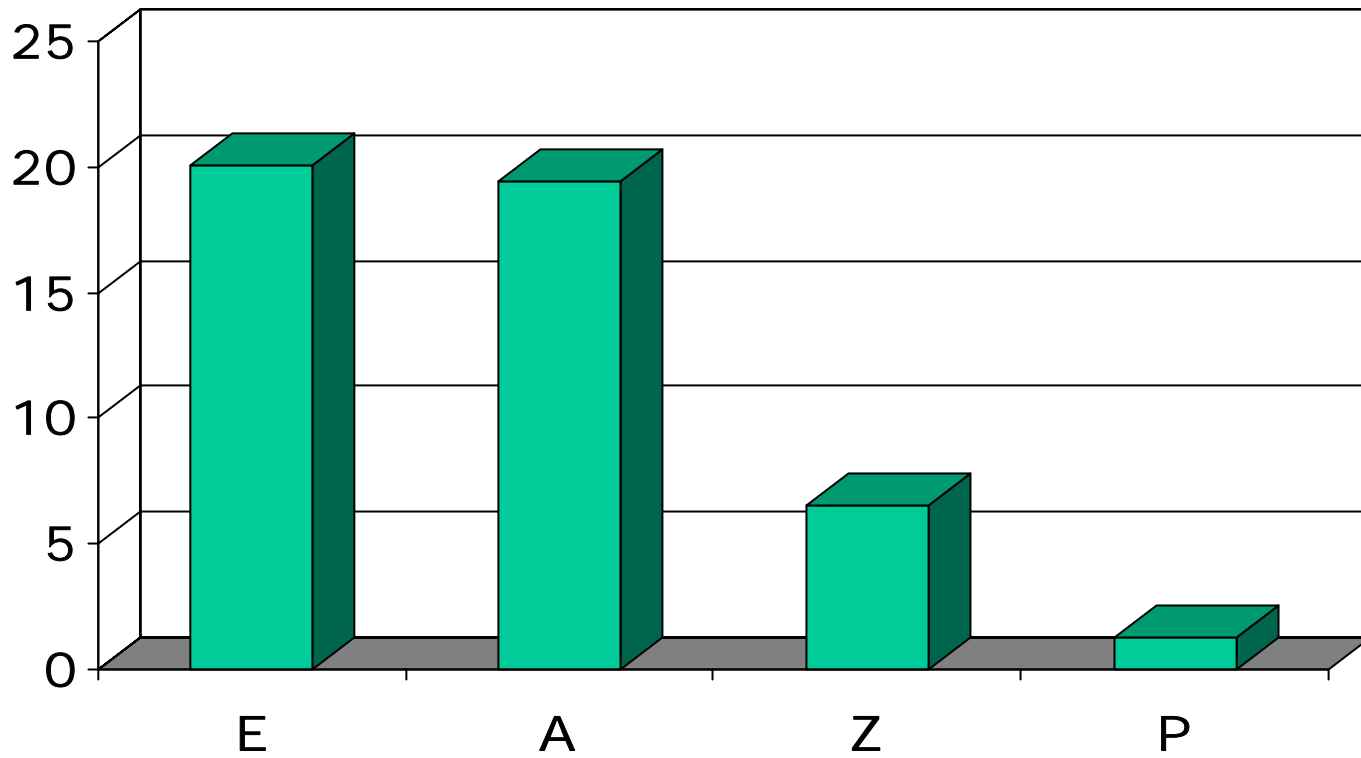


Results by Stage

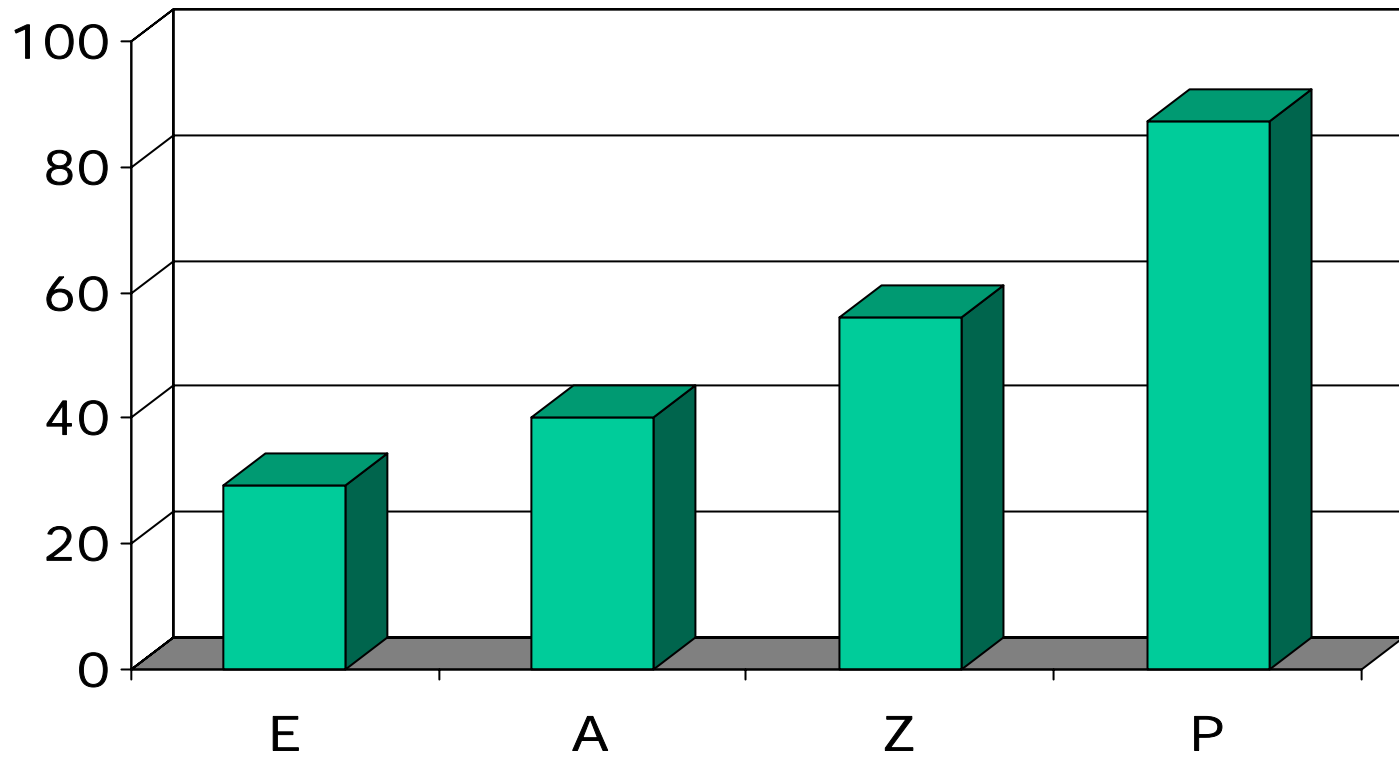
	Kappa	Brightness % ISO	Freeness CSF	% Yield
E	20.1	29.2	---	57.9
A	19.4	40.2	---	94.7
Z	6.5	56.1	587	75.3
Screening	---	---	---	98.7
P	1.3	87.4	619	93.2

Overall Yield = 38.0 %

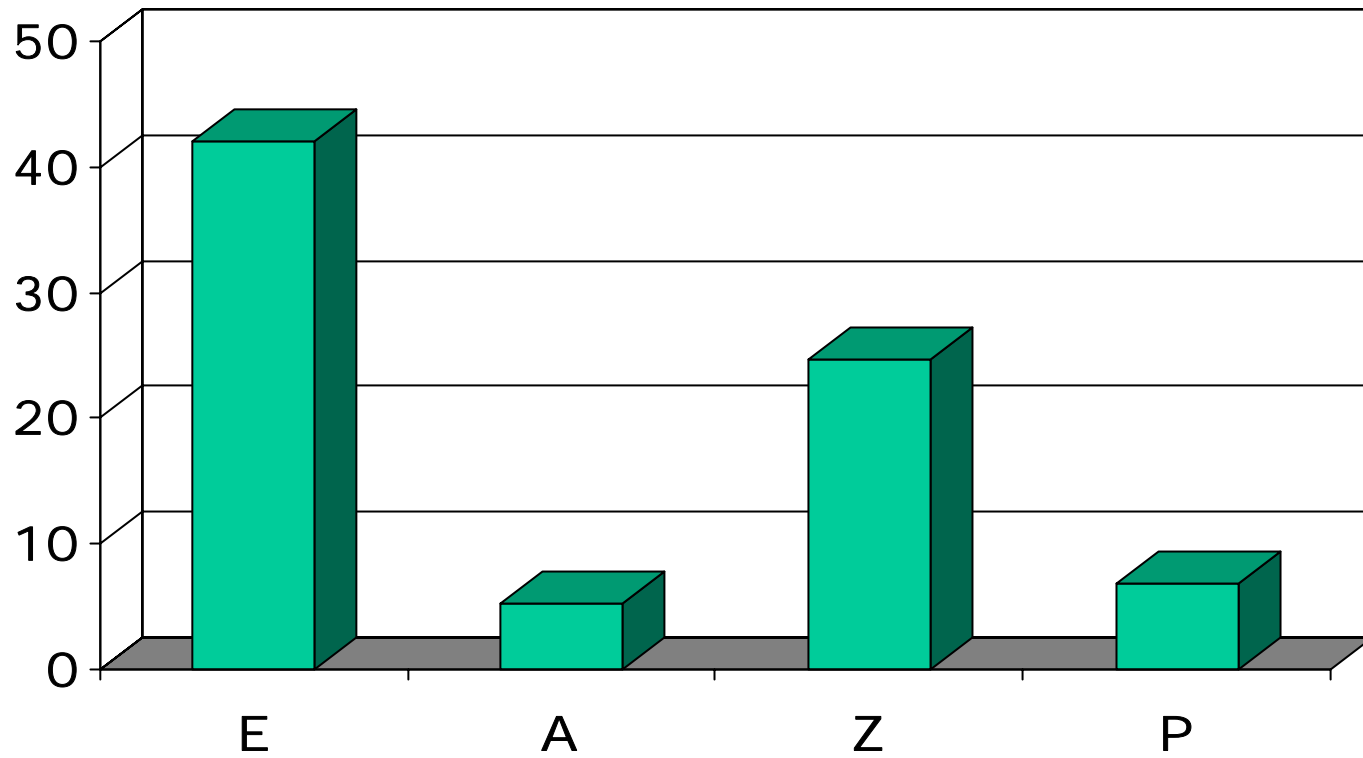
Kappa Reduction



Brightness Development



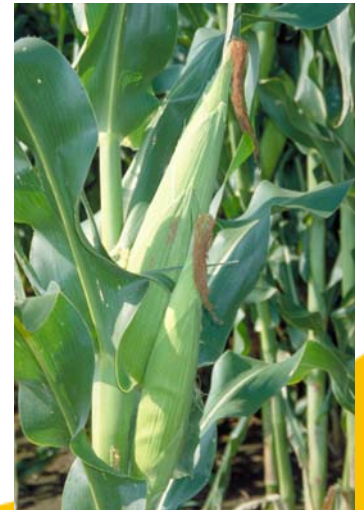
Yield Losses per Stage



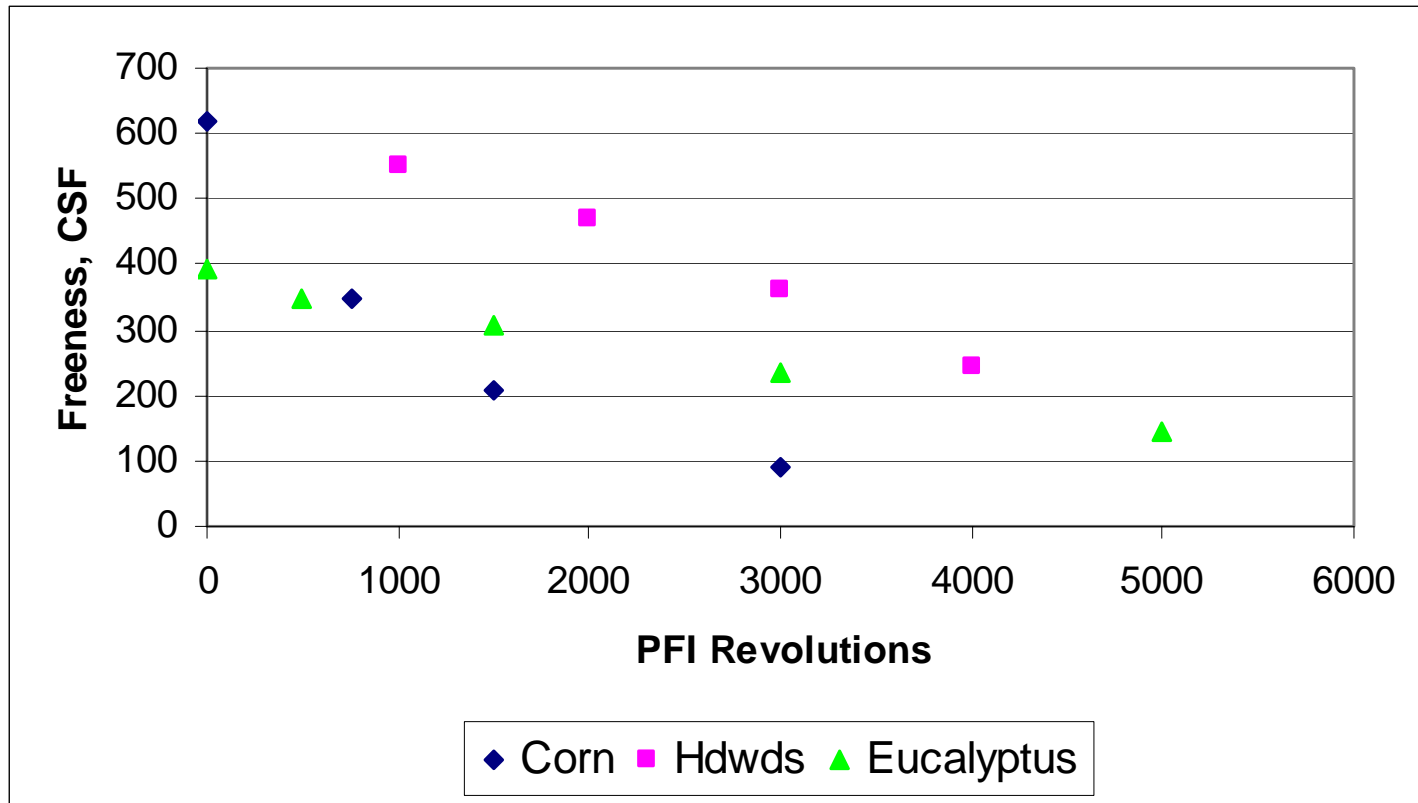
Bleached Fiber Properties

	Cornstalk	Mixed Hardwoods	Eucalyptus
Avg. Length, mm (Length-wtd)	1.09	1-1.07	0.65
Coarseness, mg/10m	1.06	1.23	0.95
Fines, % of total fibers (by number)	41.3	57.8	---

PFI Refining and Handsheets Testing



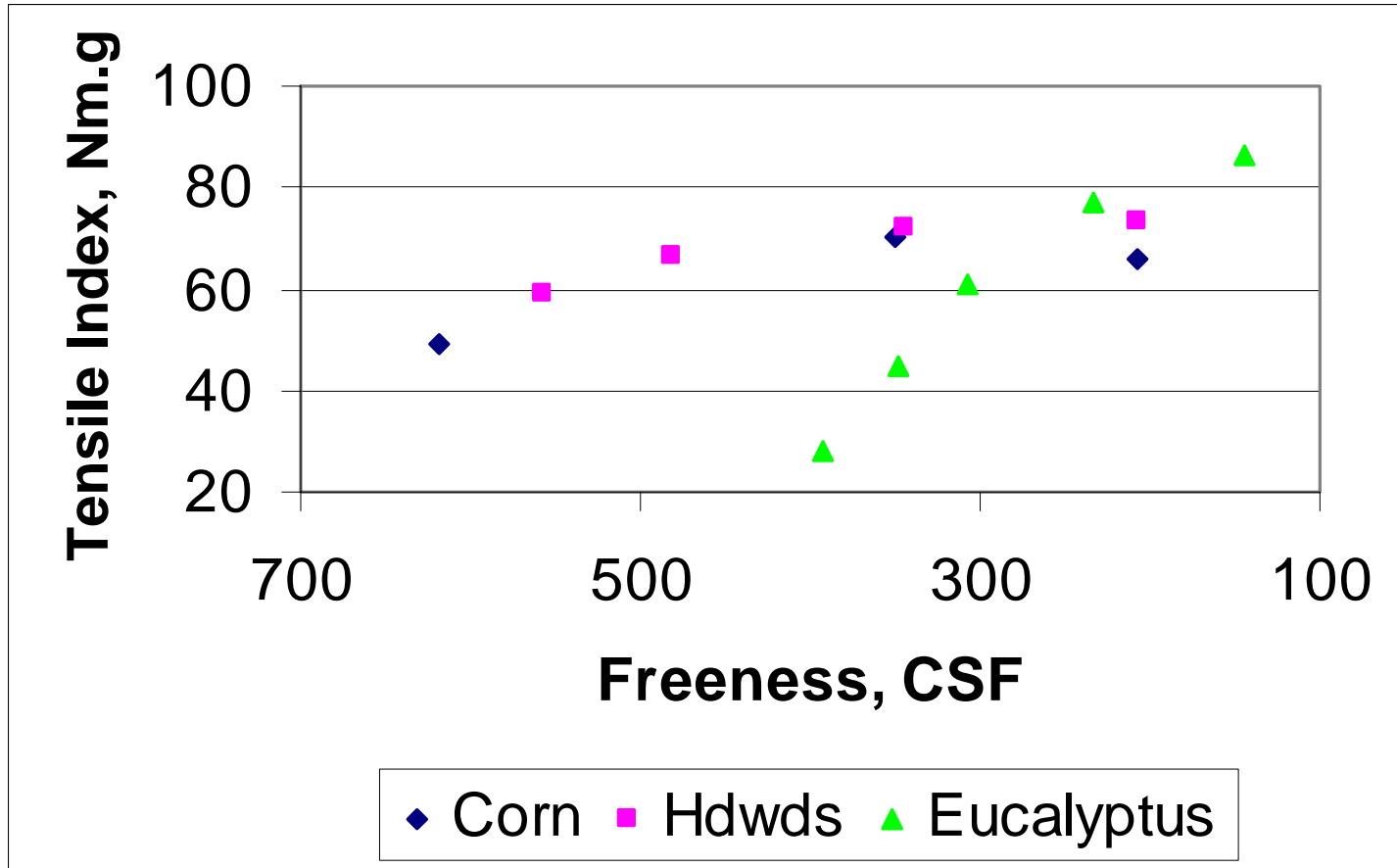
Refining Response



>Hdwds = NCSU kraft study data, mixed southern hardwoods

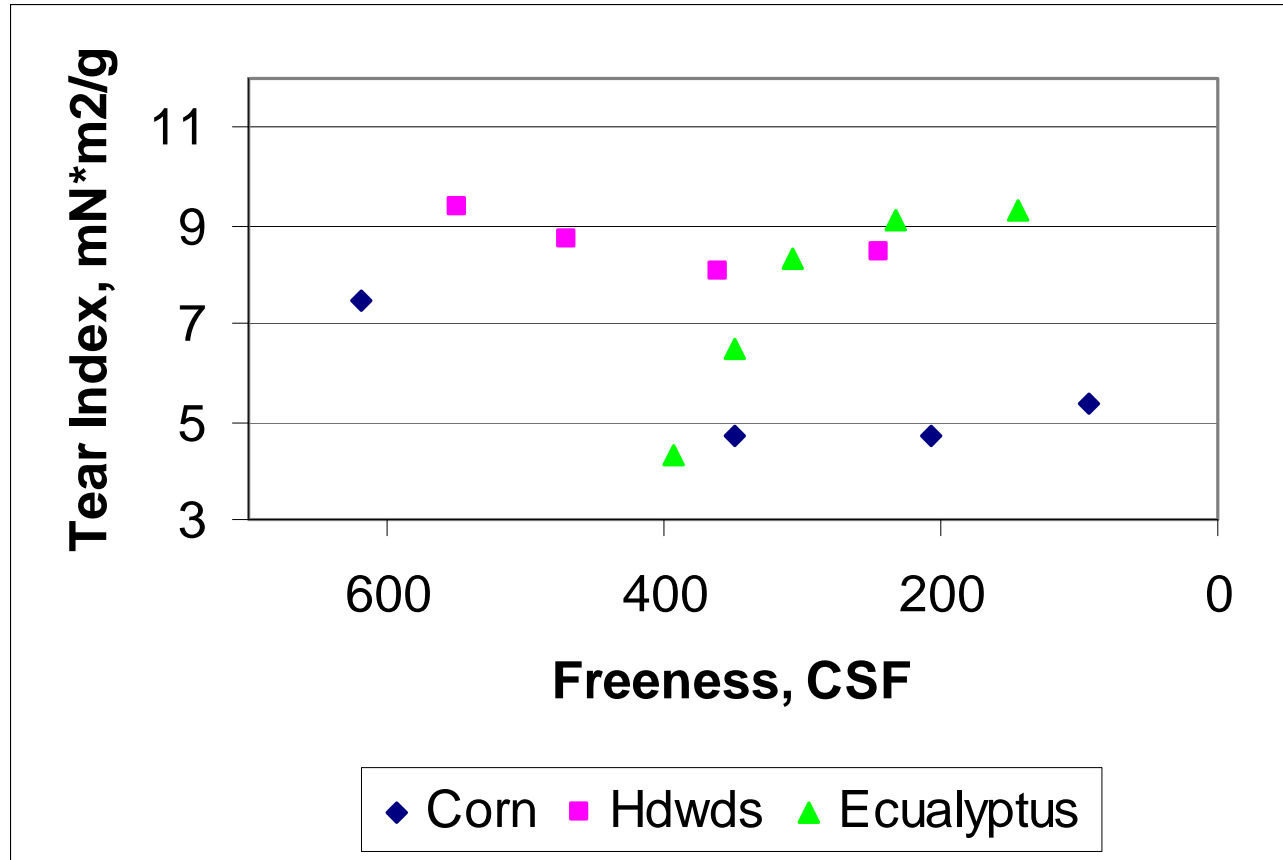
> Eucalyptus = N.I.S.T. data, Study 8496

Tensile Strength



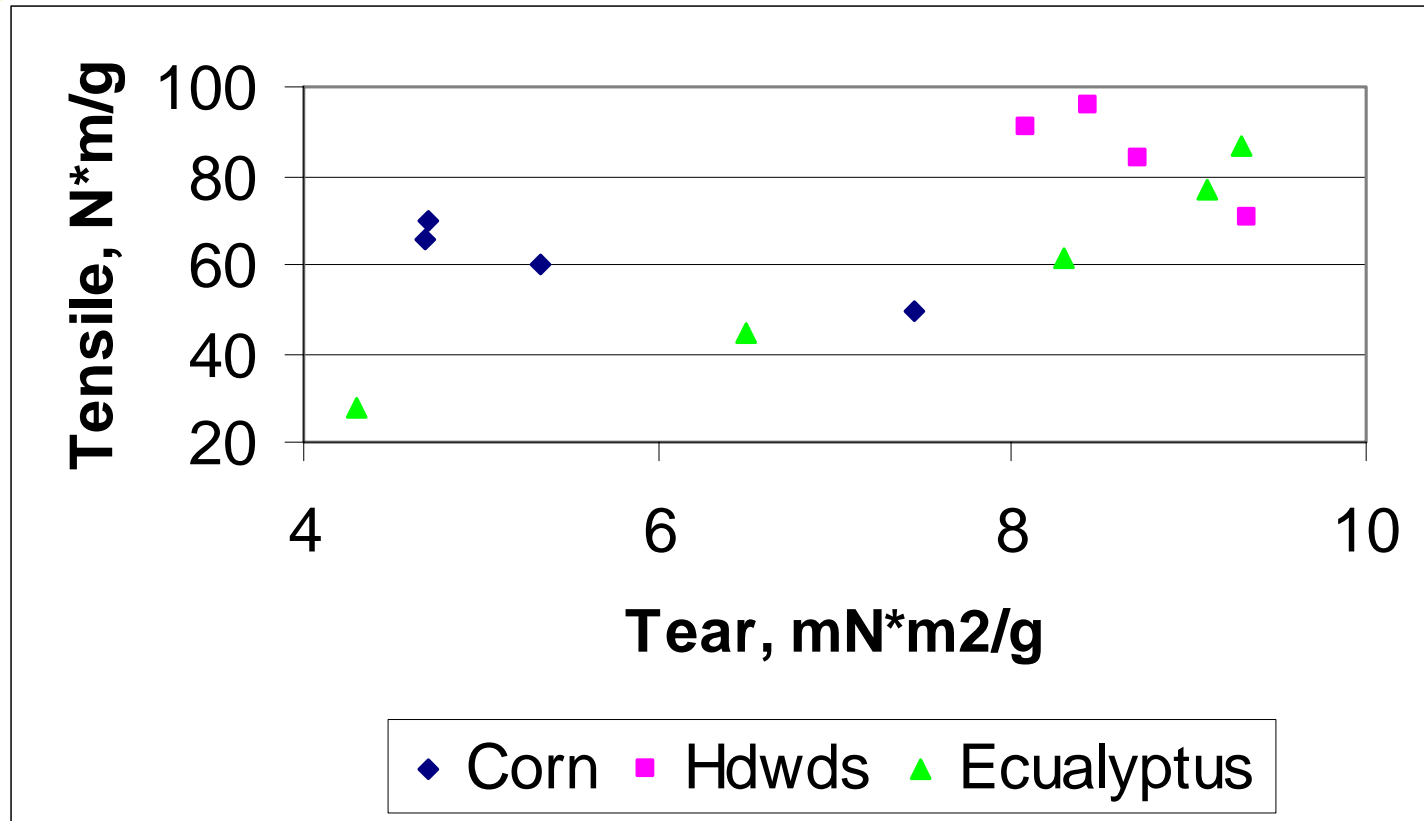
- > Hdwds = NCSU kraft study data, mixed southern hardwoods
- > Eucalyptus = N.I.S.T. data, Study 8496

Tearing Strength



- >Hdwds = NCSU kraft study data, mixed southern hardwoods
- > Eucalyptus = N.I.S.T. data, Study 8496

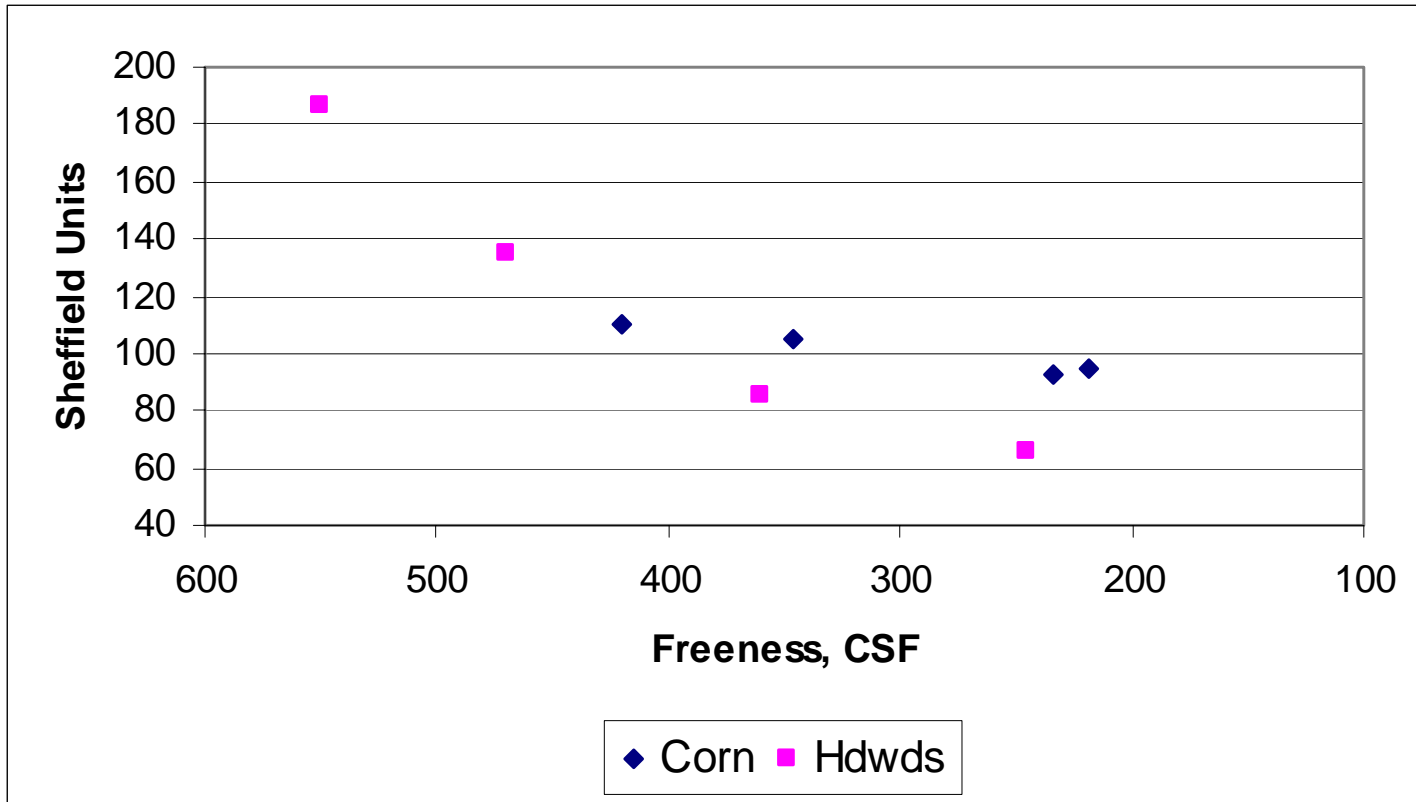
Tensile vs. Tear



>Hdwds = NCSU kraft study data, mixed southern hardwoods

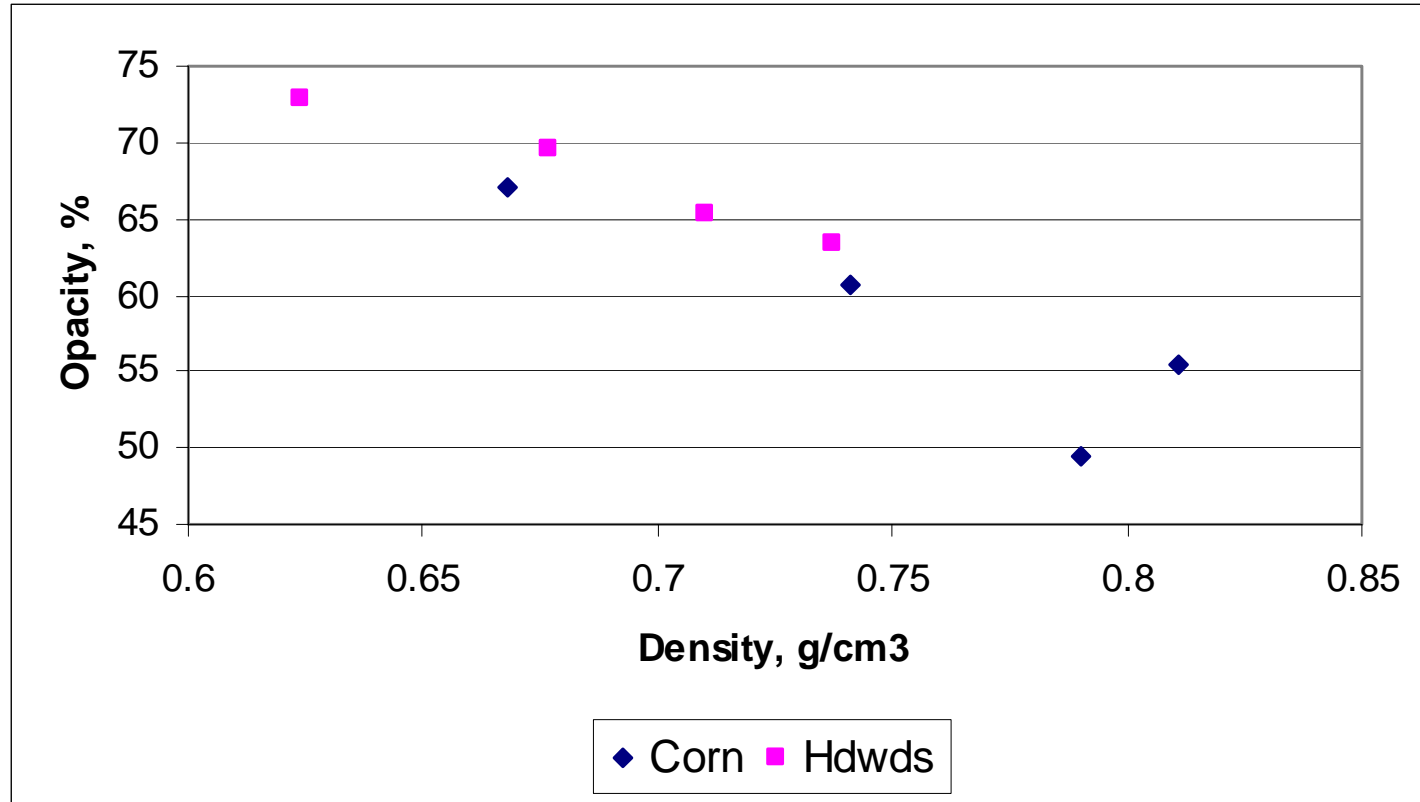
> Eucalyptus = N.I.S.T. data, Study 8496

Smoothness



>Hdwds = NCSU kraft study data, mixed southern hardwoods

Opacity (Printing)



>Hdwds = NCSU kraft study data, mixed southern hardwoods

Additional Work



Additional Work Completed

- Process works equally well with nitric, sulfuric, acetic acid
- Process works well with multiple alkali sources
- The chelant may be added into the Z stage, reducing the process to E-Z-P
- A D stage may be used instead of P

Ongoing Work

- Use of self-generated acetic acid from a pre-hydrolysis stage in the A or Z stage
- Effect of stover pre-treatment (e.g. shredding) on process results
- Application of process to other pithy nonwoods -- bagasse