

**FORTY YEARS OF  
WANDERINGS IN  
TEXTILE CHEMISTRY  
PROCESS AND  
MATERIALS RESEARCH**

**DR. FRED L. COOK  
MSE/GEORGIA TECH**

**OLNEY MEDAL ADDRESS  
AATCC INTERNATIONAL CONFERENCE  
WILLIAMSBURG, VA  
APRIL 21, 2016**

# COMBINED DESIZE- SCOUR-BLEACH

## OPPORTUNITIES

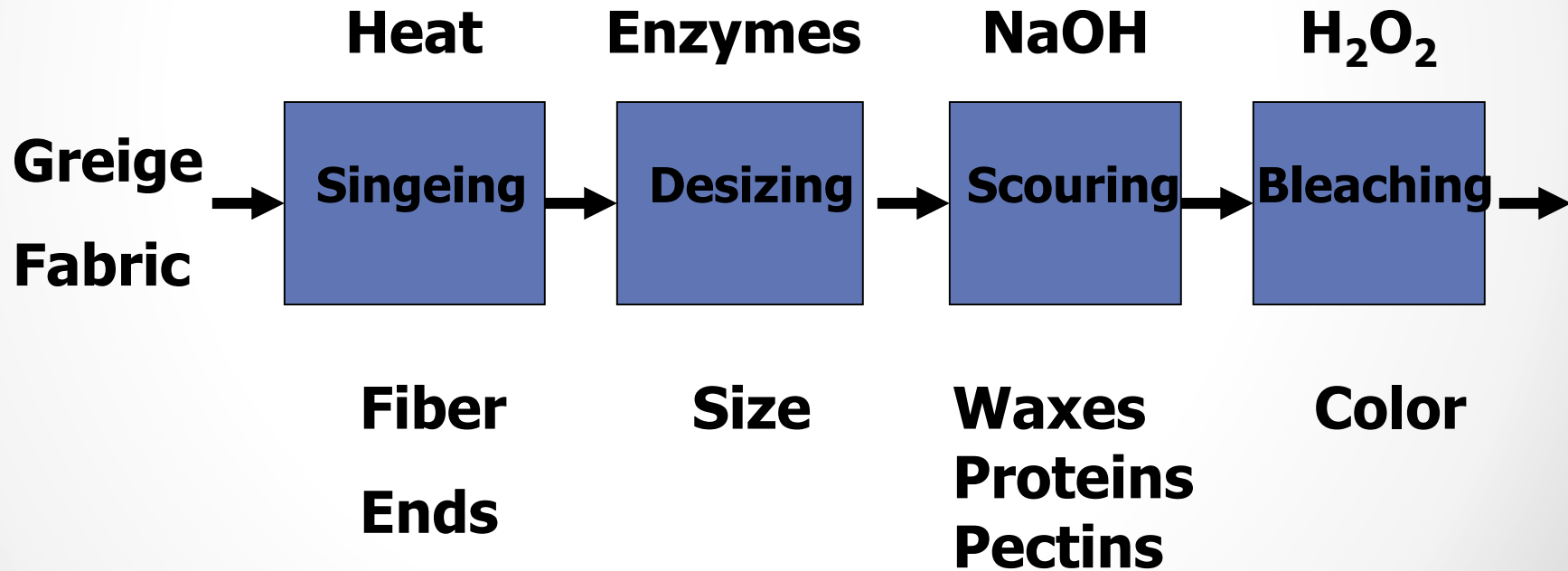
- Current Continuous Process Requires Three Separate Padding-Steam-Wash Steps/Sections
- Process is an Energy, Water, Space, Chemical, etc., “Hog”

## RESEARCH APPROACH

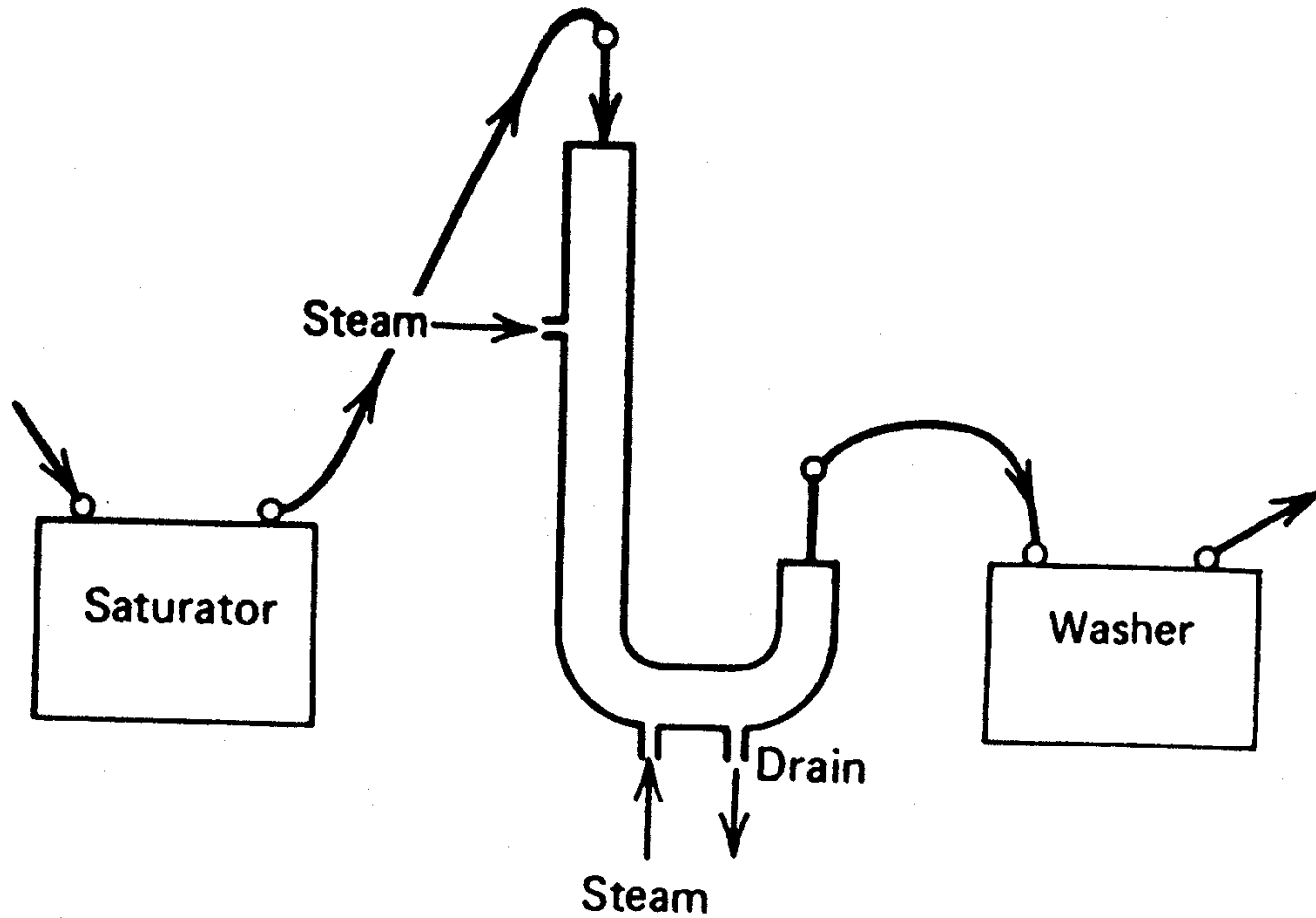
- Create a Chemical Formulation that will Allow a Single Step Path to Fully-Prepared, Cotton-Containing Woven Fabrics, i.e., a Combined Desize-Scour-Bleach Process

Cook, F. L., et al., 1982. *Evaluating Combined Preparation Processes for Energy And Material Conservation*, Textile Chemist and Colorist, 14(1), 10-23.

# ***PREPARATION RANGE COTTON AND COTTON BLEND***



# CONTINUOUS PREPARATION RANGE



# DSB FORMULATION

## INITIAL FORMULATION (50/50 COTTON/ POLYESTER FABRICS)

- |                                      |  |
|--------------------------------------|--|
| 1. NaOH                              | Comparable to Scour Formulation            |
| 2. NaSiO <sub>2</sub>                | “ “ “                                      |
| 3. Chelate                           | “ “ “                                      |
| <b>4. H<sub>2</sub>O<sub>2</sub></b> | <b>Huge Excess Compared to Bleaching!!</b> |
| 5. Surfactant                        | Comparable to Scour Formulation            |

## INITIAL RESULTS

- All properties consistent with plant standards except whiteness (achieved ~ 70 on the whiteness scale, needed ~77)

## MAGIC CHEMICAL

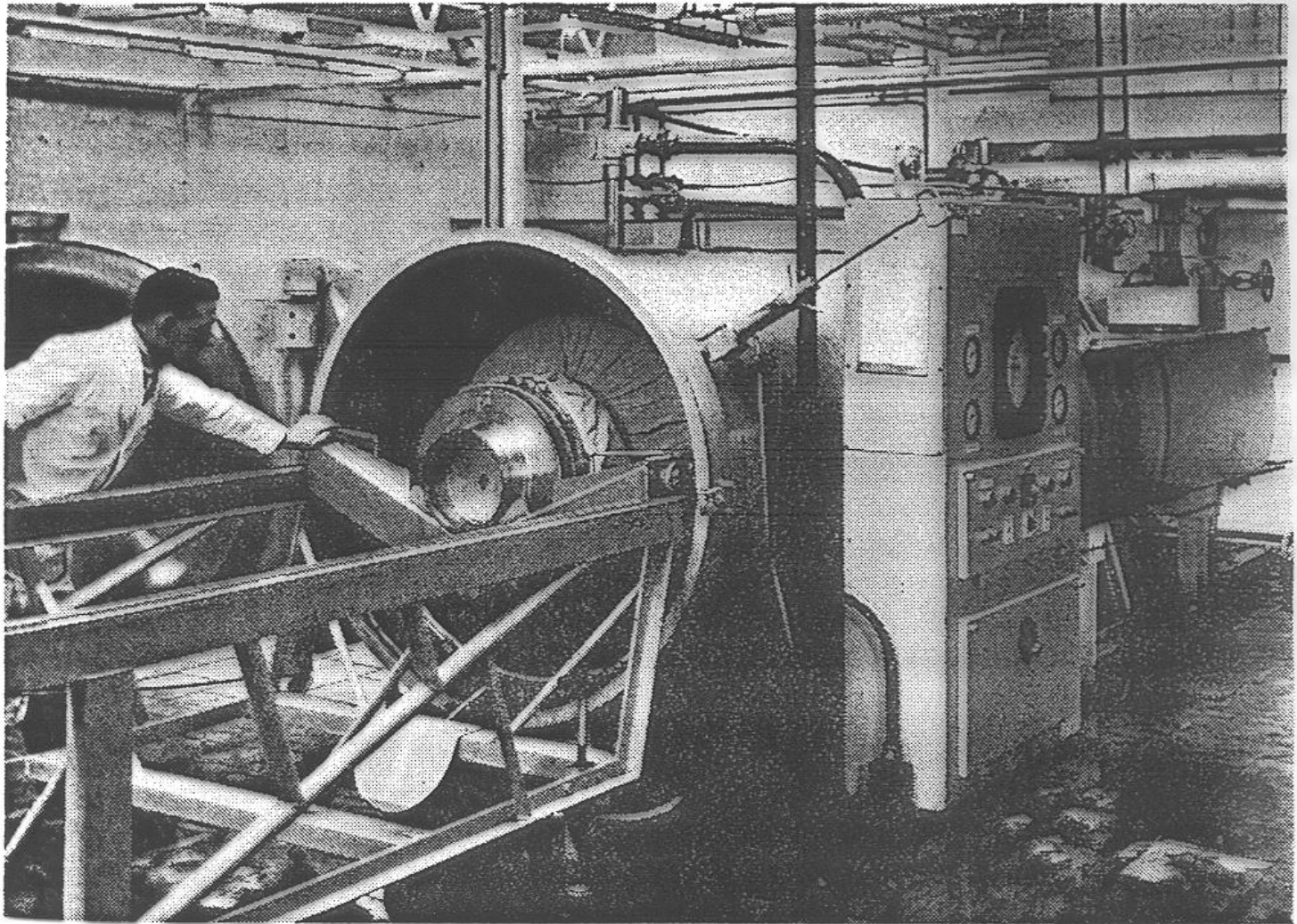
- Tetrapotassiumperoxydiphosphate, **KPP** (FMC Corp.)
- Required only ~1% w/w concentration in pad to give desired whiteness

# DYEING/PRINTING OF NOMEX®

## ALTERNATE DYEING PROCESS

- STX System from Rhone-Poulenc (France)
  - Based on 90% Perchloroethylene (PERC)/10% Methanol
  - Ionic Dyes Soluble in the Mixture, but not in PERC
  - Process is Based on the Differential Boiling Points between Methanol (~ 65C) and PERC (~121C)
  - R-P Developed the System for Acid Dyeing of Nylon (Commercialized in a Beam Dyeing Platform on Tufted Carpet in Belgium)
- 1. Cook, F. L. and Averette, M. L., 1983. *Evaluation of the STX System for Solvent Dyeing of Industrial Fabrics, Part I: Nomex aramid fabric*, Journal of Industrial Fabrics, 1(4), 33-46.
- 2. Cook, F. L. and Averette, M. L., 1983. *Evaluation for the STX System for Solvent Dyeing of Industrial Fabrics, Part II: Kevlar aramid and PBI fabrics*, Journal of Industrial Fabrics, 2(1), 11-27.

# STX BEAM DYEING OF CARPET



# STX DYEING OF NOMEX

## OPPORTUNITY

- Carrier being used by Southern Mills (now TENCATE) to Dye Nomex in Jet Machines was Acetophenone
  - Acetophenone

## APPROACH

- Investigate the Plasticization Capability of the PERC/Methanol Mixture on Woven Nomex Fabrics with Cationic Dyes
- Matched all of SM's Major Color Lines with the STX System
- Attempted Coloration of both Staple and Filament Yarn Kevlar Fabrics with Mixed Success
  - Up to Medium Shades with Kevlar Staple Fabrics, Only Tinted Filament Fabrics

## CONCLUSION

- STX System is a Viable, Closed-Loop, Waterless Process for Solid Shade Batch Dyeing of Nomex Fabrics (Type 455)
- 
-

# PRINTING OF NOMEX FABRIC

## OPPORTUNITY

- In the late 70's, the Military wanted to Expand Nomex Uniforms to enclosed personnel (tank drivers, APC Operators, etc.), but could not Develop a Viable Printing Process for Camouflage, Resulting in a NATICK RFP
- In Partnership with Southern Mills, Won the Project to Develop the First Successful Printing Process

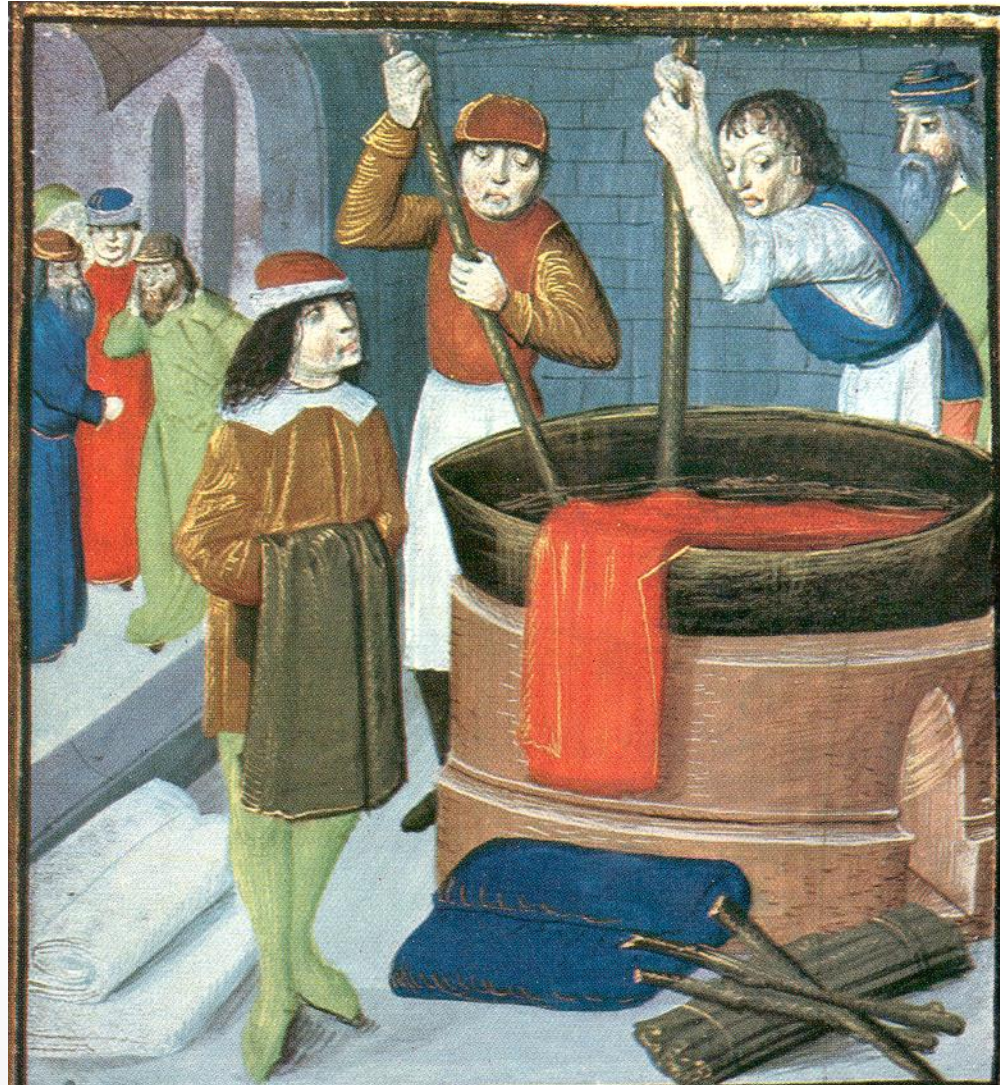
## KEY DISCOVERY

- Had to Solvent Plasticize the Nomex Fiber before the Fabric was Printed with Conventional Techniques
- Based on Solubility Parameters, Selected DMSO, Padded it onto the Nomex Fabric at 80C and then Dried

## END RESULTS

- Plasticization Followed by Printing was Incorporated Commercially by Southern Mills and Burlington Industries

# DYEING IN THE RENAISSANCE



# PLICTHO DYEING RECIPE

Liquor, or magistery that dyes each thing you want by putting in strong water of lime.

First make a strong solution in the manner below.

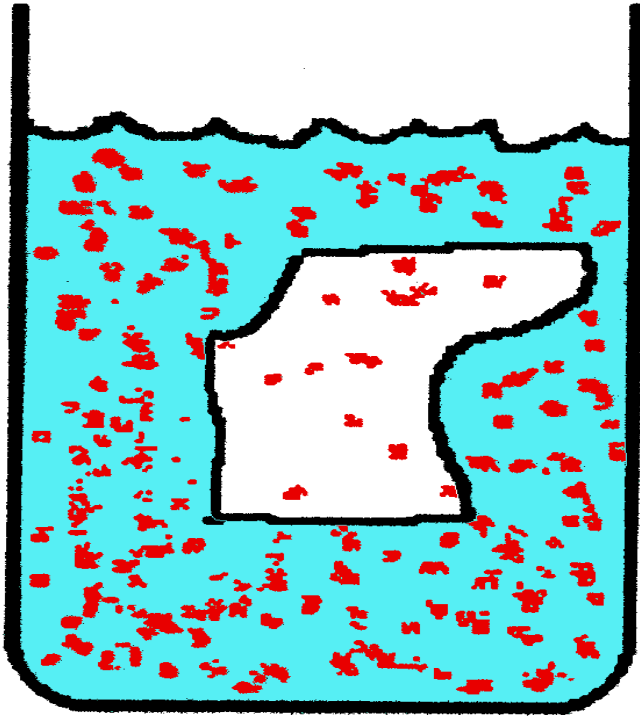
11. Measure first common water	part	half
Human urine	part	half
Strong white vinegar	part	half
Live lime	part	one
Ashes of oak	part	two
Orpiment	part	half

All these things are boiled until lowered by one third and this liquid is made to pass through a small sack or a dog-tongue-shaped felt. This you will save for your needs and in this water you will put roche alum and alum of lees.

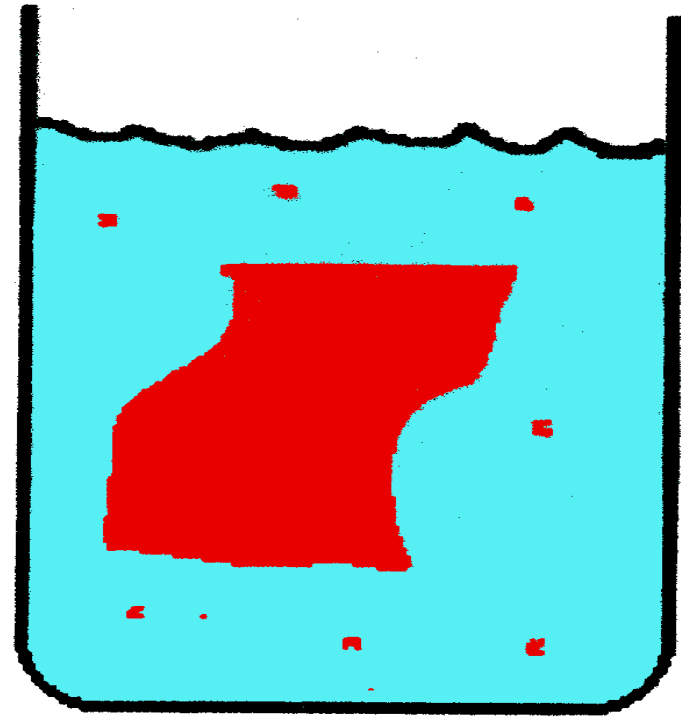
When you want to dye a color take of the color you want to work with and mix it with this magistery. Put in what you want to dye and then make it boil on the fire. This you will do in a glass vessel. Set this vessel beneath horse manure for five days or more and this dyes for ever. Know that if you add red lead it will become red, that is scarlet, and if you want violet put in brazil and thus for each other color.<sup>41</sup>



# AQUEOUS BATCH PROCESS



START



END

## EXHAUST DYEING

● Tincher, W. C., Cook, F. L., Carr, W. W., Olson, L. H., and Bergenthal, J., 1985. *Reconstitution and Reuse Of Dyebaths in Carpet Dyeing*, Journal of the Society of Dyers and Colourists, 101(11), 350-354.

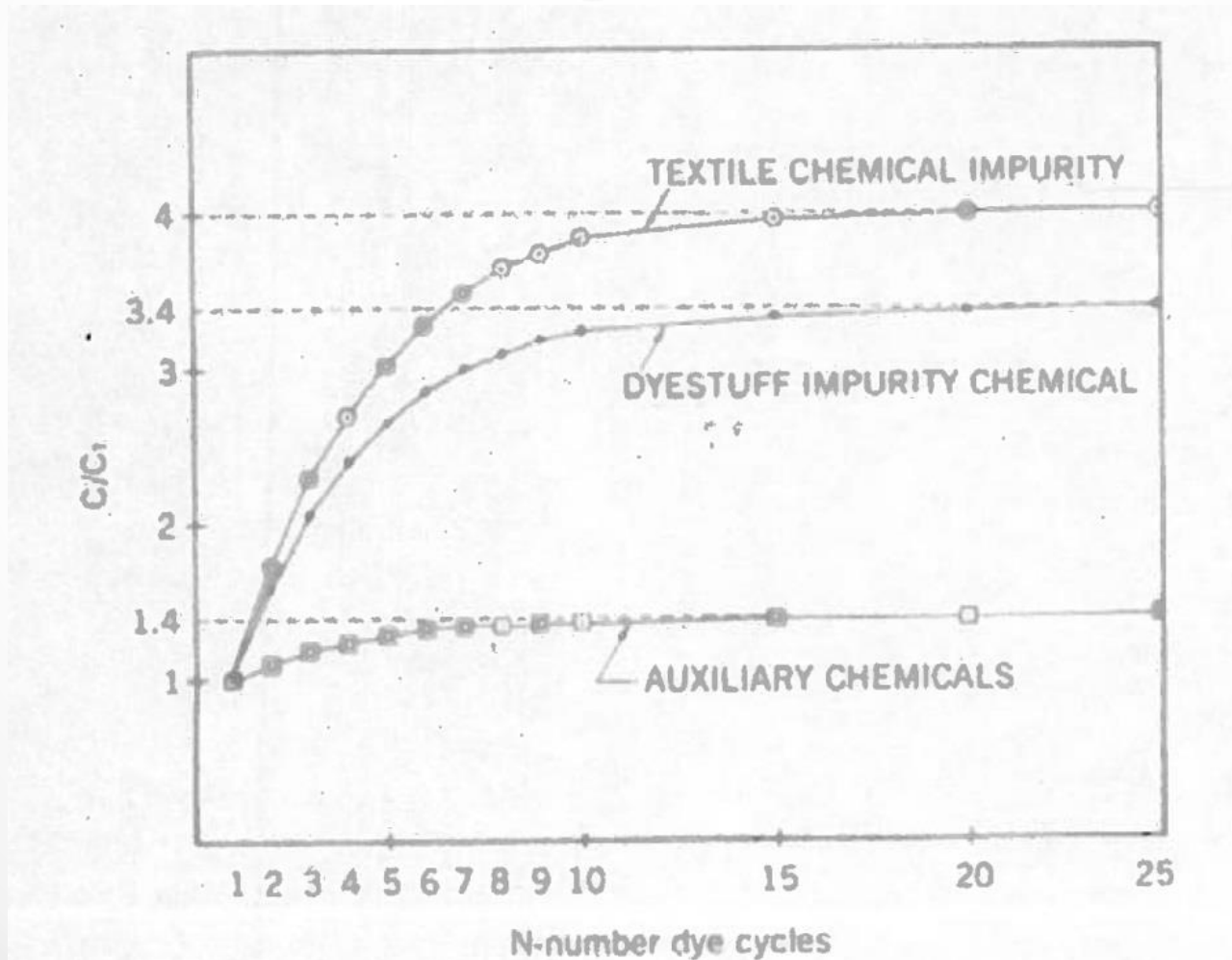


# CHANGES IN DYE BATH

	START	END
<u>Temp.</u>	Cold	Hot
<u>Dye Conc.</u>	High	Very Low
<u>Auxilliary Conc's.</u>	Higher than Required	Unchanged
<u>pH</u>	As Required	Unchanged
<u>Goods</u>	White	Dyed
<u>Liquor Ratio</u>	As Required	~ Unchanged



# IMPURITY CONCENTRATION VARIATION WITH NUMBER OF CYCLES (single-shade reuse sequence)



# SPECTROPHOTOMETRIC BASICS

$$T = I_f / I_o = \text{A Decimal}$$

$$\text{Absorbance, } A = -\text{Log } T$$

$A = abc$ , where:

$a$  = absorptivity constant

$b$  = cell path containing  
dye bath = 1 cm

$c$  = dye concentration in g/l

$$A = kc + 0$$



# MATRIX EQUATION FOR A TRICHROMATIC DYE

SERIES (yellow, red, blue)

$$A_{410} = k_{11}C_y + k_{12}C_r + k_{13}C_b$$

$$A_{510} = k_{21}C_y + k_{22}C_r + k_{23}C_b$$

$$A_{600} = k_{31}C_y + k_{32}C_r + k_{33}C_b$$



# STEPS AT END OF INITIAL DYE CYCLE

1. Drop dye bath to holding tank
2. Extract dye bath sample
3. Inject dye bath through a sintered glass filter syringe into spec cell
4. If needed, half and dilute until  $A < 1$
5. Determine absorbance at each dye's  $\lambda_{\max}$
6. Substitute 3 A's into 3 x 3 matrix equation, solve for 3 dye C's
7. Rinse goods in dye machine
8. Drop rinse bath, and remove dyed goods
9. Load next batch of goods
10. Pump in exhausted dye bath from holding tank
11. Pump in the ~ 25% bath volume needed to reach the correct LR
12. Make up the concentrate of dyes and auxiliaries as required (amount needed for 25% bath make-up for the next dyeing, plus the additional dye needed to bring the recycled bath back to full concentration for the next dyeing)
13. Pump in the small volume dye/auxiliary concentrate
14. Conduct the dye cycle as usual, starting from the cooled down composite dye bath (140-160F)
15. Return to step 1



# PROVEN ADAPTABLE TO DIRECT DYEBATH REUSE

## Georgia Tech Research

1. Disperse dyeing of polyester packages and fabrics (pressure package, jet)
2. Acid dyeing of nylon men's hosiery and carpet
3. Reactive dyeing of cotton fabrics (100% and polyester blends)
4. Cationic dyeing of type 455 Nomex fabrics (pressure jet)

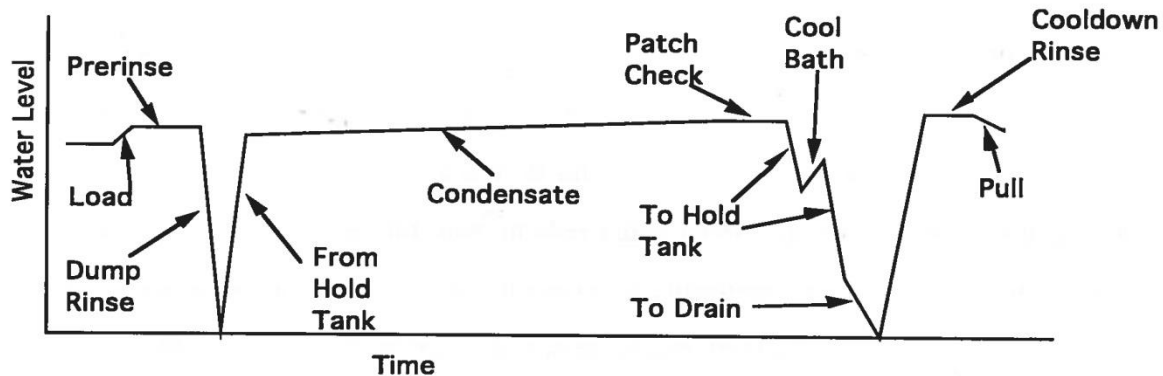
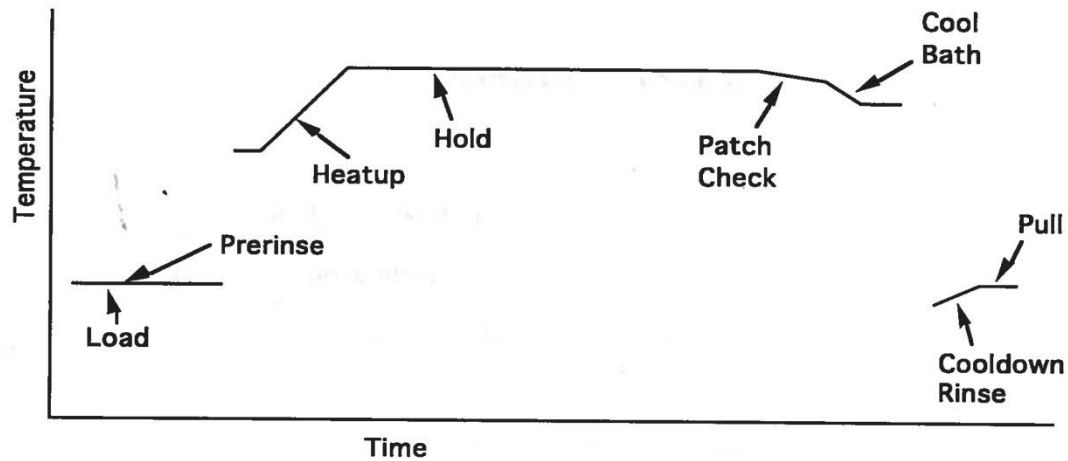
## Lessons Learned

- Had to follow the pattern of light series-medium series-dark series, could not go in reverse and maintain shade tolerance limits
- Could change one of the dyes, e.g., the red, in the middle of a sequence without violating shade tolerance limits
- Could start the dyeing hot as long as the made-up dye bath was < 160F
- Goods retain ~ 25% of spent dye bath after drop



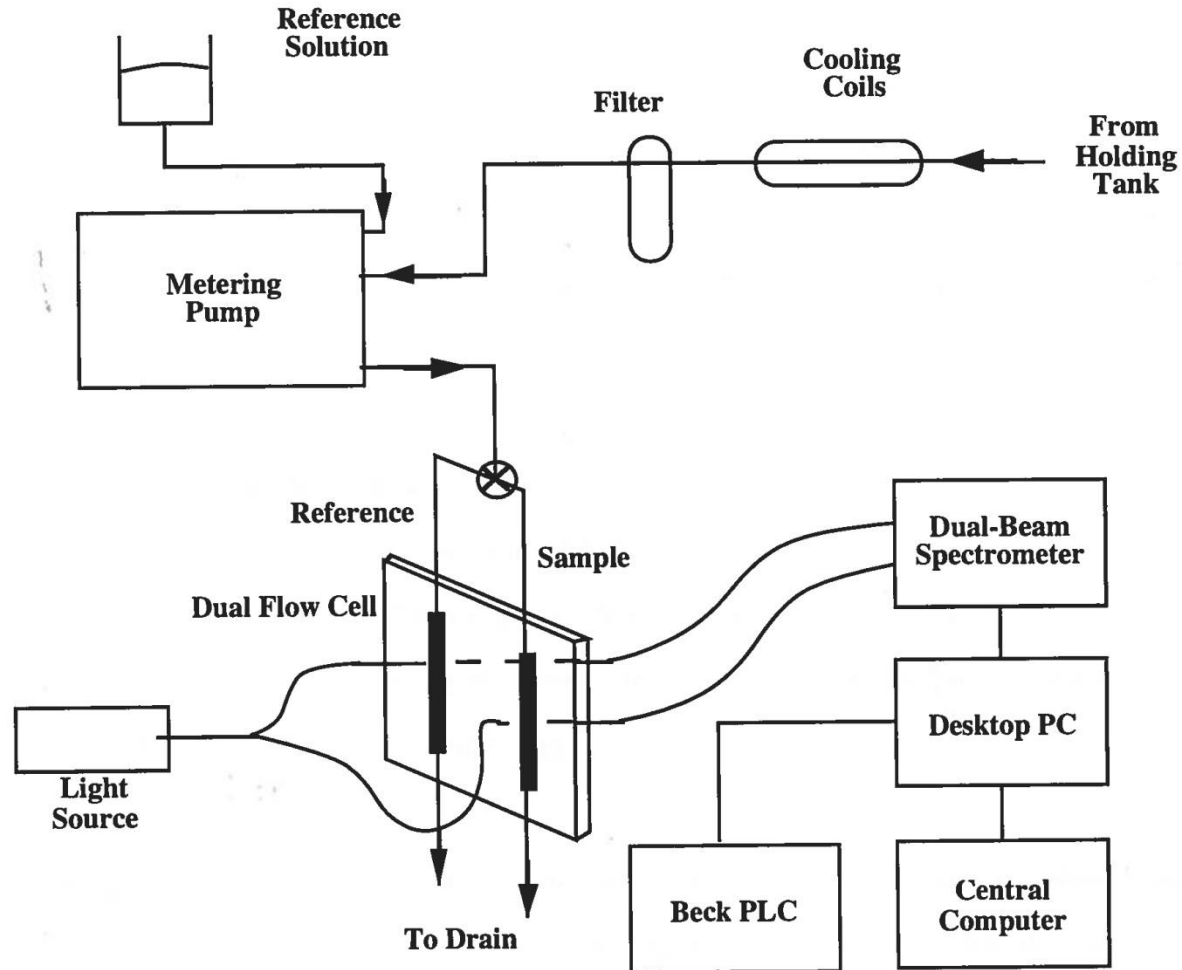
# IN-PLANT DEMO: ACID DYEING OF NYLON CARPET, SHAW IND.

## Modified Dyeing Process (22)



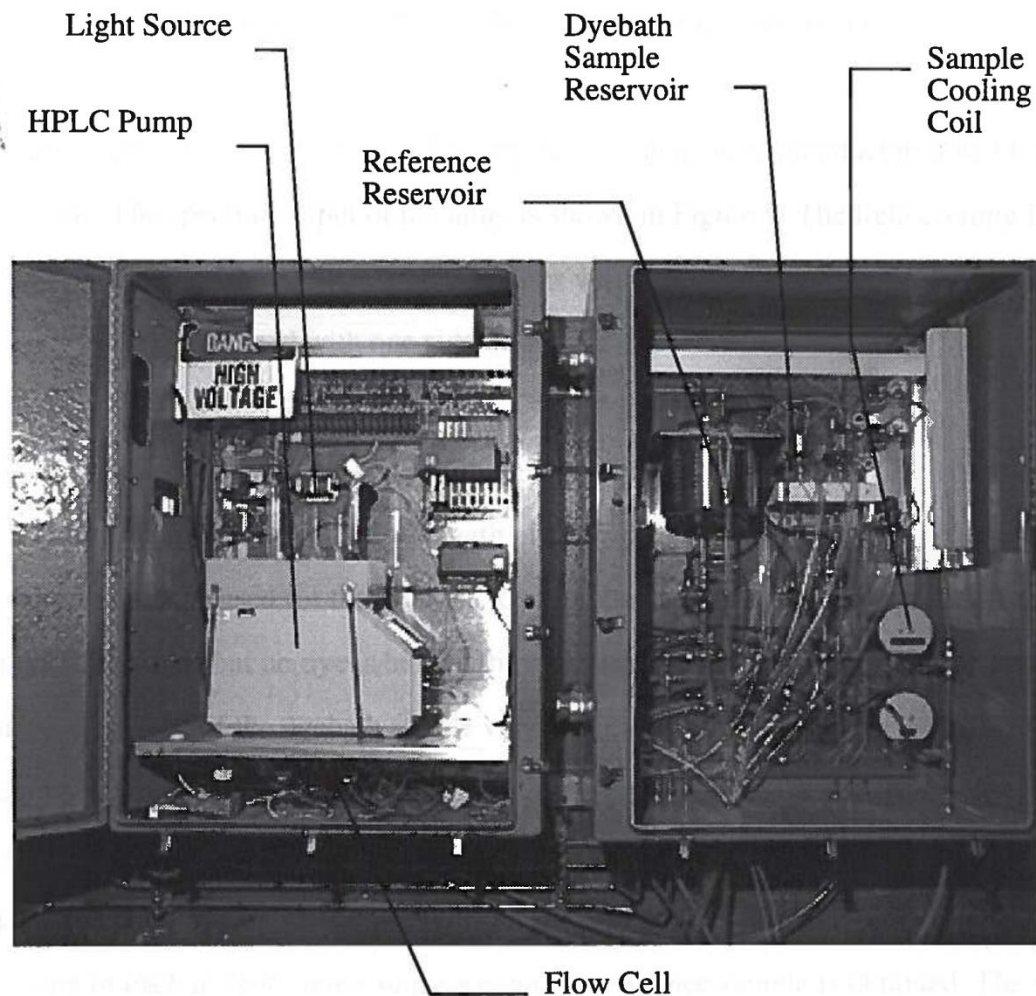
# CARPET, SHAW IND., DALTON, GA

## Analysis System (22)



# IN-PLANT DEMO: ACID DYEING OF NYLON CARPET, SHAW IND., DALTON, GA

## Analysis System and Enclosure



# IN-PLANT DEMO: ACID DYEING OF NYLON

## CARPET, SHAW IND., DALTON, GA

### Carpets Used in Automated Dye Bath Reuse Trial, Part II

Run	Style	Shade	Fiber	Result
1	51334 Affirmed	34402 Royal Blue	Nylon 66 Suessen Set	First quality, required 1 add
2	M0786 Voracious	00403 Blue	Nylon 66 Suessen Set	First quality, required 1 add
3	M0786 Voracious	00402 Blue	Nylon 66 Suessen Set	First quality
4	M0113 Graceful Impr	00504 Grey	Nylon 66 Suessen Set	First quality, required 1 add
5	51332 Seattle Slew	04532 Aged Pewter	Nylon 66 Suessen Set	Required 3 adds, then downgraded and redyed



# IN-PLANT DEMO: ACID DYEING OF NYLON CARPET,

## SHAW IND., DALTON, GA

### Analysis Results and Savings for Automated Dye Bath Reuse Trial, Part II

Run	Analysis Results (ppm)	Recovered Dye Quantity (grams)	Process Start Temp (F)	Energy Savings* (MBTU)	Auxiliary Chemical Savings (lbs)
1			Standard		
2	Yellow 0 Red 0 Blue 0.364	5	142	2.53	77.2
3	Yellow 0 Red 2.671 Blue 6.446	35 85	139	2.44	43.2
4	Yellow 3.160 Red 5.095 Blue 12.700	42 68 168	144	2.59	76.5
5	Yellow 5.121 Red 5.256 Blue 16.236	68 70 215	132	2.22	62.4



# IN-PLANT DEMO: ACID DYEING OF NYLON CARPET, SHAW IND., DALTON, GA

## Summary of Cost Savings through Automated Dye Bath Reuse

Total Savings	Cents per pound of Carpet	
	Nylon 6	Nylon 66
Auxiliary Chemicals	1.866	2.241
Dyes	0.161	0.161
Energy	0.603	0.603
Water/Sewer	0.274	0.274
<b>Total</b>	<b>2.903</b>	<b>3.278</b>



# SUCCESSFUL PLANT ADAPTATION

## Disperse Dyeing of Tufted Polyester Carpets, Becks

- Evans and Black Carpets, Dalton, Georgia
- 18 Becks on Floor, installed pump/manifold system and pumped spent dye baths to vacant beck
- Operated for several years, ~ 60 batches of carpet dyed before complete drop of bath



# SUCCESSFUL PLANT ADAPTATION

## Cationic Dyeing of Type 455 Nomex Fabrics (Jets)

- Southern Mills, Senoia, GA
- Jet machines, used holding tanks, acetophenone used as carrier
- Operated for 3 years until environmentally-friendly carrier was commercialized



# Rapid Continuous Dyeing Process for Heavy Weight Nylon 6,6 Carpet

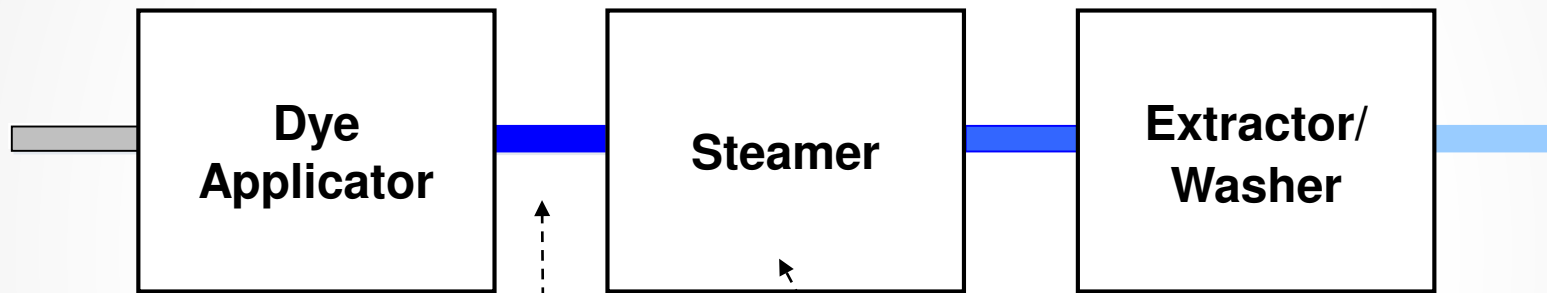
---

**Hongming Dong**

H. Dong, W. W. Carr and F. L. Cook, 2013. *Development of Rapid Continuous Dyeing Process for Heavy-Weight Nylon 6,6 Carpet*, JEFF, 8, June

# Continuous Dyeing Process of Tufted Carpet

---



*Wet pickup: 350-450%*

*Steaming Temperature: ~ 212 °F*

# Problem Addressed

---

20 oz/yd<sup>2</sup>



130 ft/min

32 oz/yd<sup>2</sup>



120 ft/min

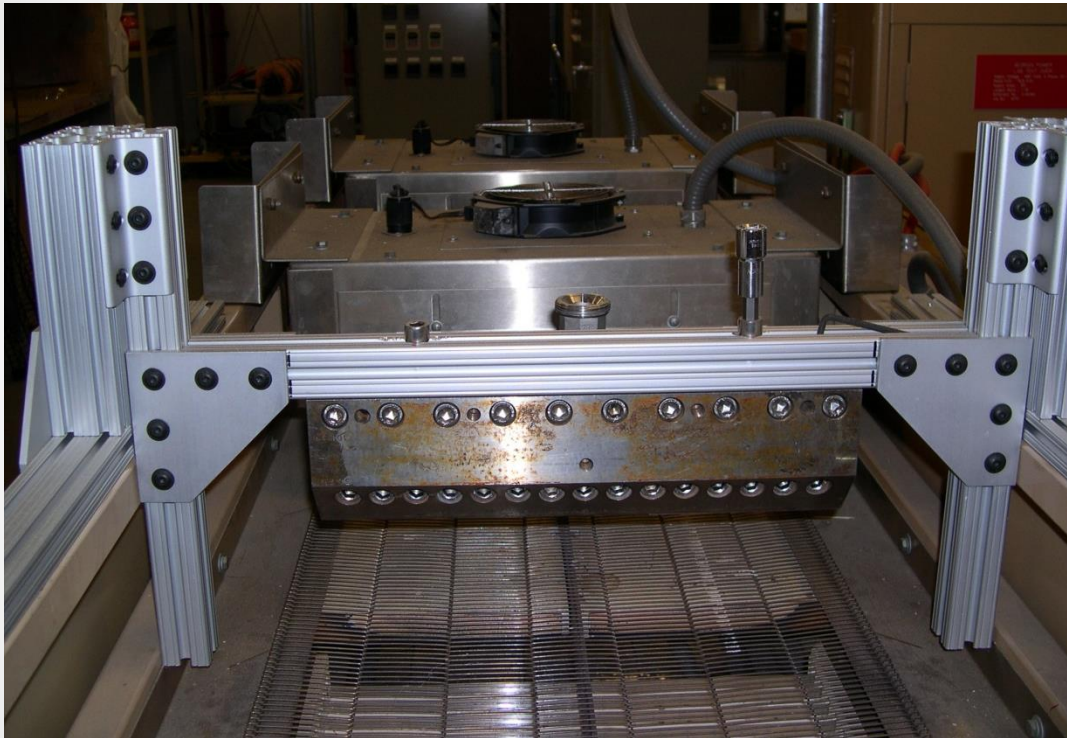
≥ 60 oz/yd<sup>2</sup>



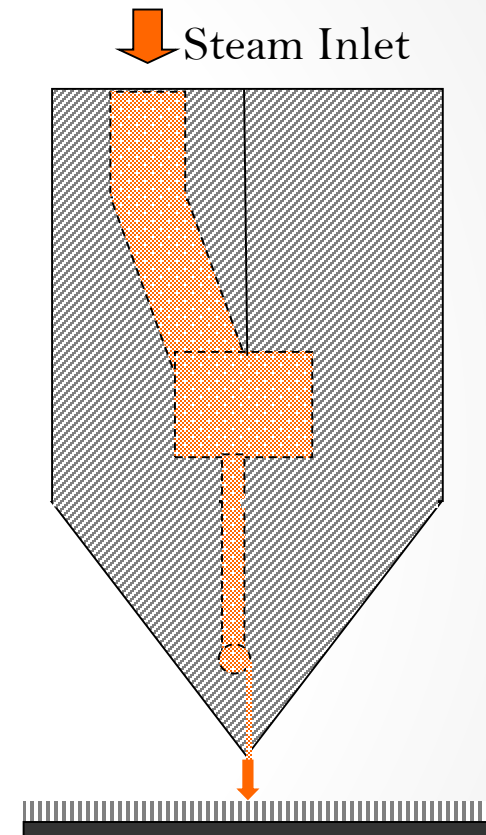
25~50 ft/min

Major bottleneck: steaming process - much longer dwell times are required for heavy face weight carpet.

# Slot Steam Applicator - Machnozzle\*



Machnozzle installed on a frame  
above conveyor belt

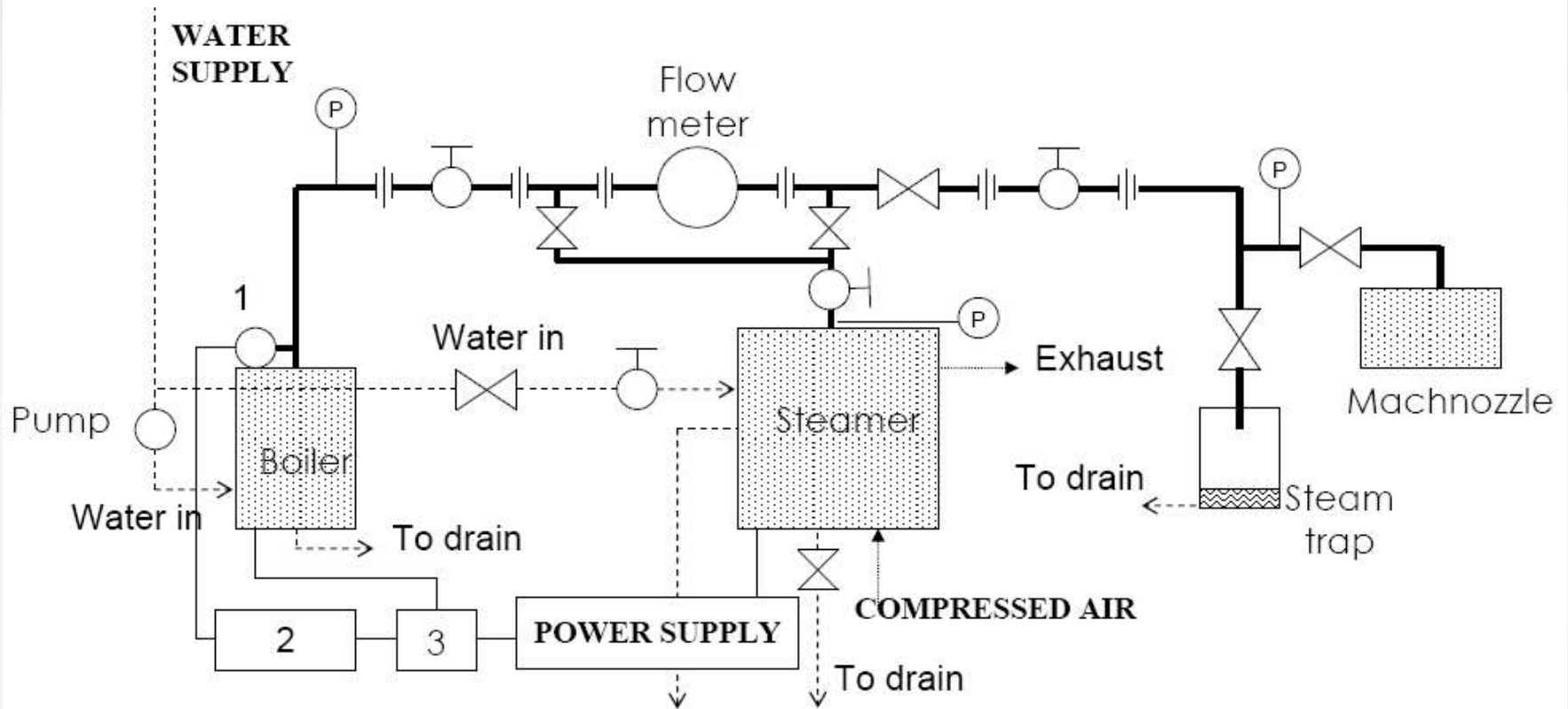


Cross-section

\* Manufactured by Brugman Company, Netherlands.

\* Julie W. Keller, Continuous Foam Dyeing of Carrier Polyester and Nylon Carpets,  
● M.S. thesis, Georgia Institute of Technology, 1984

# Experimental Setup in TAC



— Electricity cable

--- Water pipeline

— Steam pipeline



Pressure Gauge



Pressure Gauge



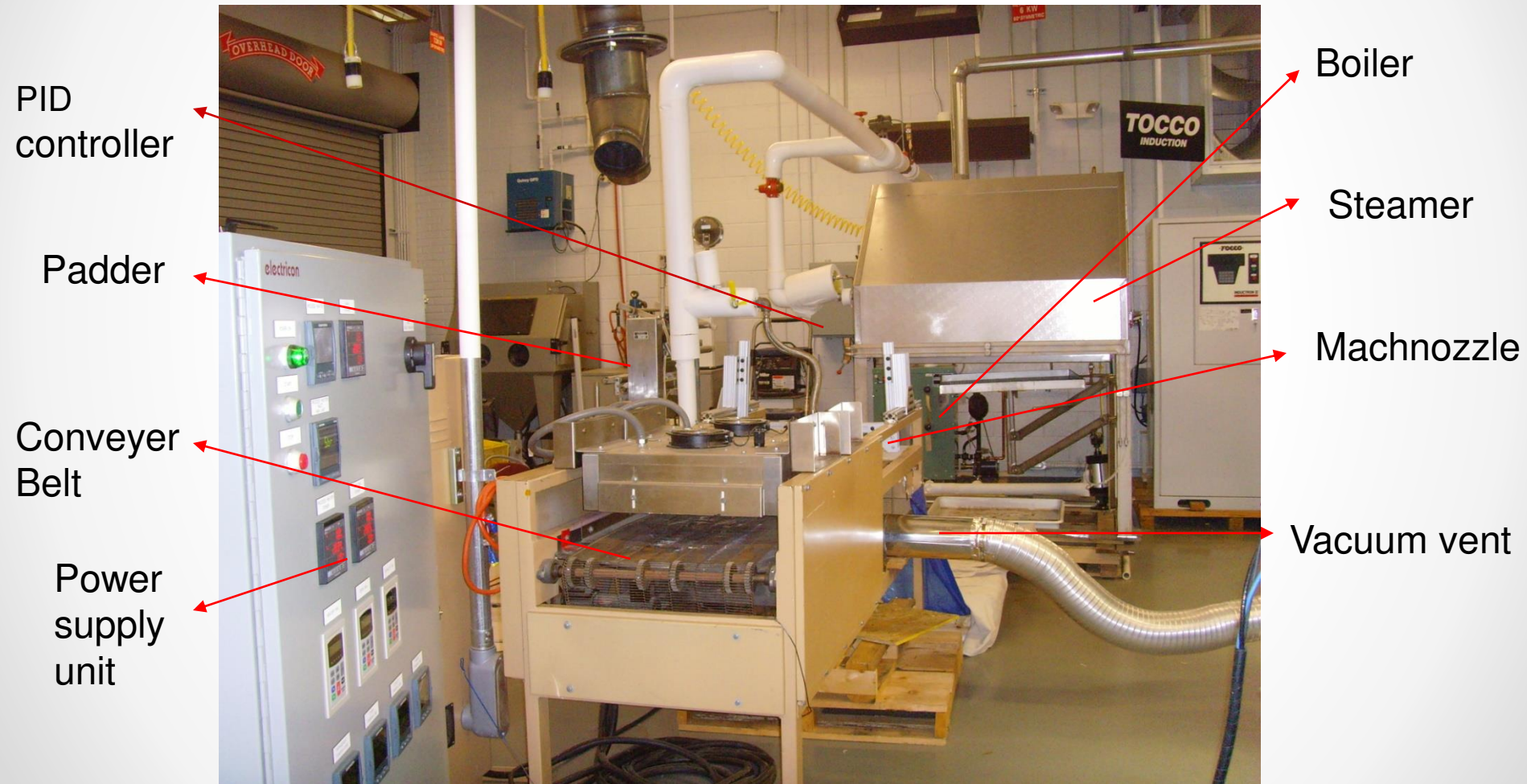
On/Off Valve



Union

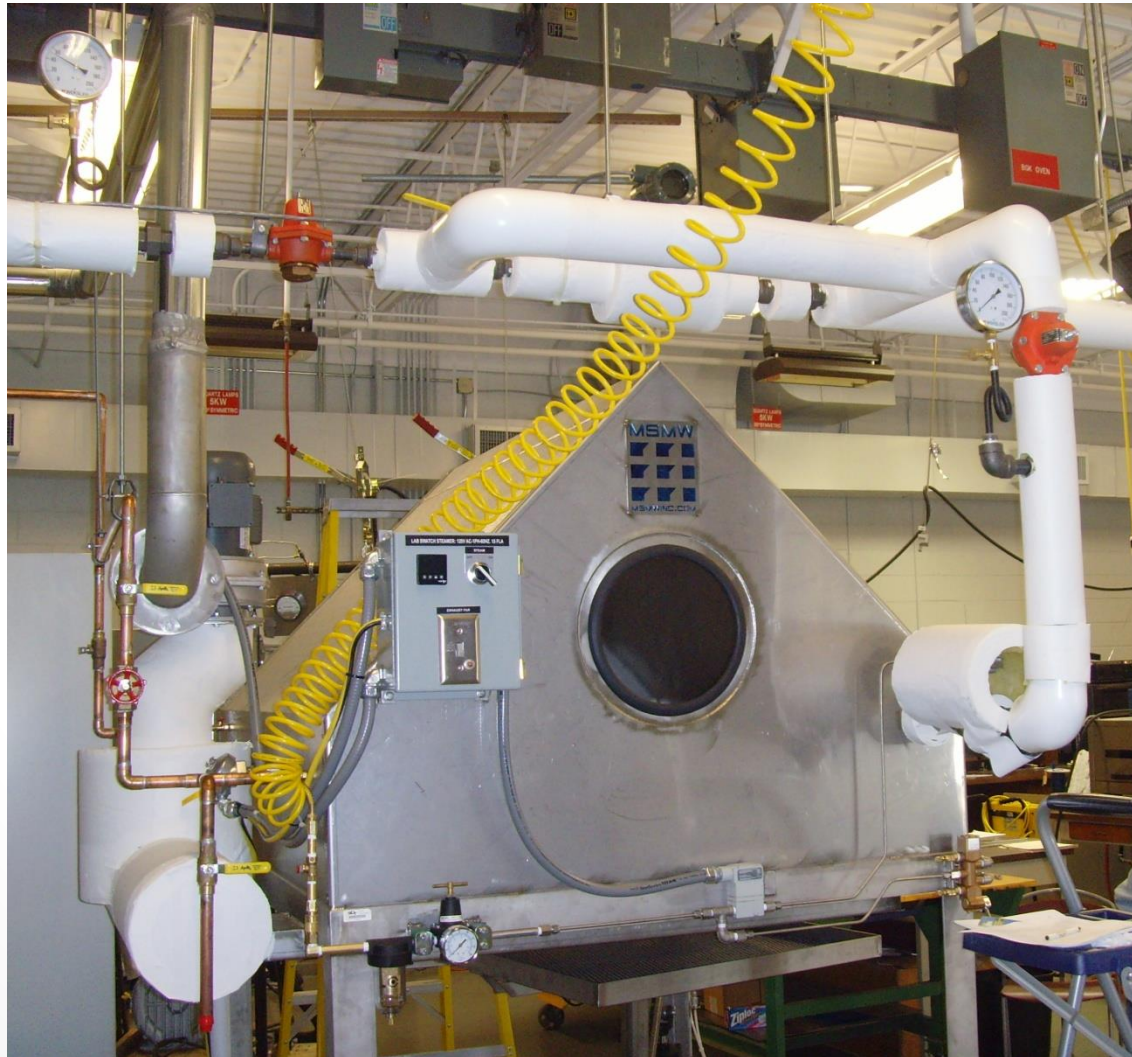
1: Pressure transducer, 2: PID Controller, 3: SCR

# Experimental Setup in TAC



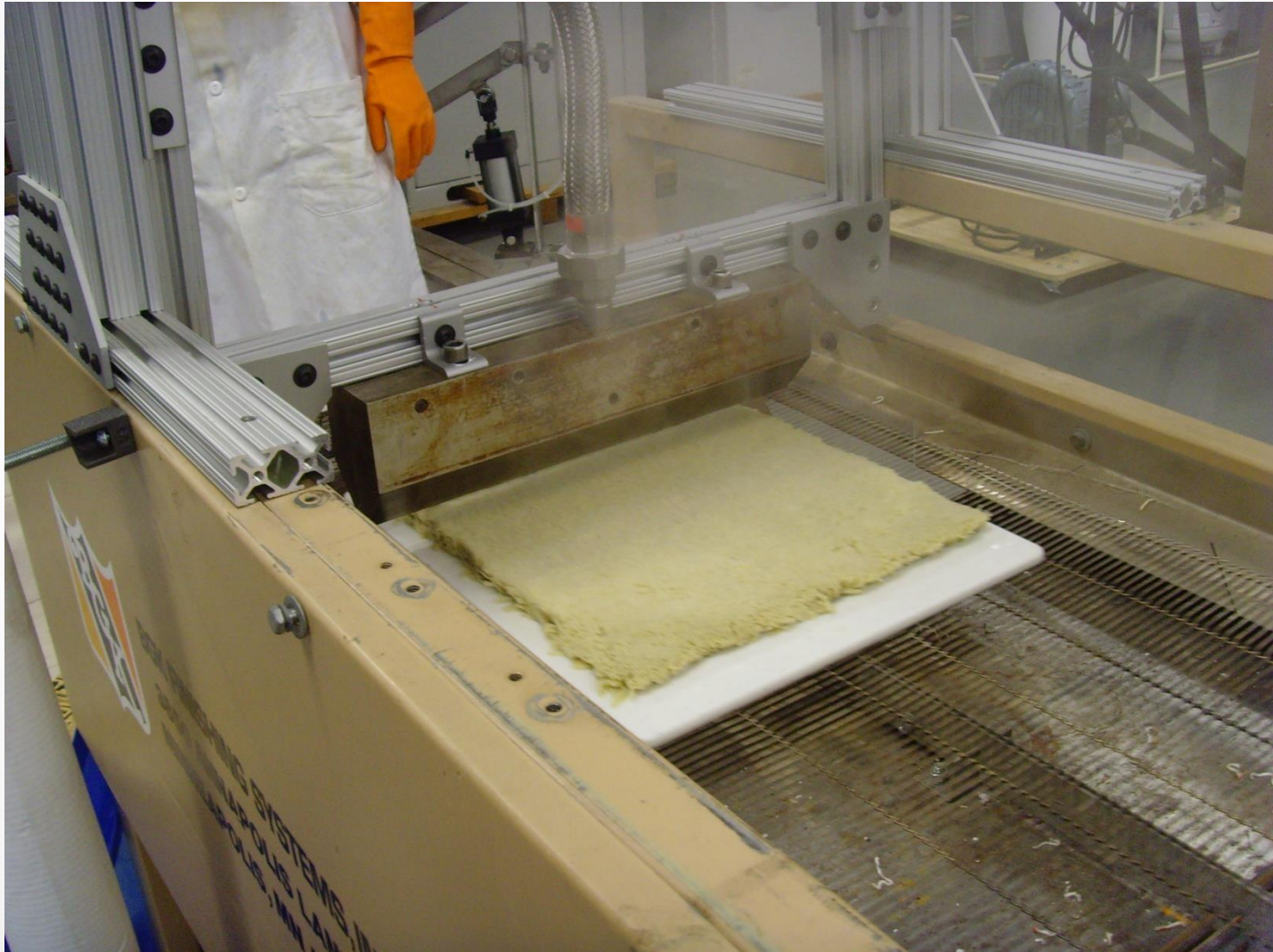
# Cloud Steamer

---

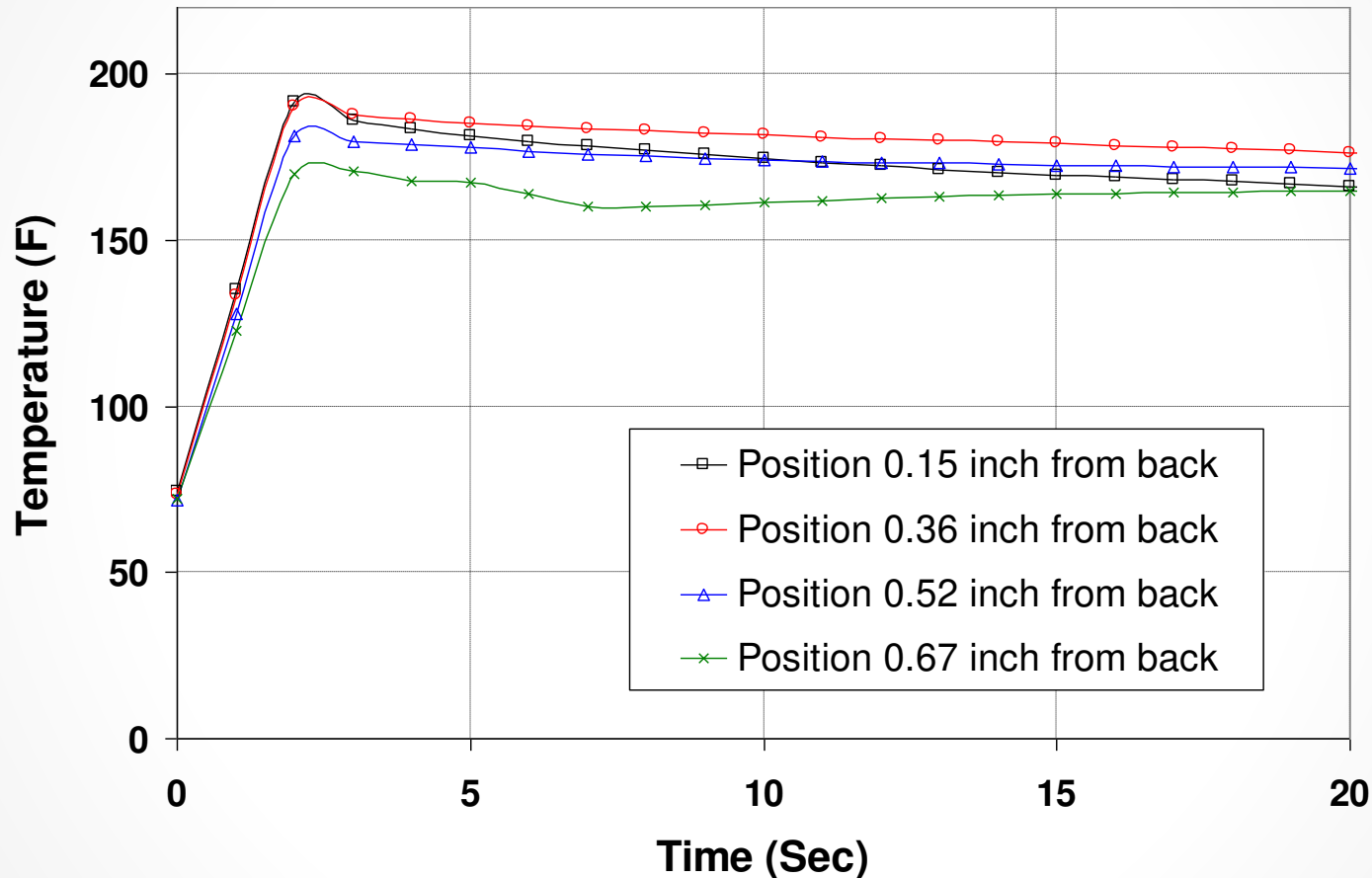


# Sample Heating using Machnozzle

---



# Sample Heating Using Machnozzle



Test conditions: Steam flow rate: 36 lb/hr  
Carpet face weight: 60 oz/yd<sup>2</sup>  
Belt speed: 10 ft/min

# Materials for Dyeing Tests

- Carpet samples: Heavy-weight nylon 6,6 carpet (face weight 65 oz/yd<sup>2</sup>) provided by Mohawk Industries, Inc.
- Acid dyes and dye formulas provided by Mohawk

		Dyes/Chemicals (g/L)		
		Dark	Medium	Light
Dyes	Telon Yellow 3RLN liq. 50% w/w	1.3912	0.2611	0.0297
	Telon Red 2BN liq. 40% w/w	0.8147	0.0542	0.0141
	Acid Blue 324 liq. 67% w/w	1.4250	0.0500	0.0104
Chemicals	Wetter (Startex EAC2)	2		
	Chelate (Starquest DCS)	0.5		
	Buffer (Buffer KAC)	1.0		

# Dyeing Test Steps



Prepare dye liquor



Immerse carpet in 3 L dye liquor



Squeeze to 375% wet pick up



Pass through machnozzle



Steam in the steamer



Wash



Vacuum

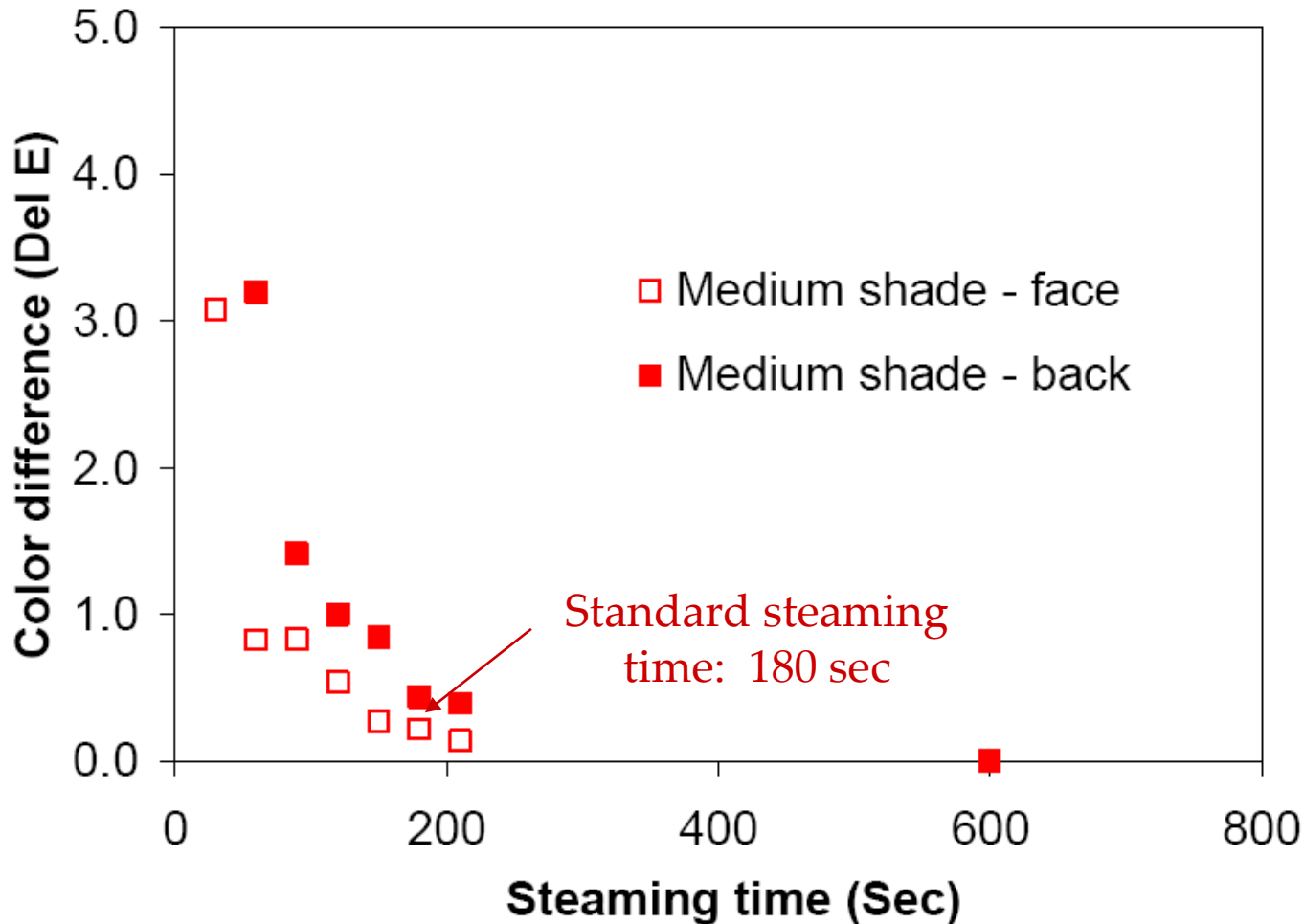


Dry

Measure color

# Dye Fixation in Steamer vs. Steaming Time

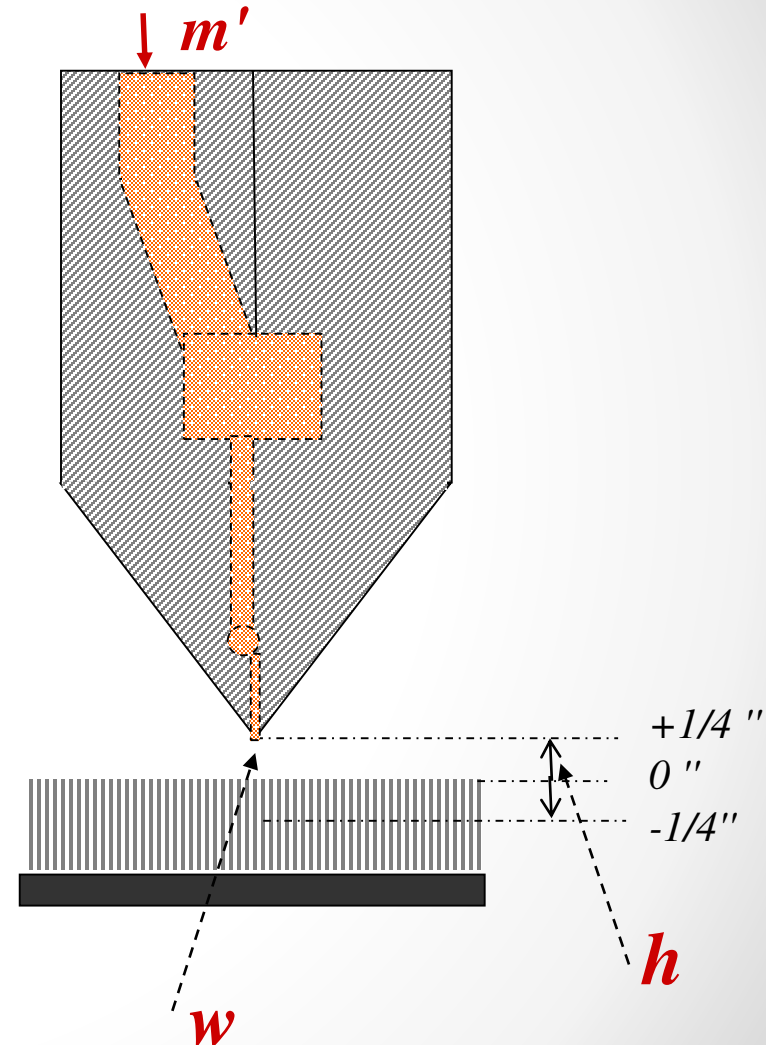
## - Medium Shade



(Del E is based on the samples dyed at steaming times of 600 and 1200 sec.)

# Operational Parameters of Machnozzle

- Mounted locations ( $h$ ) of the slot steam applicator relative to the carpet pile tips: +0.25", 0", and -0.25";
- Slot opening ( $w$ ): 6, 11.5, 17.5, and 21.5 mils;
- Steam flow rate per length ( $m'$ ): 5, 6.25, and 7.5 lb/hr-inch.



# Comparison Between Pre-steamed<sup>+</sup> Samples and Reference (Steamer-Only) Samples

---

	Steaming time (sec)	Face				Back			
		L*	a*	b*	$\Delta E^*$	L*	a*	b*	$\Delta E^*$
<b>Light shade</b>									
Steaming only	150	81.0	1.8	7.6	0.0	78.4	0.5	4.1	0.0
Pre-steaming with Machnozzle*	60	81.1	1.8	7.4	0.3	78.4	0.7	4.2	0.2
<b>Medium shade</b>									
Steaming only	180	67.5	3.4	20.4	0.0	69.5	2.0	16.5	0.0
Pre-steaming with Machnozzle*	60	67.9	3.3	20.2	0.5	69.8	2.0	16.0	0.5
<b>Dark shade</b>									
Steaming only*	600	31.8	-3.7	-1.0	0.0	35.7	-3.1	0.2	0.0
Pre-steaming with Machnozzle*	240	32.0	-3.9	-0.4	0.7	35.5	-3.2	1.0	0.8

<sup>+</sup> Machnozzle operational parameters: slot opening of 6 mil, 0" mount position, and steam flow rate of 7.5 lb/hr-inch

# Conclusion

---

Pre-steaming using the slot steam applicator significantly reduced the required dye migration/fixation times in the cloud steamer

- Demonstrated the potential to increase speeds in continuous Nylon carpet dye lines by a factor of 2.5-3X.

# PVA Size Recovery and Reuse via Vacuum Flash Evaporation

**Kishor K. Gupta**

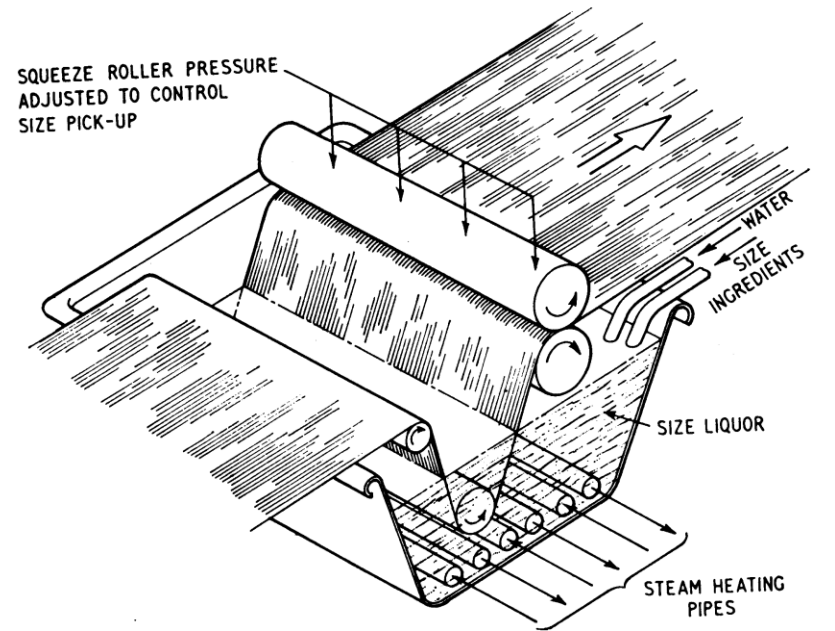
Gupta, K; Cook, FL. 2012. *Recovery and Reuse of Polyvinyl Alcohol from Desize Effluent Streams via Vacuum Evaporation*, AATCC REVIEW-Journal, 12(3), 47.



# Conventional Slashing

- Sizing Agents Widely Used

- Starch
- PVA + Starch (Most Common)
- PVA (Only with UF Recovery)



Section of Size Box\*

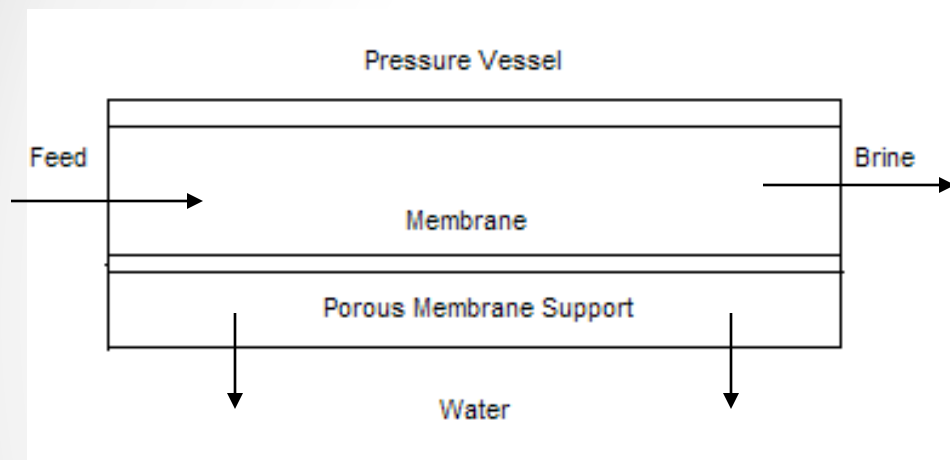


# Environmental Opportunities

- Hot water consumed by US textile industry in 1996 for desizing:  $20 \times 10^9$  liters
  - 90% of it disposed into the effluent stream
  - 10% of it recycled
  - Energy required to heat  $20 \times 10^9$  liters of water to  $95^\circ\text{C}$ :  $5.7 \times 10^{12}$  Btu
- Pollution load
  - In 1996, 90% of size used by US textile industry was disposed in effluent streams: 90 million kgm
  - Starch contributes 50% of BOD in composite textile effluent
    - 100,000 – 600,000  $\text{mg L}^{-1}$



# Ultrafiltration (UF)



Schematic of Ultrafiltration Process\*

- UF Membranes\*\*
  - Carbon Tubular Membranes
  - Stainless Steel Tubular Membranes
  - Polyvinyl Sulfone Spiral Wound Membrane
  - Cellulose Acetate Spiral Wound Membrane

\*Brandon, C.A., *et al.*, *Textile Chemist And Colorist* 5(7) (1973) 35-134.

\*\*Porter, J. J., *Journal of Membrane Science* 151 (1998) 45-53



# Disadvantages of UF PVA

## Recovery Technologies

- Based on Spiral-Wound, Cellulose Acetate Membranes
  - Membranes Foul, Must be Periodically Cleaned (Cleaning can Reduce Efficiency of Membranes)
  - Membranes Degrade with Use, Must be Periodically Replaced
- High Pump Pressure Requirements, with High US Electrical Rates and thus Costs
- Low Molecular Weight “Tail” of PVA Passes Through Membrane
  - Prohibits Unrestricted Reuse of the Exiting Hot Water, e.g., Pale Shade Dyeing Interference, Decreasing Economic Benefits
  - Loss of PVA Mass, Decreasing Economic Benefits
  - PVA Film Properties Change on Multiple Recycles, Impacting Weaving Performance



# Why This Project ?

- Threat to Environment Without PVA Recovery
  - High COD Contributor
  - Government Regulations
- Incentive to Recover PVA
  - Decrease Manufacturing Cost
  - Decrease Environmental Impact
  - Decrease Cost of Effluent Treatment
    - High Volume of Water Involved
- PVA+Starch in Slashing Cannot be Recycled
  - Starch “Fouls,” i.e., is Degraded by Starchase Enzymes
- PVA/Wax Slashing is Needed for Towel Manufacturing

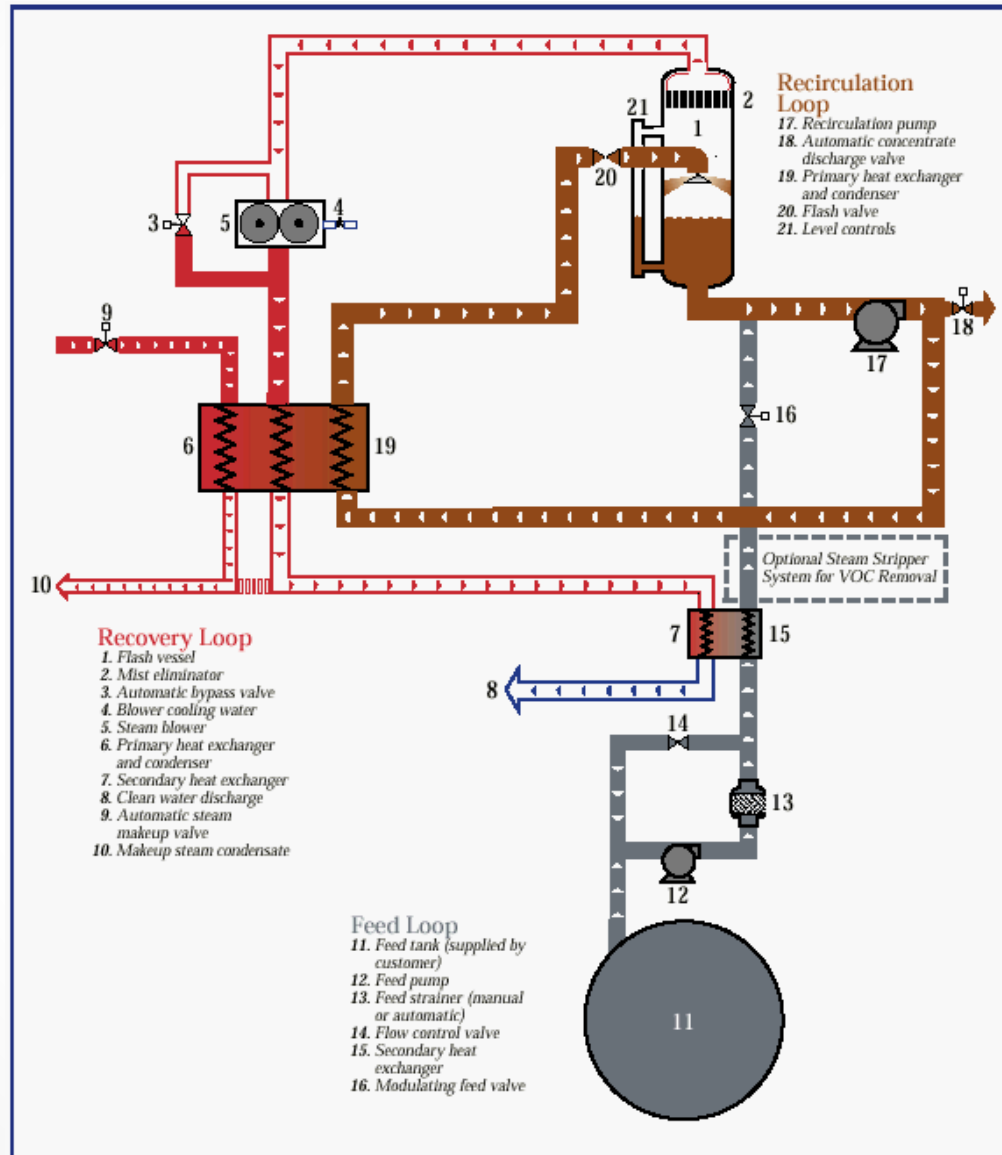


# Vacuum Flash Evaporation

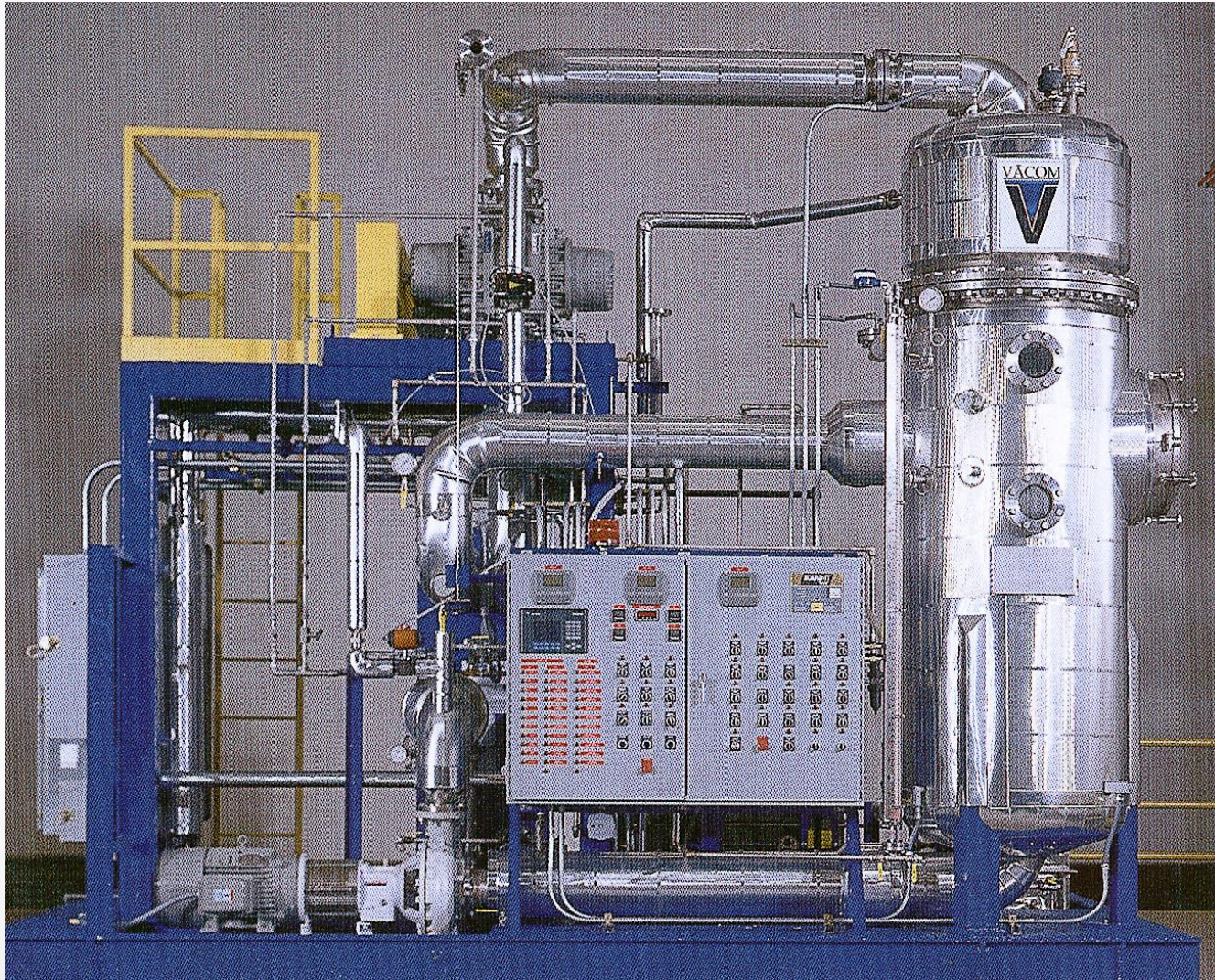
- Heated Liquid is Exposed to Sudden Pressure Change
- Heterogeneous Temperature Profiles Develop on Surface
  - Super-Heated and Sub-Cooled Areas
- Mass Transfer Rate up to 10-12 Times Greater than Normal Vacuum Evaporation



# VACOM VFE UNIT



# Vacom Commercial VFE Unit



# Advantages of PVA Concentration and Recovery Utilizing VFE

- Water Phase -Transformed During Process
  - Resulting Condensed Hot Water Reuse Unrestricted in Plant
    - Energy Required in VFE is ~a Trade-Off with Plant Boiler
    - Low MW PVA “Tail” Not Lost
- No High Pump Pressures Required, Lowering Electrical Costs
- No Cellulose Acetate Membranes to Foul or Degrade/Replace
- No Effluent (Zero Liquid Discharge)



# Objectives

- To study the technical and economic feasibilities of the VFE process in the concentration/recovery of PVA size.
- To investigate the physical/chemical properties of the recycled PVA polymer plus cotton impurities blended size, and determine the effect of size film property changes as a function of impurity levels.
- To study the effect of cotton impurities in recycled PVA size on the weaving performance of slashed yarns.

# Objectives (Cont'd)

- To study the effect of wax in recycled PVA size on the weaving performance of slashed yarns.
- To demonstrate feasibility of VFE to concentrate and recover PVA size, using the UGA ~30 end continuous pilot unit to slash and desize cotton yarns and the Vacom bench unit to concentrate/recover the size

# Vacuum Flash Evaporation and PVA Size Recovery/Recycle

- Flash Evaporation is a Zero Liquid Discharge Process, So Cotton Yarn Impurities (~2.3% of yarn weight) are Added to the Desize Water in Every Cycle of Recycling
- Effect of Impurities, Wax and Recycling on Recycled Size Properties

## Research Divided in Three Parts

- Effect of Impurities
- Effect of Wax and Both Impurities + Wax
- Small Pilot Scale Up
- 



# Lab Scale Slashing Line

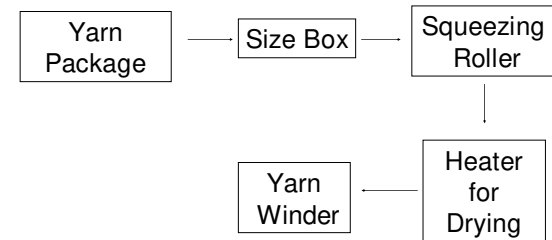


# Experimental

- Slashing Process

- Temperature of the Size Box: 70°C
- Squeeze Roller Pressure Constant
- Slashing Speed Constant
- Temperature of Drying Oven: 200°C
- Continuous Slashing
- Constant Viscosity
- Initial level of Size in the Size Box Constant

Yarn Flow on Lab. Slashing machine

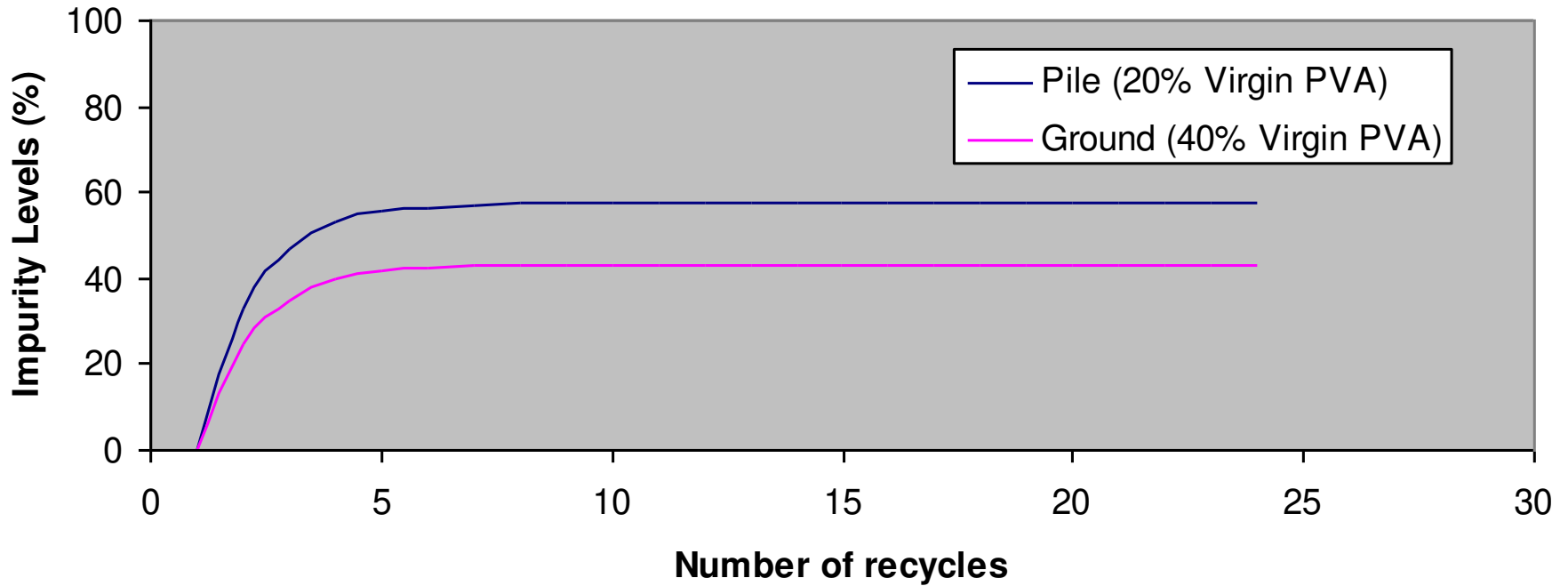


# UGA Trial

- Small Pilot Scale up
  - UGA ~30 end continuous pilot slashing unit
  - Size add-on Ground 10% and Pile 3%
  - Virgin PVA Ground 40% and Pile 20%
  - PVA size recovery from waste water
  - Size reuse on UGA slashing line, then iterate
  - Full analyses of sized yarn and PVA films
  - Pre-weave analyses of sized yarn (hairiness, abrasion resistance, tensile strength, etc.)

# Impurity Levels in UGA Trials

Impurity Levels in Pile and Ground



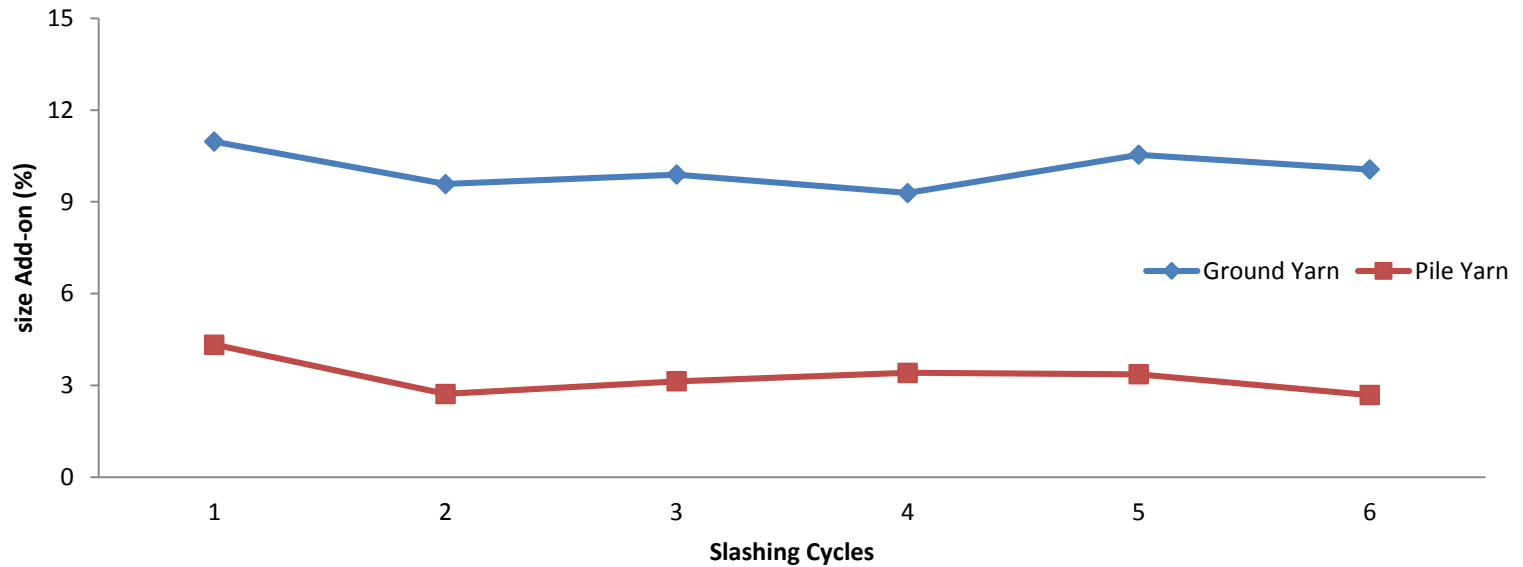
# Pilot Slasher at UGA



# Slashed Yarn Properties

## Size Add-on

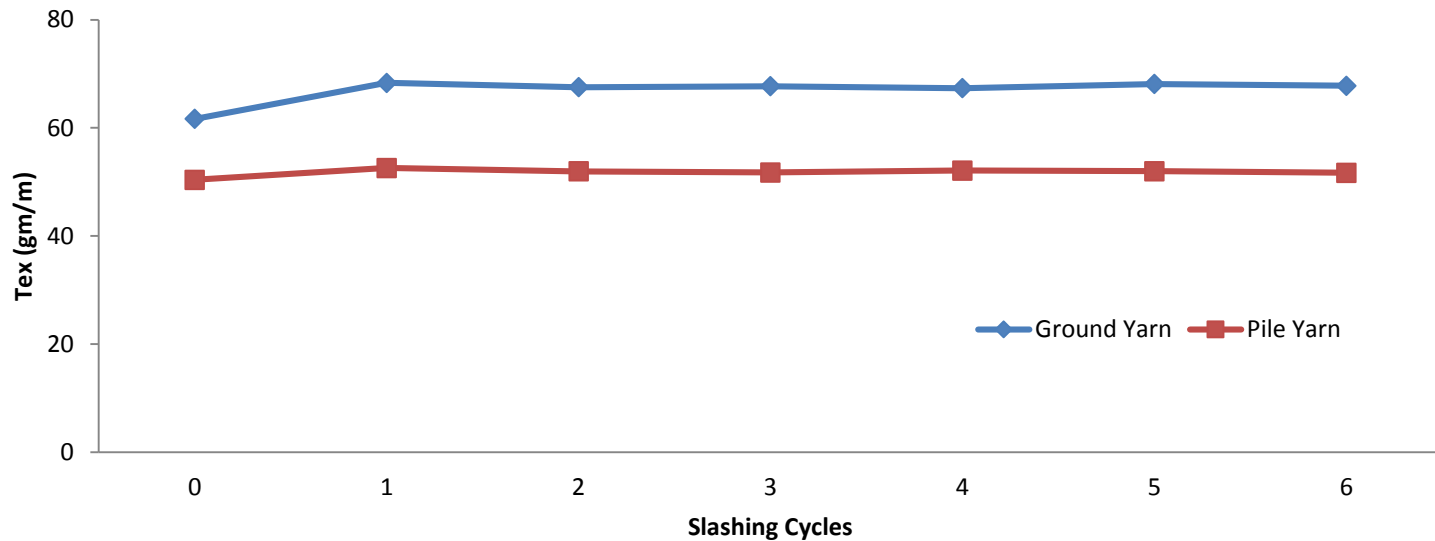
Size Add-on vs. Slashing Cycles of the Recycled Size/Slashed Yarn



# Slashed Yarn Properties

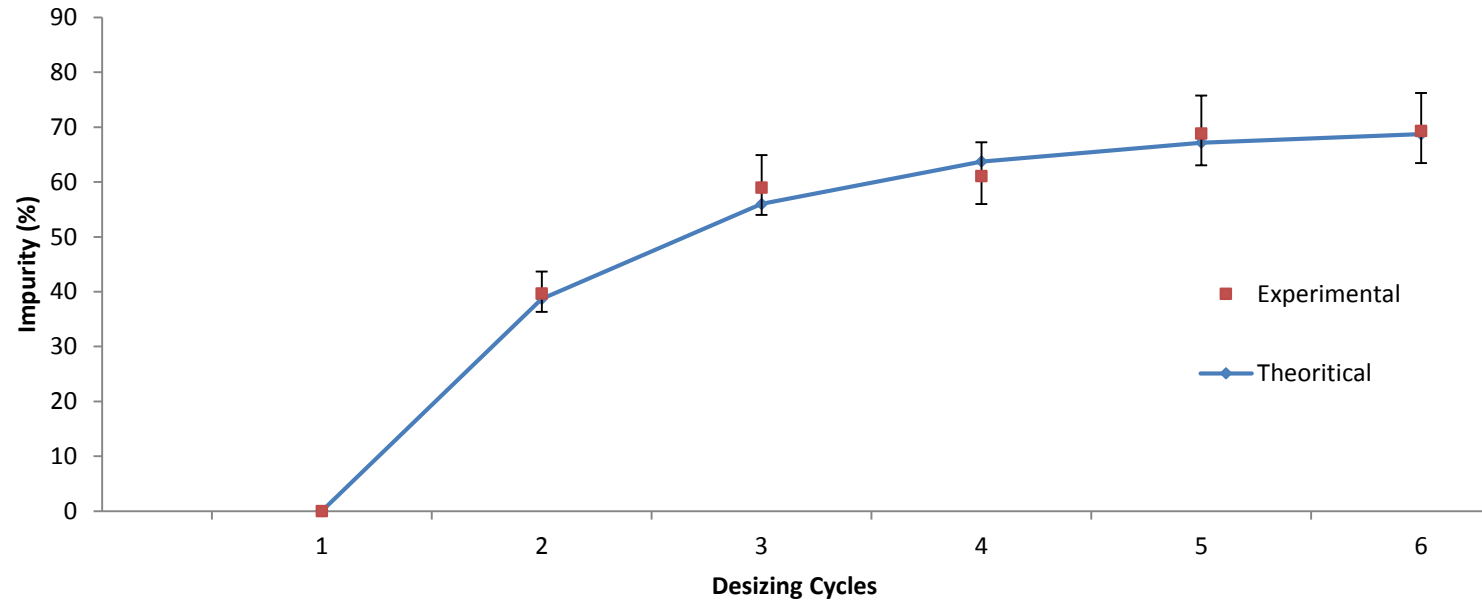
## Yarn Tex

Yarn Tex vs. Slashing Cycles of the Recycled Size/Slashed Yarn



# TGA Analysis of Impurities

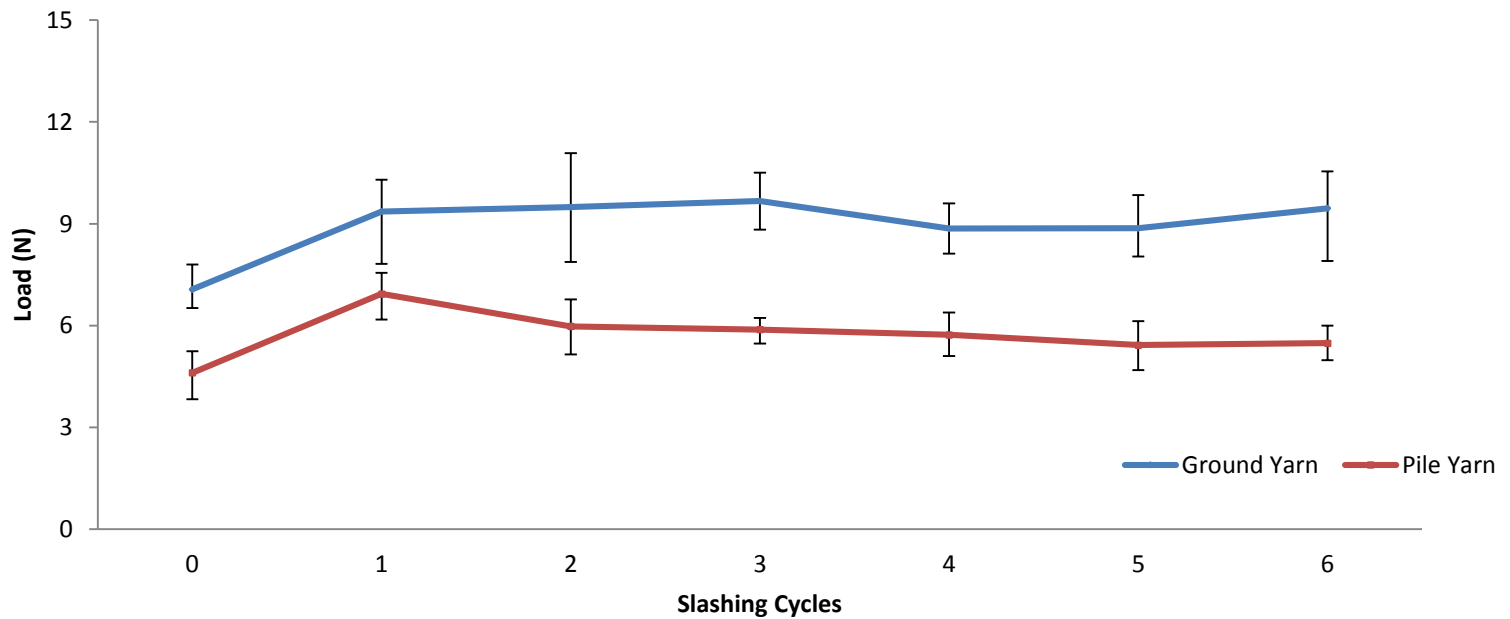
## Impurity vs. Desizing Cycles



# Slashed Yarn Properties

## Load at Break

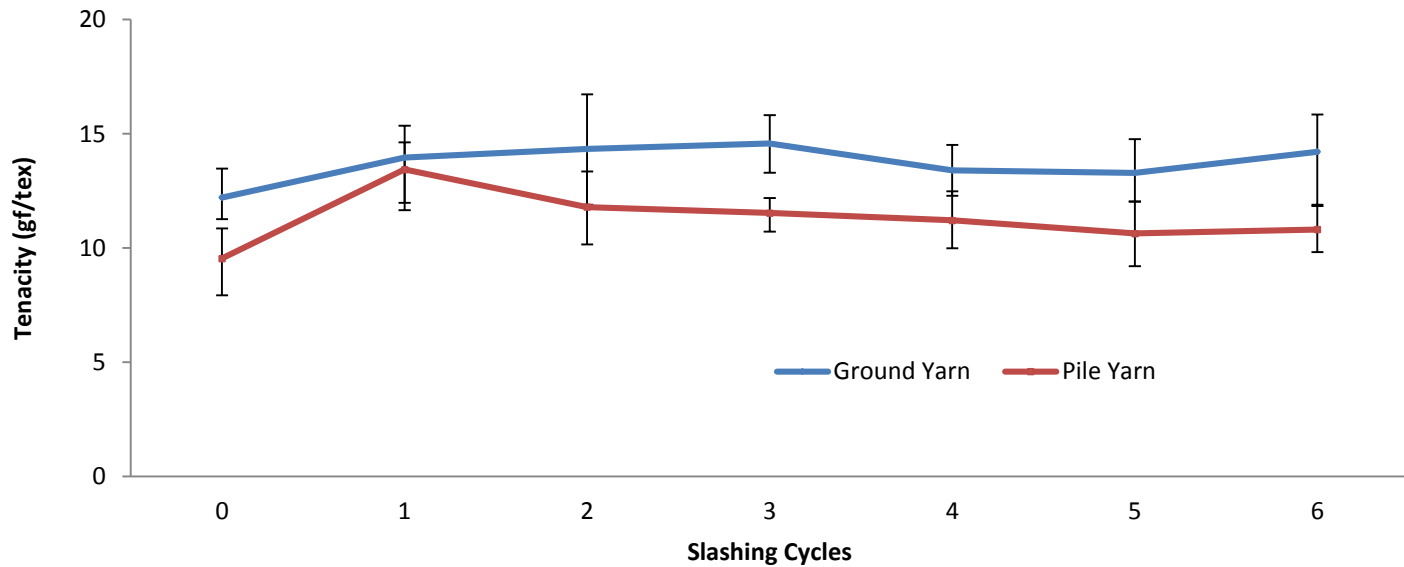
Load at Break vs. Slashing Cycles of the Recycled Size/Slashed Yarn



# Slashed Yarn Properties

## Tenacity

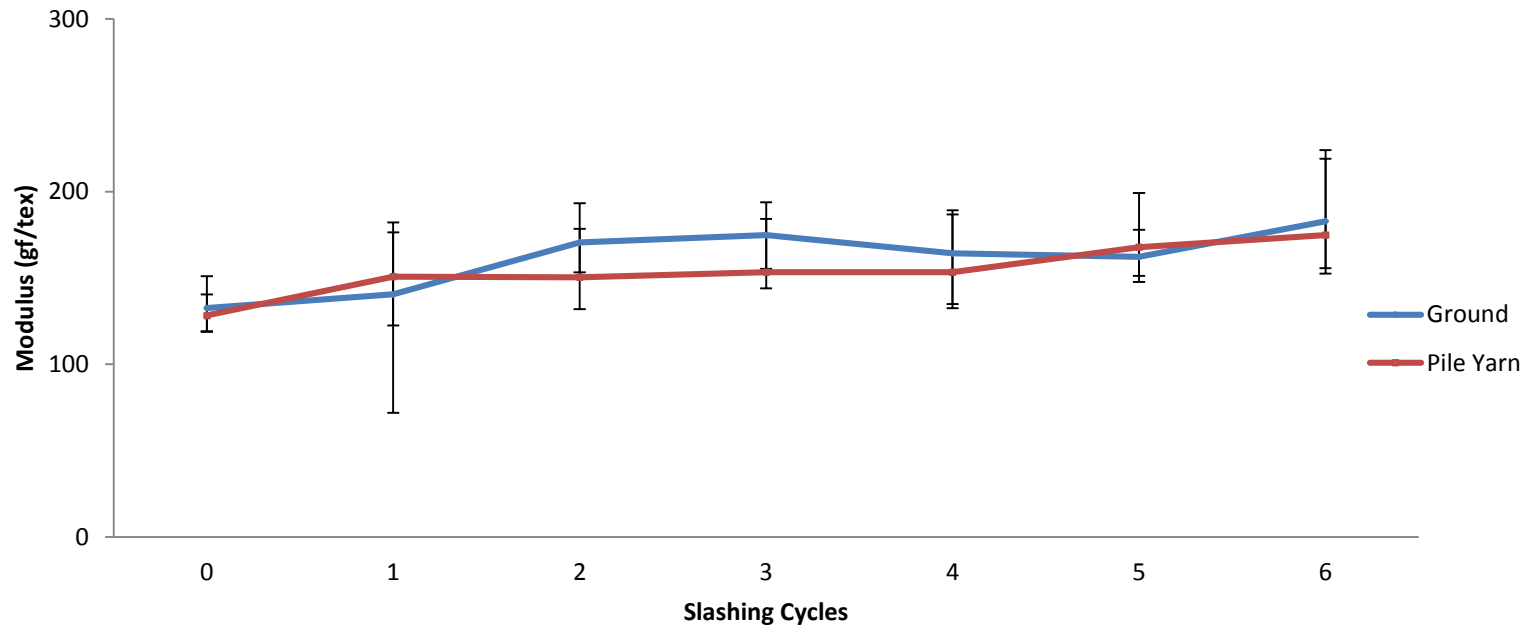
Tenacity vs. Slashing Cycles of the Recycled Size/Slashed Yarn



# Slashed Yarn Properties

## Elastic Modulus

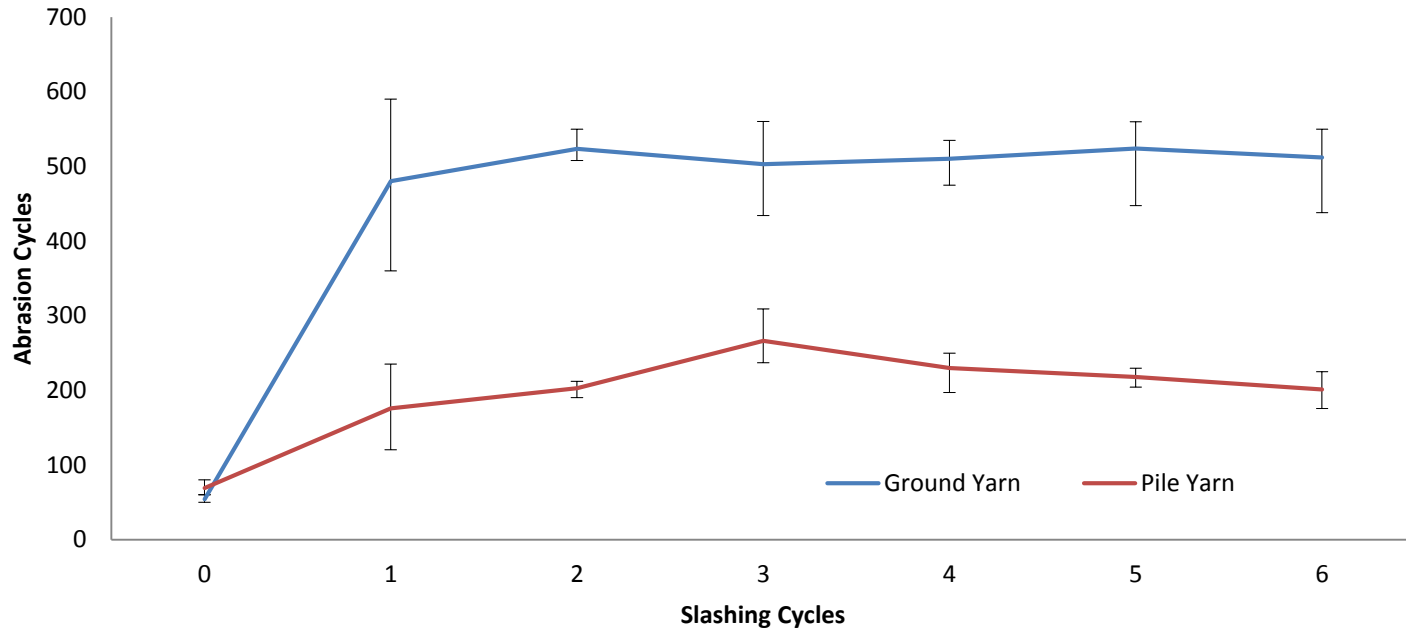
Modulus vs. Slashing Cycles of the Recycled Size/Slashed Yarn



# Slashed Yarn Properties

## Abrasion Cycles

**Abrasion Cycles vs. Slashing Cycles of the Recycled Size/Slashed Yarn**



# Conclusions

- In Vacuum Flash Evaporation, Recycled Size Entrainments Extracted Yarn Impurities (~2.3% of Yarn Weight per Desizing Cycle)
  - Apparent Molecular Weight Average of Recycled Size Decreases, as Impurities are Lower Molecular Weight than the PVA.



# Conclusions (Cont'd.)

- Slashed yarn tenacity and load at Break initially increases with increase in impurities concentration, but after 3<sup>rd</sup> slashing cycle it decreases and further increase in impurities concentration have little effect on tenacity and load at break.
- Slashed yarn work of rupture initially increases with increase in impurity concentration, but after 3<sup>rd</sup> recycle for ground yarn further increase in concentration decrease the work of rupture.
- Slashed Yarn Elongation at Break of ground yarn increases with slashing cycles, but slightly decreases after 3<sup>rd</sup> slashing cycle.



# Conclusions (Cont'd.)

- Slashed yarn hairiness initially decreases with increase in impurity concentration, but after 2<sup>nd</sup> slashing further increase in concentration increases the hairiness.
- Slashed yarn number of abrasion cycles more or less same with increase in impurity concentration.
- Bending length of the yarn decreases with slashing cycles but after 5<sup>th</sup> slashing cycles it approaches towards a constant value.

# Development of Commercial, Sustainable Processes for Dyeing Generic, Unmodified Polypropylene Fiber

**Murari Gupta**

Gupta, ML; Etters, JN; Wu, X.; Liu, H.; Cook, FL., 2010. *Aqueous Processes For Dyeing Generic, Unmodified Polypropylene Fiber*, JEFF, 5(2), 27.

# Coloration of PP Fiber: Melt Pigmentation

## Advantages:

- ✓ Excellent washing and light fastness
- ✓ Environment friendly

## Limitations:

- Not always possible to economically color small lots in different shades in a short time on the basis of customer demand.
- Inventory control
- Recovery and reuse of the polymer waste becomes difficult due to the presence of pigment particles

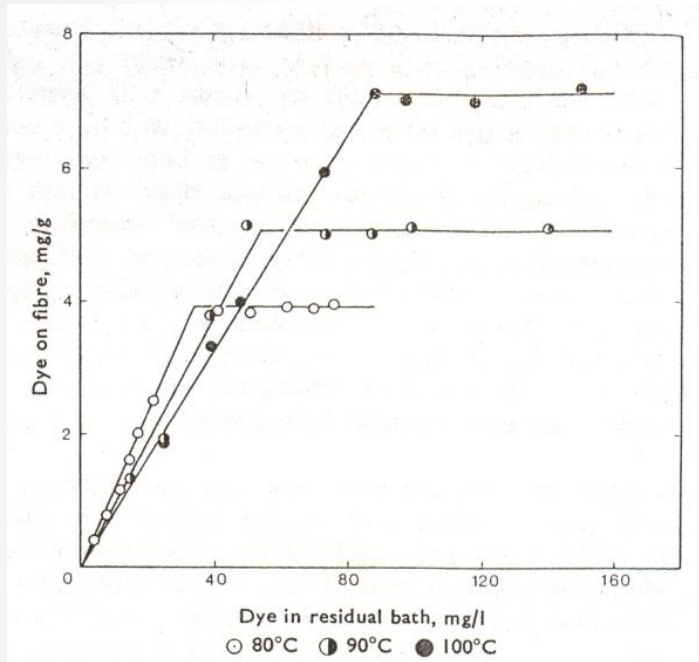
# Why Aqueous Dyeing of Unmodified Polypropylene?

- Limitations of melt pigmentation route
- Steep rise in cost/lb. with fiber modifications
  - Adverse effects on mechanical properties
- Need to develop an aqueous dyeing process for unmodified PP analogous to well-established technologies being used for other high volume fibers, e.g., PET, nylon, acrylic, etc.
- Unmodified, generic form of polypropylene is inexpensive and has good physical and aesthetic properties, making it an attractive fiber for towel, carpet, apparel and some technical products

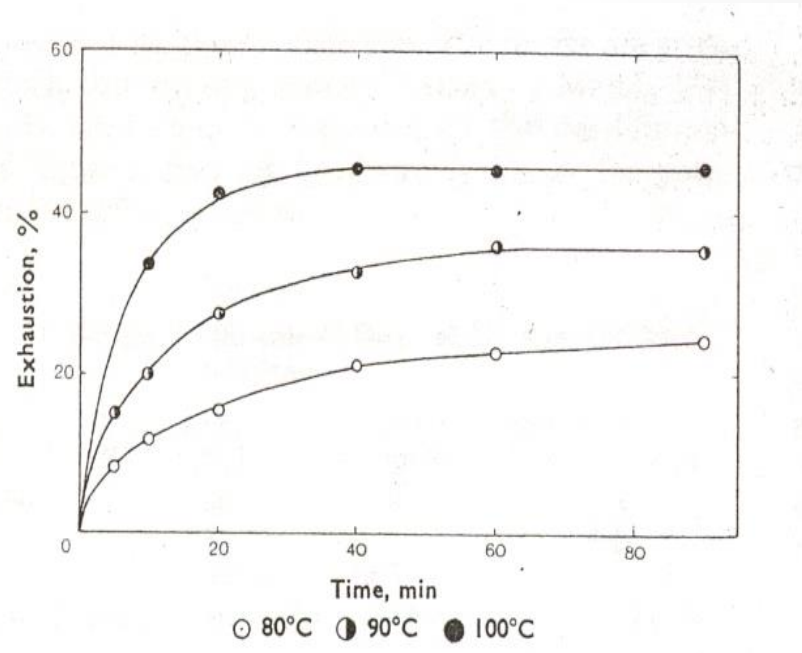
# Requirements of Dyeability

- The fiber should be dyeable or printable with the commercially available equipment and technology.
- The chemicals, dyestuffs and auxiliaries should be commercially available and not be too expensive.
- Complete or near complete exhaustion of dyebath, level dyeing, availability of a wide range of shades and adequate fastness properties

# Disperse Dyeing of Unmodified PP Fibers



Adsorption Isotherms for Dimethyl Yellow on Unmodified PP at Various Temperatures



Dyeing Rate Curve for Serisol Fast Pink RGL on Unmodified PP

Bird, C. L.; Patel, A. M., The Dyeing of Unmodified Polypropylene with Disperse Dyes. *Journal of Society of Dyers and Colourists* Nov. 1968, 84, (11)

## Acid Leuco Vat Dyeing of Unmodified, Generic PP Fiber

- Etters et al. (1997) reported several acid leuco vat dyes showing good affinity with PP knitted fabrics.
- The focus of the research was to identify and demonstrate viable vat dye candidates of a trichromatic series (red, blue and yellow colorants) plus an orange to dye unmodified PP flat fabric, towel and carpet constructions in an aqueous system.

# Solubility Parameter (SP) and Small Molecule Diffusion Theory

Flory-Huggins equation for free energy of mixing

$$\Delta G_M = kT \left[ \frac{\phi}{N} \ln \phi + (1 - \phi) \ln (1 - \phi) + \chi \phi (1 - \phi) \right]$$

Interaction parameter ( $\chi$ ) can be written in terms of solubility parameter ( $\delta$ ):

$$\chi = \frac{v_0}{kT} (\delta_A - \delta_B)^2$$

Solubility parameter ( $\delta$ ) is the square root of the cohesive energy density:

$$\delta = \sqrt{\frac{E}{v}}$$

Miscibility of a polymer and a small molecule (solvent) requires a close match in their solubility parameters ( $\pm 1.5$  (cal/cm<sup>3</sup>)<sup>1/2</sup>)

# Formation of Acid Leuco Vat Dyes

## Two-stage process (UGA)\*

- Vatting using NaOH and  $\text{Na}_2\text{S}_2\text{O}_4$
- Back-titration with  $\text{CH}_3\text{COOH}$  to achieve final pH = 7.0

## Single stage process (GIT)

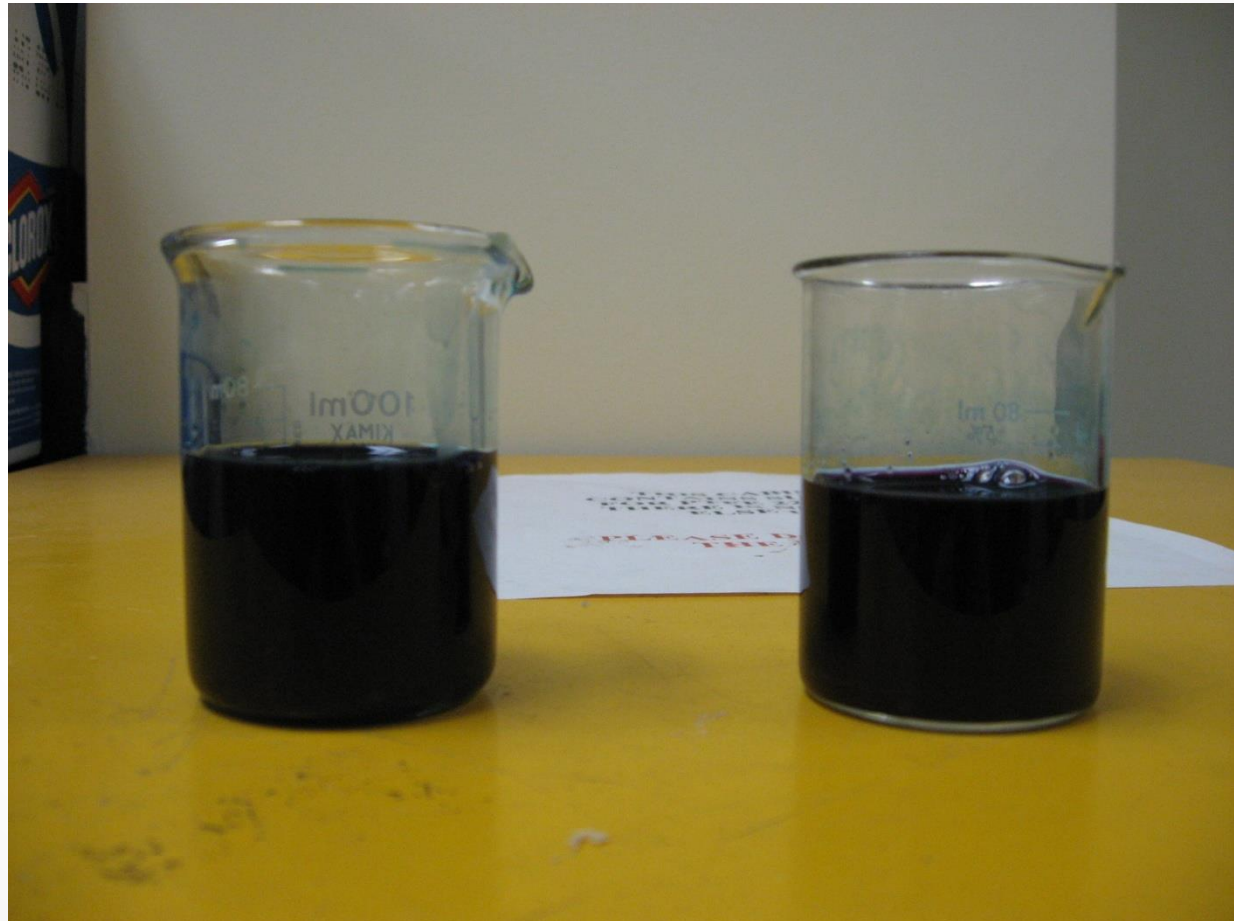
- ⊕ Add minimal amount of NaOH, but excess  $\text{Na}_2\text{S}_2\text{O}_4$ , until a pH of 7.0 is achieved
- ⊕ Analogy: 1g/l NaOH and 8 g/l  $\text{Na}_2\text{S}_2\text{O}_4$  used by Kunttou et al.\*\* for indigo dyeing of polyester fabrics at pH 7.0

Lu, Y.; Ethers, J. N.; Hardin, I. R.; Cook, F. L., Dyeing of Generic, Unmodified Polypropylene Fabrics. AATCC Review 2006 (Submitted For Publication).

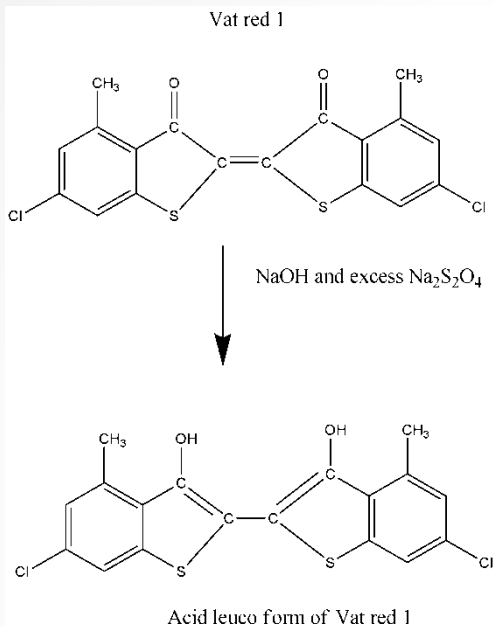
Kunttou, K.; Hongyo, S.; Maeda, S.; Mishima, K., Dyeing Polyester Fabrics with Indigo. Textile Research Journal Feb. 2005, 149-153.

# Formation of Acid Leuco Compound of Vat Green 1 (Single-Stage and Two-Stage Method)

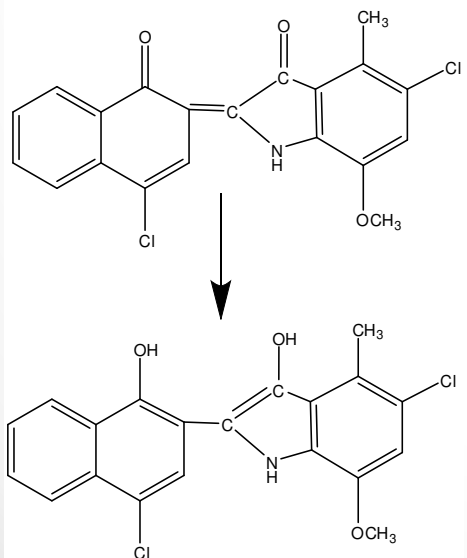
Purple-red color  
for single-stage  
(left) and two-  
stage (right)



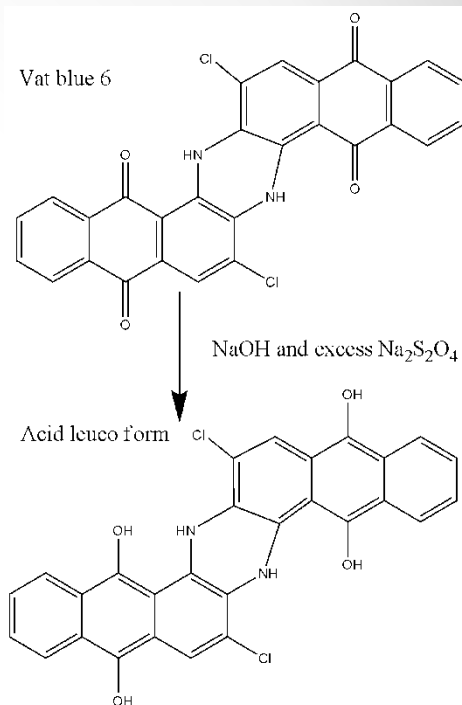
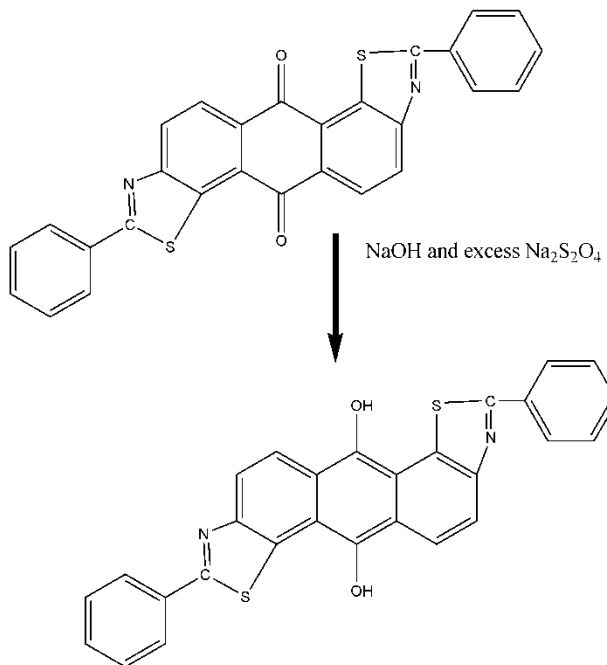
# Chemical Structures of Vat Dyes of Trichromatic Series



Vat blue 8, C.I. # 73800



Vat yellow 2



## SP (acid leuco)

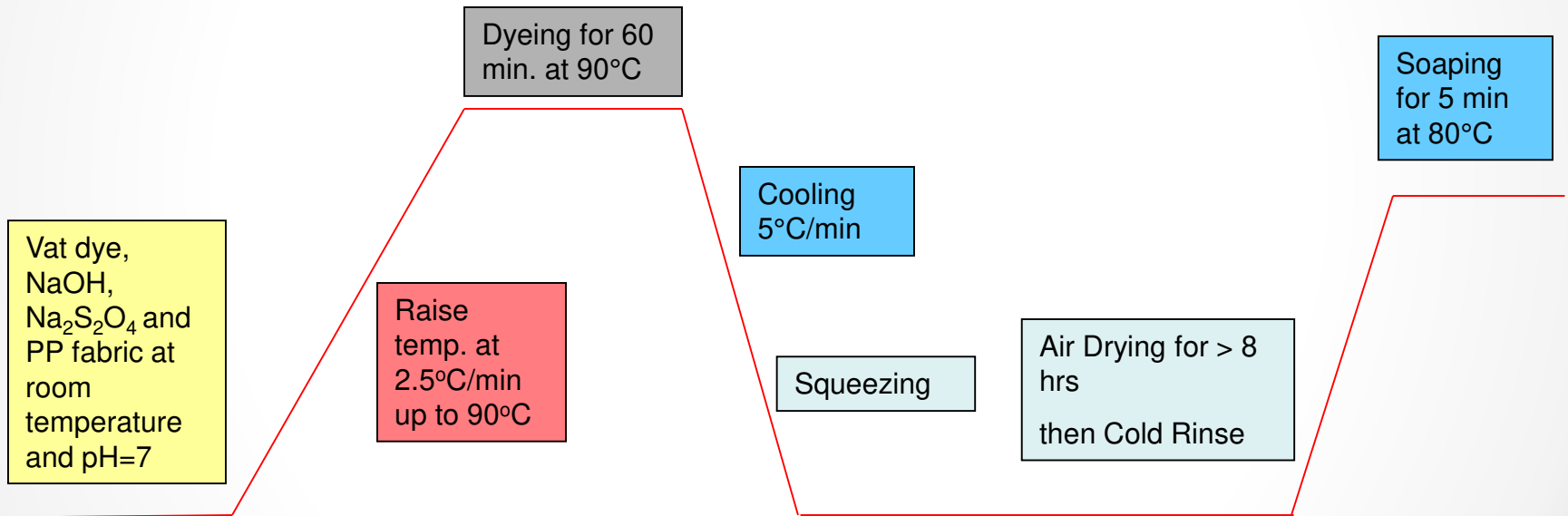
$(\text{cal}/\text{cm}^3)^{1/2}$

Vat Red 1	16.0
Vat Yellow 2	15.0
Vat Blue 6	18.6
Vat Orange 1	14.6
Vat Blue 8	15.4
Vat Blue 1	16.7
Vat Brown 1	19.3

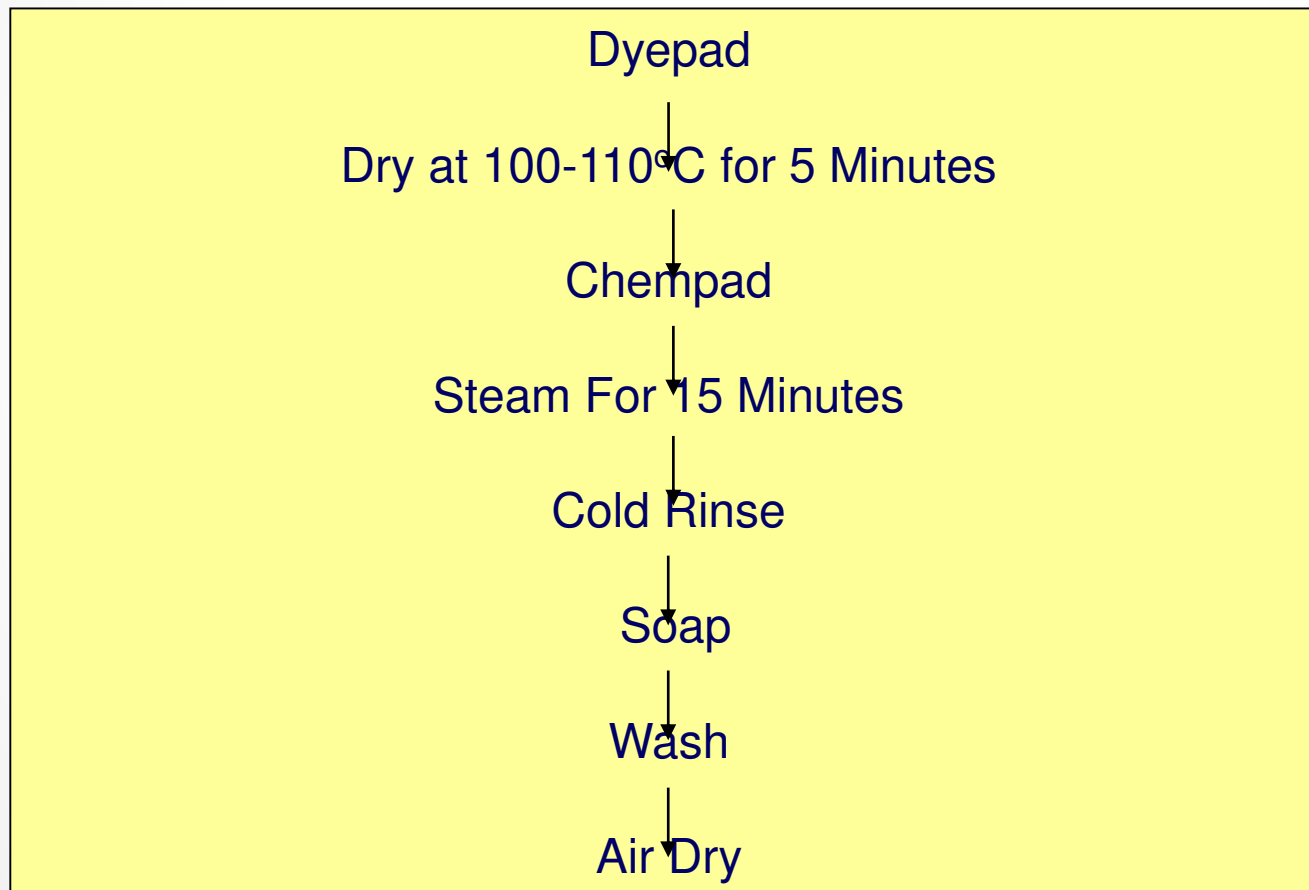
# Objectives

- To screen viable candidates from commercially-available vat dyes utilizing solubility parameter and molecular dynamics simulation approaches
- To develop an optimized, exhaust batch dyeing process to initially color PP flat fabric with a trichromatic series of vat dyes (red, yellow and blue) plus an orange
- To evaluate the fastness properties (washing, crocking and dry-cleaning) of the dyed specimens in order to certify the developed dyeing process
- To prepare exhaustion plots and adsorption isotherms for the screened dye candidates to elucidate dyeing kinetics, thermodynamics and mechanism

# One-Step Batch Exhaust Dyeing Cycle



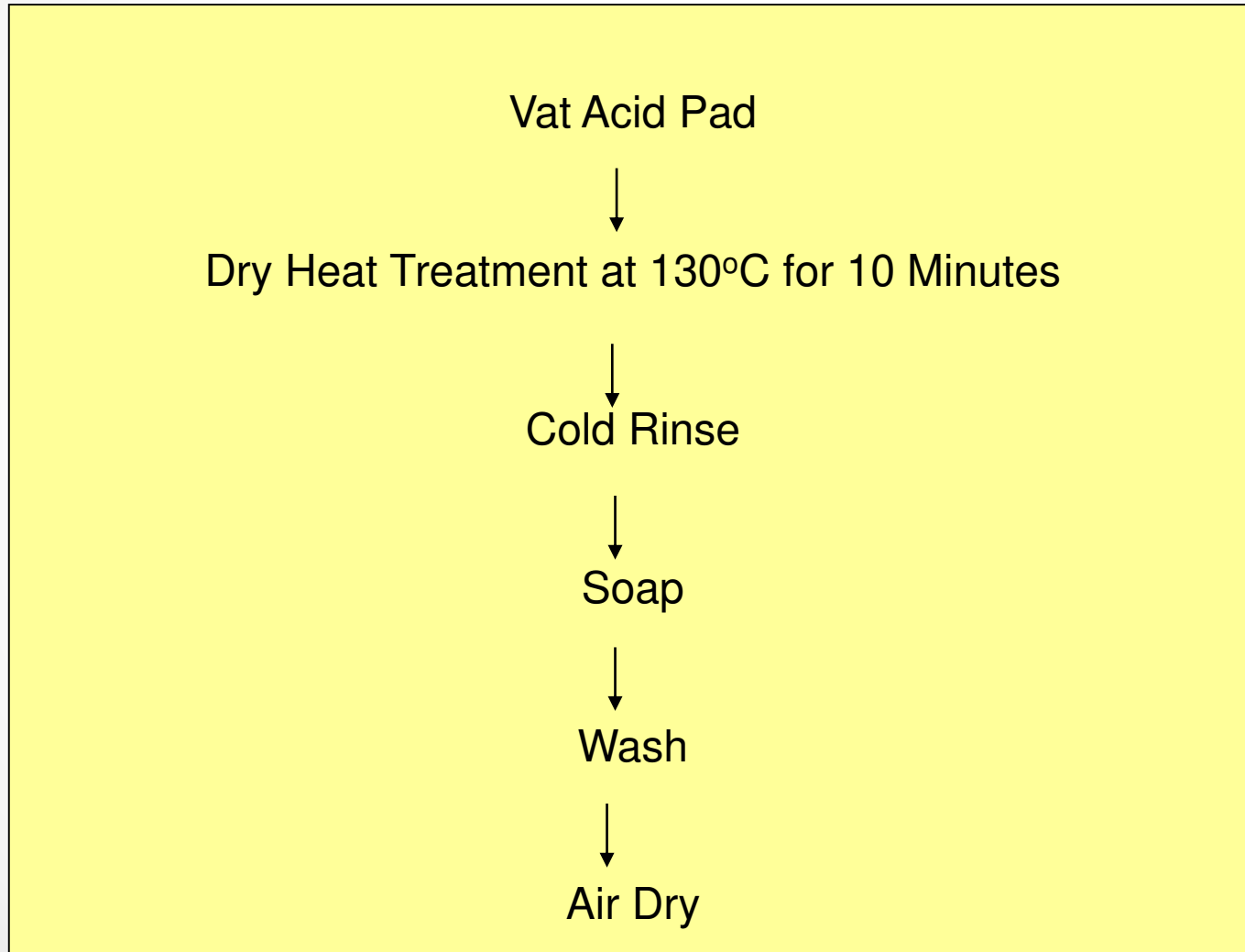
# Sequence for Simulated Pad-Steam Method



Eltz Hans-Ulrich, Lehinant Armand and Maier Hanspeter, German Patent, Hoechst AG, 1973, DE 2210878.

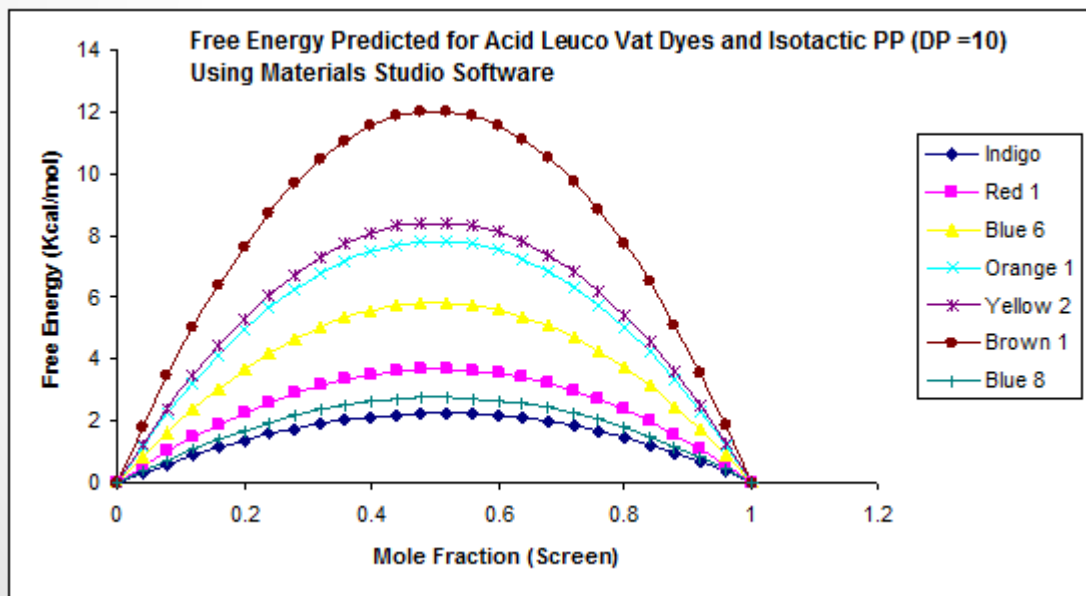
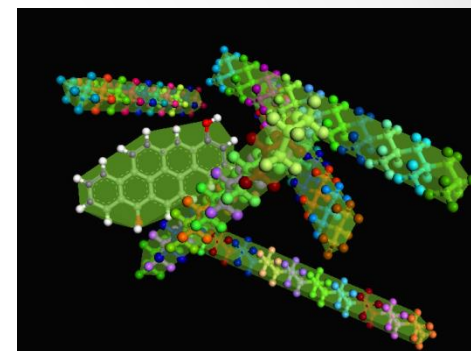
Trotman, E. R., Dyeing And Chemical Technology of Textile Fibers. Charles Griffin & Company Limited: 1975.

# Sequence for Simulated Pad-Dry Heat Method



# Molecular Dynamics-based miscibility simulation of acid leuco vat dyes with Isotactic PP (DP=10)

Predicted Free Energy of Mixing of Acid Leuco Vat Dyes with Isotactic PP at 90°C Using Accelrys' Materials Studio® Software



3-D space configuration of vat orange 1 interacting with isotactic polypropylene in Materials Studio workspace

# Reflectance Spectrophotometry

$$\frac{K}{S} = \frac{(1 - R^2)}{2R}$$

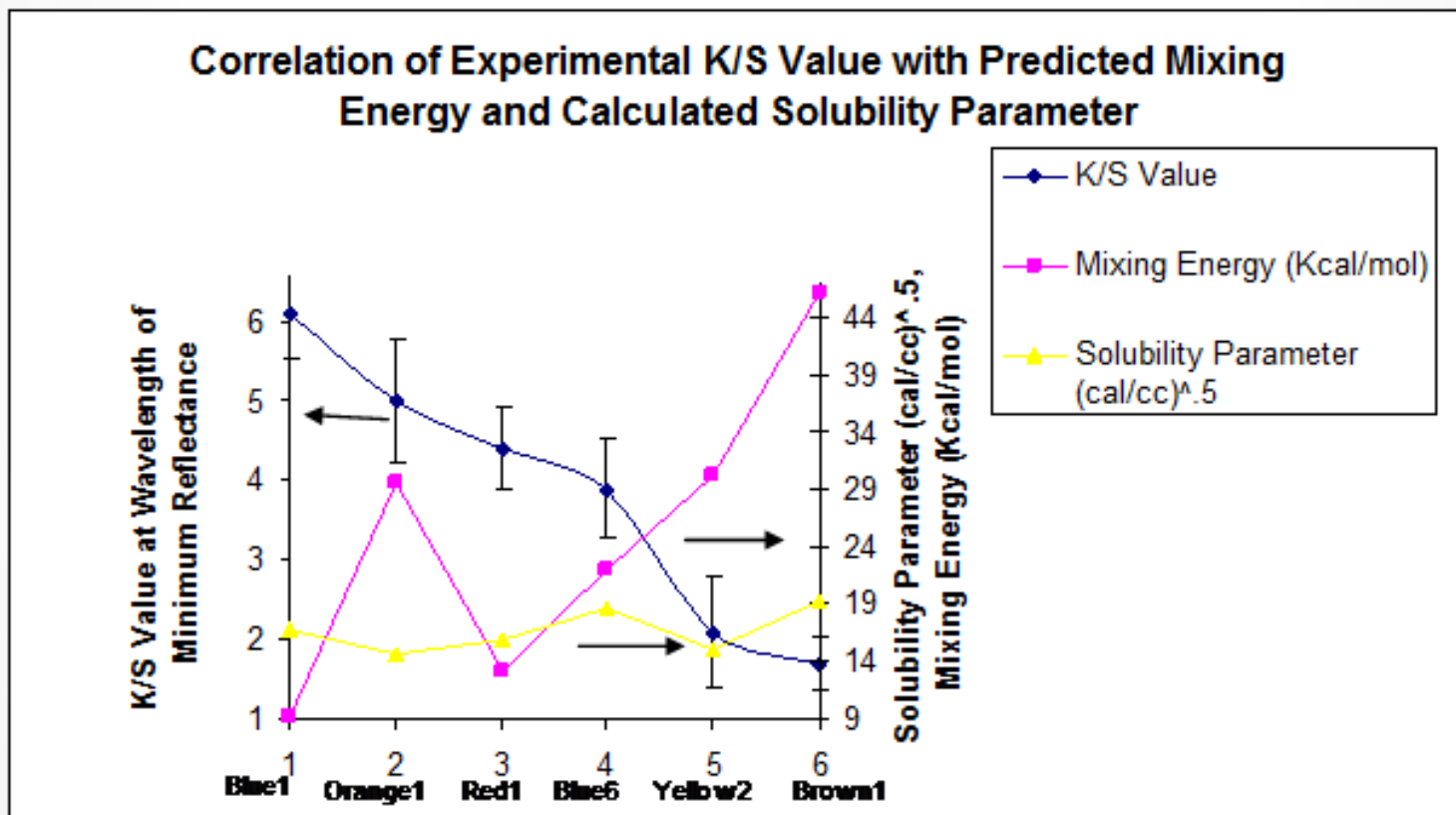
R: Reflectance

K: Coefficient of absorption

S: Coefficient of scattering

# Correlation of Experimental K/S Value with Calculated Acid Leuco

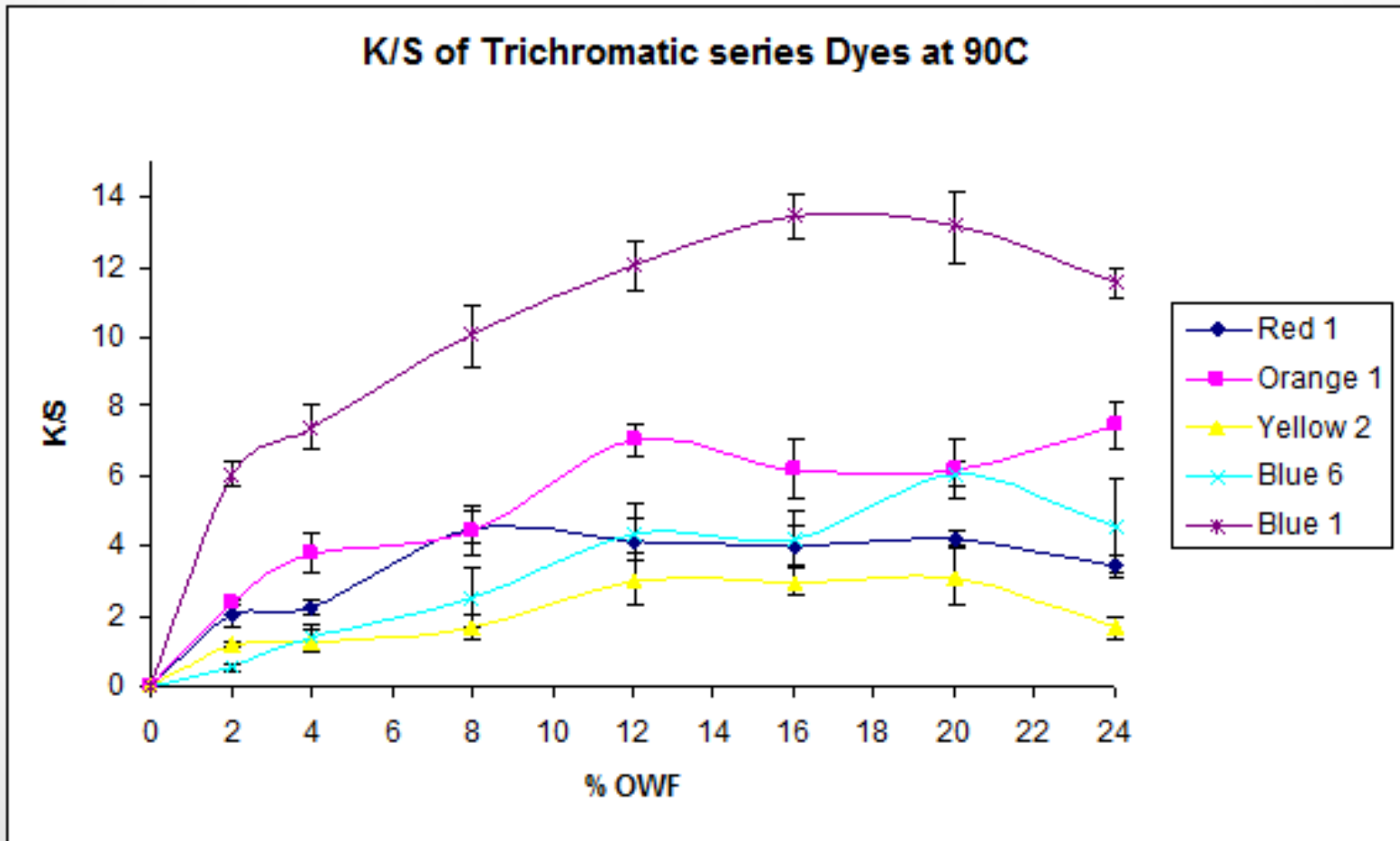
## Dye Solubility Parameter\* and Predicted Mixing Energy



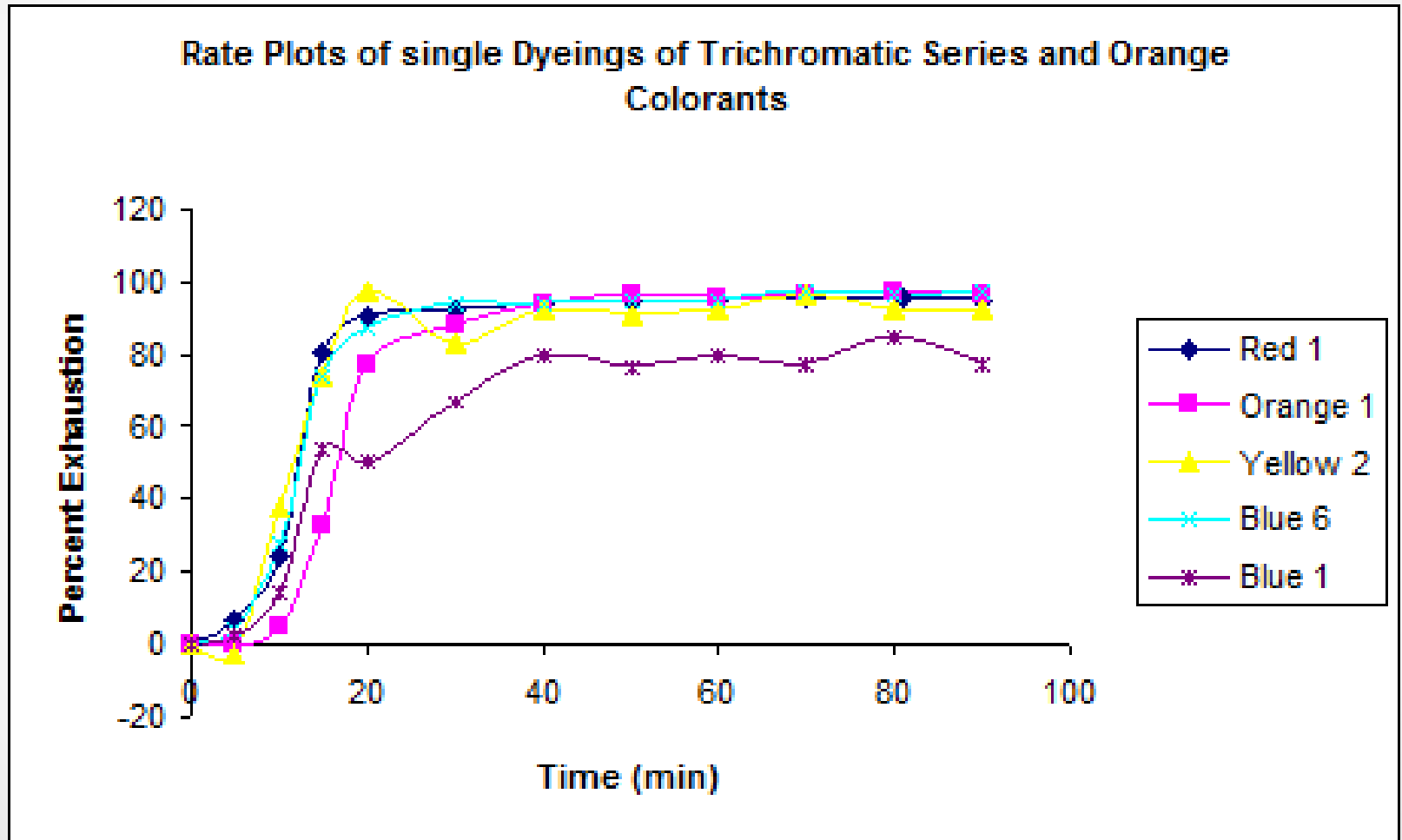
\*Solubility parameter was calculated using Fedor's method; Robert F. Fedors, Polym. Eng. Sci., Vol. 14, No. 2, 1974, pp.147-154

# K/S Values at Wavelengths of Minimum Reflectance versus %OWF

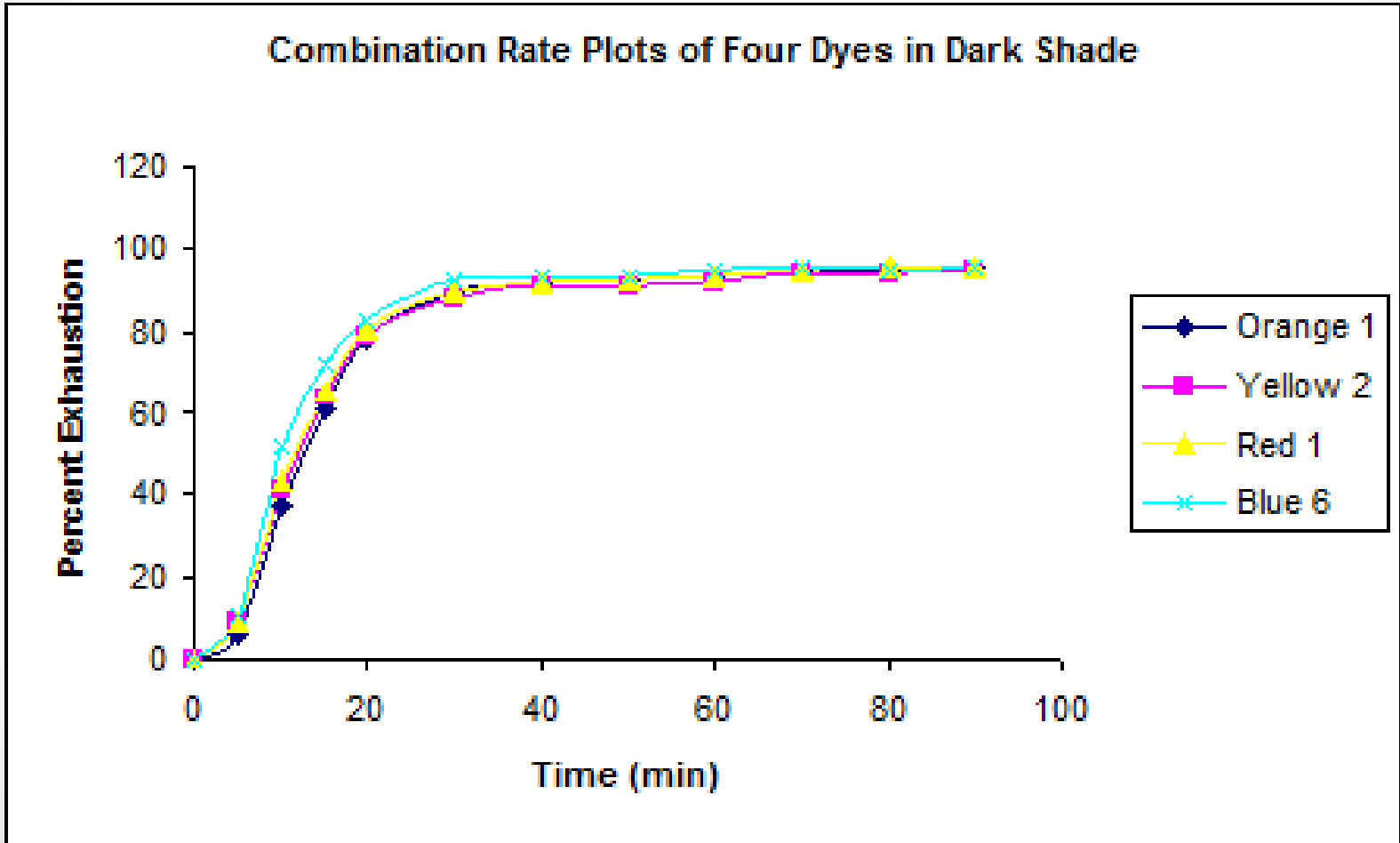
## Plots for Acid Leuco Vat Dyes at 90° C



# Dyeing Rate Plots for Single Dyeings

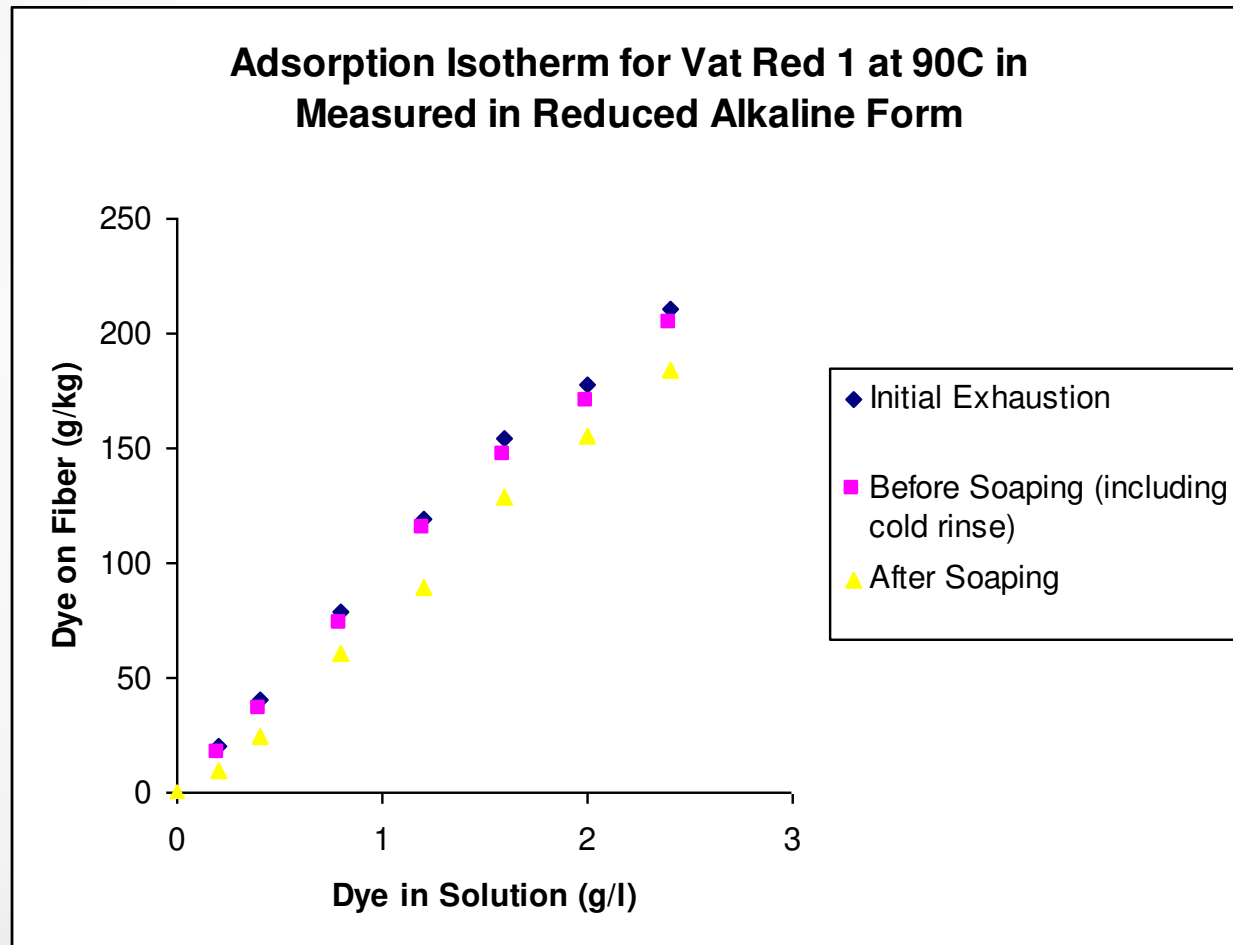


# Dyeing Rate Plots for Combination Dyeings of Trichromatic Series Plus Orange Colorants Continued...



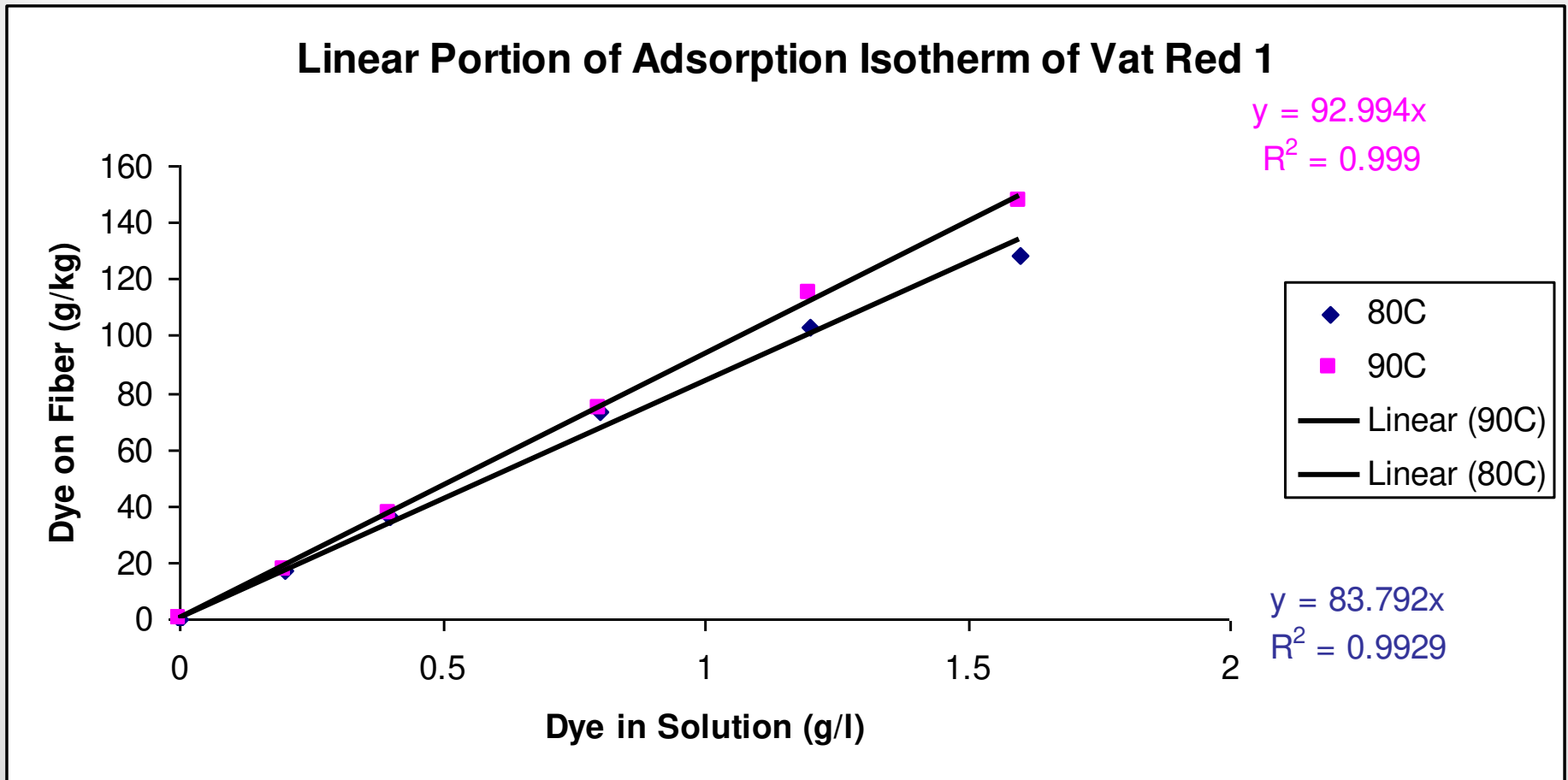
# Adsorption Isotherm for PP Acid Leuco Dyeing with Vat

## Red 1 Using Single-Stage Dyeing Method



Follows Nernst isotherm or solid solution model of dye diffusion

# Linear Portions of the Adsorption Isotherms of Trichromatic and Orange Vat Colorants at 90 and 80 C: Vat Red 1



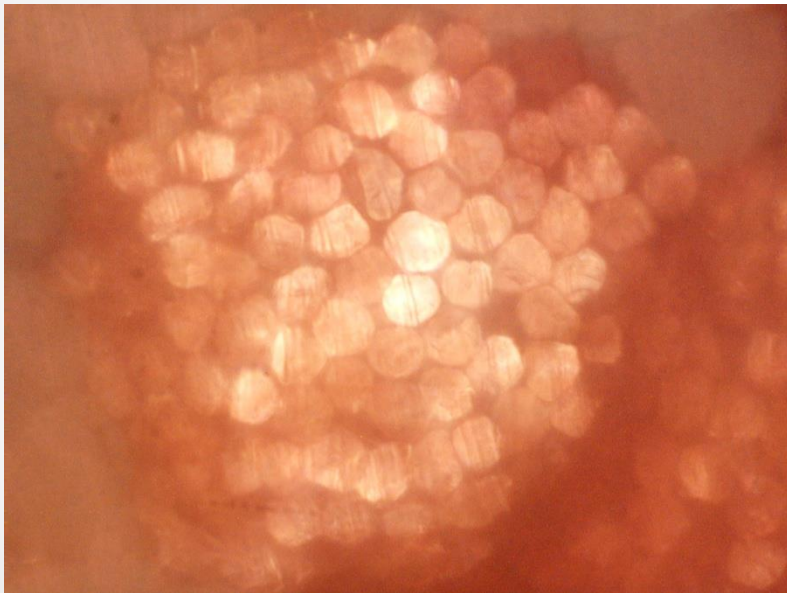
## Fastness Properties Continued

### Fastness to Washing\*

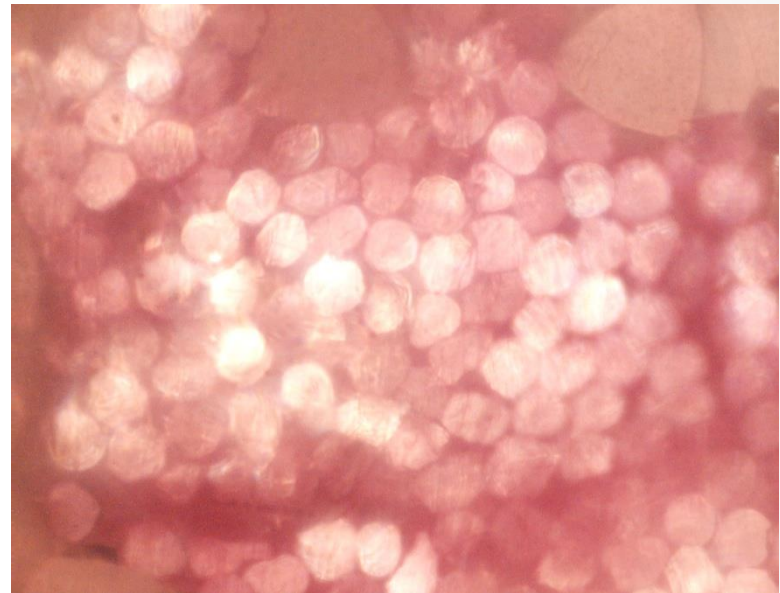
C.I. Name of Dye	Change in Color	Staining on the Various Components of Multifiber Fabric Style # 10					
		Acetate	Cotton	Nylon 66	Polyester	Acrylic	Wool
Vat Orange 1	5	5	5	5	5	5	5
Vat Yellow 2	5	4-5	4-5	5	4-5	5	4-5
Vat Blue 6	5	4-5	4-5	4-5	4-5	4-5	4-5
Vat Red 1	5	4-5	4-5	4	4-5	4-5	4-5
Vat Blue 1	5	4-5	5	4	4-5	5	4-5

\*AATCC Standard Test Method 61-2003 for Wash Fastness; AATCC Technical Manual/2005

# Cross-section Micrographs of Yarn Extracted from Acid Leuco Dyed PP Fabric



Cross-section of PP fiber dyed  
with Vat Orange 1



Cross-section of PP fiber dyed  
with Vat Red 1

# Experimentally Determined Thermodynamic

## Parameters of Dyeing

Name of Dye	Partition Coefficient at 90°C	Dyeing Affinity at 90°C (Kcal/mole)	Heat of Dyeing (Kcal/mole)	Entropy of Dyeing (Kcal/mole/°C) at 90°C	Half Dyeing Time (min) at 90°C	Diffusion Coefficient at 90°C (cm <sup>2</sup> /s)
Vat Red 1	93	-3.3	-2.5	-0.06	12	0.7x10 <sup>-10</sup>
Vat Blue 6	61.4	-3.0	-1.0	-0.04	12	0.7x10 <sup>-10</sup>
Vat Yellow 2	88.3	-3.2	-1.8	-0.06	11	0.7x10 <sup>-10</sup>
Vat Orange 1	81.9	-3.2	-0.4	-0.04	16	0.5x10 <sup>-10</sup>

# K/S Values of PP Fabrics Dyed with Pad-Steam Dyeing

Name of Dye	K/S at Wavelength of Minimum Reflectance
Vat Red 1	2.7
Vat Yellow 2	3.4
Vat Blue 6	3.6
Vat Orange 1	2.1
Vat Blue 1	8.9

## Results of Pad-Dry Heat Process

### K/S Value Obtained at Wavelength of Minimum Reflectance

Name of Dye	K/S at Wavelength of Minimum Reflectance
Vat Red 1	2.5

### Crock Fastness Ratings of PP Fabric Dyed with Vat Red 1

C.I. Name of Dye	Staining rating	
	Dry	Wet
Vat Red 1	4-5	4-5

### Wash Fastness Ratings of PP Fabric Dyed with Vat Red 1

C.I. Name of Dye	Change in Color	Staining on the Various Components of Multifiber Fabric Style # 10					
		Acetate	Cotton	Nylon 66	Polyester	Acrylic	Wool
Vat Red 1	5	5	5	4-5	4-5	4-5	5

# Conclusions

- ✓ Acid leuco forms of low SP/ low mixing energy vat dyes exhibited good affinities toward unmodified PP fiber at pH 7.0 in the developed single-stage, acid leuco vat dyeing process.
- ✓ Solubility parameter and blend miscibility approaches provided good correlations with experimental color parameters, thereby providing a scientific basis for prediction of the dyeability of acid leuco vat dyes on unmodified PP fibers based strictly on their chemical structures.
- ✓ Vat Red 1, Vat Yellow 2 and Vat Orange 1 dyes demonstrated good color yields and fastness properties with unmodified PP fiber, and thus are certified members for the trichromatic series + orange colors. Vat Blue 6 is a marginal candidate for the trichromatic series, but the best-performing blue colorant currently available .
- ✓ C. I. Vat Blue 1 exhibited good color yield and adequate fastness properties to coloring PP but is not compatible with the colorants of trichromatic series plus orange because of its faster air-oxidation.

# Conclusions Continued

- ✓ C. I. Vat Blue 8 yielded low SP and mixing energy, indicating the possibility of its having better affinity with PP than Vat Blue 6. However, Vat Blue 8 is currently not in commercial production.
- ✓ Vat dyes possessing high solubility parameters/mixing energies (e.g., Vat Brown 1) exhibited lower color yield than low solubility parameter/low mixing energy vat dyes.
- ✓ Experimentally-determined thermodynamic parameters such as affinity of dyeing and heat of dyeing quantified the significant degree of interaction between the certified acid leuco vat dyes and generic PP fiber, e.g., the negative heats of dyeing and low entropies of dyeing.

# Conclusions Continued

- ✓ Adsorption isotherms confirmed high saturation values of the certified dyes in the fiber phase, resulting in good color yields on the dyed fabrics.
- ✓ Dyeing rate plots with colorant combinations exhibited similar profiles to those of the component dyes, confirming the compatibility of the certified vat dye mixtures (critical for wide shade development of products).

# Conclusions Continued

- The simulated pad-steam method for all dyes produced fabrics exhibiting good dye levelness.
- The developed, simulated pad-dry heat (modified Thermosol®) dyeing method for Vat Red 1 on generic PP produced a fabric with a similar K/S value to that generated by the simulated pad-steam method with the same dye at the same pad bath formulation.
- Dyed fabrics produced by both the pad-steam and pad-dry heat processes exhibited adequate fastness to wet/dry crocking, washing and dry cleaning.

# Next Generation Broadloom Carpet Construction

**Anthony Casio**

Anthony John Casio, *Development of Next Generation Carpet Backings for Facile Recyclability* (2006)

# Next Generation Carpet

- Opportunity: 7B lbs. of post-consumer carpet (PCC) are landfilled in the US each year!!!!
- States are warning that they may ban PCC from landfills, e.g., as they have tires.
- Carpet and Rug Institute (CRI) signed in 2007 a MOU with 15 states which sets a goal of reducing PCC going into their landfills by 40% within 5 years.



# Current Broadloom Carpet (BLC) Construction

- Consists of a soft composite:
  - Two thermoplastic polymers (nylon (NY) face yarns, woven polypropylene (PP) primary and secondary backings).
  - One thermoset polymer (styrene-butadiene rubber (SBR), applied as a latex foam, consolidates the construction).
  - Calcium carbonate (CaC) fine particle filler in the SBR.
- Construction is unfriendly to recycling:
  - Cured SBR rubber is extremely strong and resistant to cutting/shredding.
  - CaC filler is a hard abrasive that wreaks havoc on knives/cutters.
  - Tufted yarns are imbedded and locked in the backing.

# Restrictions Set By Industry

- Next generation carpet (NGC) must be cost and performance neutral to current PCC construction.
- Current manufacturing lines must be kept intact.
- NGC must possess simple, economical recycle capability, with near-total material utilization.

# Main Opportunity Points of Attack

- PP primary and secondary backings:
  - Primary backing is woven PP tape (15 picks/inch) that yarn is tufted into.
    - The tufting bar operates like a multi-needle sewing machine.
    - The impact forces exerted on the backing by the rapidly moving needles are extremely large.
  - Secondary backing is woven PP tape warp, with staple yarn PP filling (five picks/inch).
  - PP is extremely cheap (~45 cents/lb.).
  - PP properties, however, are great for the application.
- What fiber could possibly meet both the cost and property attributes of PP in the two primary backings?????

# Owens Corning Fiberglass Non-woven Mat

- Fiberglass is produced from silicate (sand).
  - An OCF mat is currently utilized in carpet tile (CT) production.
  - Mohawk Industries is currently producing a tufted carpet line utilizing a polyester fiber non-woven primary backing.
  - OCF supplied the project with a variety of fiberglass non-woven mat fabrics designed to withstand tufting needle forces.
- 
-

# First: Identify SBR/CaC Substitute

- Identified nylon 6 as a performance substitute for SBR.
  - NY6 is melt compatible with NY6,6, plus has reasonable adhesive capability with PP.
  - Economic trade-off was in utilization of recycled nylon fiber from “shaving” PCC and CT.

# Optimization of NGC

- Added to the optimized glass primary backing a thin film (5 mil) of NY6.
  - Ran the “layered structure” through heated calendar rolls (210C, softening point of NY6).
  - Tufting successful in generating targeted tuft properties and vastly reduced glass particle ejection.
- Secondary backing was a lighter weight OCF NW mat, and the additional NY4 was added at the consolidation stage via a melt slot extruder adapted from the carpet tile line of the plant.

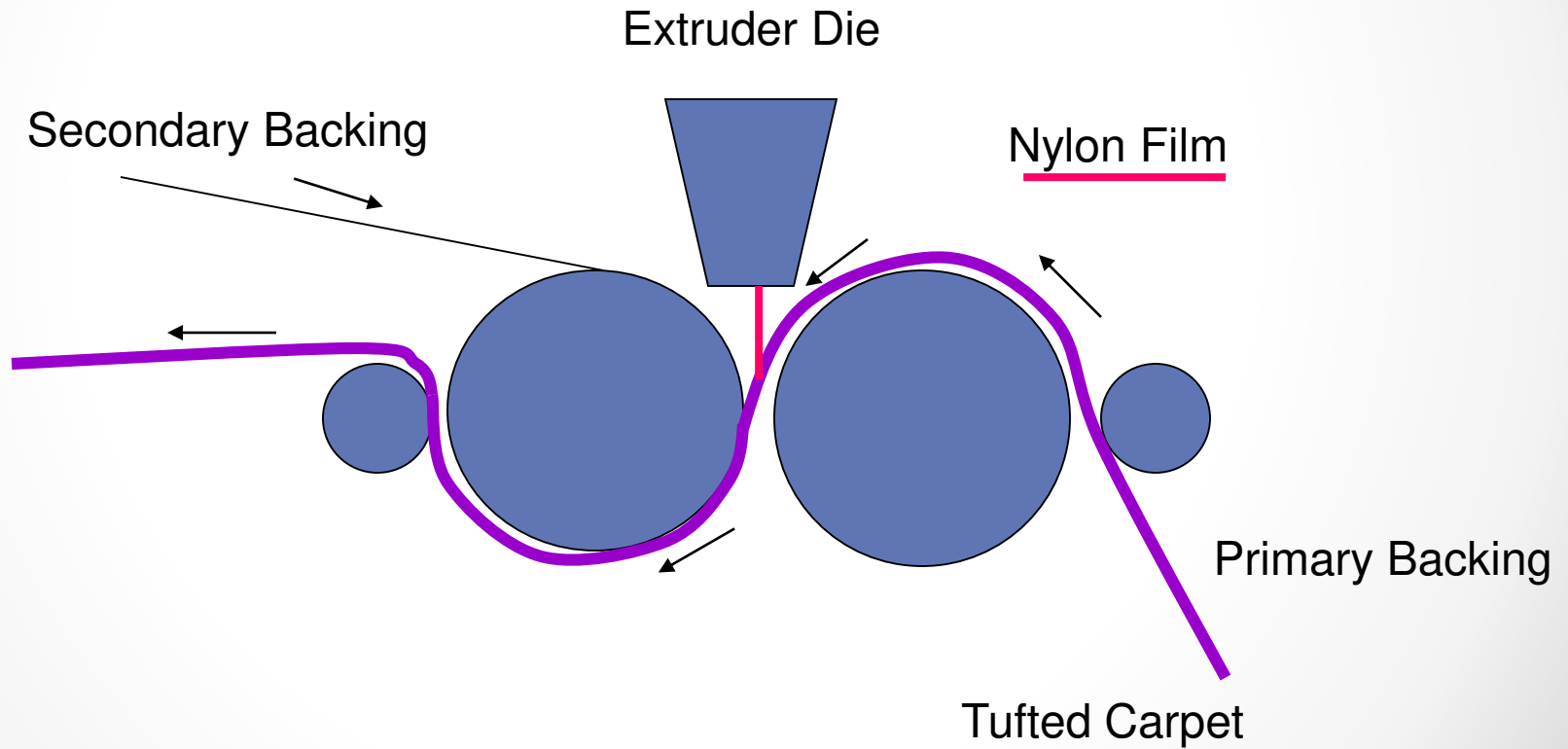
# Simple, Economical Recycling Scheme

- NGC is composed of two materials, glass and nylon.
  - NY6 melting point (MP) is ~240C, NY6,6 MP is ~260C.
  - Glass MP is ~1250F.
- Heated (280C) continuous melt belt line with holes in the belt to allow molten nylon to flow to a collection chamber below the belt will separate the two materials.
  - Nylon melt recycled into new products (fibers, plastics, composites, etc.).
  - Glass fiber reformed/recycled (either by shredding/reforming articles, or by melting/extruding into fibers) into carpet backings or other products (e.g., composite reinforcement fabrics).

# Alternate NGC Developed on Project

- Replaced the SBR/CaC binder/adhesive with recycled NY6 (applied by slot melt extrusion as before).
- Kept the same PP primary and secondary backings as in the current construction.
- Utilized previous research findings that PP and NY can be made melt compatible by the addition of ~5% by weight maleated PP to the mixture.
  - Mix of the two materials will be ~90% weight NY/10% weight PP (will change more toward PP if the nylon face fiber is first shaved off the PCC).
  - NGC can then be cut/shredded and fed directly into a melt extruder, formed into new articles of lower properties.

# Extrusion Coating



# Nylon 6 Carpet coated with Nylon 6 Contains PP Primary Backing



# Conclusions

- Extrusion coating viable alternative to latex binding
  - Nylon copolymers work
  - Nylon 6 works
  - PP backing fabrics are viable with nylon 6 coatings

# Permanent Colorants and Fiber Finishes via Ionic Self Assembly-Covalent Fixation

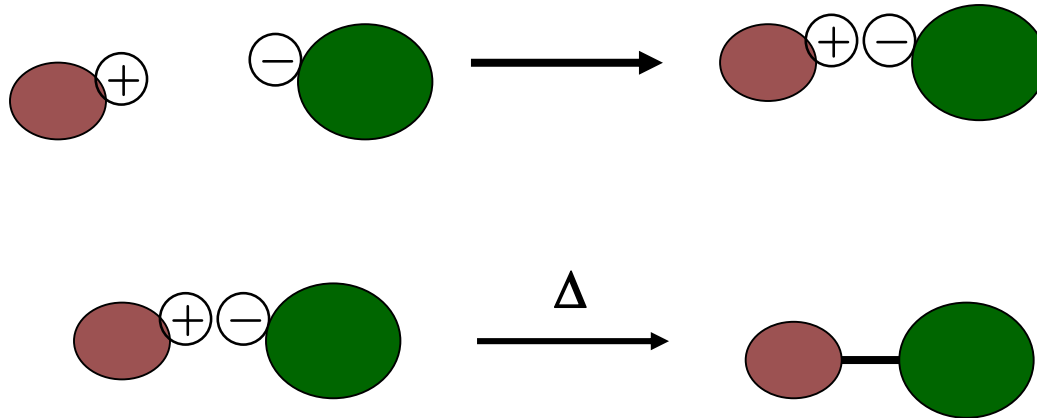
Christopher A. Hubbell

1. Christopher Hubbell, *Surface Modification and Chromophore Attachment via Ionic Assembly and Covalent Fixation*, Co-Advised with Haskell Beckham (2009)
2. Foston, M; Hubbell, C; Park, D-Y; Tezuka, Y; Beckham, H; Cook, FL., 2012. *Surface Modification by Electrostatic Self-Assembly Followed By Covalent Fixation*. *Angewandte Chemie International Journal*, 51(8), 1849.

# Research Goals

---

- Incorporate a novel, ionic functional group in textile finishes and colorants, leading to high-yield add-on and permanency



- Apply the chemical containing the reactive ionic group and optimize application conditions for various substrates
- Compare the reactive ionic chemical to analogous conventional chemicals in terms of fixation yield, salt requirements, etc.

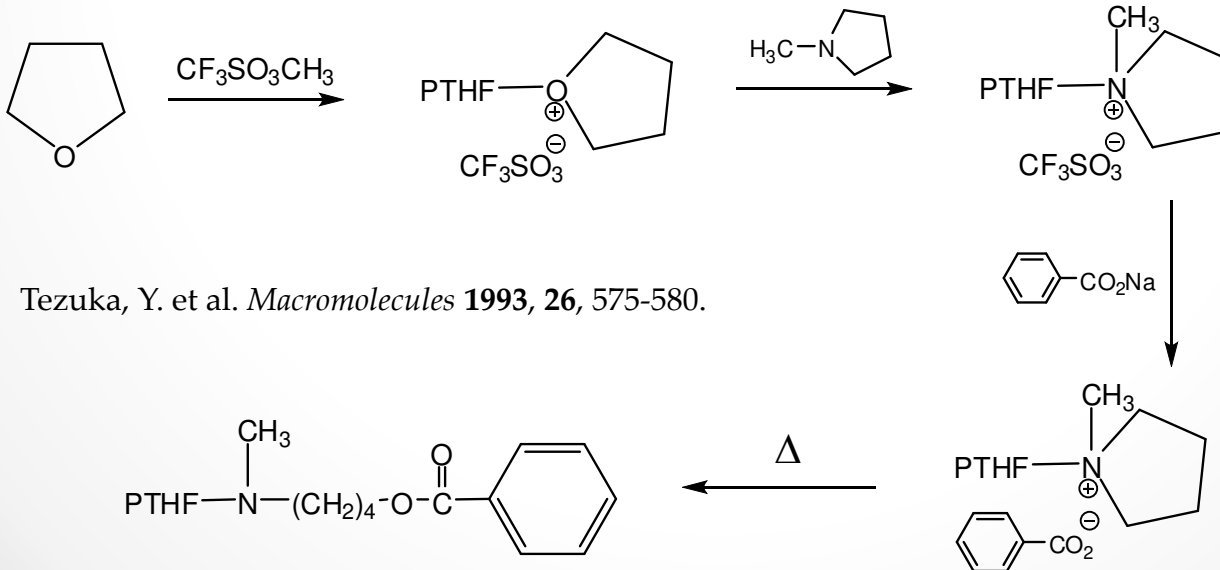
# Introduction to Ionic Self-Assembly

## and Covalent Fixation

Macromolecular, ion coupling reactions for complex polymer topology

Cyclic “onium” salt end groups attach ionically to negatively charged carboxylate groups

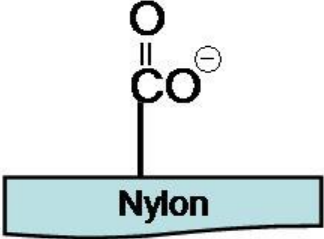
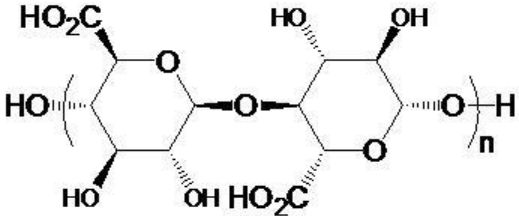
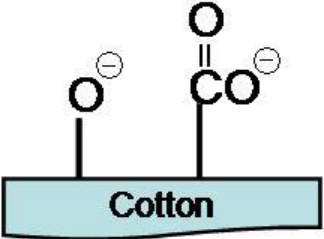
Conversion of ionic bond to a covalent bond via ring-opening reaction at elevated temperatures



Tezuka, Y. et al. *Macromolecules* **1993**, *26*, 575-580.



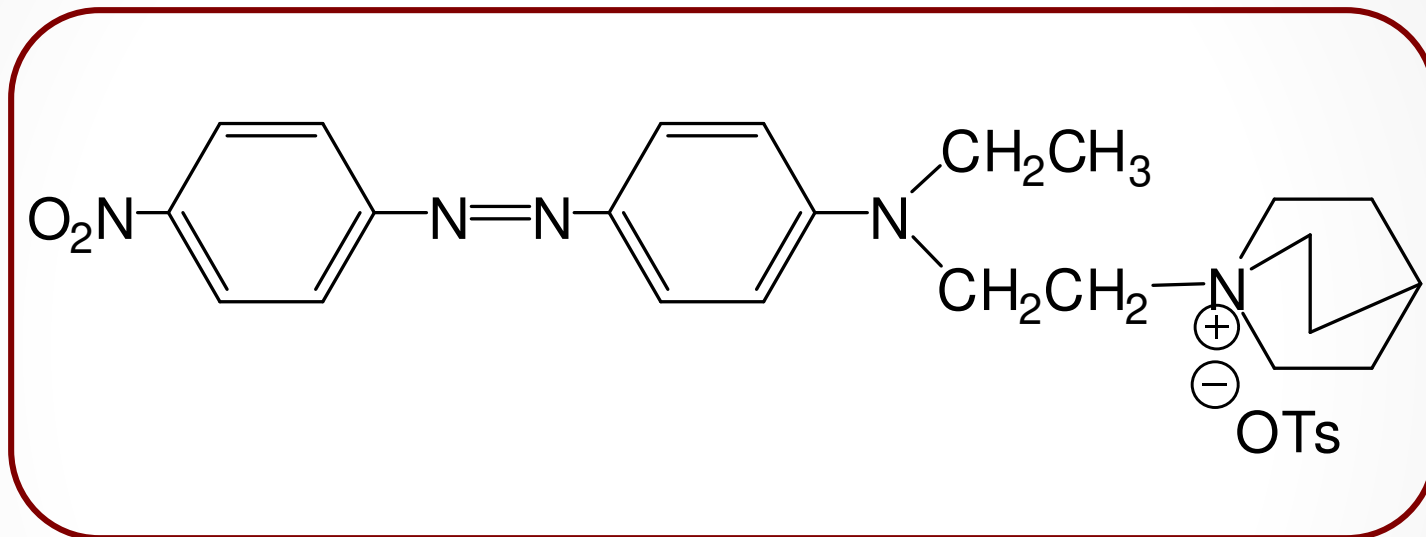
# Suitable Substrates

<p><b>Nylon</b></p>	$\text{H} \left( \text{N} \begin{array}{c} \text{H} \\   \end{array} \text{---} (\text{CH}_2)_5 \text{---} \text{C} \begin{array}{c} \text{O} \\    \end{array} \right)_n \text{---} \text{OH}$	<p>Acid chain ends at the surface form carboxylate ions in basic conditions</p>	
<p><b>Bleached Cotton</b></p>		<p>Carboxylate and alkoxide ions are formed under basic conditions at different pH values.</p>	

Nylon (MW = 20 kg/mol) has approximately 5 mmol/100 g of chains and thus the same number of carboxyl end groups. Cationic dyeable nylon contains up to twice this number.

Bleached cotton can contain up to 14 mmol/100 g of carboxyl groups due to oxidation of primary alcohols along cellulose chain during bleaching process.

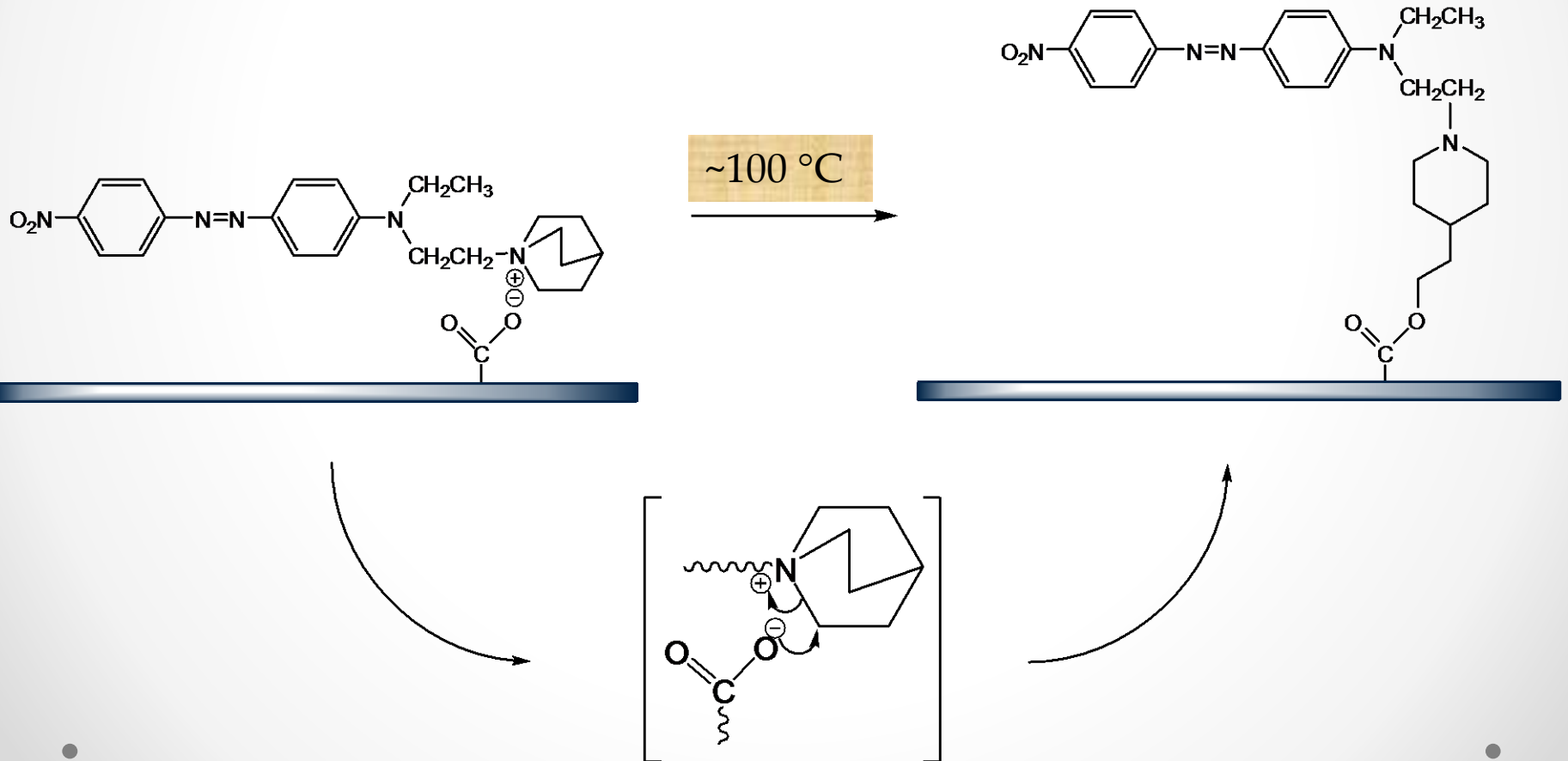
# Structure



Reactive-Ionic Dye

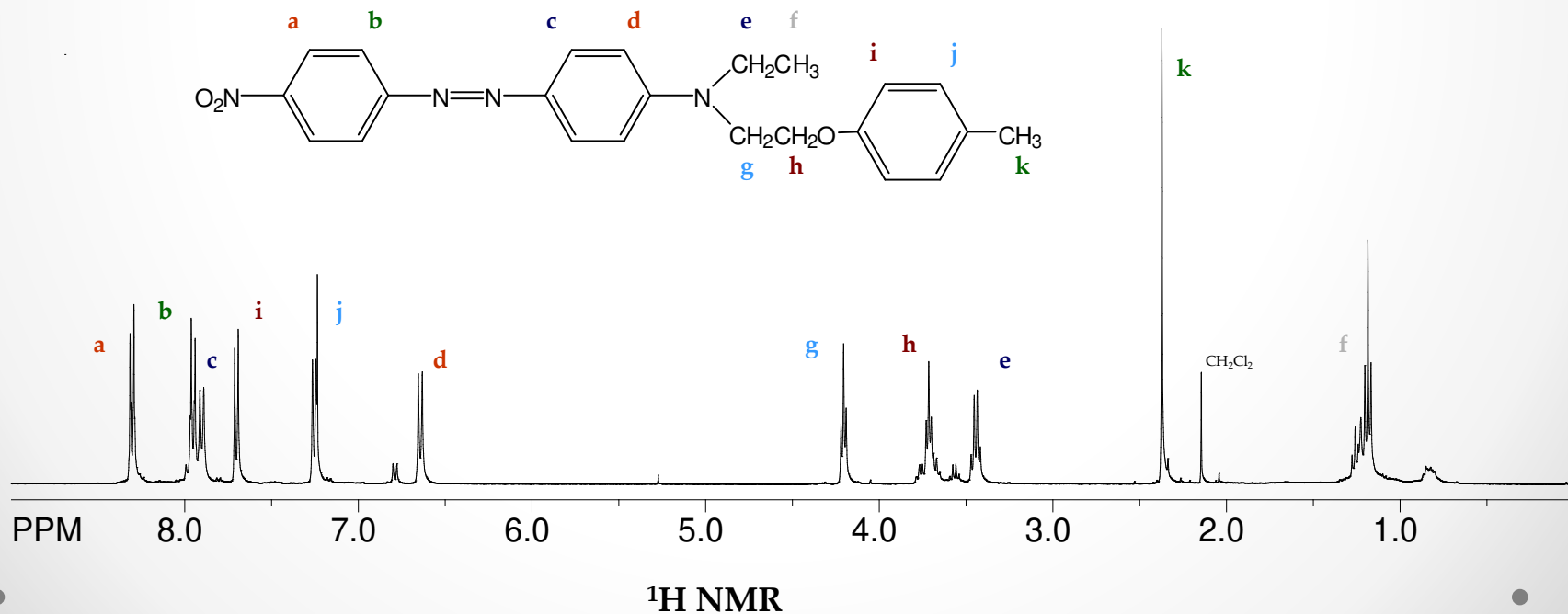
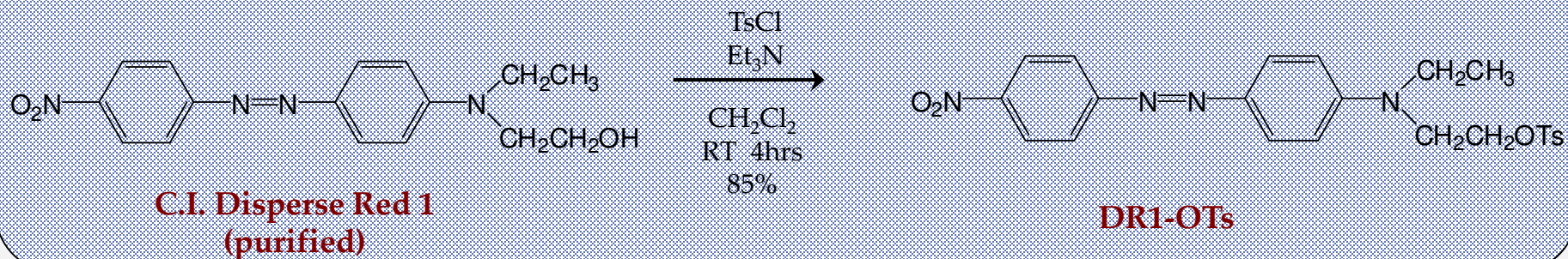
# Covalent Fixation of Dye on Surface

- Electron attack on  $\alpha$ -carbon of ring at elevated temperatures
- Leads to ring-opening and covalent fixation



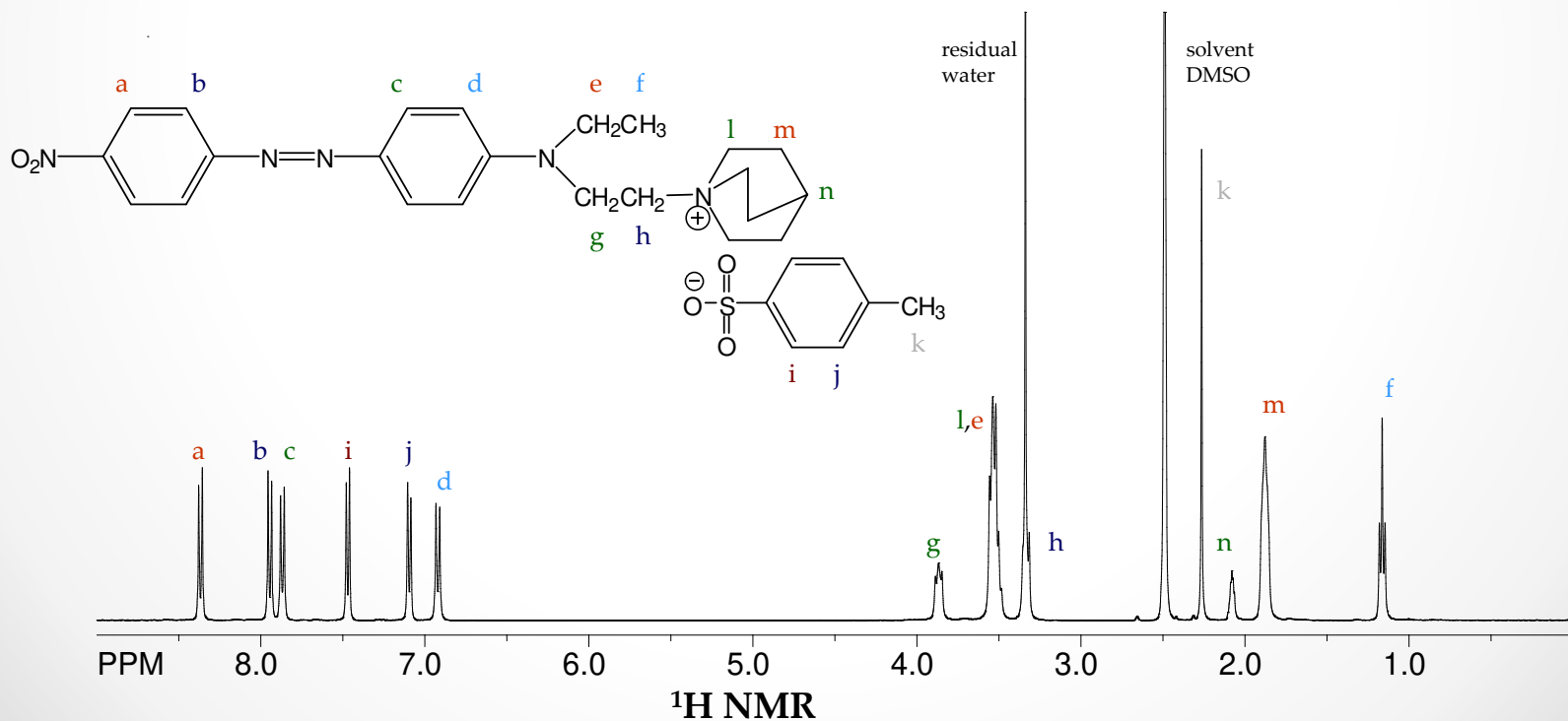
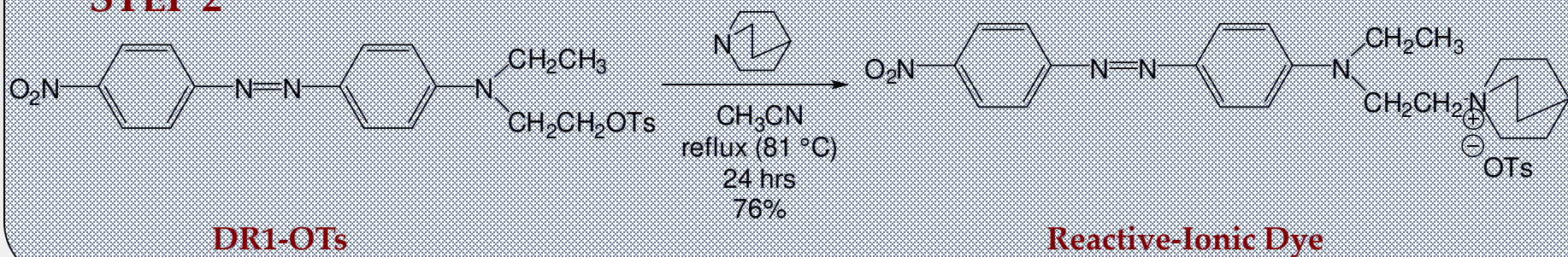
# Reactive-Ionic Dye Synthesis

## STEP 1

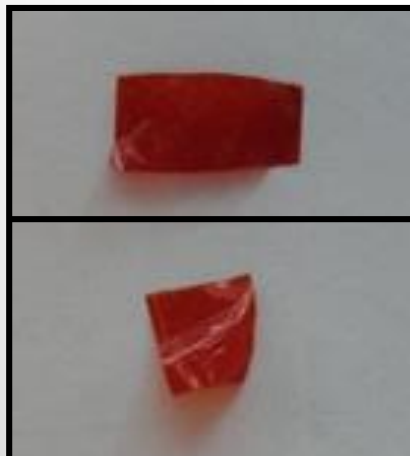
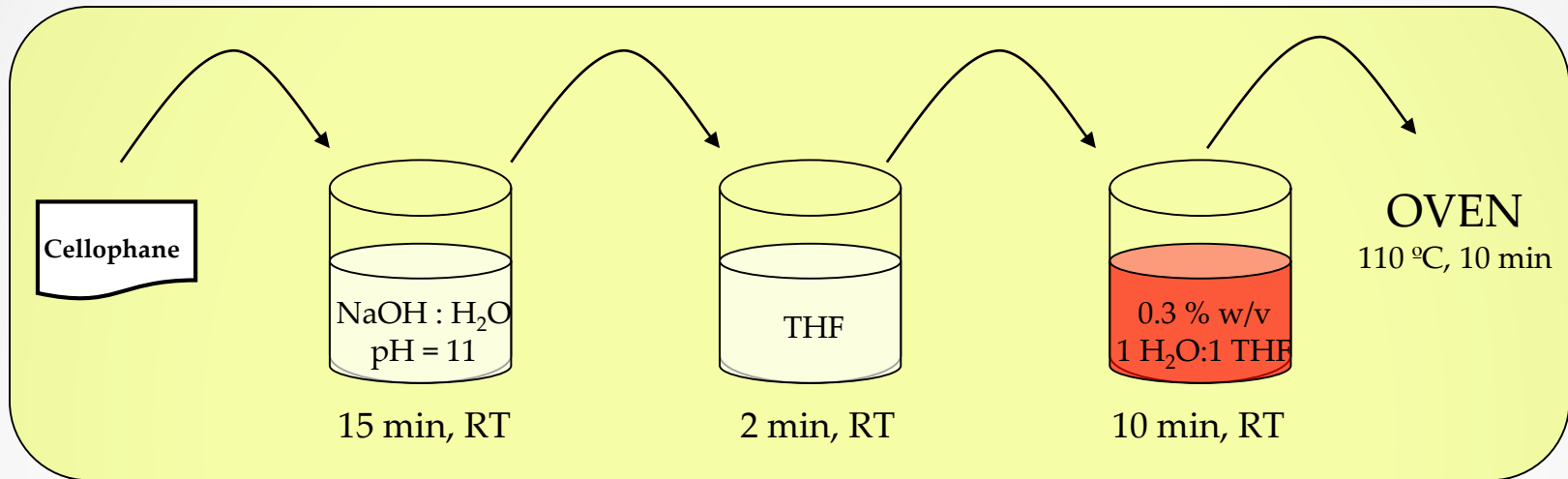


# Reactive-Ionic Dye Synthesis

## STEP 2



# Application of RI Dye to Cellophane



**RI Dyed Sample**

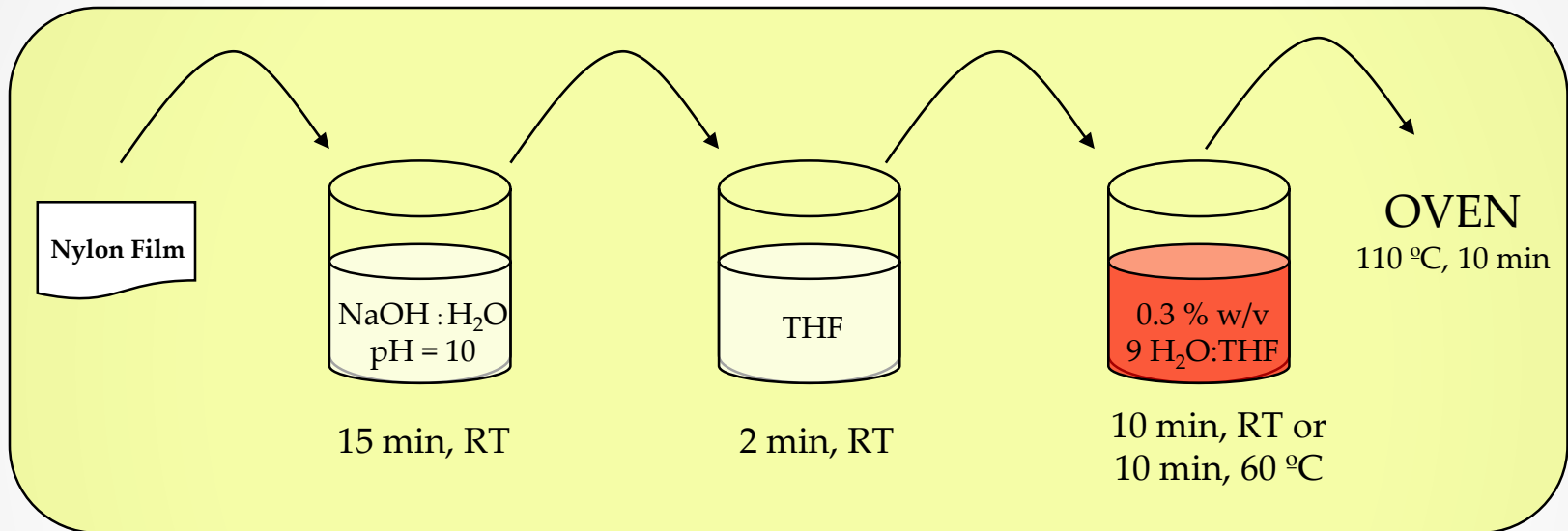
**RI Dyed Sample  
w/ 15 min extraction  
(THF @ boil)**

**DR1  
Dyed Sample**

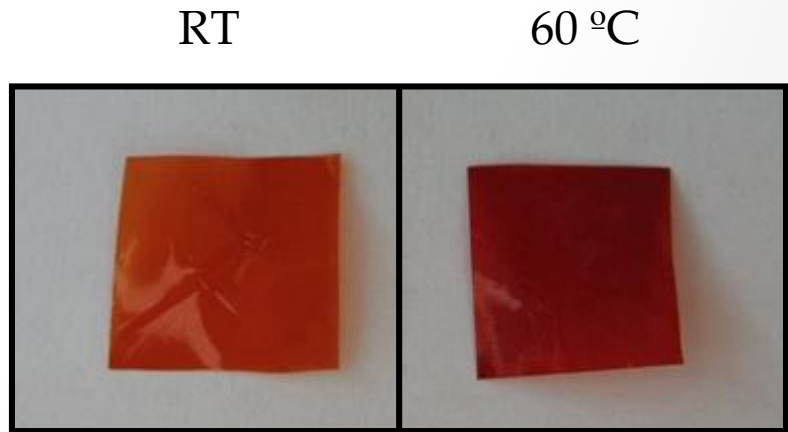
**DR1-OTs  
Dyed Sample**



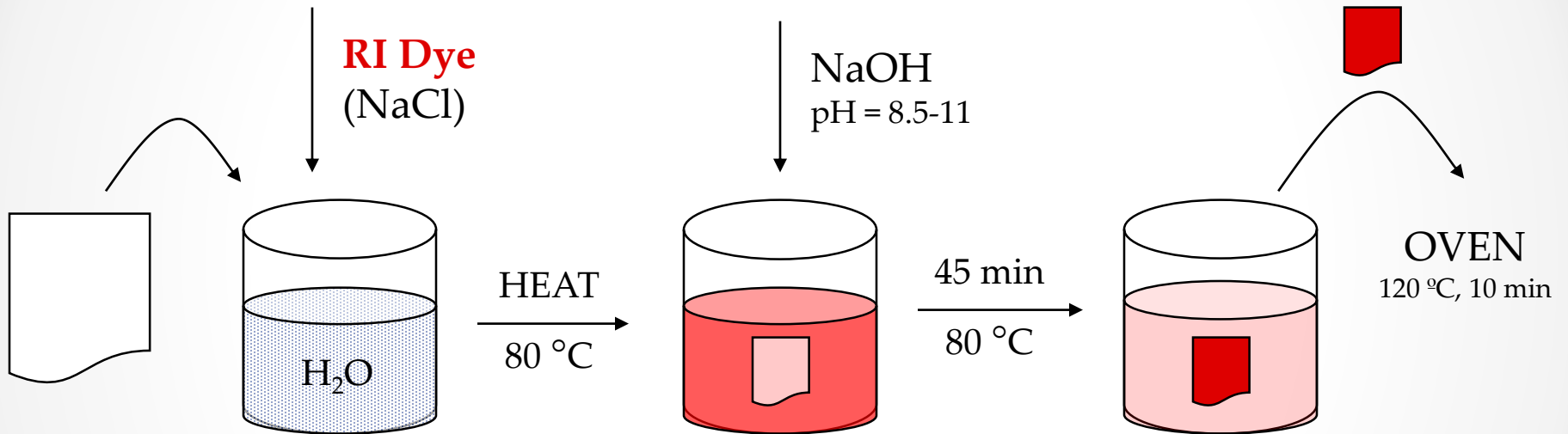
# Application of RI Dye to Nylon 6,6 Film



Post-dye Extraction:  
15 min THF boil



# Application of RI Dye to Fibers and Fabrics



# RI-Dyed Multi-Fiber Strip

## Dyebath

LR = 30:1, pH = 8.5

0.5% owf RI dye

80 °C, 30 min

Types of functional  
groups?

pH dependence  
for cellulose?



Cellulose Acetate

Modacrylic

Cellulose Triacetate

Cotton

Acrylic (Creslan 61)

Polyester (Dacron 54)

Polyester (Dacron 64)

Nylon 6,6

Acrylic (Orlon 75)

Silk

Polypropylene

Rayon

Wool

# How Permanent Is RI Dye on Nylon?

## Dyebath

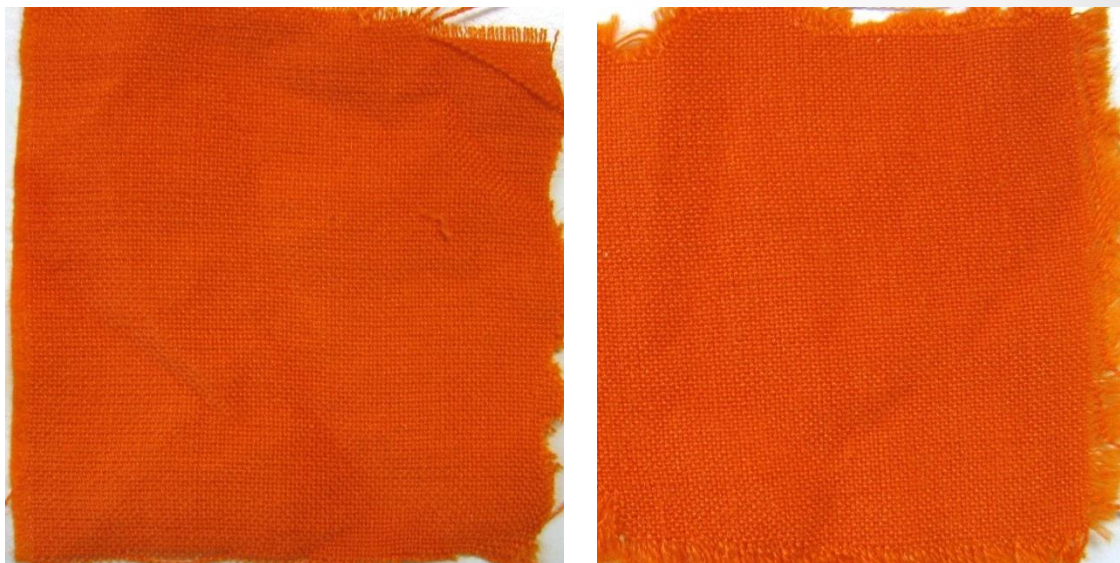
LR = 30:1, pH = 8.5

1% owf RI-Dye

80 °C, 30 min

## Fixation

120 °C, 20 min



No Extraction

15 min THF

Reflectance Data:

K/S:

15.1

6%  
color  
loss

14.2

		L	a	b	dE
unextracted extracted	std	50.08	50.22	45.35	
	sample	50.70	48.63	45.13	1.72
	$\Delta$	-0.62	1.59	0.22	

# Is RI dyeing a high exhaustion process ?

Silk Fabric



1% owf

2% owf

**K/S: 16.7**

**24.0**

Dyebath

LR = 50:1, pH = 9

80 °C, 30 min

Exhaustion =

$$(C_0 - C_t) / C_0 =$$

$$(A_0 - A_t) / A_0 =$$

$$(1.65 - 0.04) / 1.65 =$$

**Exhaustion ~ 98%**

For 1% owf  
RI dyeing  
of silk fabric

# Conclusions

---

- Novel method for high-yield add-on and permanency of colorants
- A RI dye has been prepared from C.I. Disperse Red 1
- RI dyeing of cellophane and nylon films, bleached cotton, silk and nylon fabrics
- High salt levels do not benefit the adsorption of RI-dye on bleached cotton fiber
- Dyebath exhaustion of 98% for 1% owf RI dyeing of silk

# Development of Novel Synthetic Turf Infill Materials

Richard E. Harper, Jr.

Richard Harper, *Development of Novel Synthetic Turf Infill Materials*, Co-Advised with John Muzzy (2015)



# What is synthetic turf?

Natural turf is grass growing from loose soil.



AP Photo/AI Behrman

Synthetic turf is polymer “grass” with **infill** acting as loose soil.



AP Photo/Gene J. Puskar

In the worst weather or during hard play, natural turf breaks apart sooner than synthetic turf. Consistent performance reduces injuries like sprains or concussions.

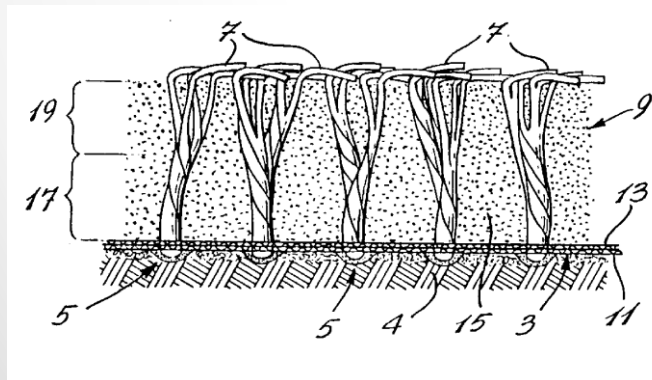
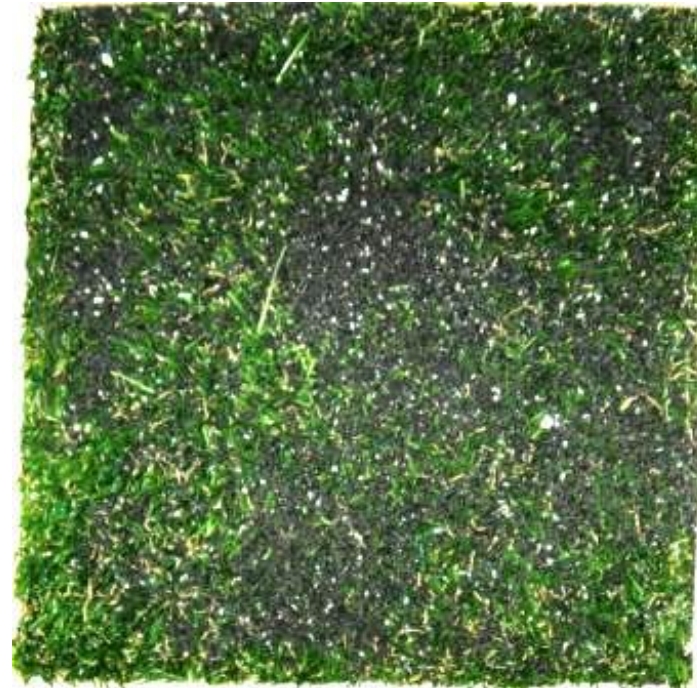
# What is infill?



AP Photo/J.D. Pooley



Discarded automotive tires is ground into granulated crumb rubber that fits between the tufts of STF.



US Patent 6,723,412 B2 (Figure 2.1)

# Objectives of Study

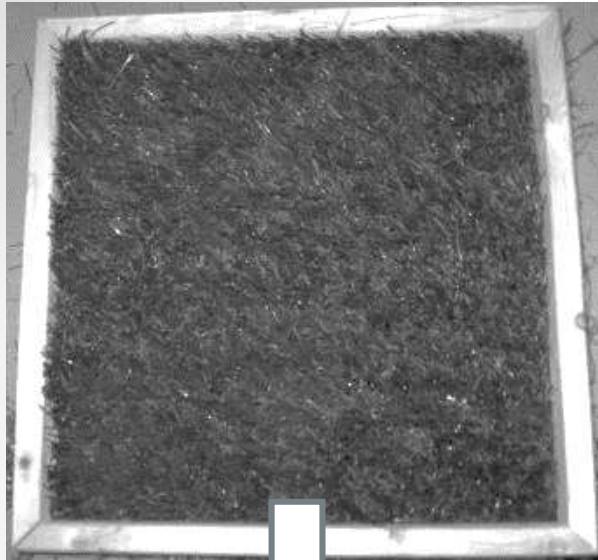
- Develop a fundamental understanding of how standard granulated crumb rubber infill (GCRI) and STF operate synergistically to meet safety performance goals.
- Identify and evaluate polymeric waste streams that lack the environmental and health issues of GCRI as potential infill replacements in STF.
- Compare the economics between GCRI and the alternative polymeric material(s) that are shown to be technically feasible as STF infills.

# Impact Instrument

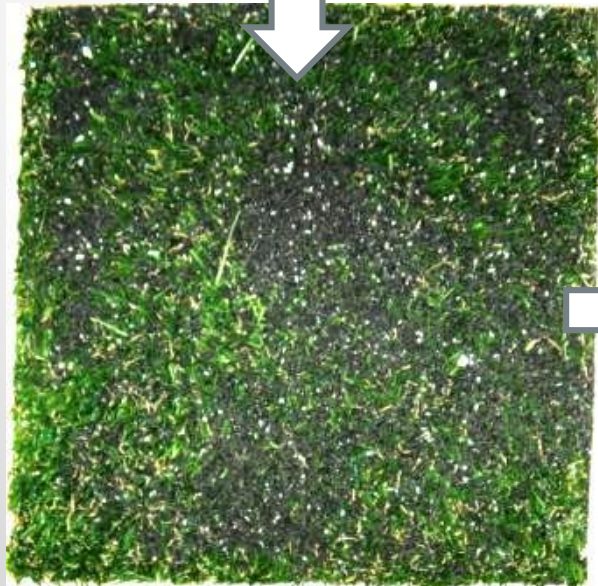


Shorten, M. *Sports Surface Impact Test System Instruction Manual*,  
©2001-2003 Biomechanica, LLC (Figure 4.8)

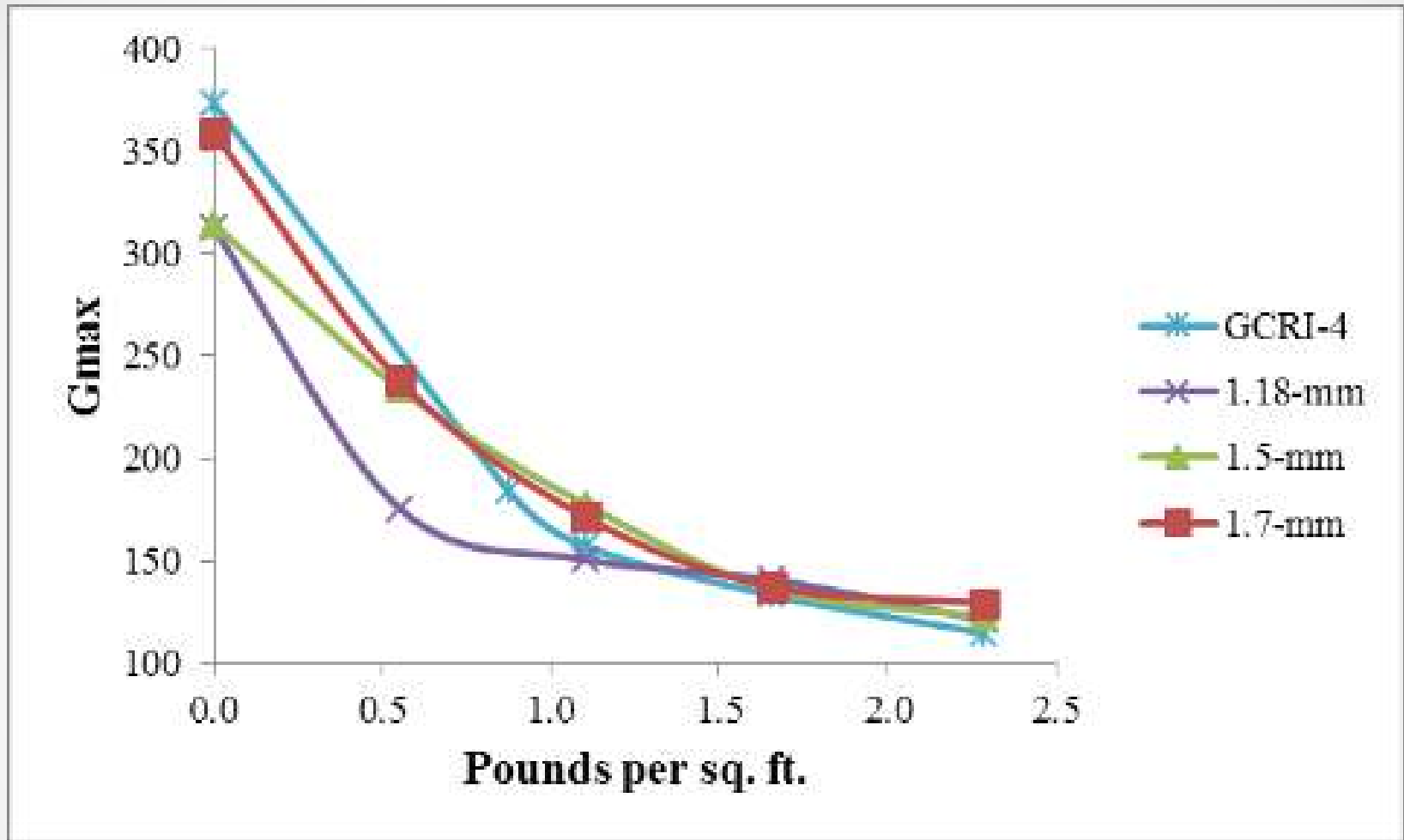
# Impact Test Setup



- (Counter clockwise)
- 12 x 12 square inches of turf stapled to wooden frame. (Fig. 4.9)
  - Infill integrated into turf.
  - Temperature and level of infill monitored. (Fig. 4.12)
  - Impact instrument placed on level surface prior to missile fall. (Fig. 4.10)



# Refined GCRI on Impact



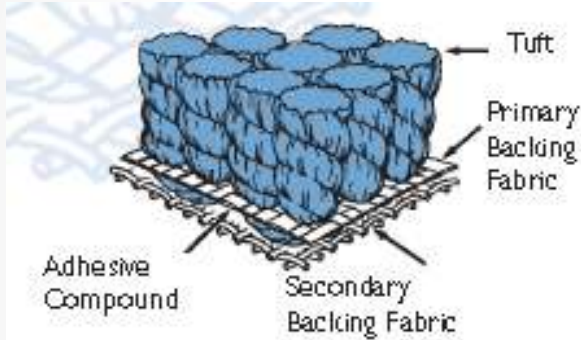
(Figure 5.16)

All three sizes displayed non-linear curves similar to GCRI-4 , but none ultimately exceeded the unrefined standard. Thus, particle size cannot be refined at least between 0.5 – 2.5 mm .

# Alternates to infill

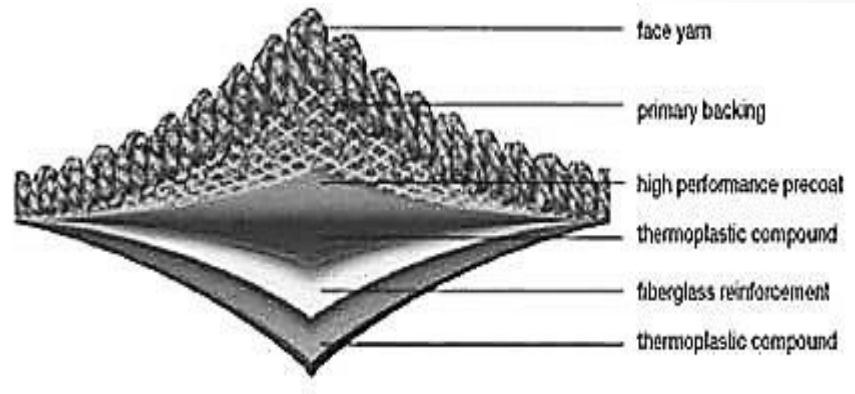
A viable replacement would be benign to human exposure while performance and cost still match GCRI. Logistics made carpet reclaim available.

Shaved backing from post-consumer carpet broadloom (**PCCB**) is flexible and cross-linked.



Primer, Carpet and Rug Institute, 2008 (Figure 2.5)

Post-consumer carpet tile (**PCCT**) yields a firm, thermoplastic composite after shaving the face yarn.



Shaw EcoWorx (Figure 2.7)

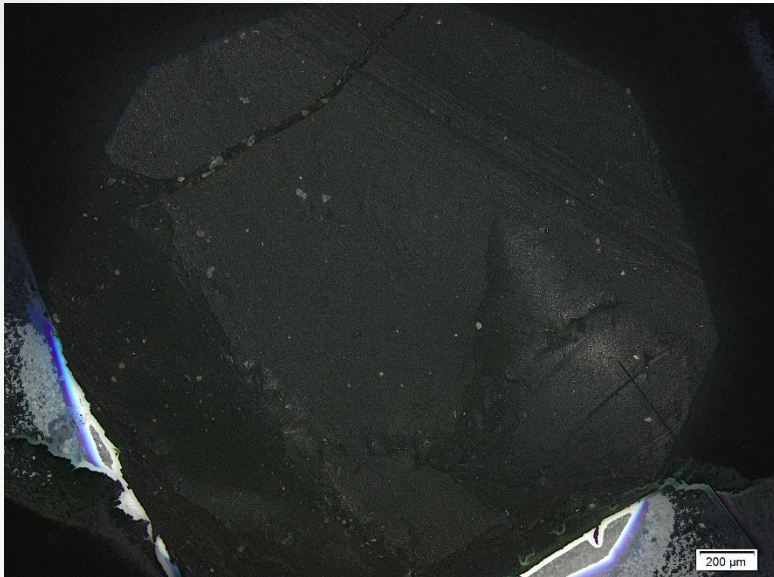


(Figure 4.6)

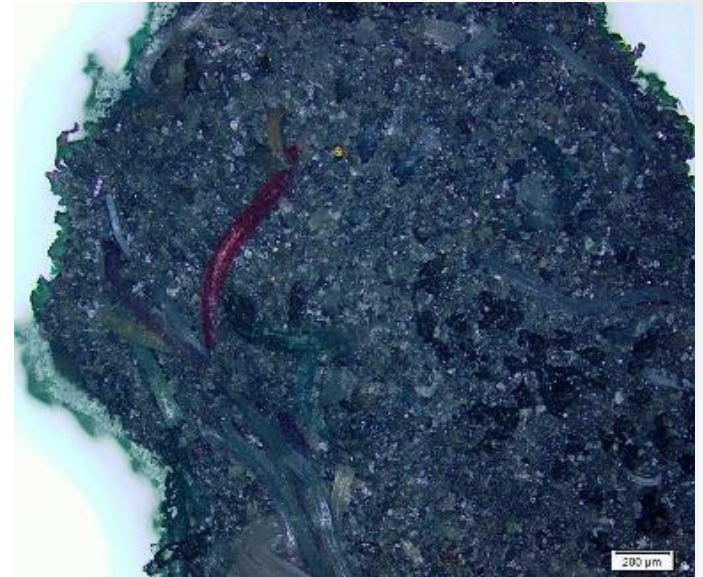
Green polyethylene terephthalate (**PET**) from drinking bottles:

1. Recycled very successfully into many products like more bottles, fibers and even carpet.
2. Green tint restricts fiber to six shades.
3. The green shade can match STF.

# GCRI vs. PCCB vs. PCCT



GCRI-4 (Figure 5.6)



PCCT-5 (Figure 5.50)



PCCB-1 (Figure 5.29)

# Study of PCCT

The face fibers are shaven for recycling, thus leaving a carcass of hardened backing. Reclamation for InterfaceFLOR grinds the carcass into PCCT crumb.



PCCT-1 (Figure 5.39)



PCCT-4 (Figure 5.40)

## Observations:

1. Figure 5.39 came from PCCT Lot 1 and represents the amorphous crumb form of five lots.
2. PCCT-4 in Figure 5.40 was the only pellet form.

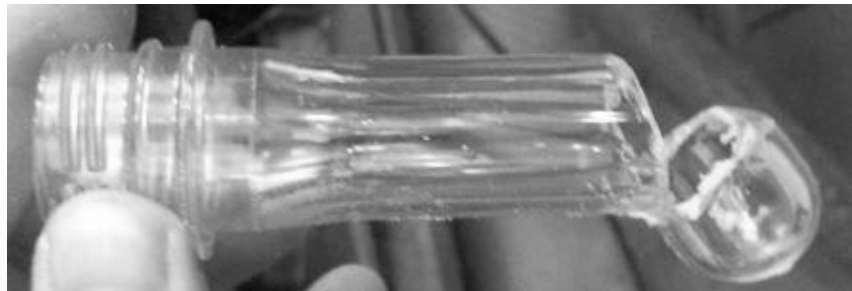
# Study of PET

The “green” PET stream reclaimed from soda bottles can only be over-dyed in fiber form to six acceptable shades for the commercial carpet market.



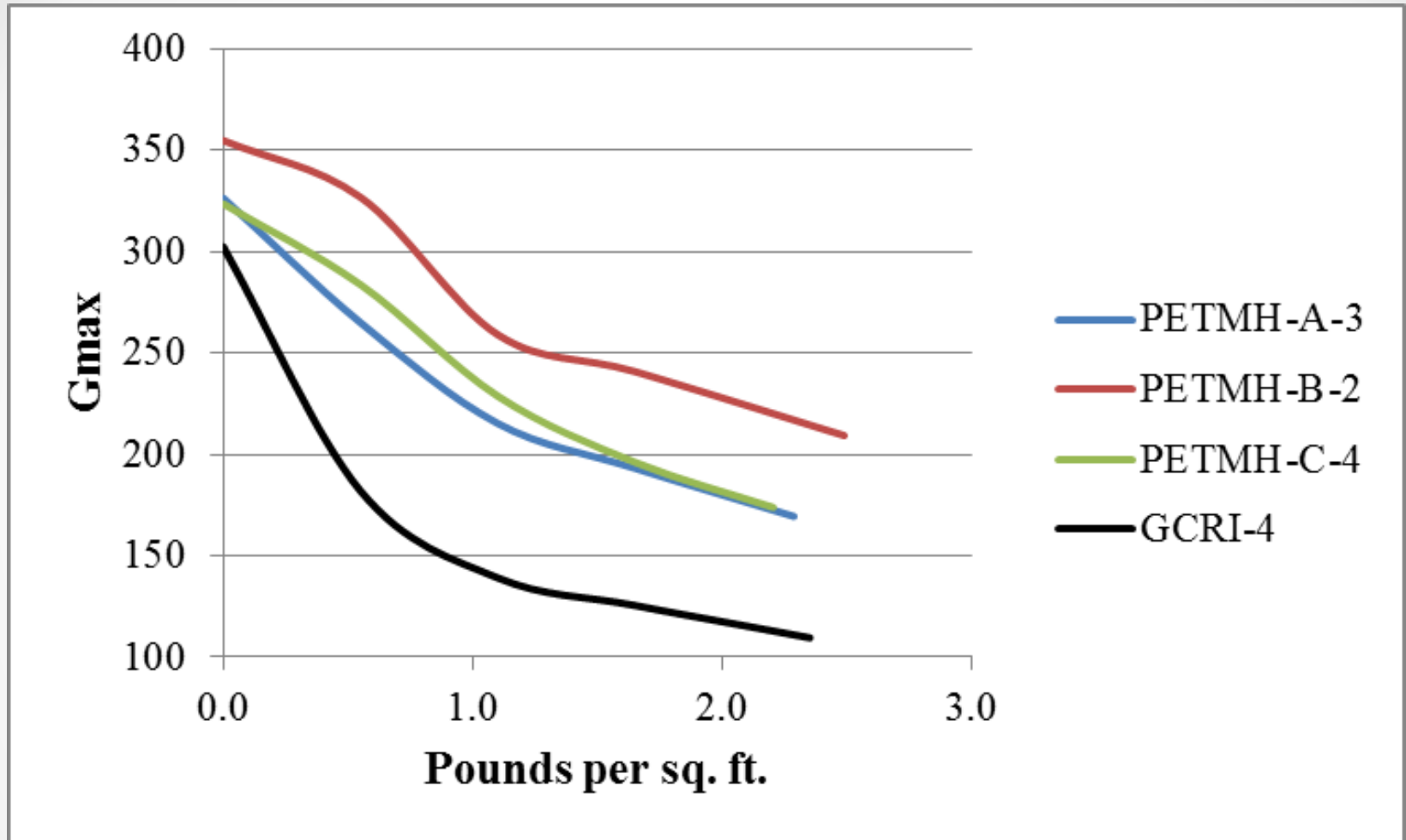
Green PET (Figure 5.61)

Soda bottles come from preform that is molded into a desired shape and size.



Clear Preform (Figure 4.7)

# PET Impact



(Figure 5.69)

Observations:

1. PETMH-A-3 had the largest average particle and the lowest Gmax at 169.
2. The g-vs-loading curves are closer to linear instead of non-linear like GCRI.

# Conclusions: PCCT

- The PCCT lots with the lowest particle size averages have the lowest bulk densities and achieves the lowest Gmax values.
- 4th generation synthetic turf improved impact performance of the PCCT-5 and PCCT-6.

# Conclusions: PCCB

For the infill alternate post-consumer carpet from broadloom (PCCB):

- In the turf, PCCB proves less effective than GCRI due to:
  - A high amount of powdered sodium carbonate filler content
  - The detached fibers prevented sufficient packing in the turf.
  - The combination of both factors led to insufficient impact at fatal Gmax and HIC levels, respectively.
- Refined PCCB-1 lot raised bulk density from **0.25 to 0.44 gm/cc**.
  - By removing the majority of small particles and loose fibers.
  - Improving impact absorption from **208 to 177 Gmax** values.

# Conclusions: PET

- With synthetic turf, the semi-crystalline PET particles could not match GCRI Gmax values.
  - Particle size distributions and bulk densities mirrored GCRI.
- Since the physical form of the PET particles matched GCRI, the polymer properties became a major factor.
- PET is more rigid than the tire rubber and cannot dissipate as much impact energy.
  - Glass transition of PET is above the impact testing temperature.
- Particle size and bulk density were significant factors: