TO THE COURSE:

TECHNOLOGY OF TEXTILE PREPARATION AND FINISHING

(TXL-241)

About the course……

3 credits (3-0-0); Pre-requisites: TXL110 OR TXL111 OR PHL110 OR MAL110 OR TXN101 OR TXL111L AND TXL111P

A. Preparatory Processing

Natural and added impurities in textiles, singeing, desizing, scouring, bleaching, mercerization and optical whitening of cotton. Combined preparatory processes, carbonization, scouring and bleaching of wool, degumming of silk. Heat setting. Machinery for preparation of textiles. Surfactants and their application.

B. Finishing

Introduction to chemical and mechanical finishes. Chemical finishes for hand modification. Bio-polishing, easy care, oil, water and soil repellent finishes. Fire retardancy, antimicrobial finishes. Finishes for wool. Mechanical finishes like shrink proofing and calendering; Raising, sueding and emerising. Low liquor application techniques and machinery; Stenters and dryers.

Instructor……

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Instructor……

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Introduction to Preparatory Processes

Reference Book

Books: Part A

- Chemical Technology in the pre-treatment process of textiles, S. R. Karmakar, 1999, Elsevier Science

- Textile Scouring & Bleaching by E. R. Trotman, B.I. Publications, New Delhi

- Handbook of Fibre Science and Technology- Volume I: Chemical properties of fibers and fabrics fundamentals and preparation Part-A and B. Ed. Mena Chem Lewin and Stephen B-Sello. Marcel Dekker Inc. New York

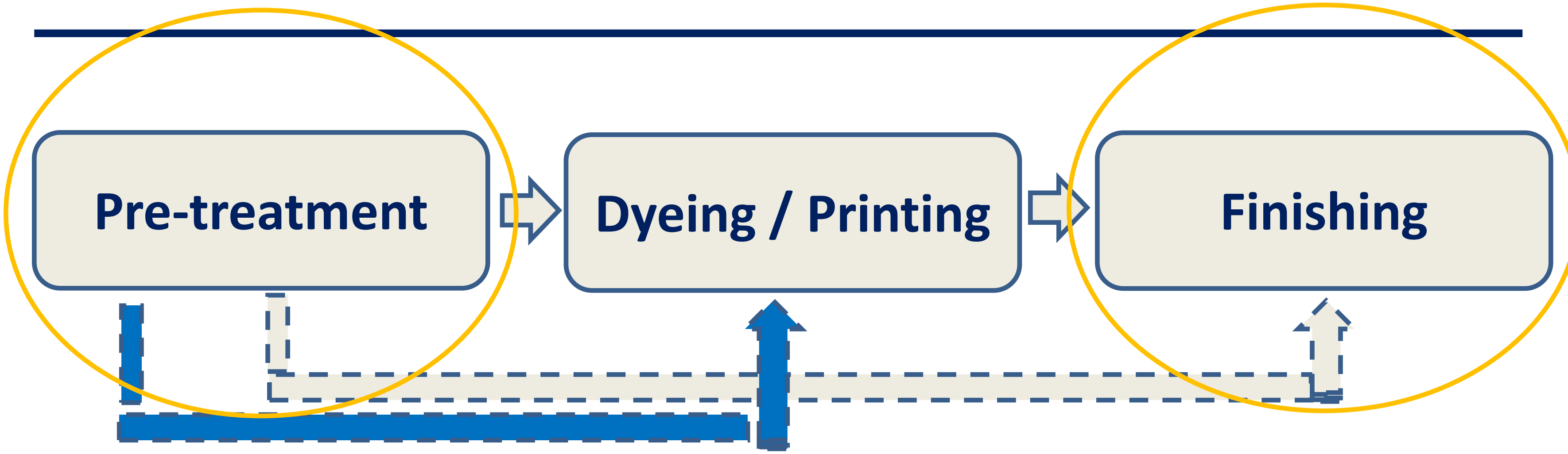
Major Textile Fibres

Natural:

- Cotton
- Wool
- Silk

Synthetic / Man made:

- Polyester
- Nylon
- Acrylic
- Viscose

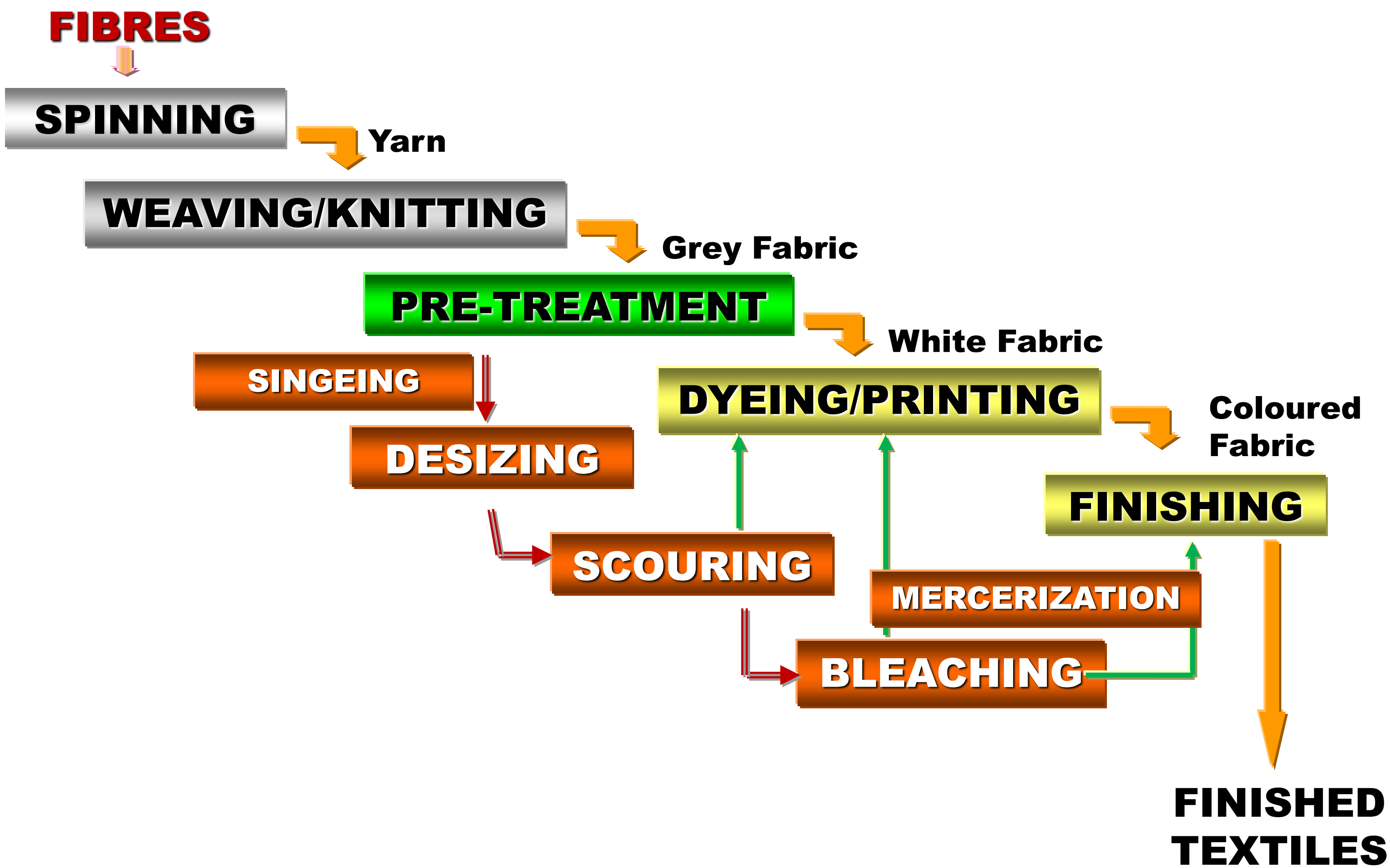


Final step of fabric manufacturing processes

- **Improved comfort**
- **Improved downstream processing**

- Appearance (aesthetics)
- Special functionalities (case basis)

- **Appearance (aesthetics)**
- **Special functionalities**
- **Feel (touch)**


FIBRES
SPINNING
Yarn
WEAVING/KNITTING
Grey Fabric
PRE-TREATMENT
White Fabric
SINGEING
DESIZING
SCOURING
BLEACHING
MERCERIZATION
DYEING/PRINTING
Coloured Fabric
FINISHING
FINISHED TEXTILES

Define – Pretreatment

Textile pretreatment is a series of cleaning operations that removes impurities which might adversely affect downstream processes like dyeing, printing and finishing.

- Natural impurities
- Added impurities

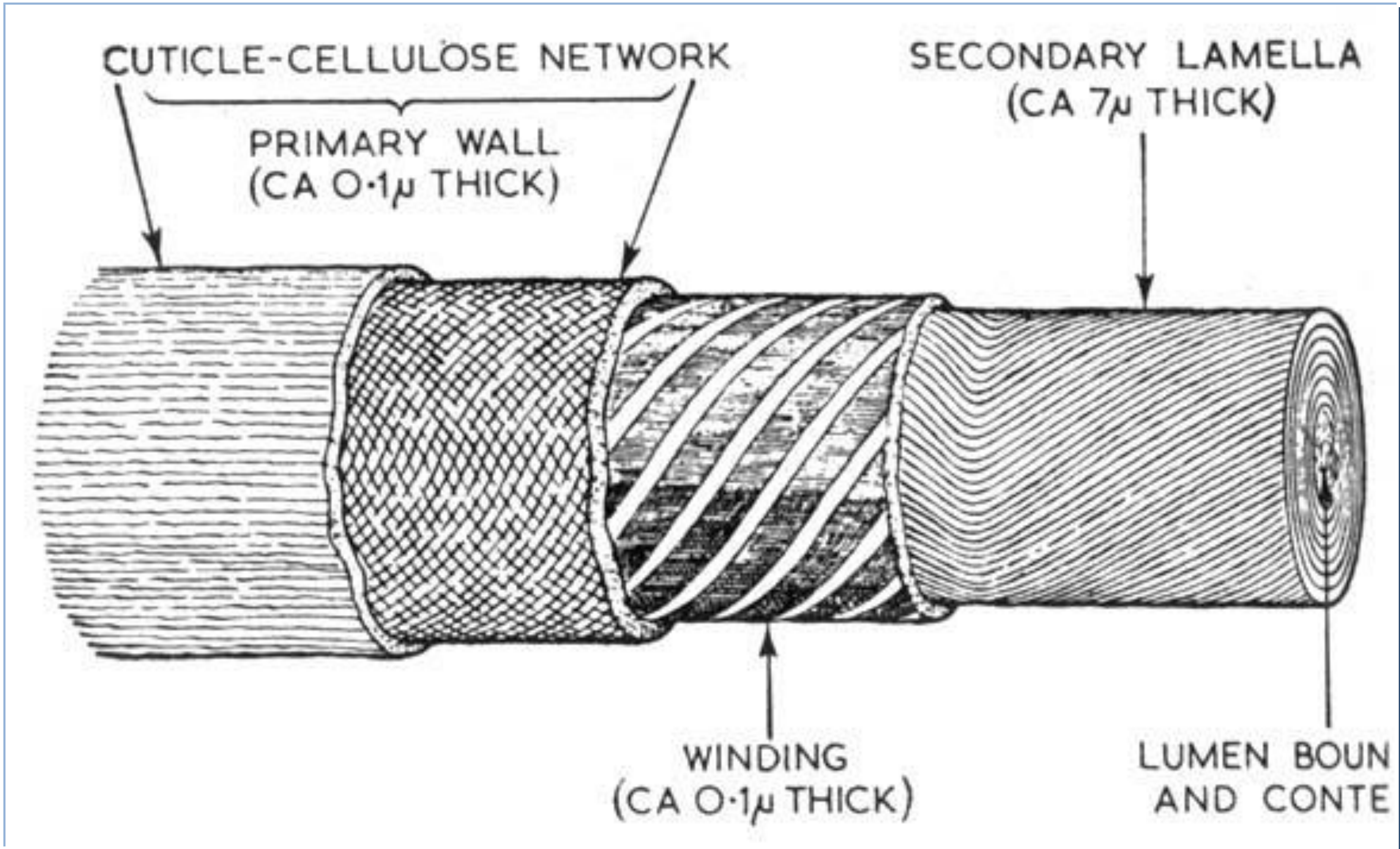
Pretreatment Objectives:

(Should be carried out with Minimum damage to the material)

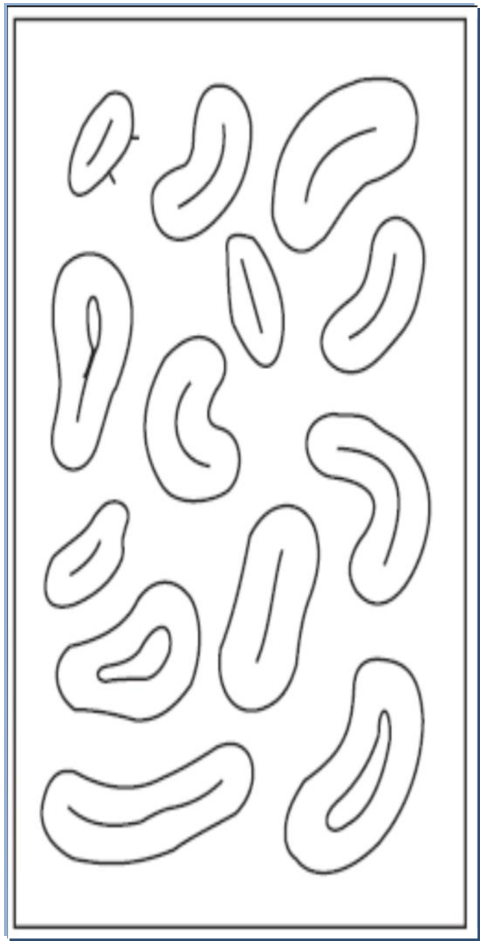
Improvement in absorbency

- Improved comfort
- Improved downstream processing (dyeing / finishing)

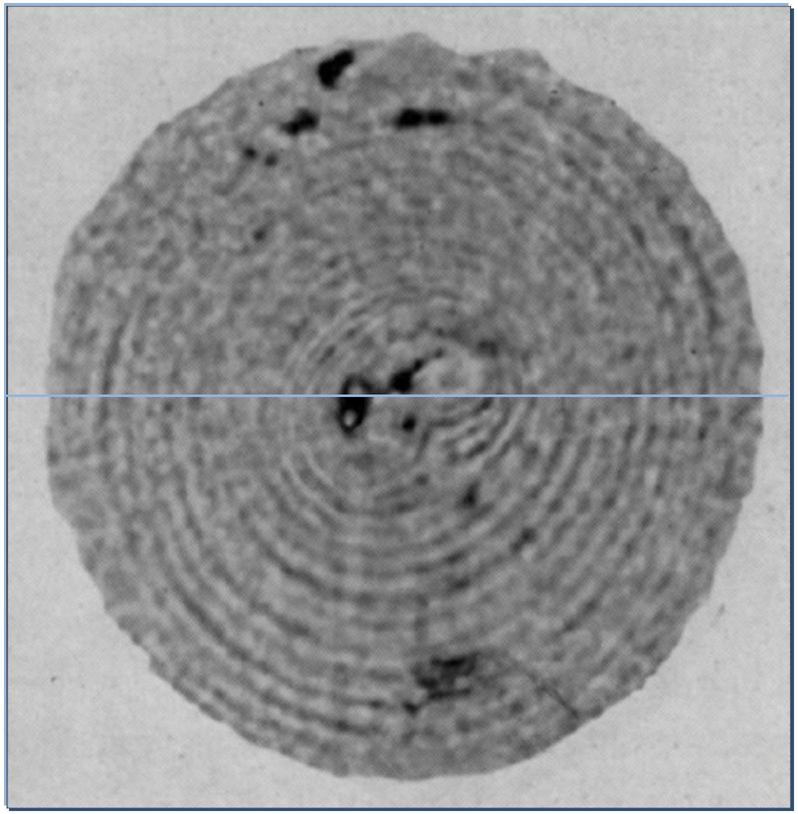
Layered components of a cotton fibre cell wall



Cotton fibres have a typical bean shaped x-section, convoluted, collapsed tube like structure

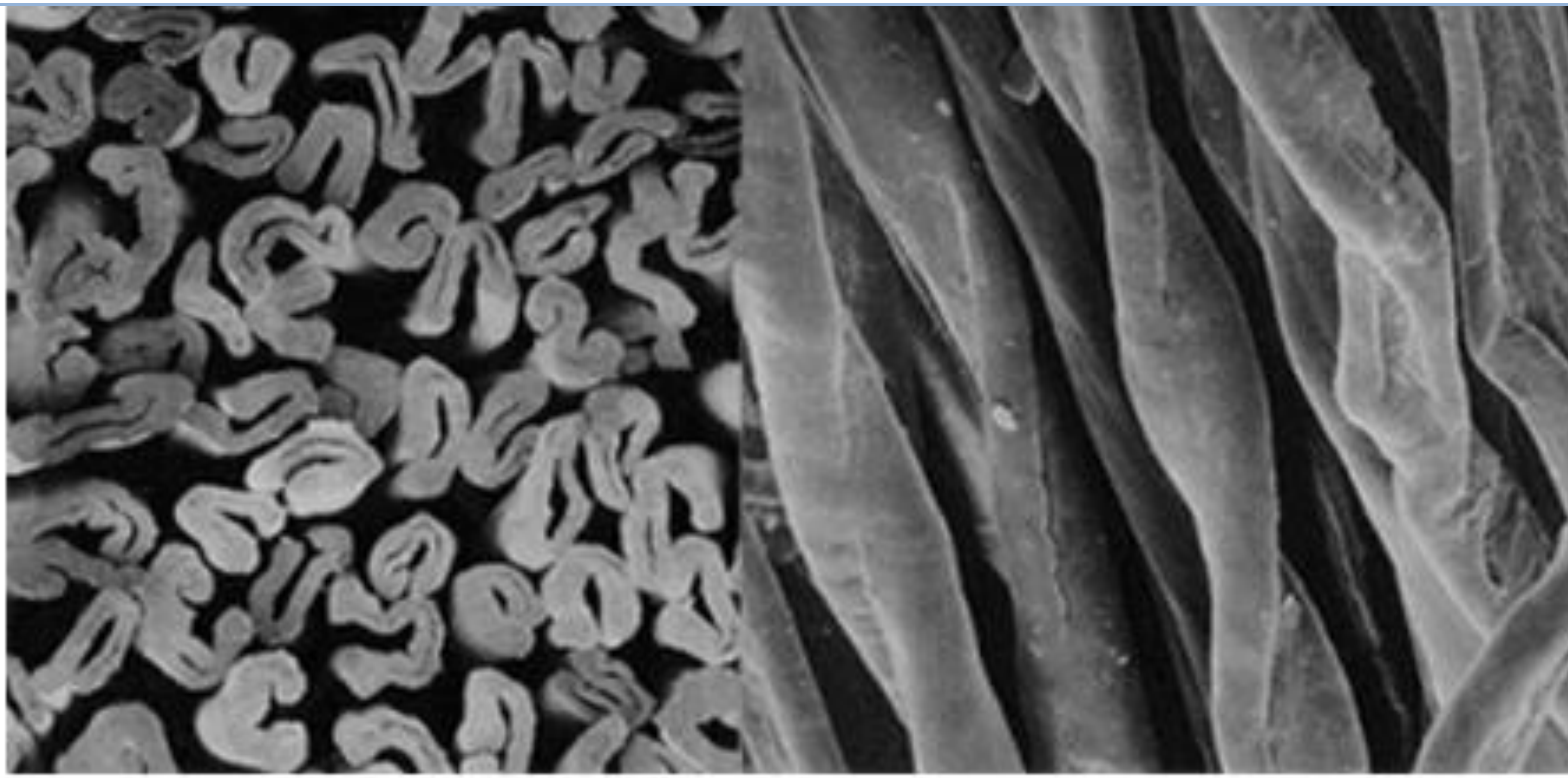


Typical cross-sections of matured cotton fibres

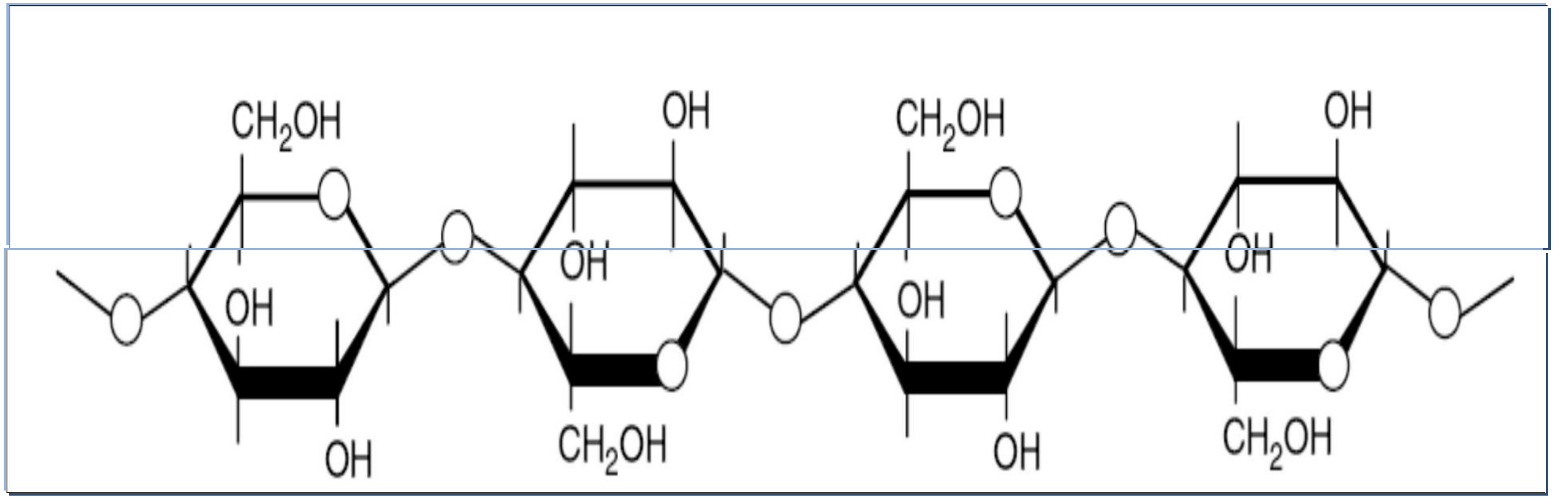


Cross-section of swollen cotton fibre showing daily growth rings

Microscopic view of cotton

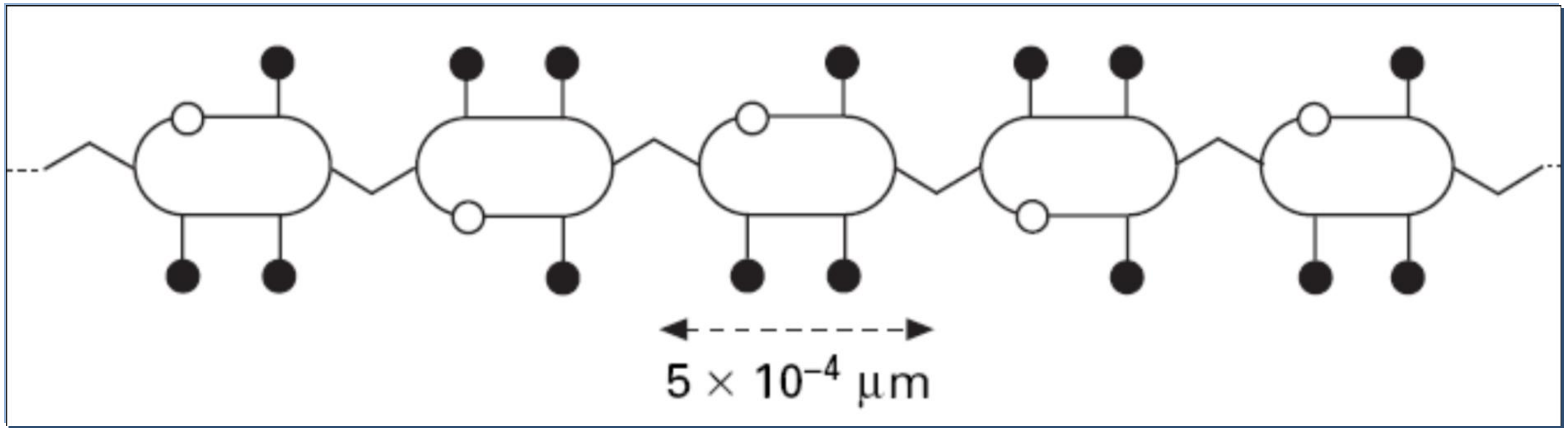


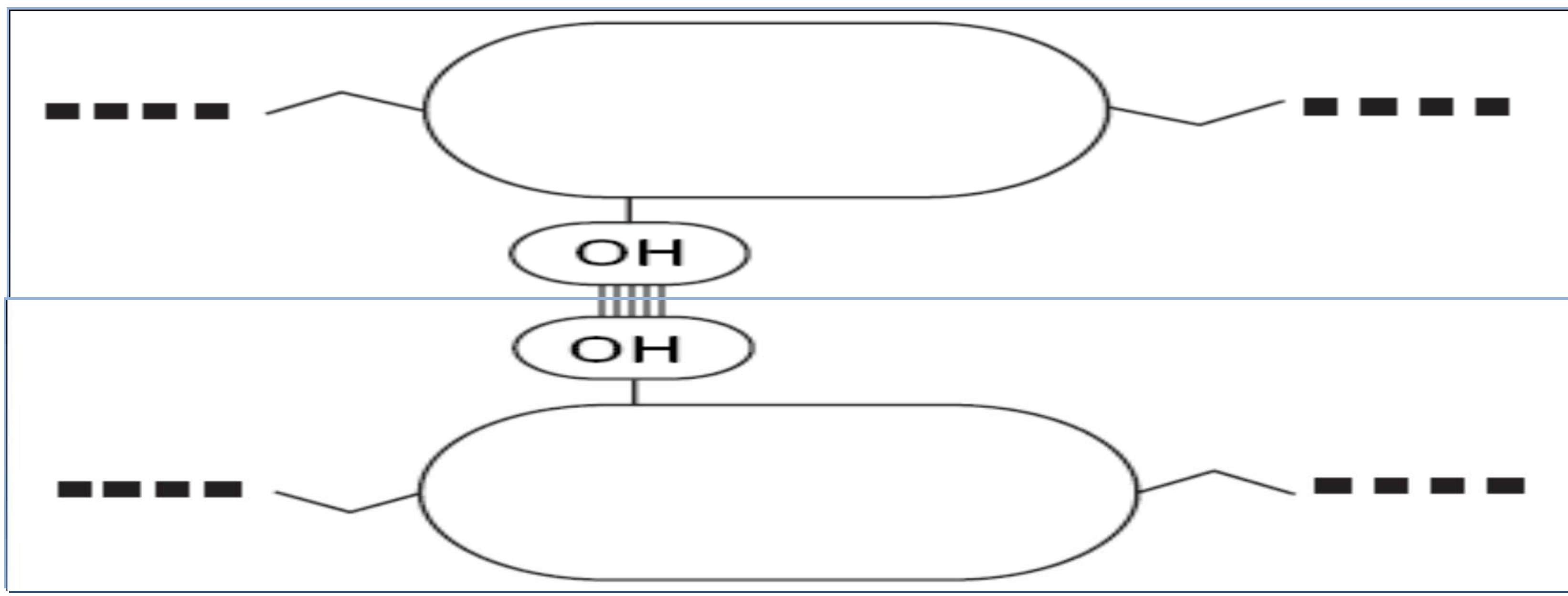
Chemical drawing of Cellulose



Polymer made up of a long chain of glucose molecules linked by C-1 to C-4 oxygen bridges with elimination of water (glycoside bonds). The anhydroglucose units are linked together as **beta-cellobiose**; therefore, anhydro-beta-cellobiose is the repeating unit of the polymer chain

Features of the cellulose molecule chain





Cotton Fibre

Constituents	Composition of a fibre		
	Typical (%)	Low (%)	High (%)
Cellulose	94	88	96
Protein	1.3	1.1	1.9
Pectic matter	0.9	0.7	1.2
Wax	0.6	0.4	1.0
Mineral matter	1.2	0.7	1.6
Malic, citric and other organic acids	0.8	0.5	1.0
Total sugars	0.3	-	-

Proteins

Nitrogenous compounds:

- Present in primary cell wall and lumen
- Yellow colour of cotton is due to presence of proteins and some colouring matter

Some amino acids:

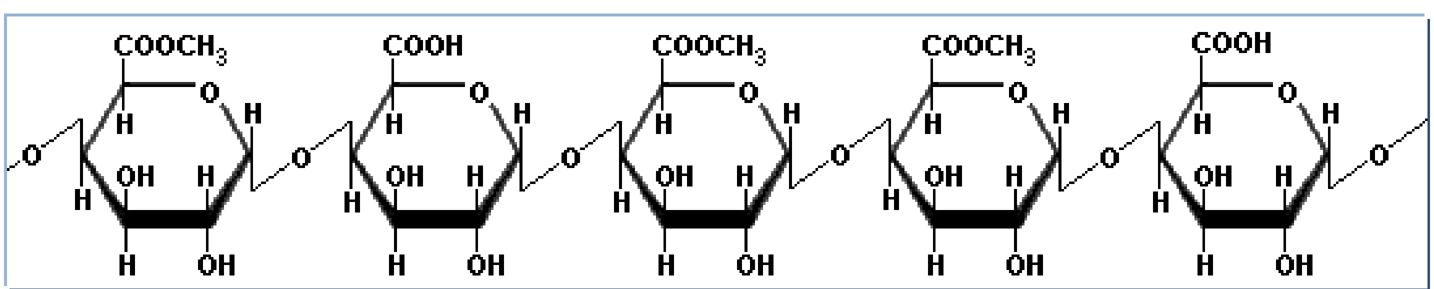
- Leucine
- Valine
- Proline
- Alanine

Pectins

- Derivatives of pectic acid
- Found in the cover of citrus fruits
- Polymer of high molecular weight

- Pectin is a <u>polysaccharide that acts as a cementing material in the cell walls of all plant tissues</u>. It is a polymer of <u>α-Galacturonic acid</u> with a variable number of methyl ester groups.

- Some COOH groups are present as Ca and Mg salts.



Components of Fats and Waxes

- The wax present in the primary cell wall of cotton protects the fibre from environmental agencies
- Responsible for the smooth handle and is a source of hydrophobicity
- In the presence of wax, cotton has poor wettability
- The wax consists of <u>long chains of fatty alcohols, fatty acids, their esters, cholesterin & hydrocarbons</u>

Fats and Waxes

Fats and waxes:

- Fatty acids
- Stearic acid
- Palmitic acid
- Oleic acid
- Fatty alcohols
- Cetyl alcohol ($C_{26}H_{53}OH$)
- Montanyl alcohol ($C_{28}H_{57}OH$)
- Gossipyl alcohol ($C_{30}H_{61}OH$)

Mineral Matters

- Depends on soil composition
- Determined by ash analysis

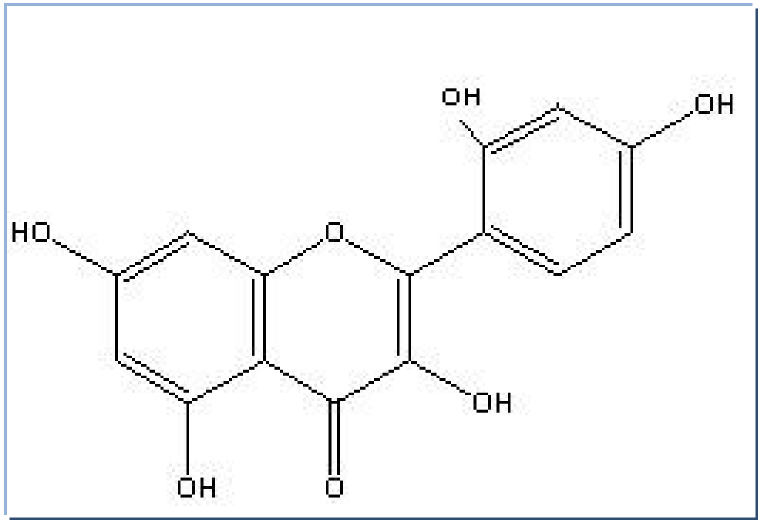
Potassium carbonate	44.8
Calcium carbonate	10.3
Potassium chloride	9.9
Potassium sulphate	9.3
Calcium sulphate	9.0
Magnesium sulphate	8.4
Aluminum oxide	5.0
Ferric oxide	3.0

Colouring Matter

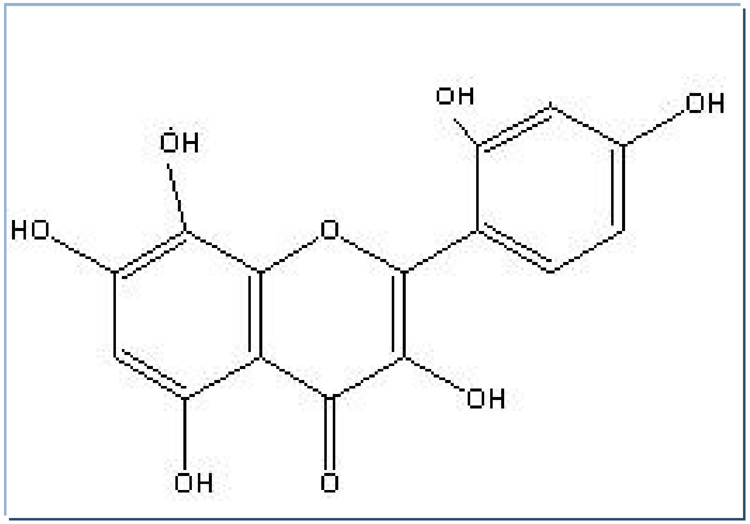
Colored pigments:

Flavones (flavus—Latin for yellow)

3,5,7,2',4' penta hydroxy flavone (Morrin)



3,5,8,2',4' Hexa hydroxy flavone (gossypetin)



Added / acquired impurities

- Mainly sizing matter (protective coating for warp yarns)

- Machine oils, lubricants, grease, etc.

- In knitting-coning oil

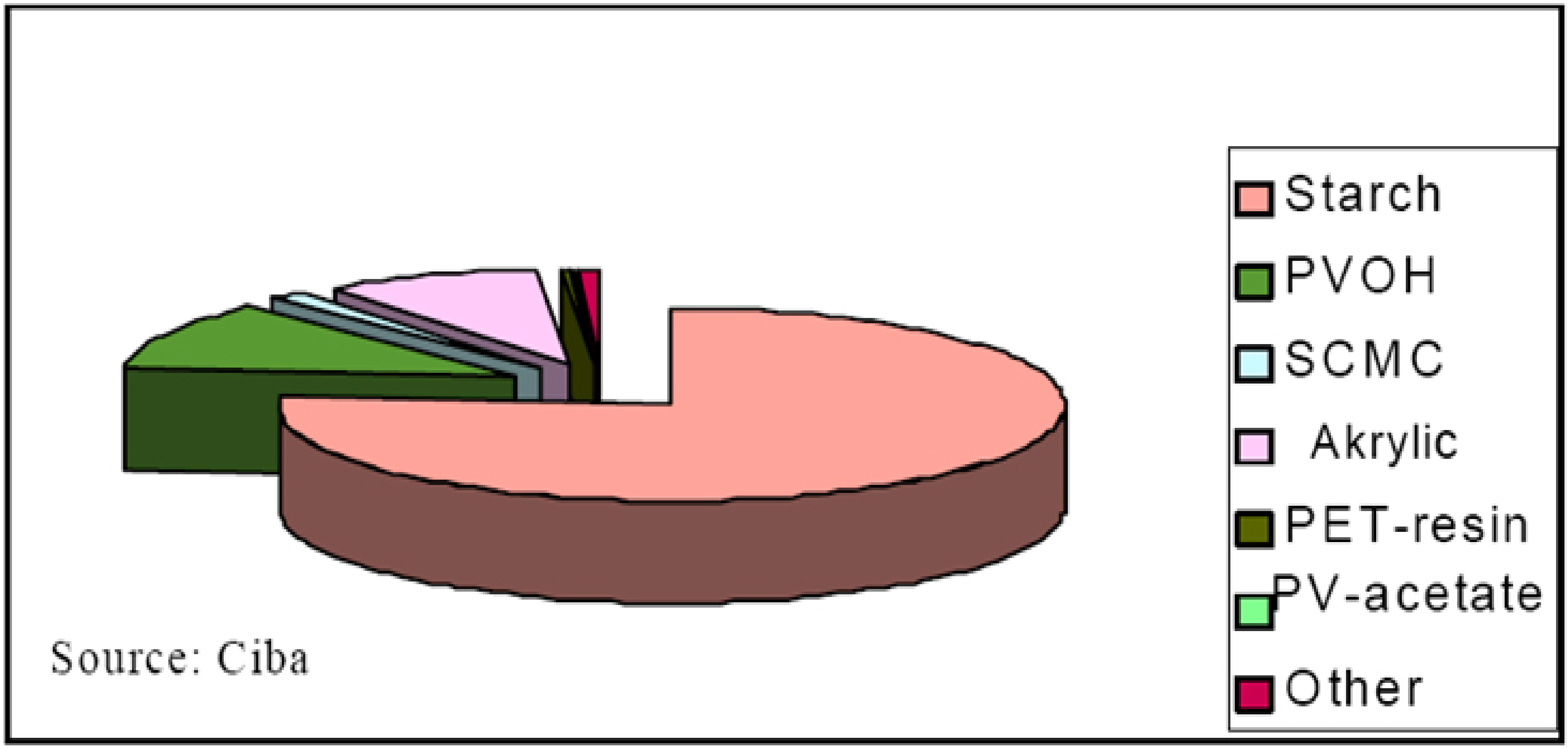
Chemistry of Size

Sizes are mainly formulations, with high molecular weight film forming polymers being the main components.

The size material applied on the warp yarns for facilitating weaving process.

- Natural / their derivatives
- Synthetic
- Blend of all these

Various sizing materials



Sizes based on Natural polymers/derivatives

Starch and its derivatives (75%)

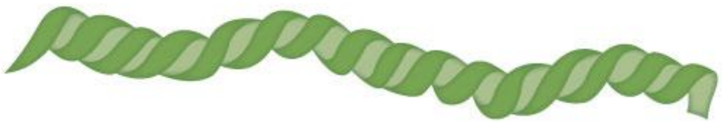
Starch has two components:

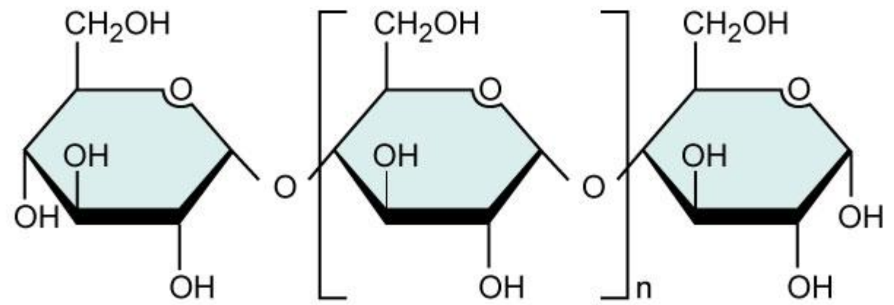
- ❑ Amylose: Relatively lower mol. wt. & water soluble (20%)
- ❑ Amylopectin: Higher mol. wt. and difficult to remove (80%)

In case of sizes based on natural polymers, add-on is of the order of ~ 15% and they generally require removal by chemical degradation. This results in high pollution of discharge.

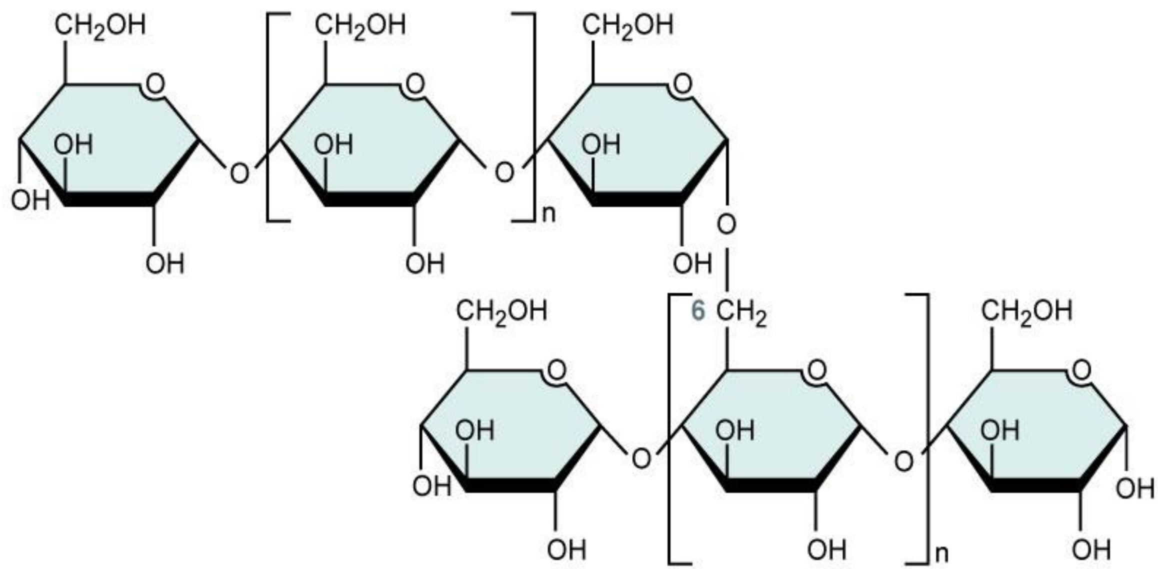
Amylose and Amylopectin

Amylose





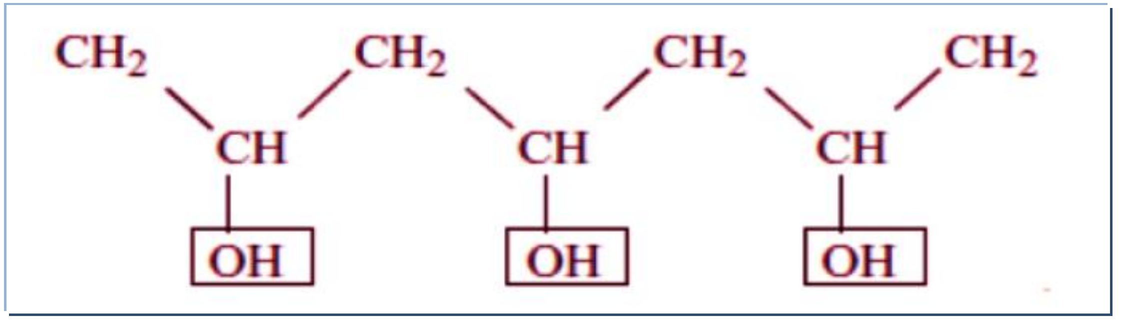
Amylopectin



Sizes based on Synthetic Polymers

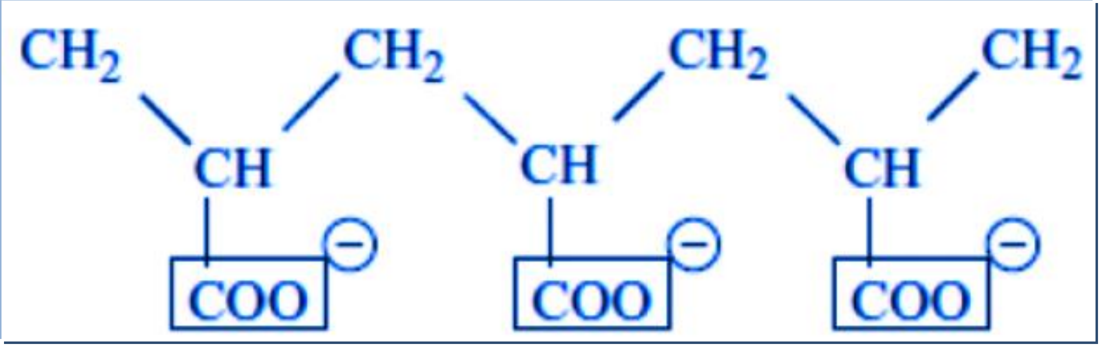
Polyvinylics:

(PVA)

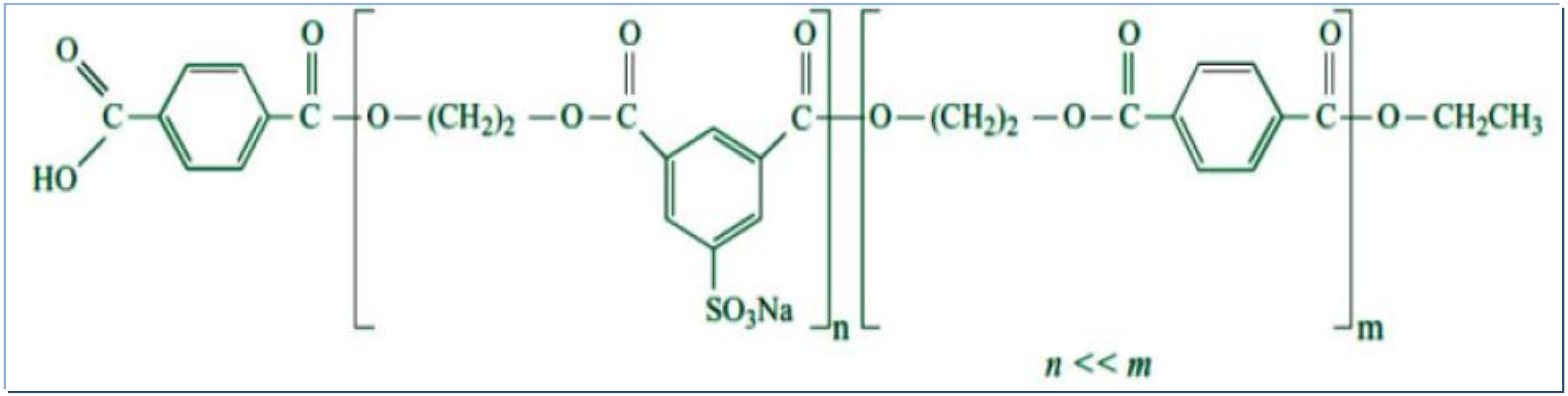


Polyacrylates:

(Polyacrylate)



Water dispersible Polyester



Mixed sizes:

These sizes are generally either water soluble or water dispersible. This results in lower pollution of discharge waters (recovery and recyclability)

Removal of impurities

- Solubilization
- Emulsification
- Chemical breakdown by
  - Hydrolysis
  - Oxidation

Important parameters:

- Temperature
- pH
- Time
- Circulation
- M:L ratio

Major Preparatory Processes

Singeing

↓

Desizing

↓

Scouring

↓

Bleaching *(optional)*

↓

OBA treatment *(optional)*

↓

Mercerization *(optional)*

IMPURITIES REMOVED DURING PRE-TREATMENT

Short Fibres → **Singeing**

Applied Impurities (Size Material) → **Desizing**

Natural Impurities (Oil, Wax, Pectins, Proteins) → **Scouring**

Artificial Impurities (Oil, Stains, Dust, Dirt) → **Scouring**

Colour Pigments (Naturally present in cotton) → **Bleaching**

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Singeing

Protruding fibres on the surface of a fabric

- Create a fuzz which might obscure sharpness of a print or a colored strip on the garment
- Can attract soil
- May aggravate pilling

Singeing: Objective

Many cotton materials are valued for their smooth appearance, e.g. lustrous sateen and satin weaves.

Hence, it is desirable to remove these surface fibres.

One of the most common methods to do this is just **burn off these fibres! (Singeing)**

Now a days, it is also done using **enzymes for cellulosic** fibre fabrics.

Fabric appearance after Singeing

Singeing Process:

Singeing usually involves passing/exposing one or both sides of a fabric over a gas flame to burn off the protruding fibers

- Other methods of singeing include infra-red singeing and heat singeing for thermoplastic fibers

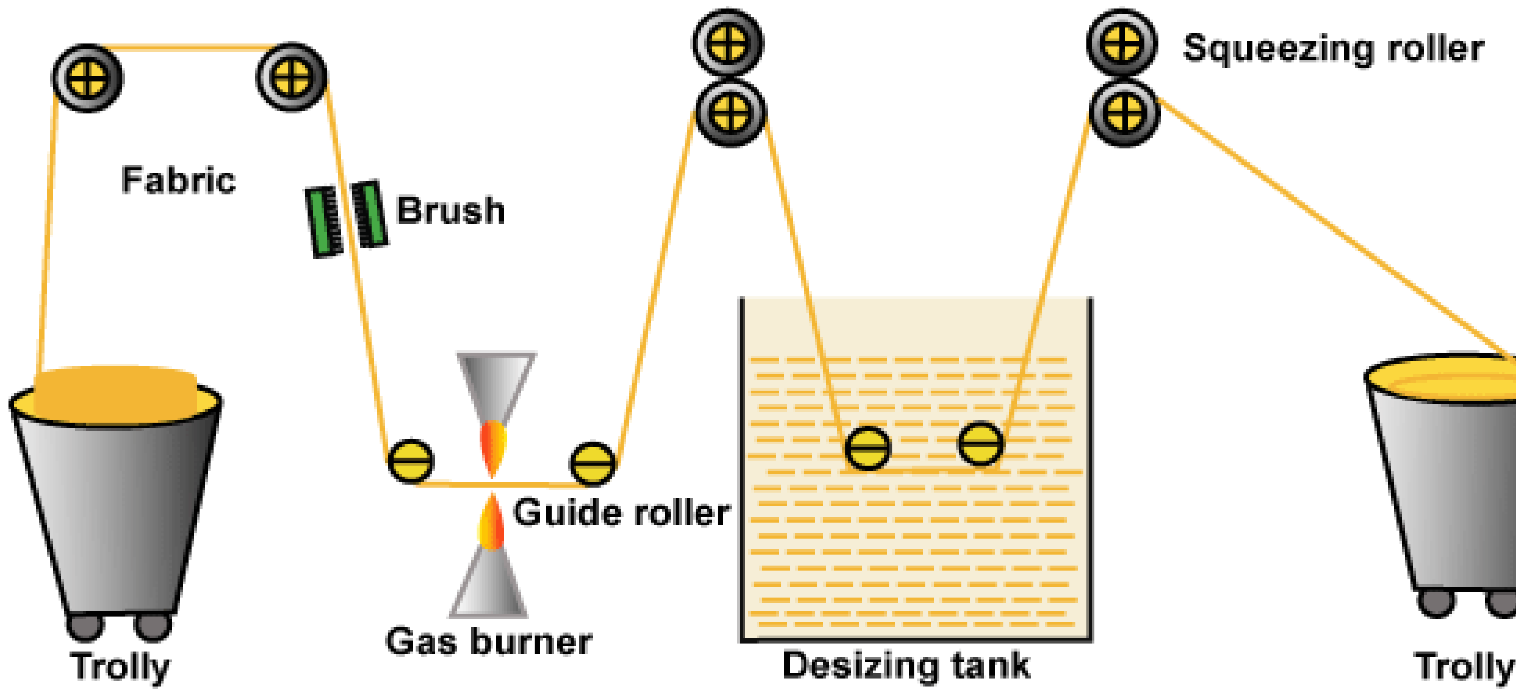
- Singeing of yarns is called “gassing”

- Cellulosic fibers such as cotton are easily singed because the protruding fibers burn to a light trace ash which is easily removed

- Thermoplastic fibres are harder to singe because they melt and form hard residues on the fabric surface

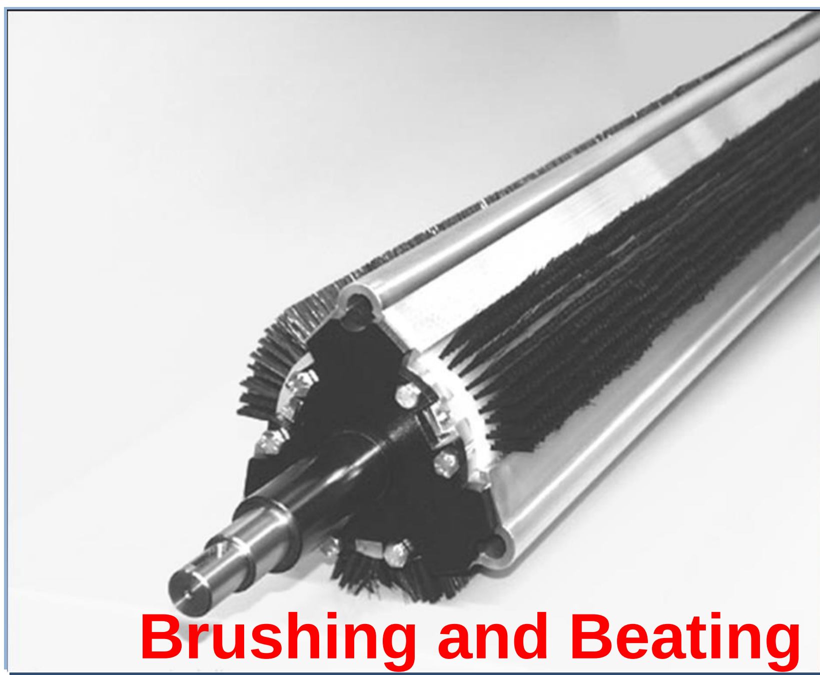
A gas burner used for singeing of textile materials

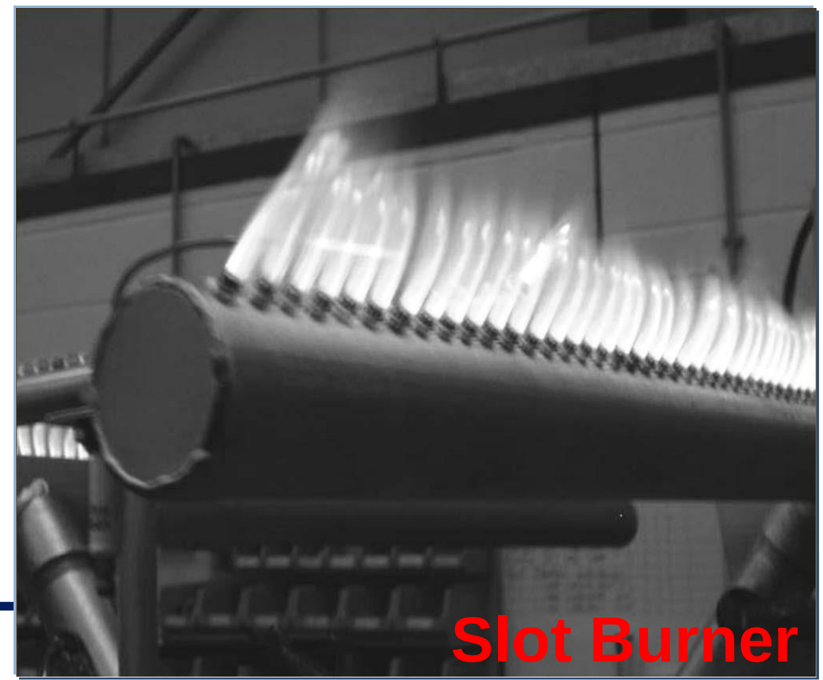
Typical singeing arrangement




Brushing


Beating


Brushing and Beating


Slot Burner

SINGEING PROCESS

COTTON & OTHER CELLULOSE FIBERS

- In Grey State (Economical) - As one dry step is avoided
- Slight yellowing occurs - Removed on bleaching
- Singed with maximum burner intensity

POLYTESTER

Melts at 260-280 ^oC, but burns at 500 ^oC.

- Normal flame forms fused polymer at fabric surface
- So, powerful flame for quick burning

Singeing Parameters

- **Machine Speed**
- **Flame Intensity**
- **Burner Position**
- **Flame Distance from Fabric Surface**

SINGEING SYSTEMS

SYSTEMS

DIRECT

- ✓ Hot Plate
- ✓ Rotary Cylinder
- ✓ Gas

INDIRECT

Radiation Singeing

- ✓ No Flame Contact
- ✓ Uniform Singeing

PLATE SINGEING

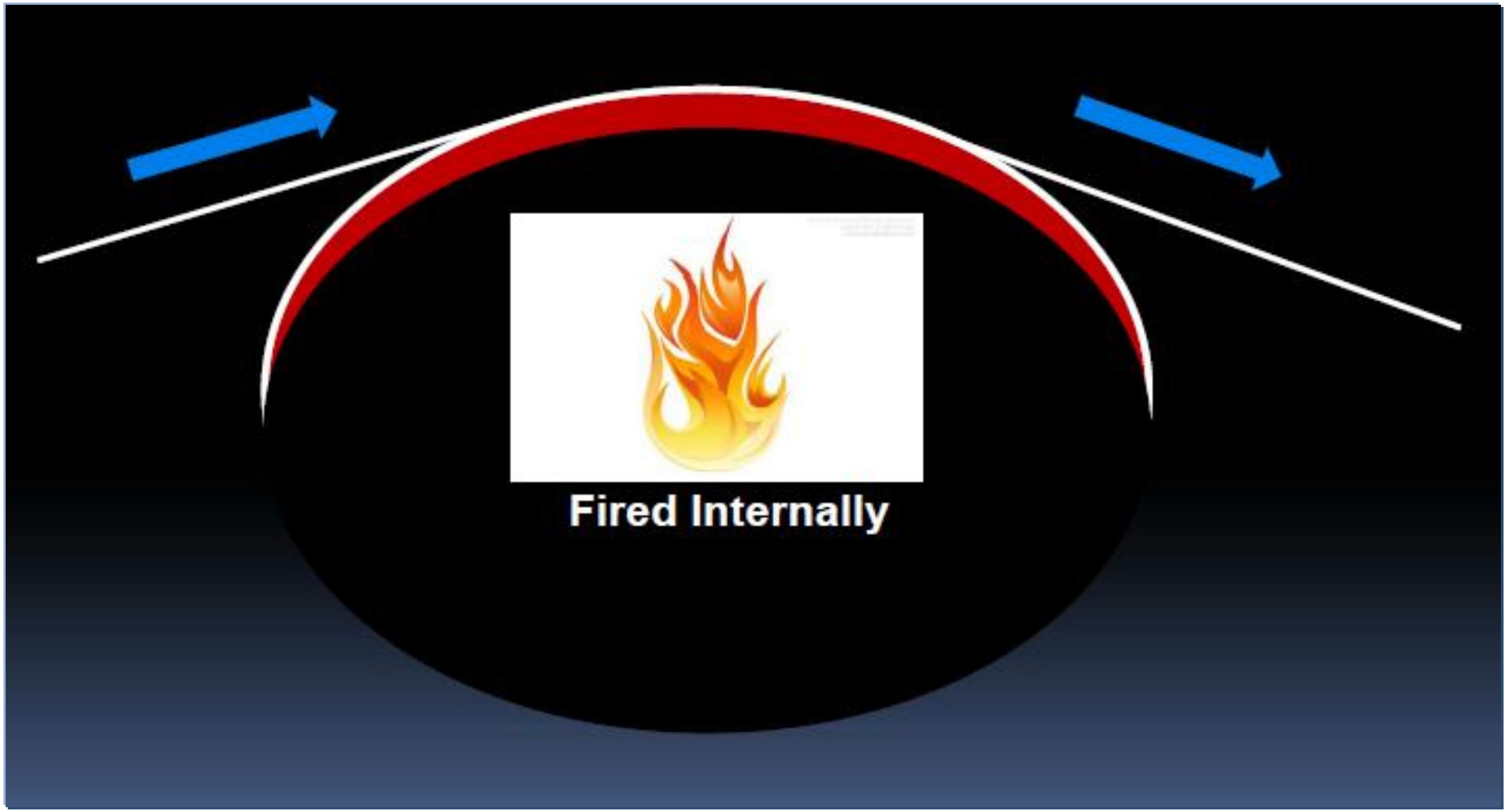
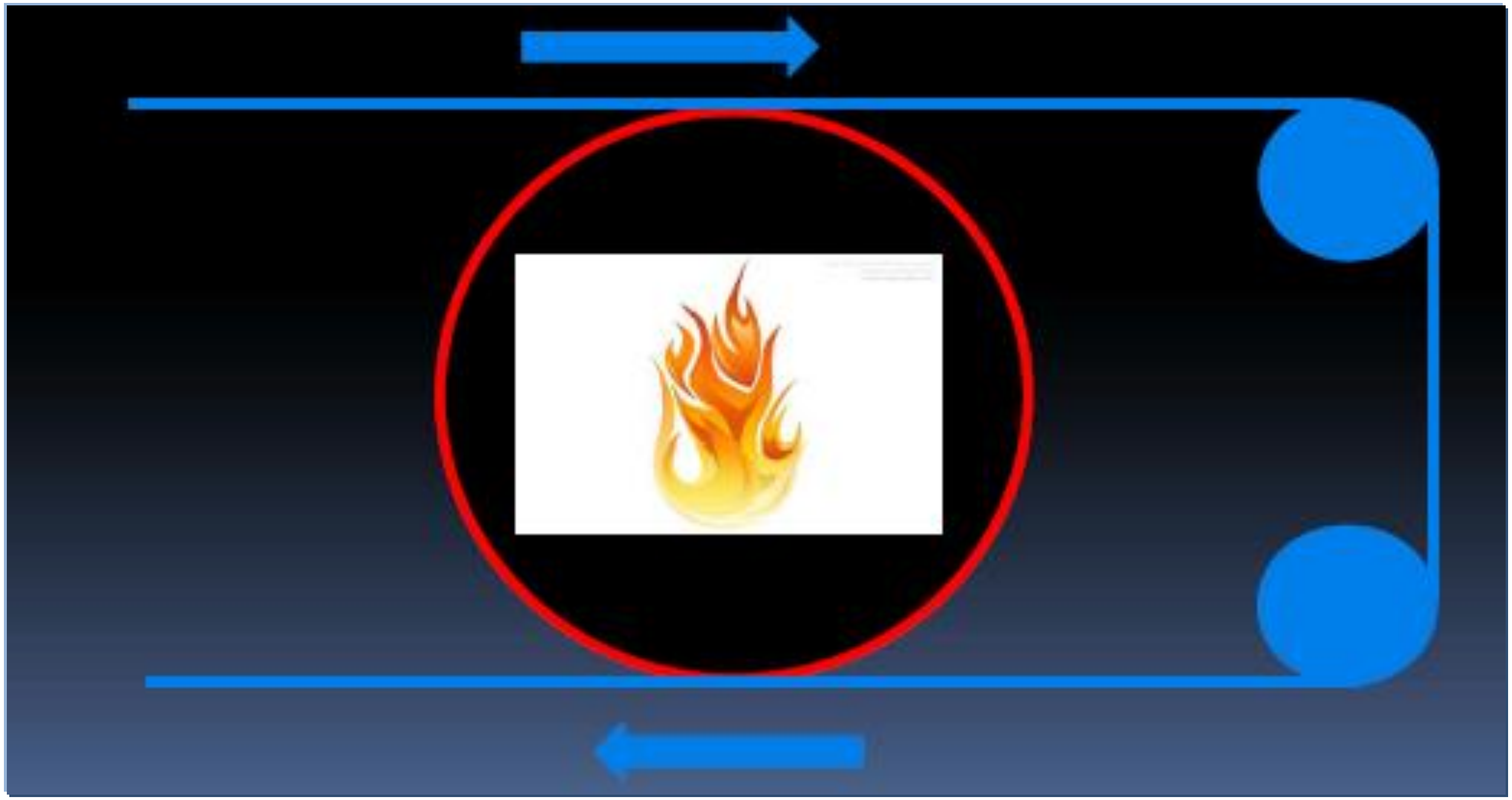


PLATE SINGEING

- Curved copper plate of 1-2 inch thickness
- Heated to bright red from opposite side
- Heavy petroleum oil is burnt
- Cloth is dried and passed over the plates
- Speed up to 200 yard/min
-

Rotary cylinder



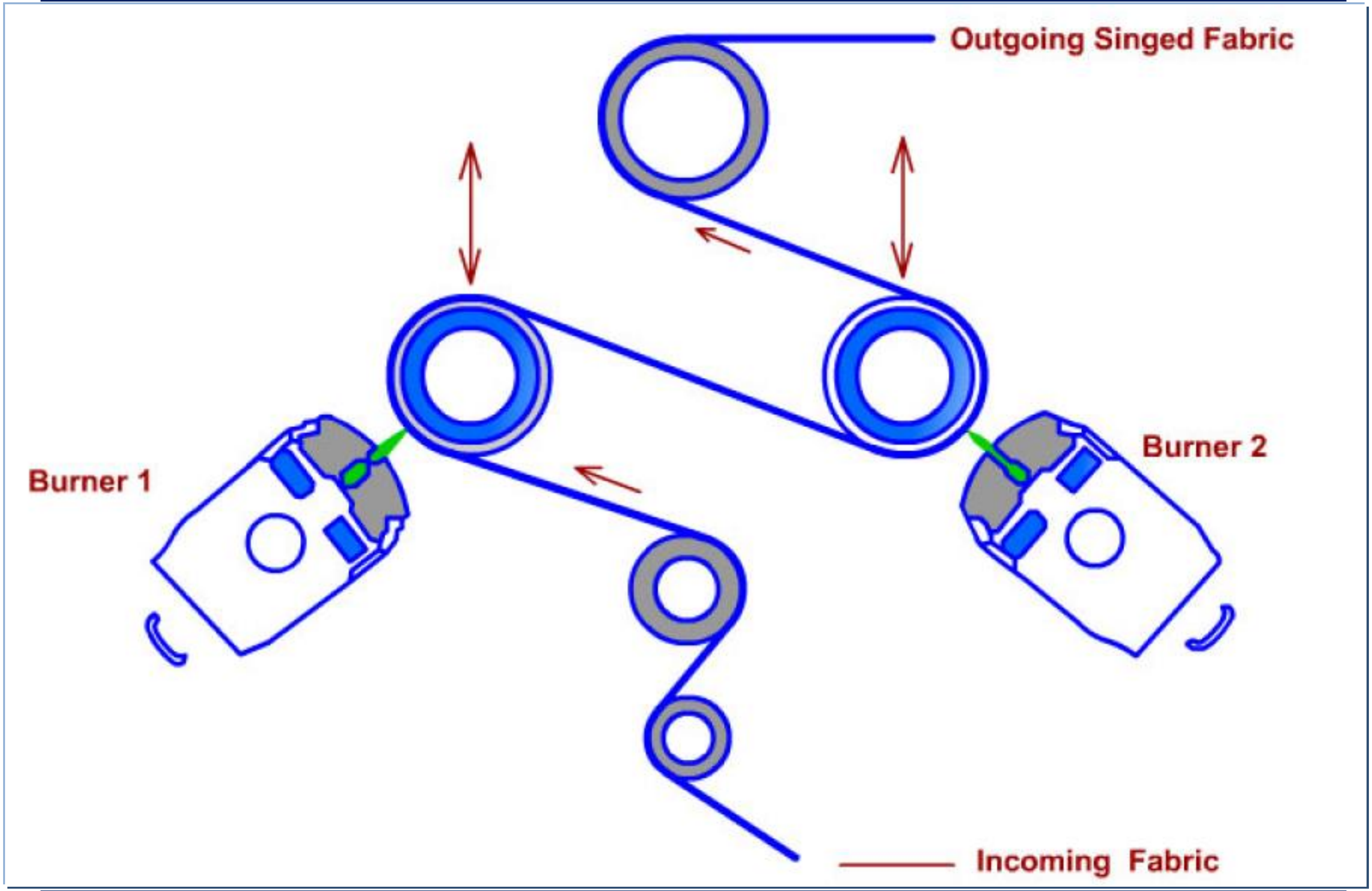
- Hollow cylinder rotates slowly in opposite direction
- Fired internally

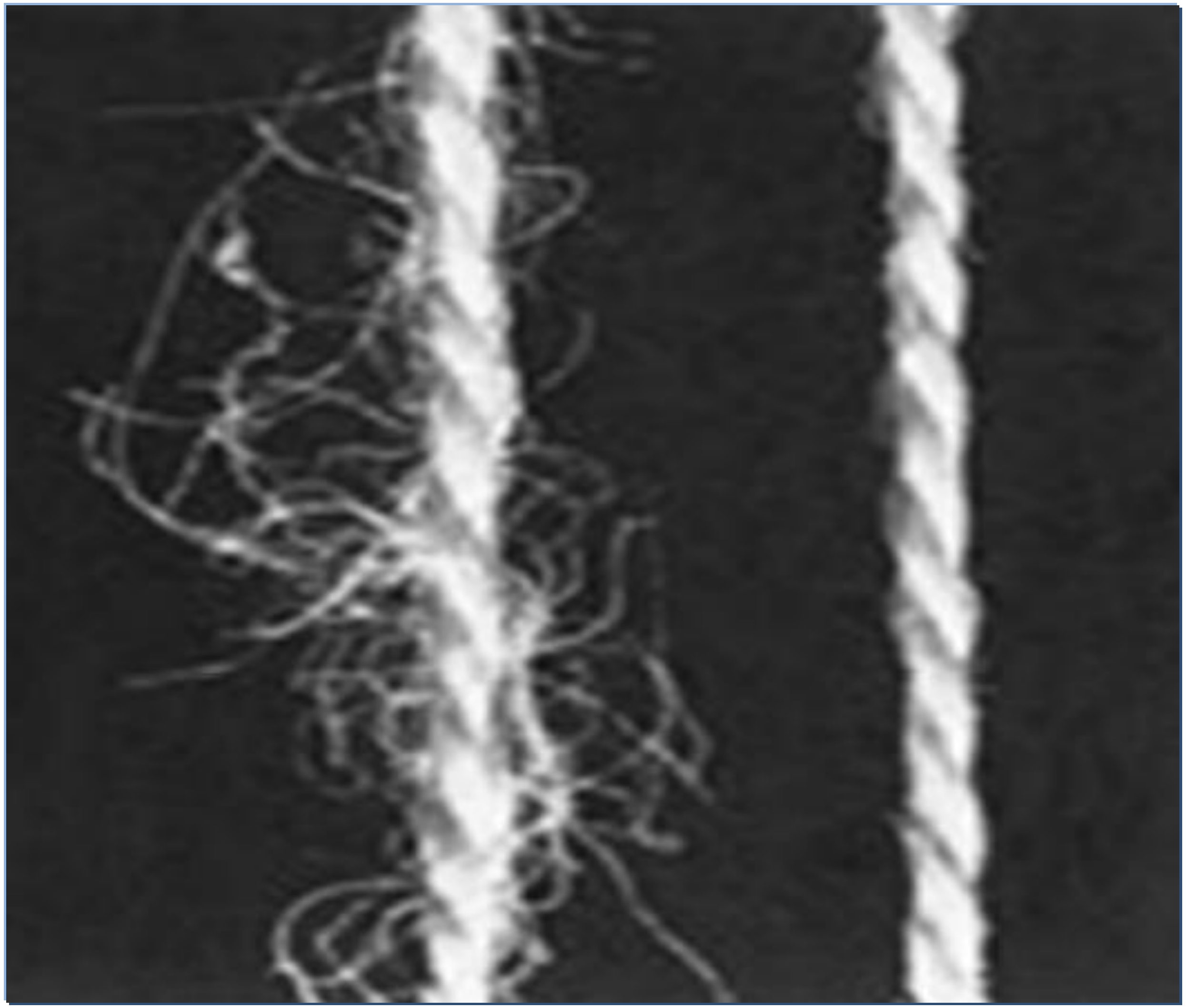
Gas Singeing

Gas singeing

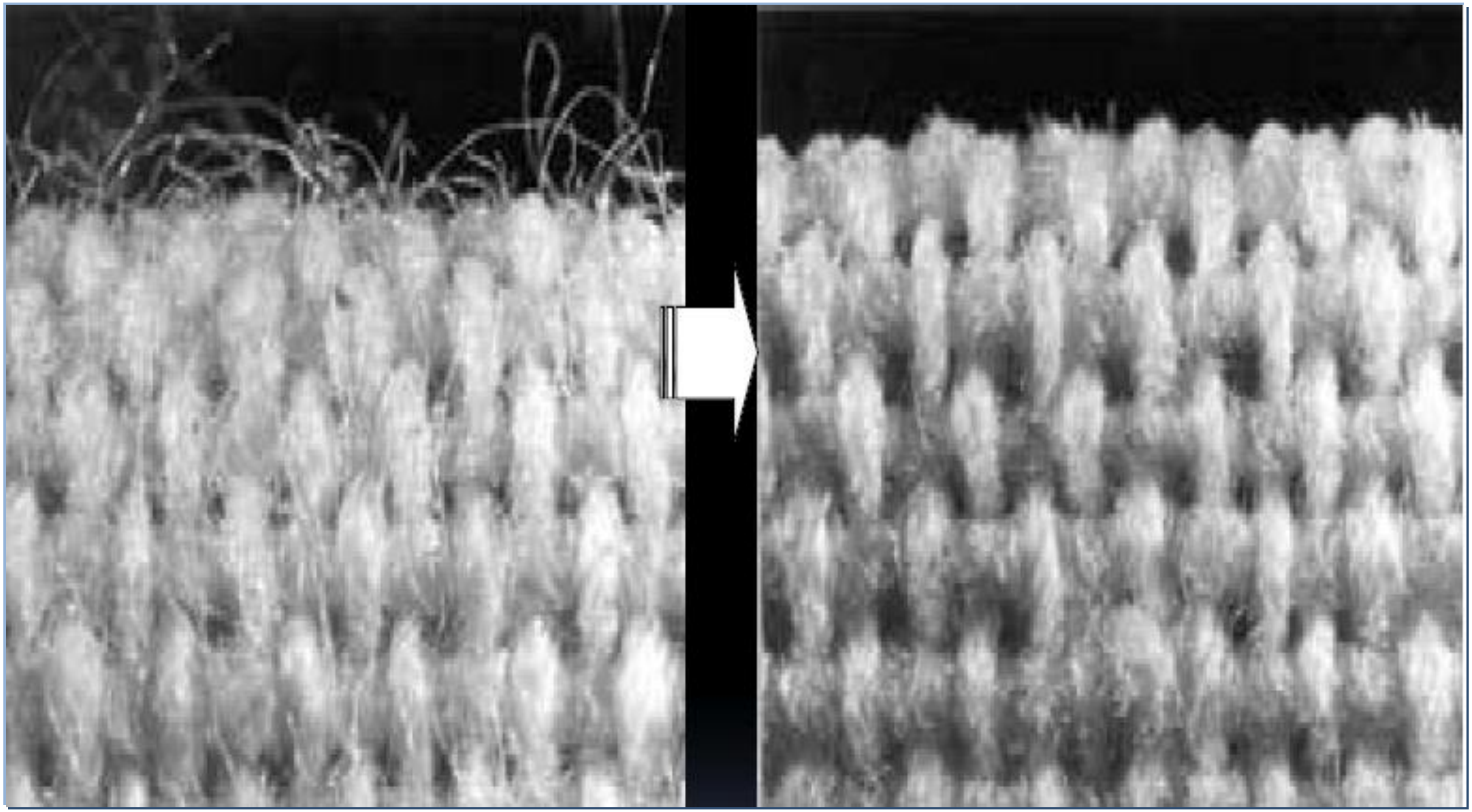
- Flame has high thermal and mechanical energy
- Can loosen the fibre ends
- Flame temp: 1300º Celsius (approx.)

Schematic of Gas Singeing

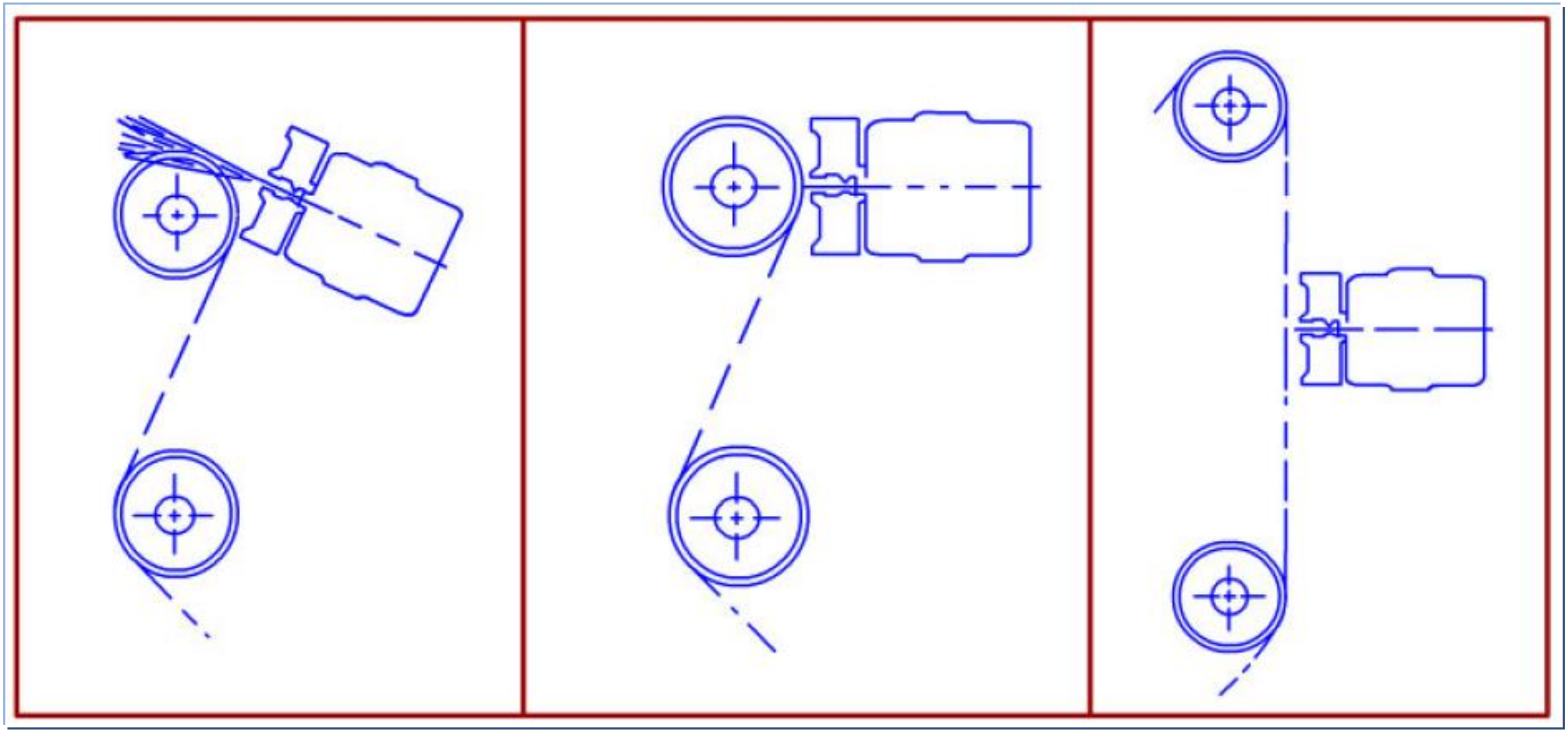




Fabric Appearance: Before & After Singeing



POSITION OF BURNER



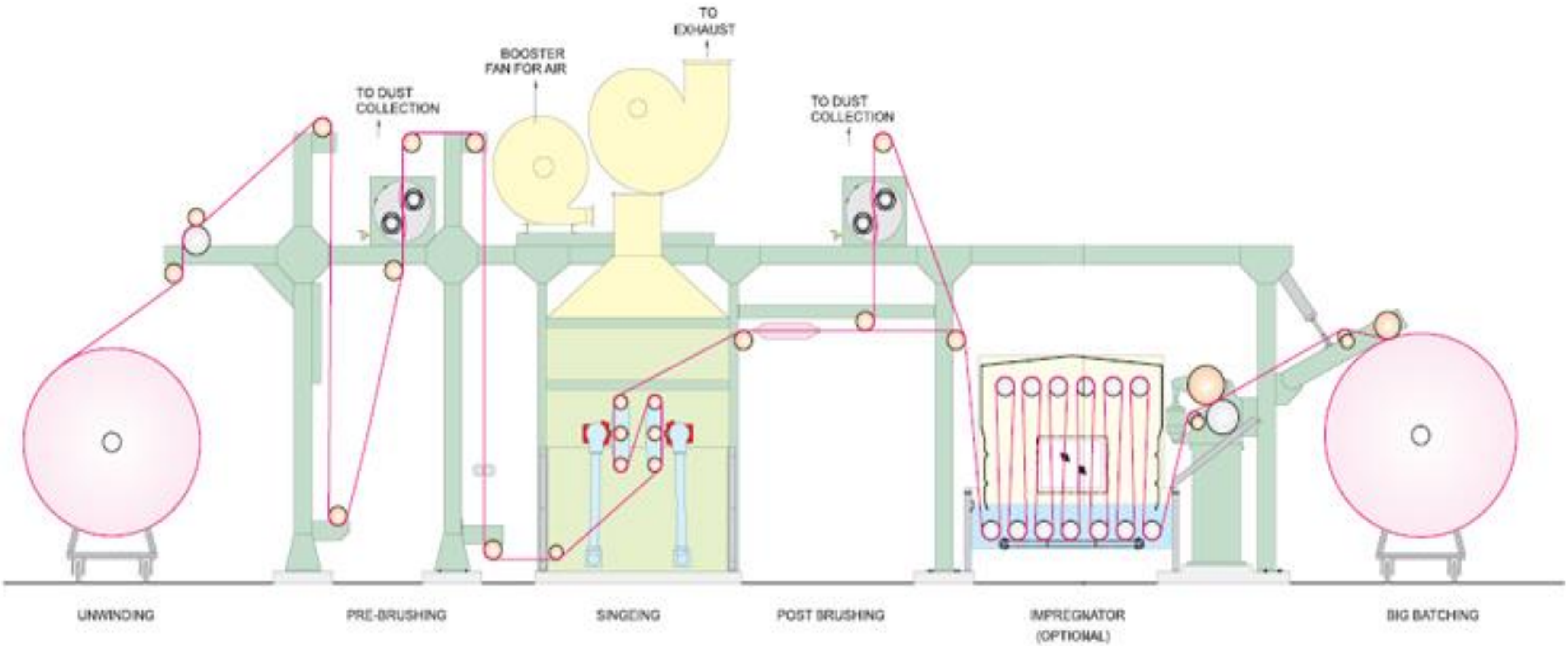
Tangential Singing	Singing onto a water cooled roller	Singing into the fabric

POSITION OF BURNER

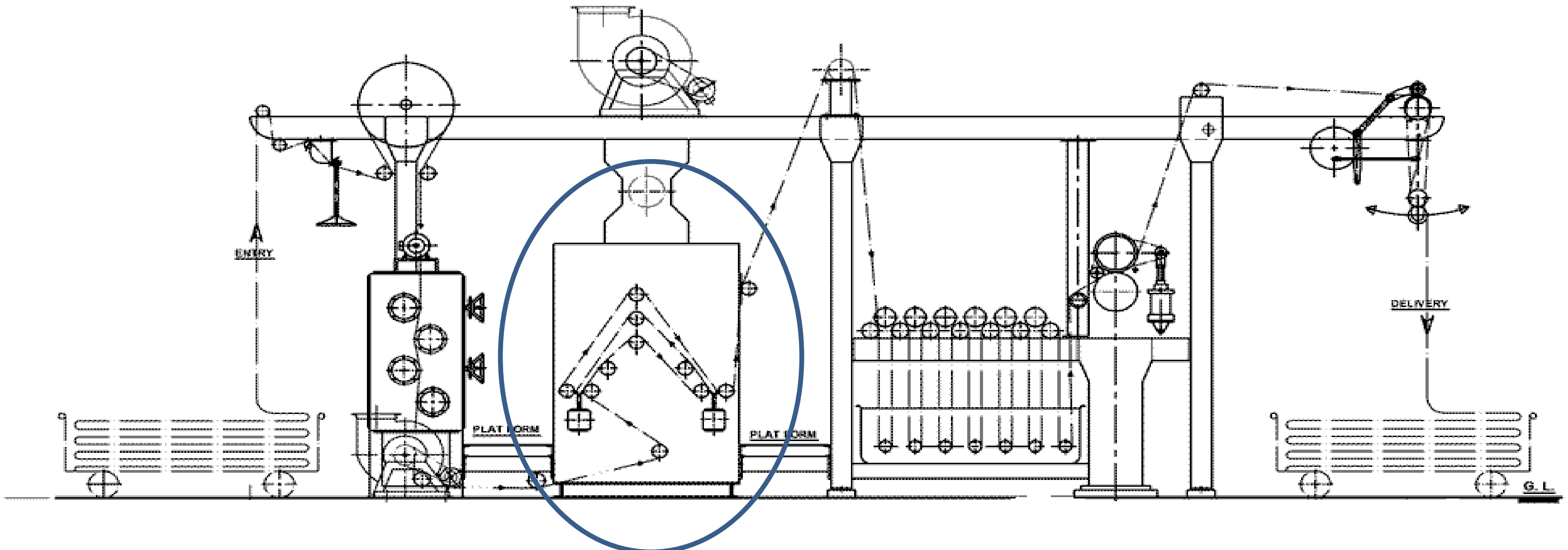
- Flame position tangential to fabric for the fabrics which cannot tolerate direct exposure to flame (delicate, light weight & heat sensitive fibre).

- Flame at right angle to fabric for temperature sensitive fibers, open weave structures, synthetic & blends with gsm < 125 g/m^2

- Flame at right angle to the fabric freely guided between two rollers- for natural fibres and blends with gsm > 125 g/m^2 (heavy technical fabric of blends)



TO EXHAUST
BOOSTER FAN FOR AIR
TO DUST COLLECTION
TO DUST COLLECTION
UNWINDING
PRE-BRUSHING
SINGEING
POST BRUSHING
IMPREGNATOR (OPTIONAL)
BIG BATCHING



GENERAL ASSEMBLY OF SINGEING M/C.
(FIX BURNER) WITH DESIZING TANK .

PROBLEMS

- ❑ Uneven singeing (dyeing streaks)

- ❑Thermal damage (due to high flame temperature)

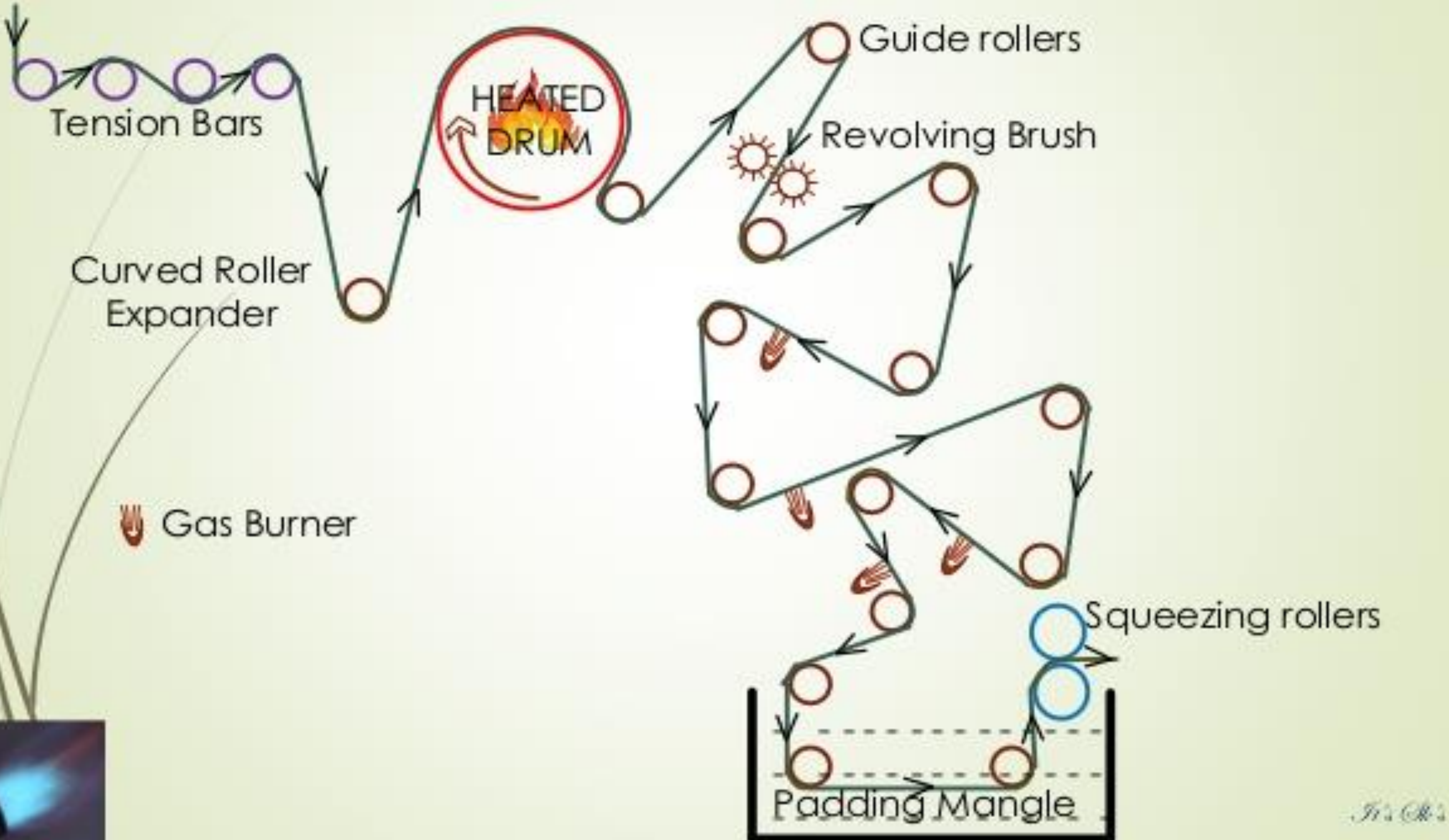
- ❑ Stop Offs (whenever the machine stops, excessive localized heating may result in Heat Bars)

- ❑ Creasing (dyeing streaks)

Size hardening --- Difficulty in desizing

15

A Typical Schematic Diagram of Gas Singeing



Yarn Singeing Machine

Desiging

Define - Desiging

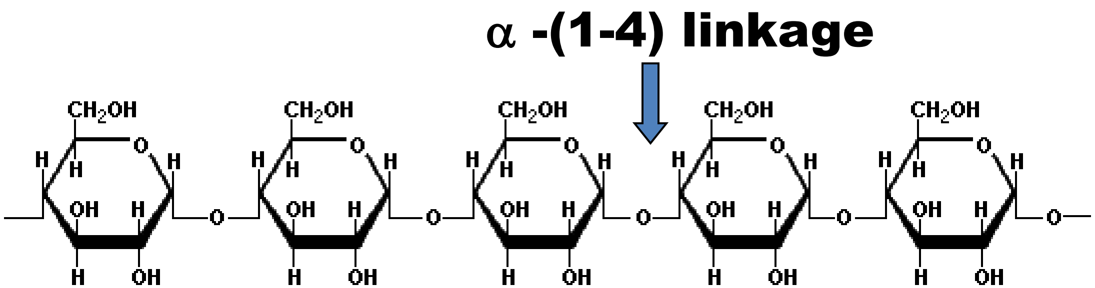
(Size forms a stiff, hard and smooth coating on warp yarns to enable them to withstand the cyclic tensions during weaving and reduce breakage)

Desizing is the process of removal of size material applied on warp threads of a fabric to facilitate the process of weaving.

Sizing Agents

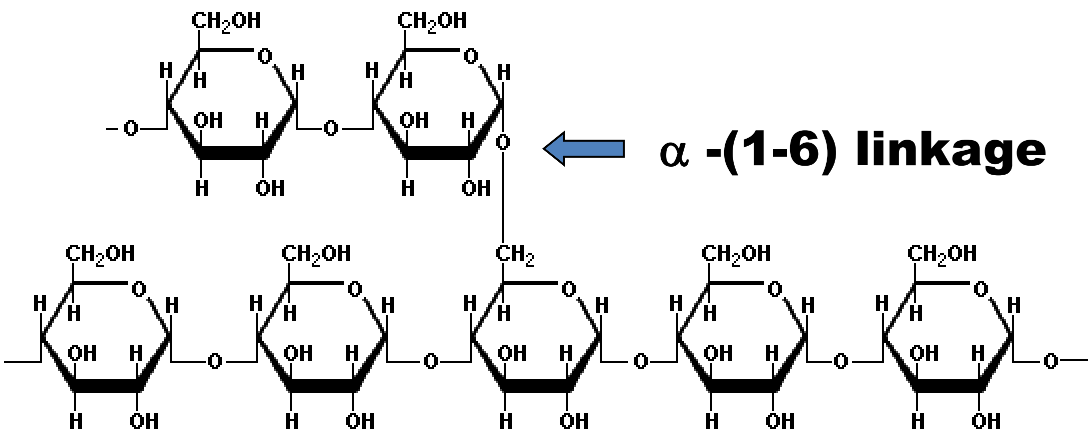
1. **Starch sizes**
2. **Modified starches**
3. **Cellulose derivatives**
4. **CMC (carboxymethyl cellulose)**
5. **Sizes based on polyvinyl alcohol (PVA)**
6. **Polyacrylates (PAC)**
7. **Galactomannans (GM)**
8. **Polyester sizes (PES)**
9. **Vinyl, acrylic and styrene homo- and copolymers**
10. **Waxes and fats**
11. **Paraffins, silicones, softeners, fungicides and others**

Starch



Representative partial structure of amylose

(20-30%)



Representative partial structure of amylopectin

(70-80%)

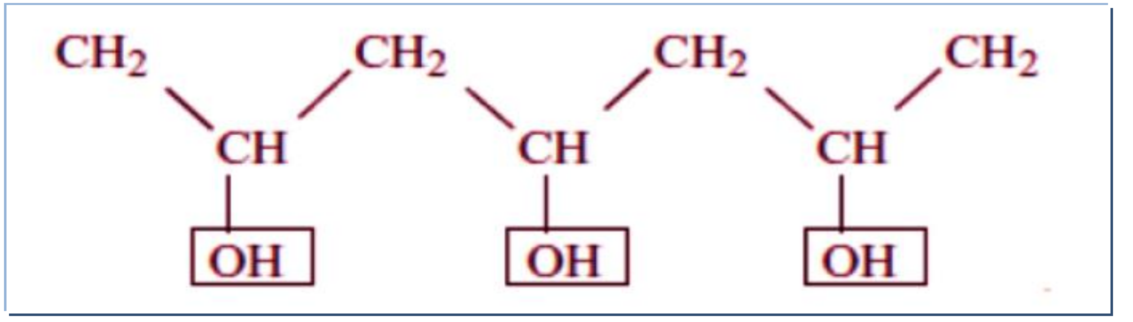
Structural difference: Amylose and Amylopectin

Properties	Amylose	Amylopectin
Structure	linear	Branched
	α-1,4 glucoside	α-1,4 glucoside
		α-1,6 glucoside
Molecular weight	10,000 to 50,000	50,000 to 10,00,000
Iodine reaction	blue	Violet
Film-forming properties	elastic	not very elastic
Enzymatic degradation	complete	partial (60%)
Structure	crystalline	amorphous

Sizes based on Synthetic Polymers

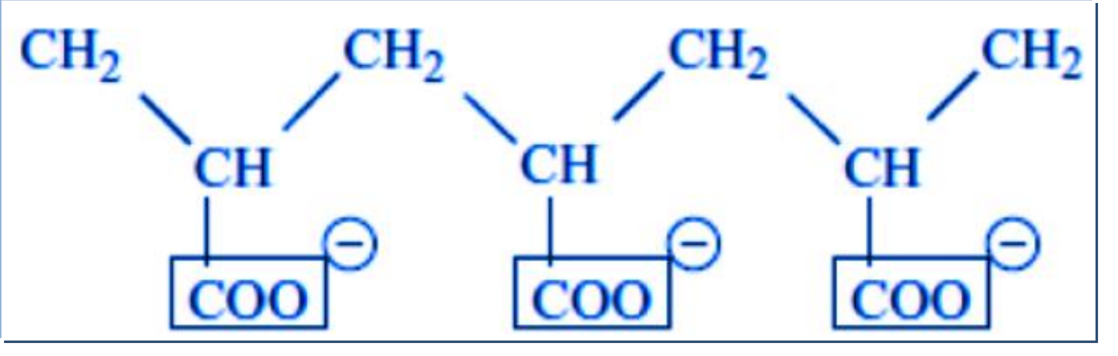
Polyvinylics:

(PVA)



Polyacrylates:

(Polyacrylate)



Water Jet Weaving (preferable):

$COONH_4$ (form)

Desizing – Mechanism

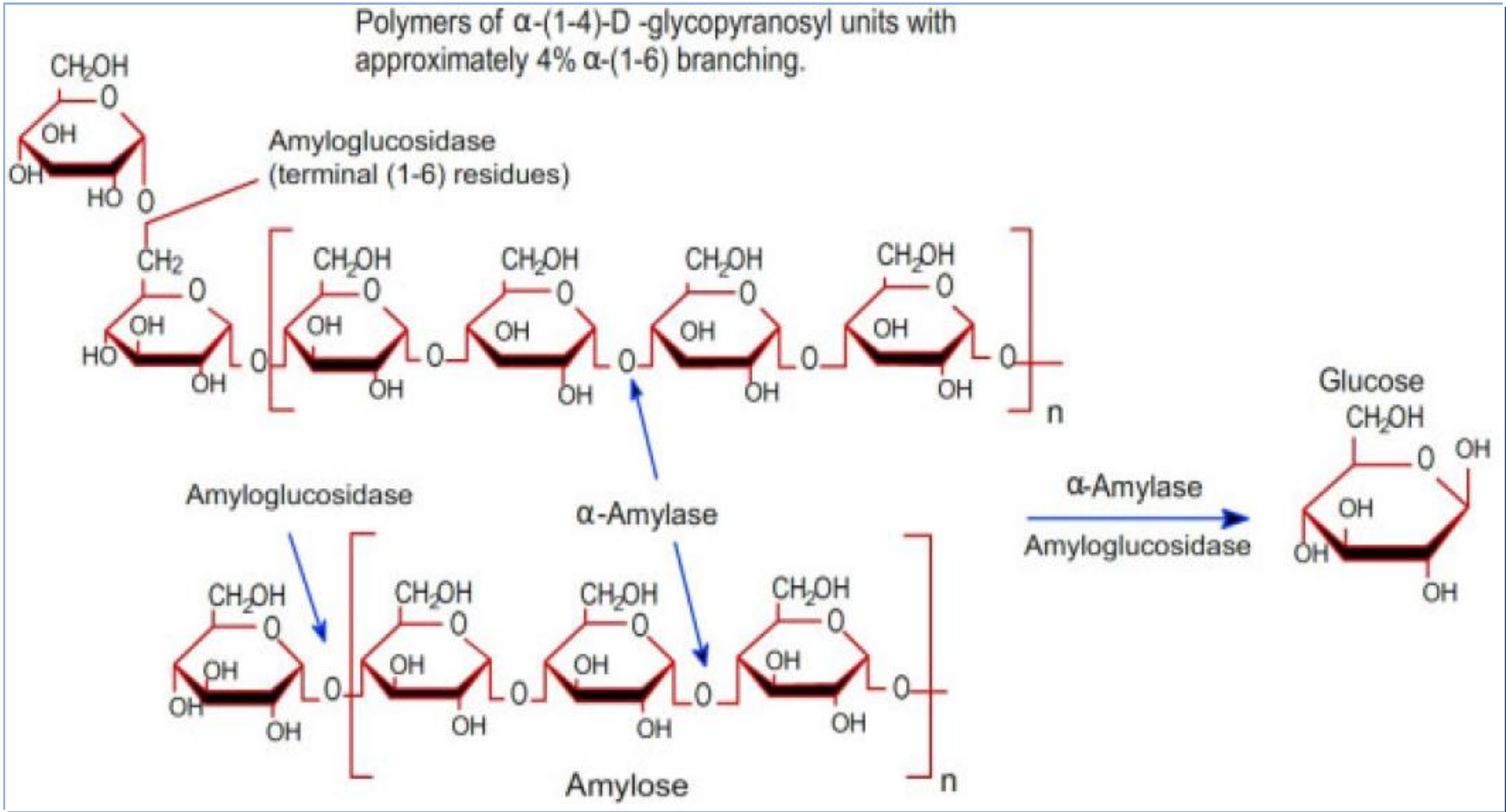
- **HYDROLYTIC DESIZING**
- **OXIDATIVE DESIZING**

Hydrolytic Desizing

When the ‘**1-4 linkage**’ is attacked and broken by hydrolysis, the desizing is called hydrolytic desizing.

Initial starch degradation product is insoluble dextrin, which on further hydrolysis is converted to soluble dextrin and finally glucose.

HYDROLYTIC DESIZING



Hydrolytic Desizing Methods

ROT STEEPING

- Steeping in water at 30 - 40^{0}C, starch swells
- Swollen starch is attacked by enzymes secreted by microorganisms in environment
- Hydrolyzed starch is removed by normal washing
- Low capital investment
- Slow, low reproducibility, risk of cellulose being attacked

Rot steeping has become obsolete because of poor process control and slowness of the process.

ACID DESIZING

❖The 1-4 linkage can be broken by acid hydrolysis also
❖ Mineral acids are used

- H_2SO_4/ HCl (5 – 10 gpl) is needed at 40 ^{0}C for 3-4 hrs. (> 40 ^{0}C & 10 gpl acid concentration, degradation of cotton cellulose itself may occur)

- The fabric is padded with acid solution and stored (batched)

ACID DESIZING…………..

Action of acid will result in gradual degradation of starch. But not all starch is degraded to the extent where it all becomes water soluble.

Hence the action of acid will result in fabric having a range of starch molecular weights. Some with high water solubility, some with medium and some with poor or no solubility.

Hence desizing should always be followed with hot water washing to remove maximum amount of starch

Precaution: Local drying during storage should not be allowed to take place at any cost. It may result in high concentrations of acid at localized places which can cause cellulose degradation.

ENZYMATIC DESIZING: Enzyme means yeast (Greek word)

The term was coined in 1876 by German biochemist Willey Kuhne

- Organic biocatalyst, highly specific in reaction catalyzed and choice of reactants/substrate and hence safe to the substrate
- Physically colloidal in nature & High molecular weight proteins (3D)
- Work under specific conditions of temp. & pH (better process control is needed)
- Lose activity gradually with time
- Generally, act under mild conditions
- Have to be cultivated and nurtured like crops (from fungi)
- Genetic engineering allows enzymes to be designed for specific processes

Enzyme Sources for Desizing

Vegetable, Bacterial, Animal
(Diastase, Amylase, Rapidase, etc.)

Developments:

(1900) Malt Diastafor, by Diamalt Co. of Munich, Germany for commercial Desizing (vegetable)

(1912) Enzymes from slaughter-house waste (animal)

(1919) From bacterial sources (bacterial)

Amylase

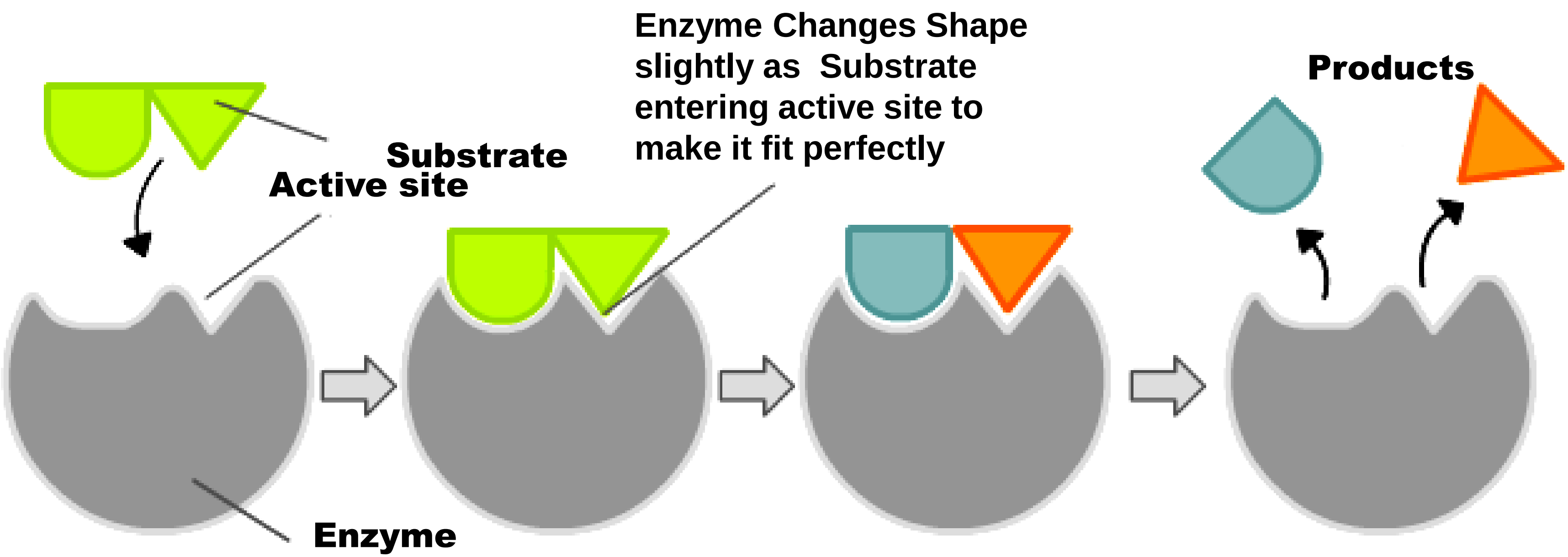
The enzymes are classified according to the manner in which the glycosidic bond is attacked:

- **α- Amylase** attacks the chain at random yielding dextrins, oligosaccharides and monosaccharides.

- **β -Amylase** attacks the chain end & produces maltose

- **Glucoamylase** cleaves the alpha-1, 4 and alpha 1,6 glycosidic linkages of amylose and amylopectin to yield glucose

MECHANISM OF ENZYME ACTION



Enzyme + Substrate entering active site

Enzyme/Substrate Complex

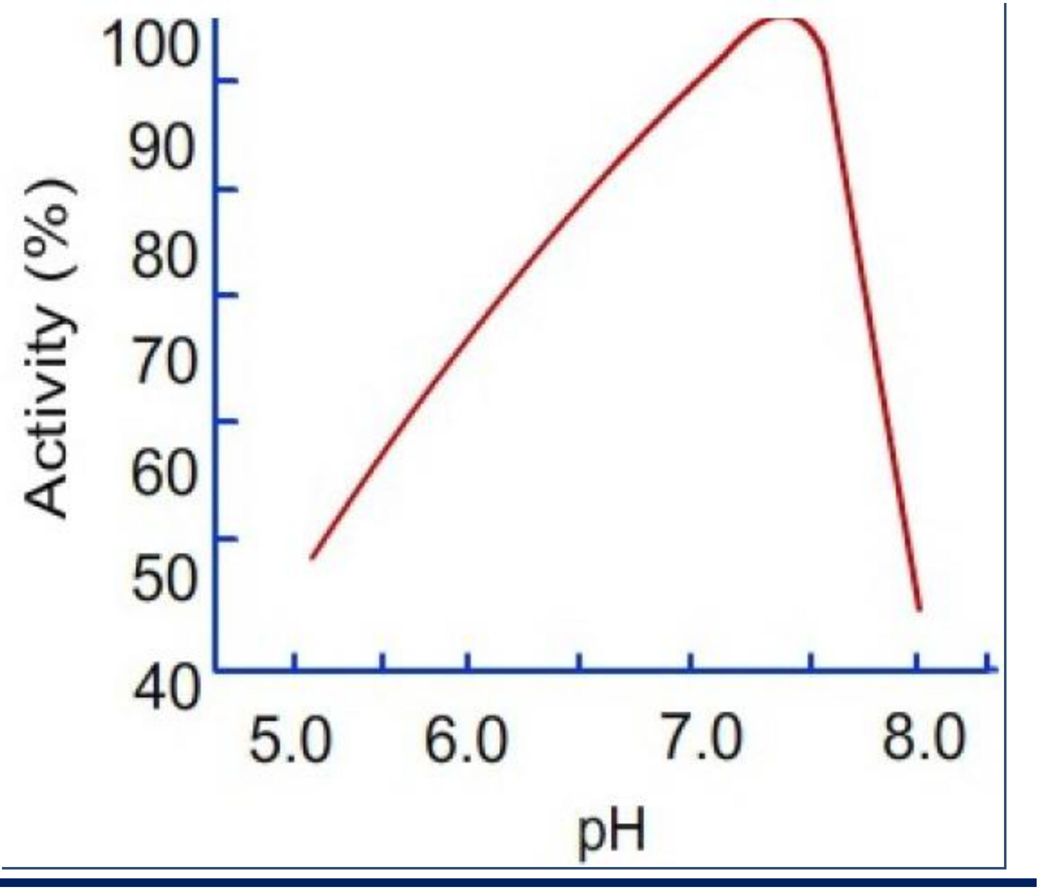
Enzyme/Product Complex

Enzyme +Products leaving active site

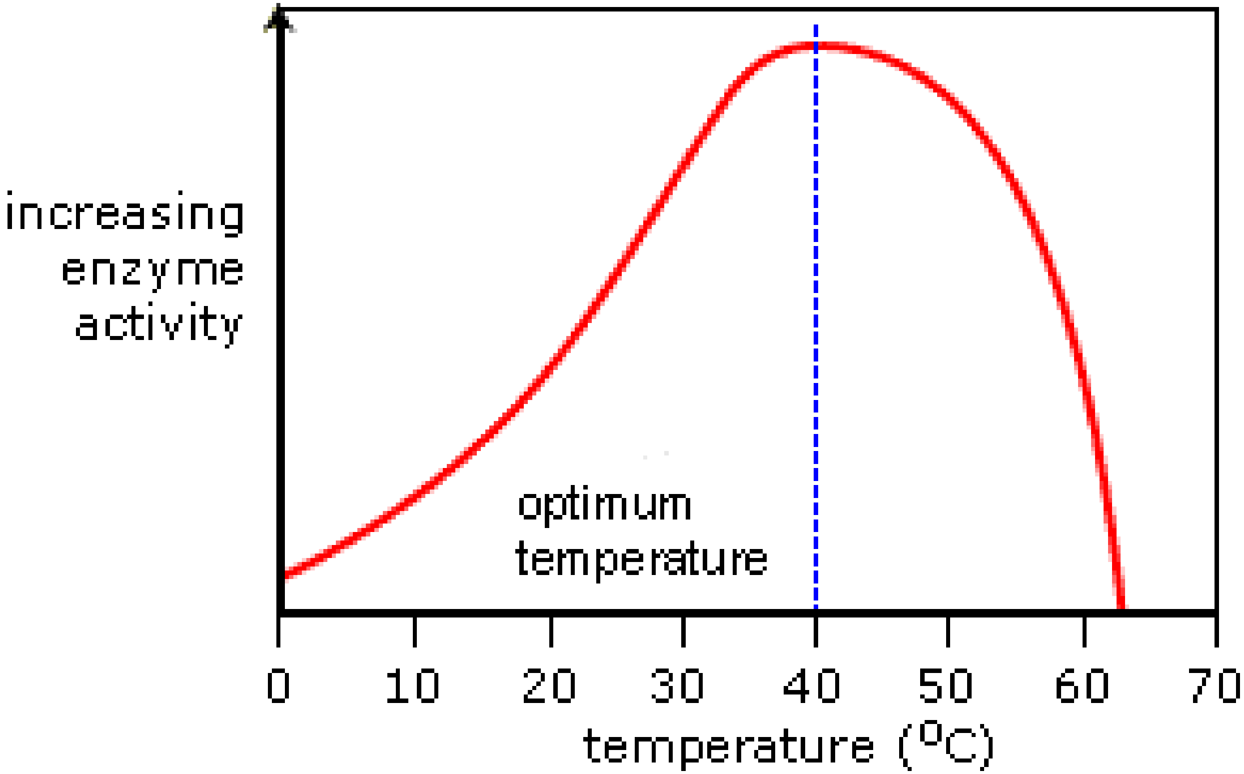
Enzymatic Desizing Parameters

Since enzymes act optimally at specific conditions of temperature and pH, it is important to maintain narrow range of conditions for best results.

Effect of pH on enzyme activity



Effect of temperature on enzyme activity



Stability of various amylases

- Sodium & Potassium chloride (0.2-1%) improve the activity of pancreatic enzyme

- Ca^{++} improves the thermal stability of malt amylase

- However, heavy metal ions like copper, iron, etc. inhibit their activity

Water Quality ?

Use of Surfactant

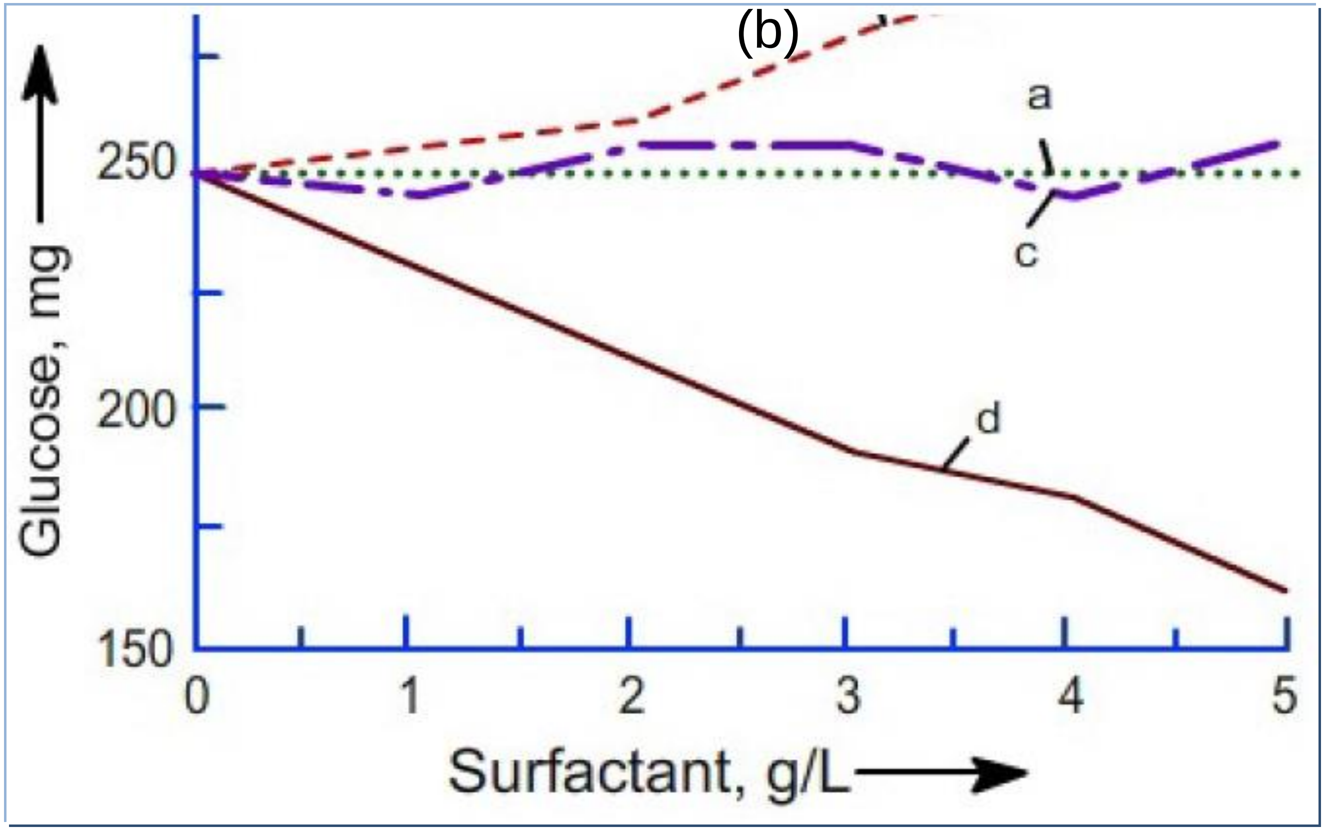
Grey cotton has poor absorbency

(Surfactants can be used to improve the wetting of fabric and dispersion of hydrophobic impurities)

- In general, anionic surfactants inhibit enzyme action

- Non-ionic surfactants are generally preferred

Effect of surfactant type of starch degradation into glucose



a) No surfactant
b) Octaphenyl ethoxylate
c) Mixture of nonionic and anionic surfactants
d) Ester of sodium sulfosuccinic acid

The optimum process conditions for various enzymes

The table given below compares the best process conditions for these enzymes

PROCESS	CONCENTRATION (gpl)	TIME (hr.)	TEMPERATURE (°C)
Rot Steeping	-	10-16	30-40
H_2SO_4	5-10	3-4	40
Malt Diastase	3-20	4.5-5.5	50-60
Pancreative Diastase	1-3	6.8-7.5	50-60
Bacterial Diastase	0.5-1	6.5-7.5	60-70

High temperature enzymatic desizing

Novozymes, an enzyme producing organization, introduced an alkaline amylase with a broad activity spectrum, capable of application over the pH range 5 – 10 and from 20 - 85° C. This has enabled combined desizing and bio-scouring to achieve commercial acceptance in a number of textile mills.

Enzyme product	Temp. (°C)	pH
Aquazym® PS	85-115	5.5-6.5
Aquazym® SD	20-85	5.5-6.5

Oxidative desizing

In oxidative desizing, the size is removed by **oxidative degradation**

Oxidative desizing

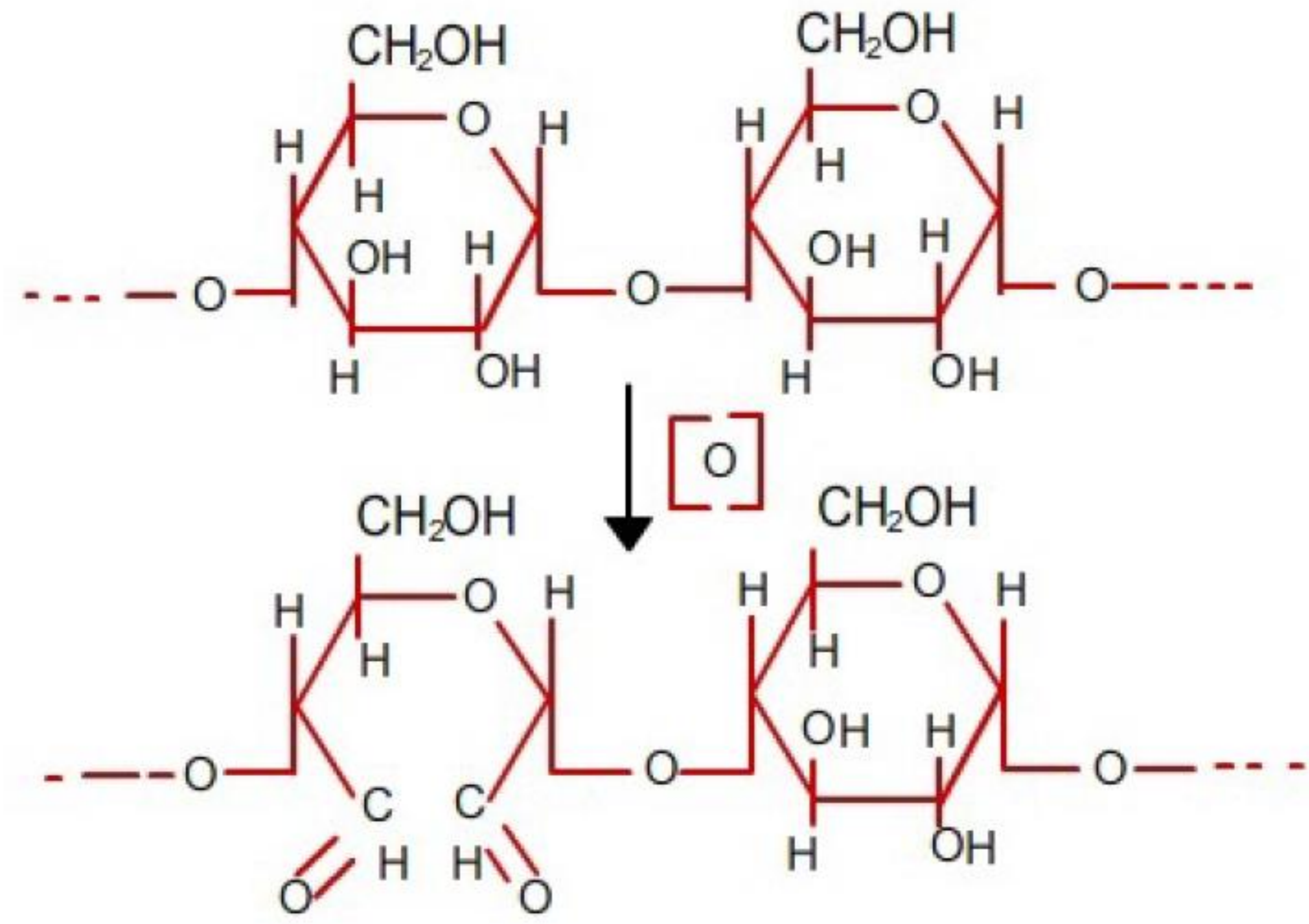
Hydrolytic desizing is successful in case of natural starch but it does not remove sizes based on synthetic polymers.

- It is particularly useful when the size is based either on a synthetic polymer like high MW. PVA (poly vinyl alcohol) or is a mixture of synthetic and natural polymers.

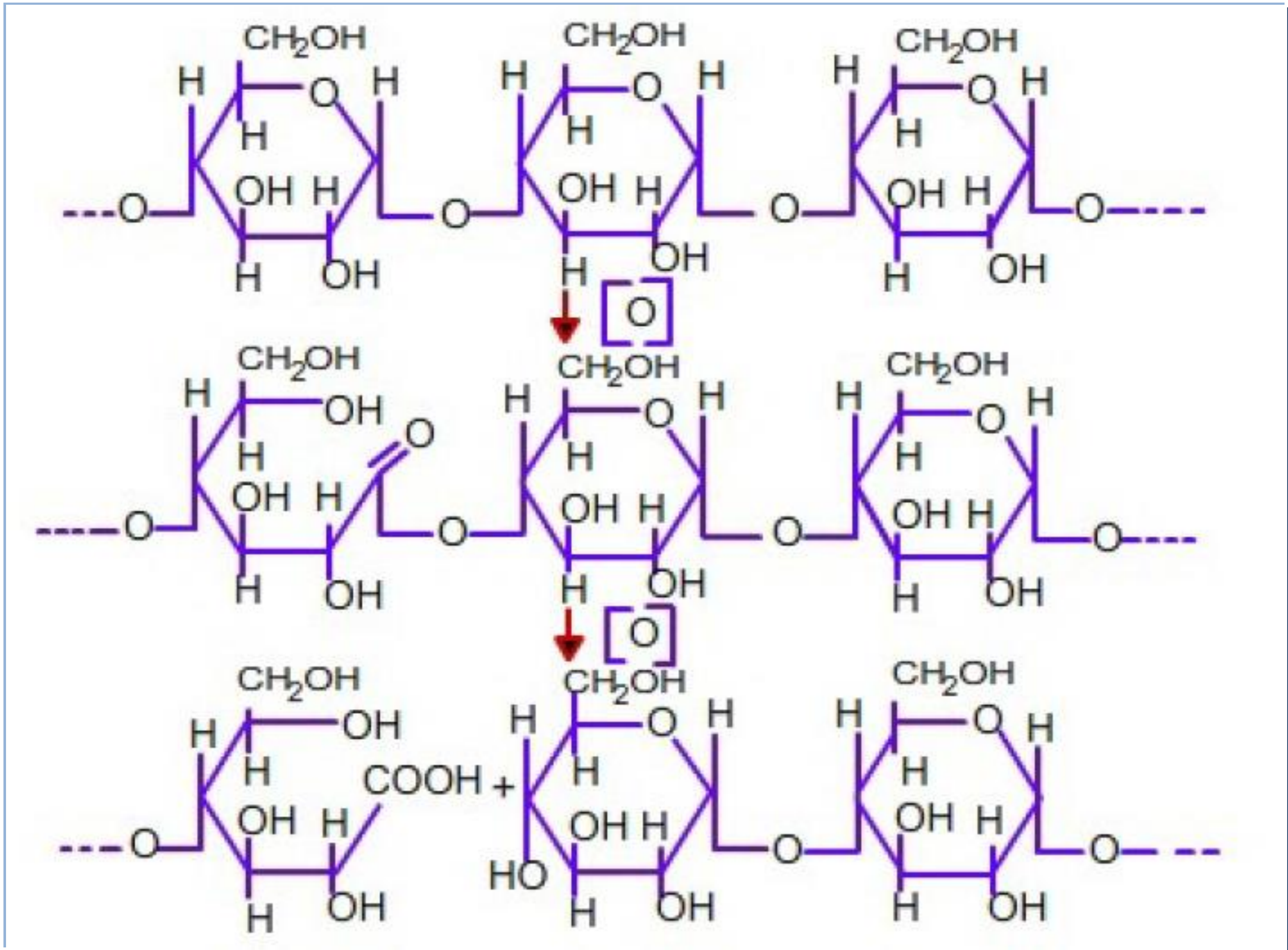
Oxidative agents used in oxidative desizing:

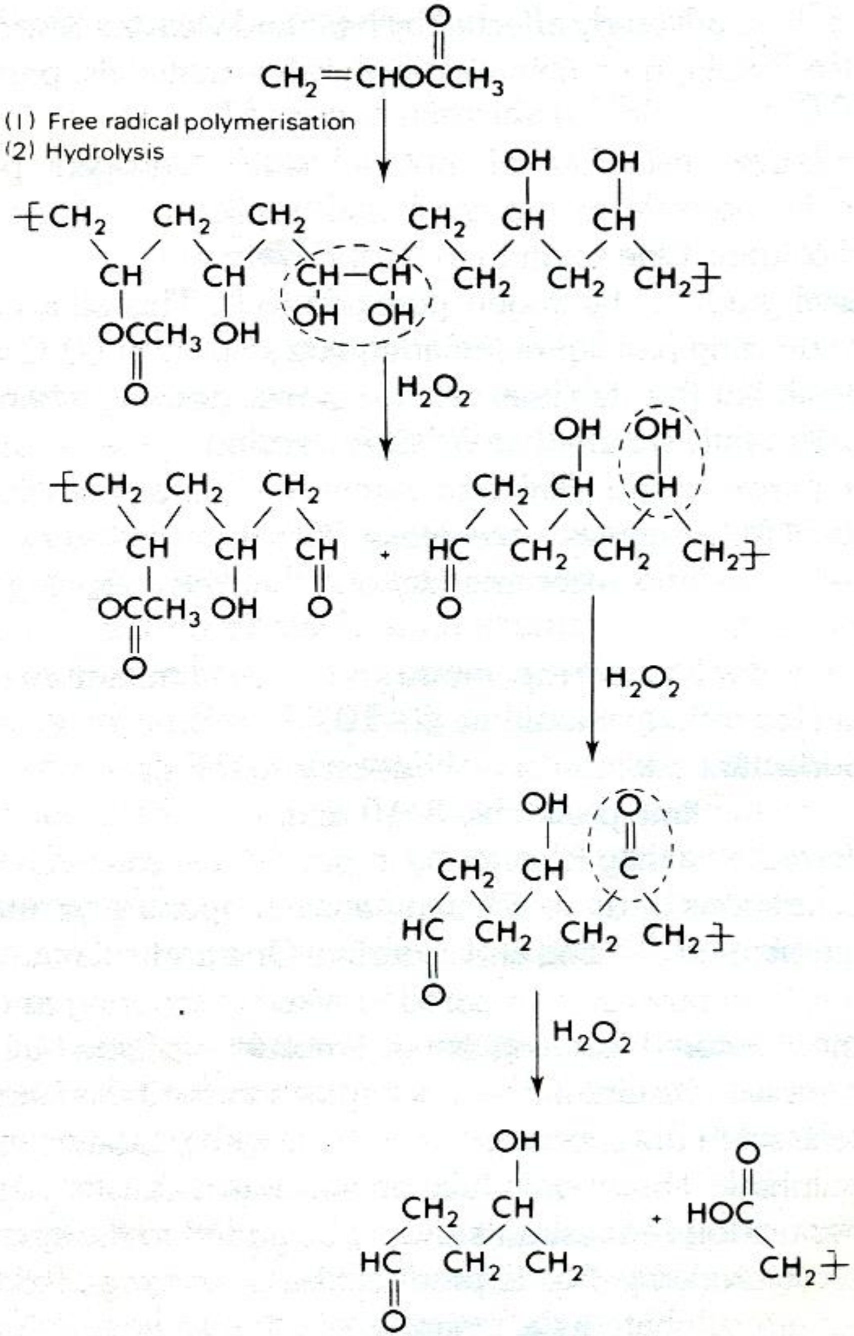
- Sodium chlorite ($NaClO_2$)
- Sodium bromite ($NaBrO_2$)
- Hydrogen peroxide (H_2O_2)
- Sodium and Potassium persulphate ($Na_2S_2O_8$, $K_2S_2O_8$)
- Peroxy monosulphuric acid (H_2SO_5)

(a) Oxidation of starch – ring opening



(b) Oxidation of starch – chain scission





Mechanism of oxidation of Poly vinyal Alcohol with Hydrogen Peroxide during Desizing Process

Sodium chlorite ($NaClO_2$)

With sodium chlorite, the desizing is carried out under alkaline conditions.

Sodium bromite ($NaBrO_2$)

- Sodium bromite is a powerful oxidant for starch
- Breaks glucose ring by breaking C_2-C_3 link and produces a dialdehyde. The dialdehyde dissolves in alkaline solution. hence desizing is followed by an alkaline wash
- Acts best at pH 10
- Works at room temperature for short durations and is highly efficient
- However, 50% of the bromite reacts with impurities and increases pollution problem of the effluent

Sodium and potassium persulphate ($Na_2S_2O_8$, $K_2S_2O_8$)

With persulphates, desizing is carried out in alkaline medium.

Peroxy monosulphuric acid (H_2SO_5) / Hydrogen peroxide (H_2O_2)

Peroxy compounds are sensitive to presence of heavy metal ions, a sequestering agent is usually employed.

Hydrogen peroxide may be used in a similar way. Conditions are generally kept mild (pH-6-8) and washing is done in alkaline conditions.

Desizing Conditions

Methods	Chemical	Temp. (ºC)	Time	pH
Rot	None	30 (RT)	16 -24 hrs.	7.0
Acid	HCl (0.5 -1.0 %)	30 - 60	2 -8 hrs.	1.0 -2.0
Enzyme	0.5 -1.0%	60 - 70	1 -2 hrs.	6.0 -7.0
Oxidative (Persulphate)	0.3 -0.5%	100	10 min	14.0

Assessment of desizing efficiency

TEGEWA violet scale (industrially)

The TEGEWA Association (established in 1951 in Wiesbaden) comprises of manufacturers of the following:

Textile, paper, leather & fur auxiliaries, colourants, surfactants, complexing agents, antimicrobial agents, polymeric flocculants, cosmetic raw materials, pharmaceutical excipients and allied products

The name being an amalgam of these key activities:

“TExtilhilfsmittel” (textile auxiliaries)
“**GErbstoffe” (**tanning agents**)**
“WAschrohstoffe” (detergent raw materials**)**

Reagent preparation

Put potassium iodide [10 g of KI (100%)] in 100 ml of water; then add 0.6358 g of iodine (100%).

Stir well until iodine is completely dissolved in the KI solution. After this, add 800 ml of ethanol.

Then by adding water the volume should be raised to 1000 ml.

Testing Method

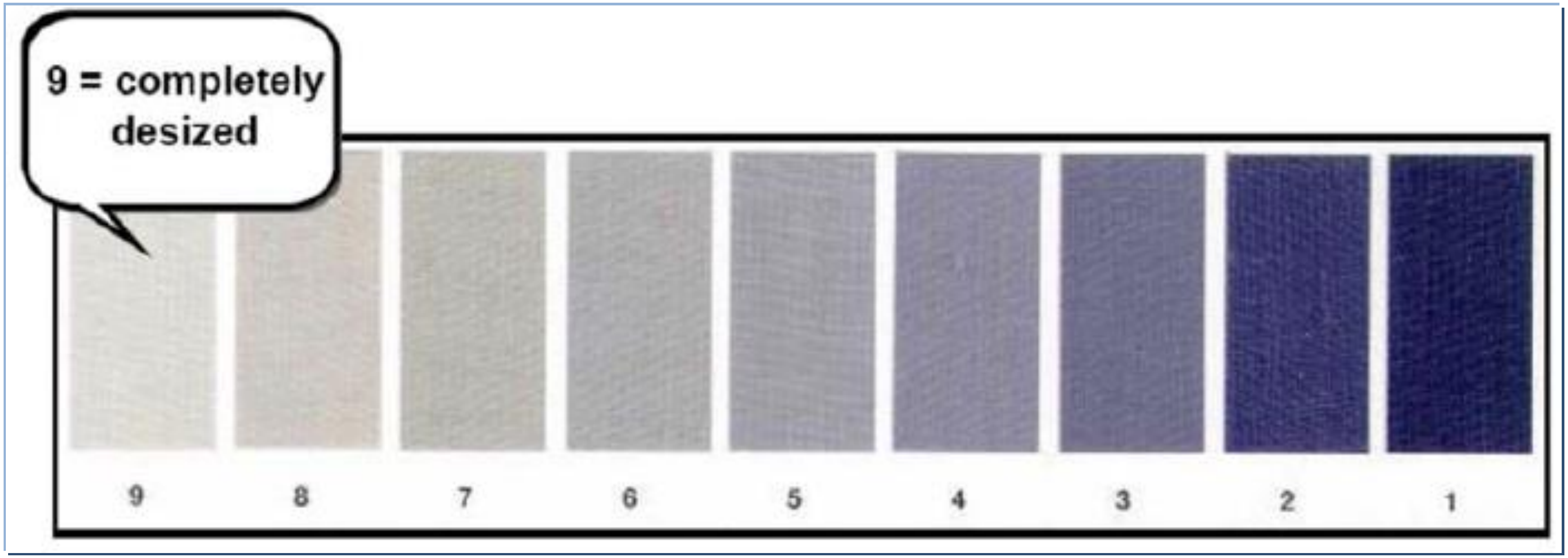
Put one or two drops of the above solution on a fabric. Rub it gently and then assess the colour change as per the TEGEWA scale. Before testing, the fabric should be cold and there should not be any residual alkalinity in it

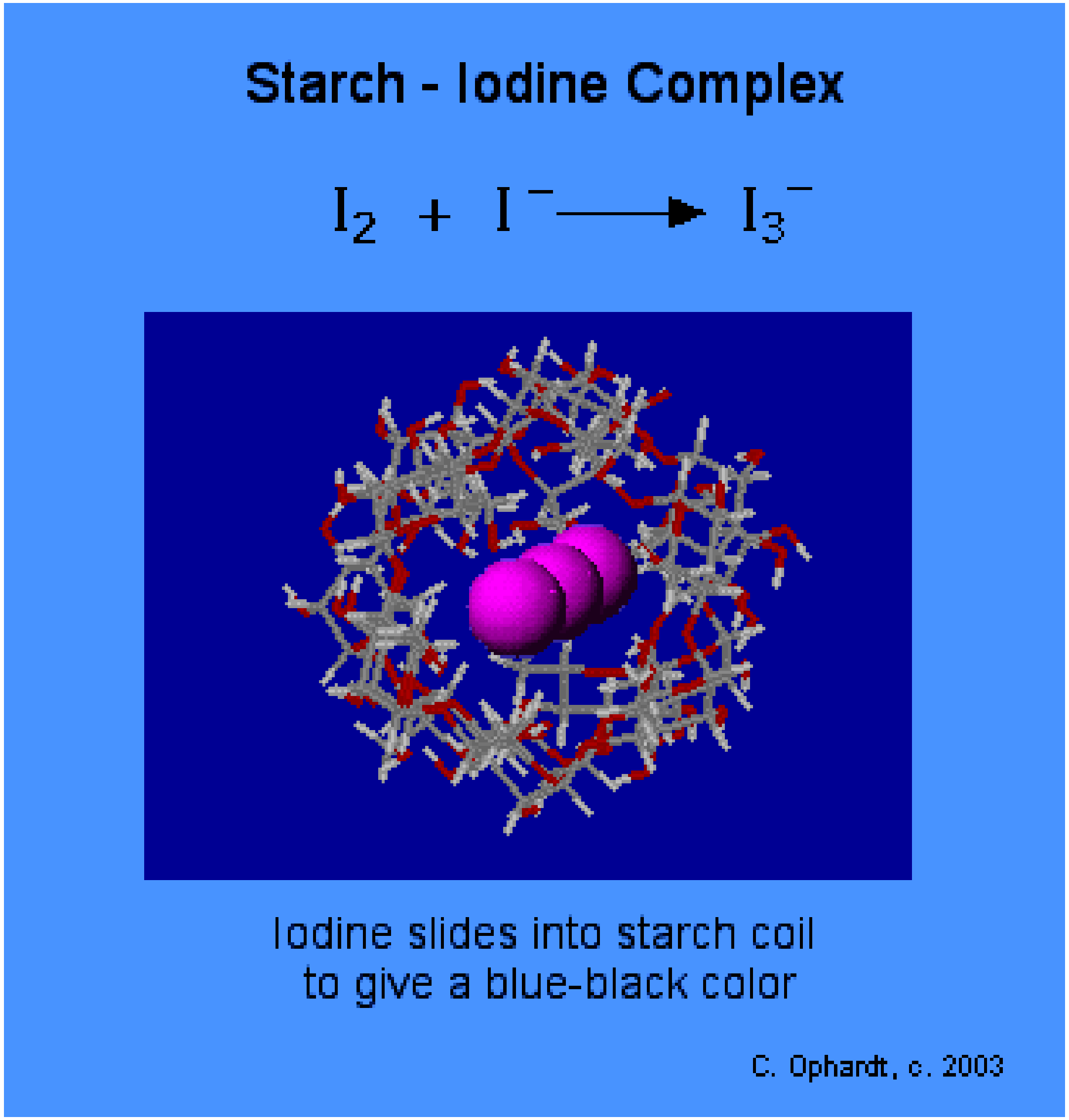
Assessment: TEGEWA violet scale (1-9)

No Colour Change = No starch is present

Pale blue to bluish violet = Presence of starch size or a blend of starch+ synthetic size

Brown = Presence of modified starch or a blend of modified starch/PVA size





Starch Test:

Add Iodine-KI reagent to a solution of starch or to starch. A blue-black colour results if starch is present. If starch amylose is not present, then the color will stay orange or yellow. Starch amylopectin does not give the colour, nor does cellulose, nor do disaccharides such as sucrose in sugar.

Scouring

Define - Scouring

Scouring is an important pre-treatment stage especially for natural fibres which tend to have a significant presence of natural impurities such as oil, wax, fat, hand dust, etc. They are removed to produce hydrophilic and clean textile material.

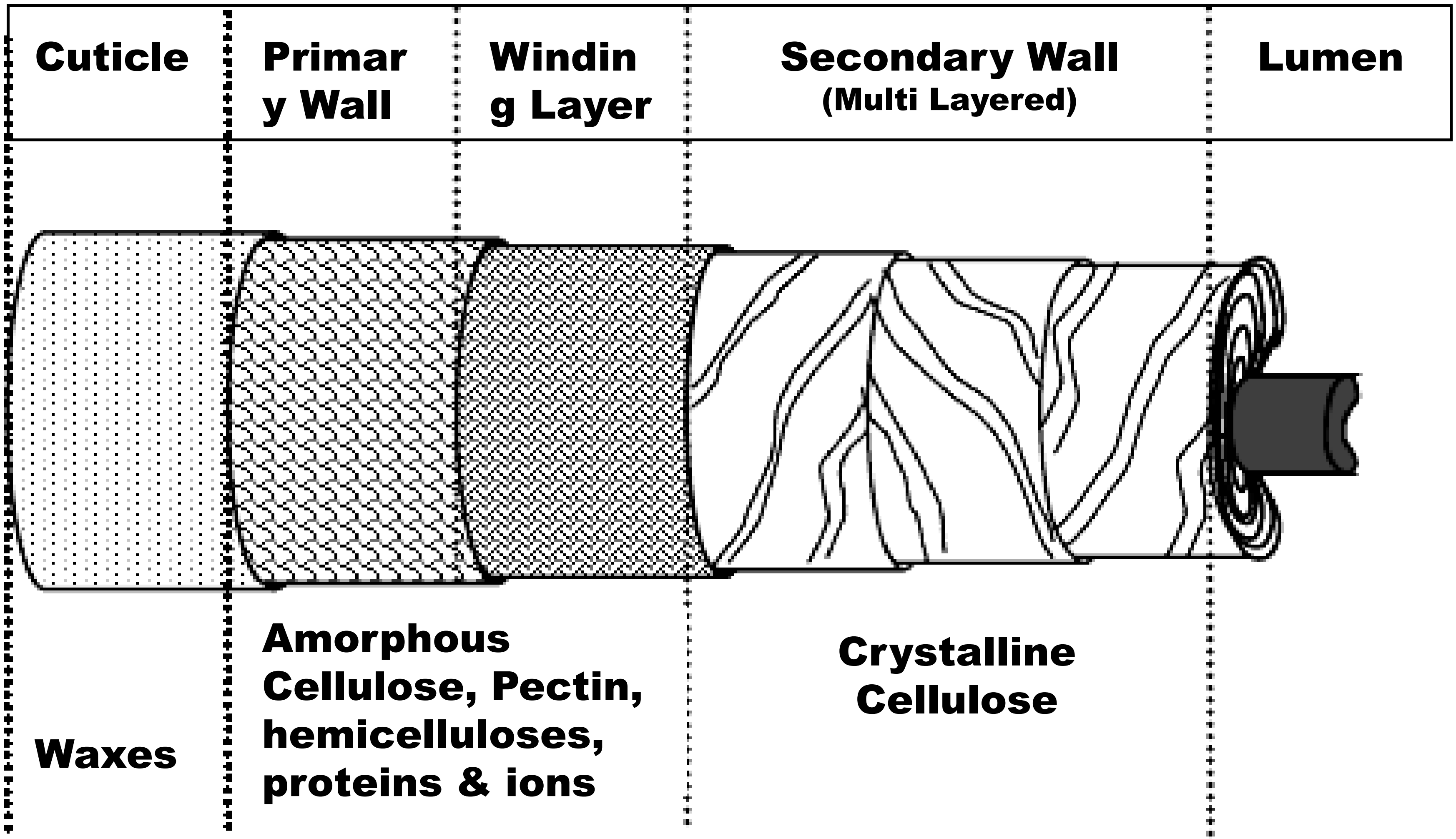
Objective:

To reduce the amount of impurities sufficiently to obtain level results in dyeing & finishing operations by bringing the substrate to a **highly absorbent state.**

Hence the emphasis is on removal of hydrophobic impurities

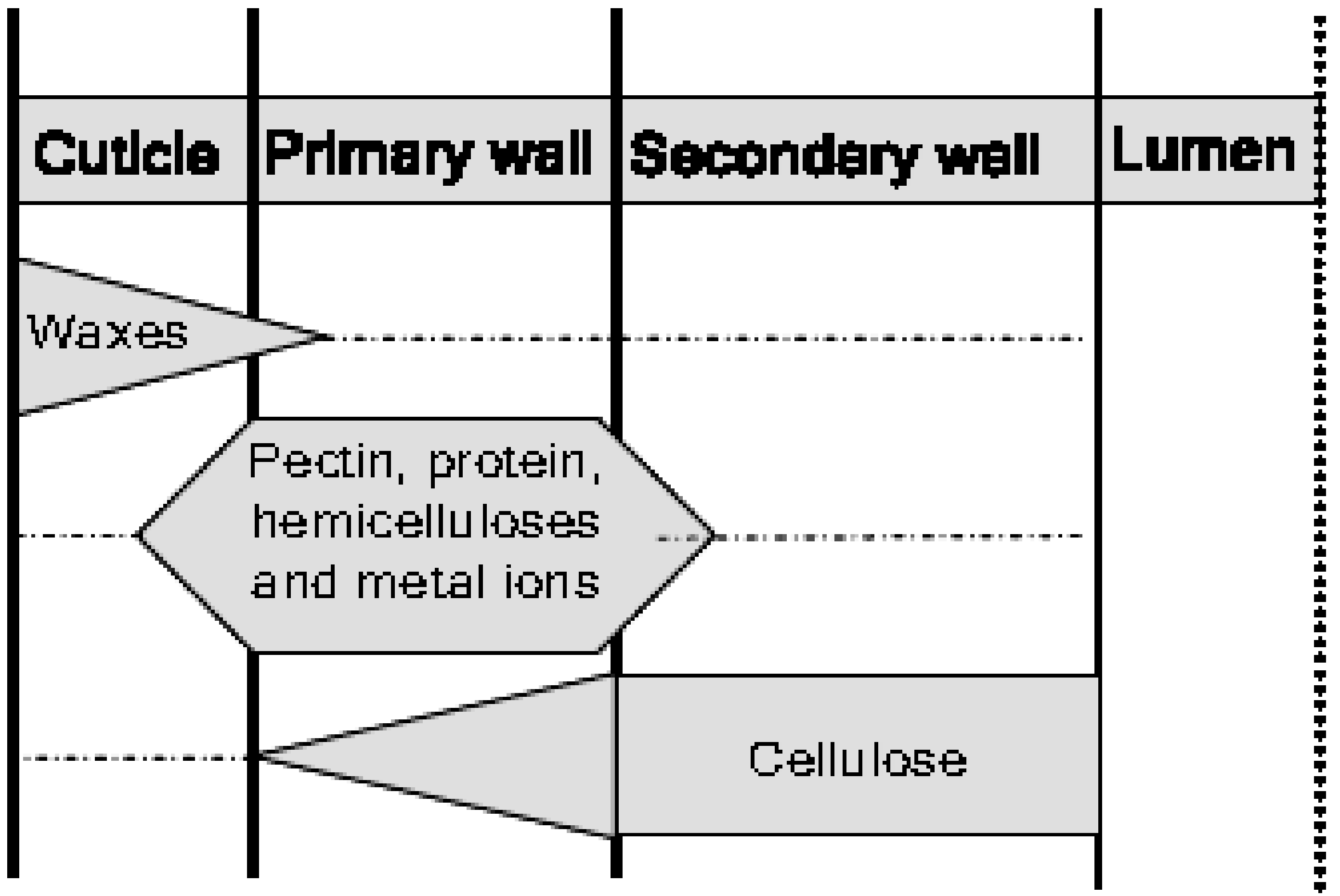
Impurities in Cotton

Impurities	Percentage (%)
Waxes	0.4-1%
Nitrogenous Matter (Proteins)	1-2.8%
Pectic Matter	0.4-1%
Minerals	1-1.8%
Motes (seed coat fragments)	-
Natural Coloring Matter	Variable
Added	Lubricants/Knitting oils, grease stains



A schematic representation of nature of cotton fibre showing its various parts

A Schematic Representation of the Cellulosic and Non-cellulosic Materials in the Cotton Fibre



Scouring Systems for Cotton fibres

The wax is present in the outermost layer of cotton fibres.

- It is **non-absorbent in its natural state**
- Wax present on a fibre is low (0.4- 1%)
- It is not easy to remove

WAX:

- Higher Monohydric aliphatic alcohols, C_{24} to C_{30}
- Fatty acids, C_{16} to C_{34}
- Esters
- Hydrocarbons, C_{30}

Scouring Systems for Cotton fibres

- Conventional system
- Solvent based system
- Enzyme based system

Conventional System

Chemicals used in Scouring Process:

Different Agent	Descriptions
Alkali	Mainly Sodium hydroxide (NaOH), sometimes mix of NaOH and Na_2CO_3 (washing soda)
Wetting agent	To reduce the surface tension of scouring liquor so as to wet out the goods uniformly, generally anionic surfactants are used
Emulsifier	To emulsify non-saponifiable wax, generally nonionic surfactants are used

How are the impurities removed ?

Impurities	Mechanism of removal
Fats and waxes	**Saponification:** The saponifiable parts of waxes (fatty acid, glycerides and other esters) are converted into soap.
	Emulsification: The non-saponifiable parts of the waxes such as alcohols and hydrocarbons are emulsified by the soap formed.
Pectin & related Substances	Pectins are converted to water soluble salts of pectic acid.

How are the impurities removed?

Impurities	Mechanism of removal
Proteins and Amino acids	Proteins are hydrolyzed with the formation of soluble sodium salts of amino acid. Amino compounds are hydrolyzed to ammonia by alkali.
Minerals & heavy metals	• Partially dissolve in NaOH • By use of sequestering or chelating agents

How are the impurities removed ?

Impurities	Mechanism of removal
Hemicelluloses	Dissolution: Hemicelluloses with low DP are dissolved in NaOH
Motes	• Cellulose of low crystallinity swells in alkali and becomes sodium cellulosate, water soluble. • Residual motes are destroyed in bleaching.
Other organic compounds	Cellobiose, Cellotriose, Organic acid (i.e. Malic acid, etc.)

Mechanism of Saponification

- Wax, fats, oils and lubricants are esters in the form of triglycerides
- The triglycerides form **glycerin** and **soap** when reacted with NaOH

$$
\begin{array}{llll}
CH_2OOCR & & CH_2OH & \\
| & & | & \\
CHOOCR \; + \; 3\ NaOH & = & CHOH \; + \; 3\ RCOONa & \\
| & & | & \\
CH_2OOCR & & CH_2OH &
\end{array}
$$

Soap (water soluble)

Glycerol (miscible in water)

$$
\begin{array}{rcl}
\text{Oleic Acid,} \quad R & = & CH_3(CH_2)_7(CH)_2(CH_2)_7 \\
\text{Palmitic Acid,} \quad R & = & CH_3(CH_2)_{14} \\
\text{Stearic Acid,} \quad R & = & CH_3(CH_2)_{16}
\end{array}
$$

Soap also acts as a surfactant

Mechanism of Saponification……..

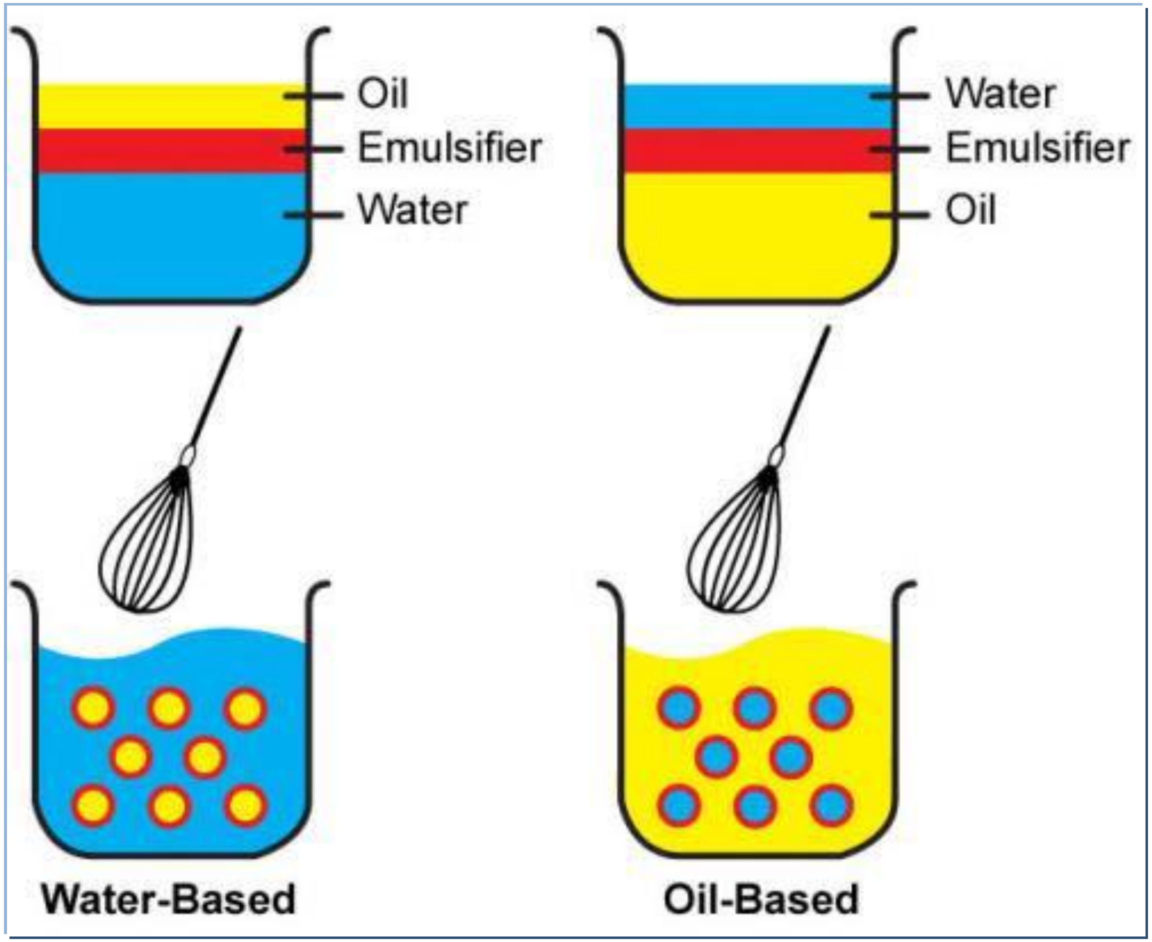
- Hence fat/oil forms glycerol (miscible in water) and soap (RCOONa)

- Soap is soluble in water and acts as a surfactant to reduce the surface tension of scouring liquor

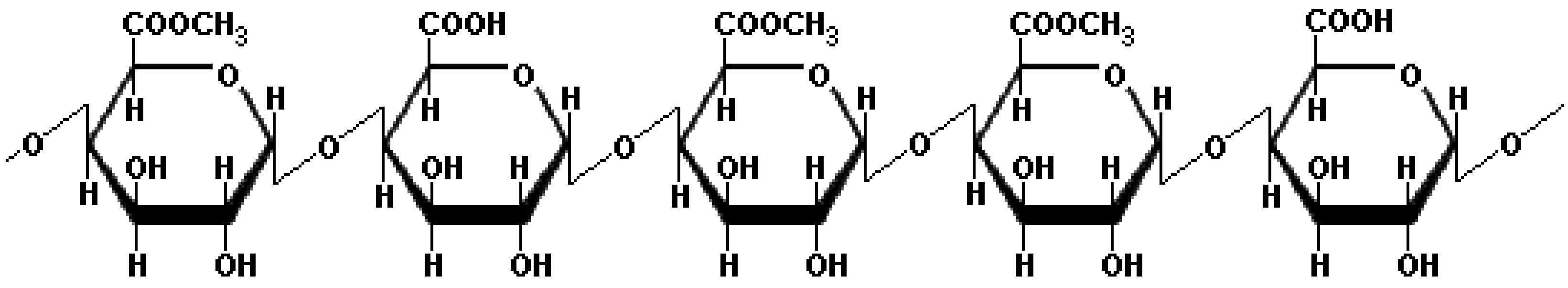
- However, since the amount of saponifiable matter present in cotton may be very low (< 0.5%) one has to make additions of wetting agents in the liquor

Mechanism of Emulsification

An emulsion is a dispersion of two immiscible liquids



Pectin and related substances

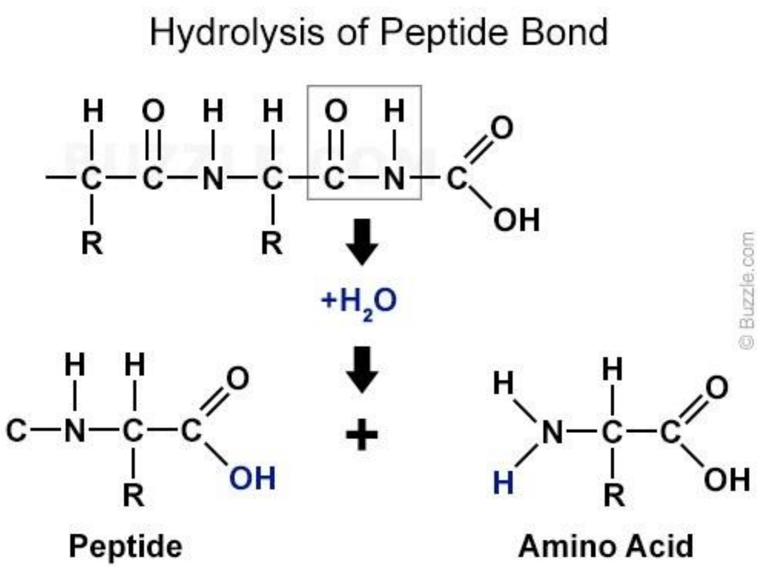


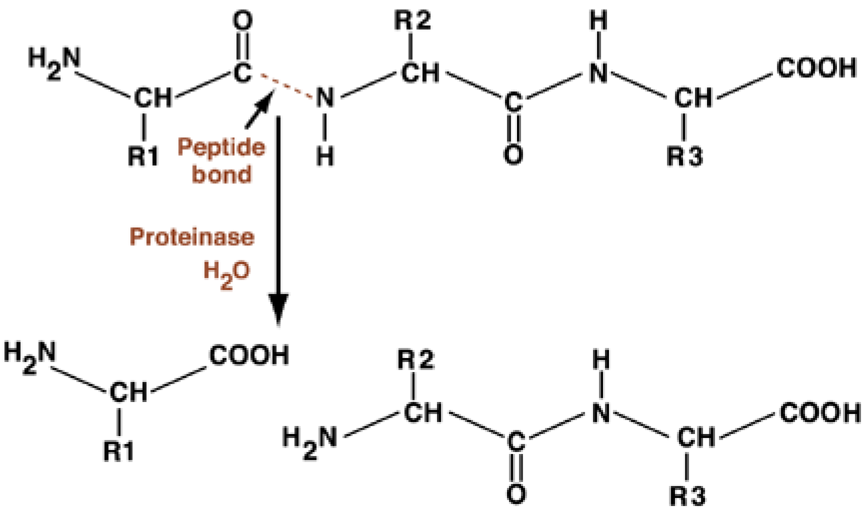
Pectin molecules have a linear backbone composed of units of (1,4)-linked α-D-galacturonic acid and its methyl ester.

- **Pectins are converted to water soluble salts of pectic acid**
- **Solubilisation: by the action of alkali, which also acts as a swelling agent to facilitate removal**

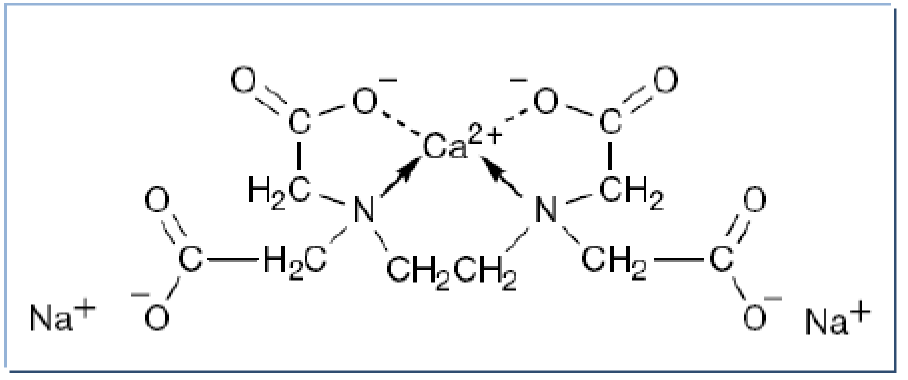
Protein substances

Compounds of Aspartic acid and Glutamic acid (Amino acids)

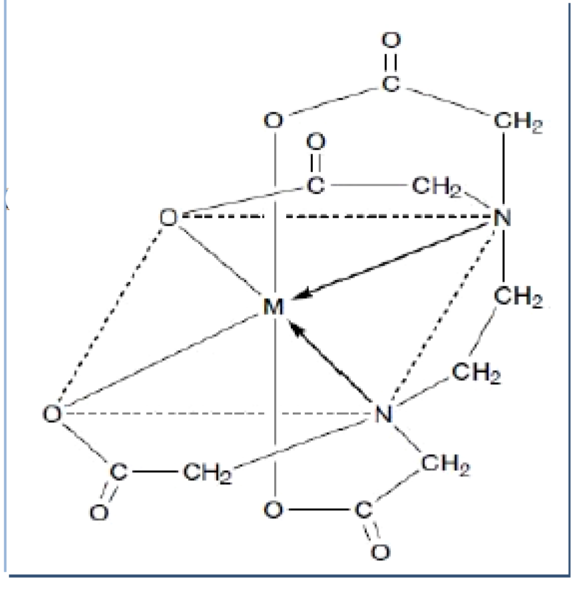




Sequestering Agent

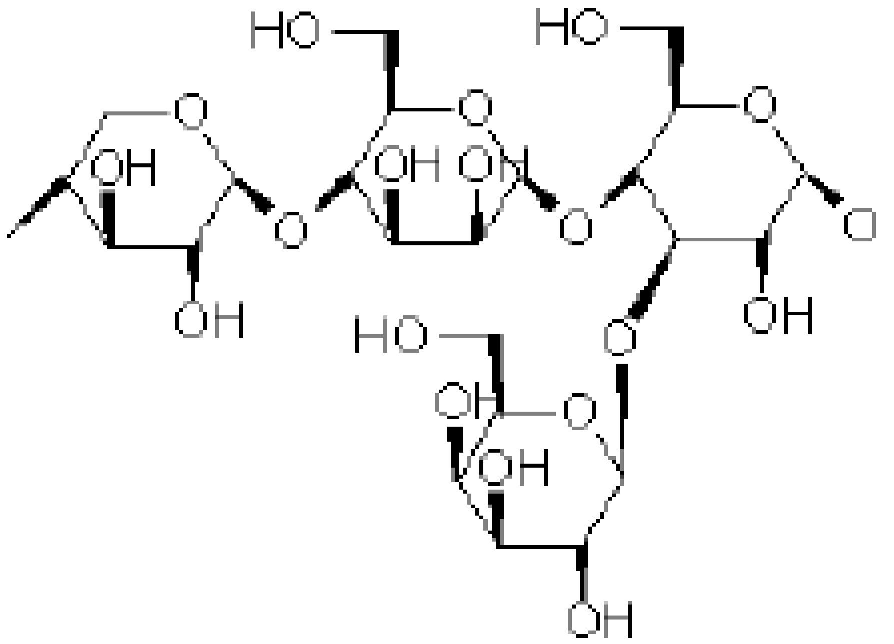


Simplified 2-D structure of EDTA-metal complex



More elaborate 3-D EDTA-metal complex

Hemicellulose



\- Xylose - ß(1,4) - Mannose - ß(1,4) - Glucose -
\- alpha(1,3) - Galactose

Cellulose is crystalline, strong, and resistant to hydrolysis. Hemicellulose has a random, amorphous structure with little strength and is easily hydrolyzed by dilute acid or base as well as hemicellulase enzymes

Conventional Scouring Recipe

Chemicals	Amount
NaOH	4% owf for normal fabric 6% owf for heavier fabric
Wetting agent	1 – 3 gpl
Emulsifying agent	Non-ionic surfactant (1-3 gpl)
Temperature	130 ^{o}C

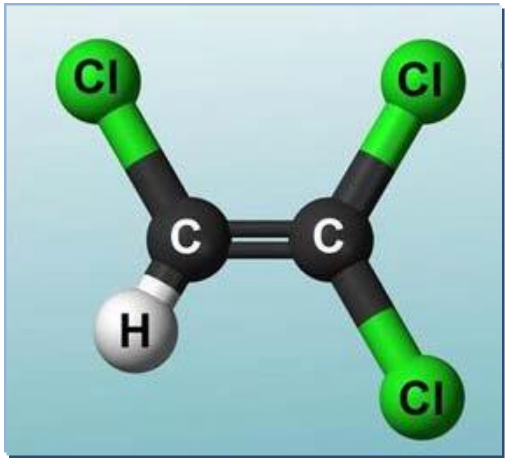
Solvent System

- Developed in 1970s in Europe and given up towards the end of 70s.

- Certain organic solvents dissolve oils, fats and waxes and these solvents can be used to purify textiles

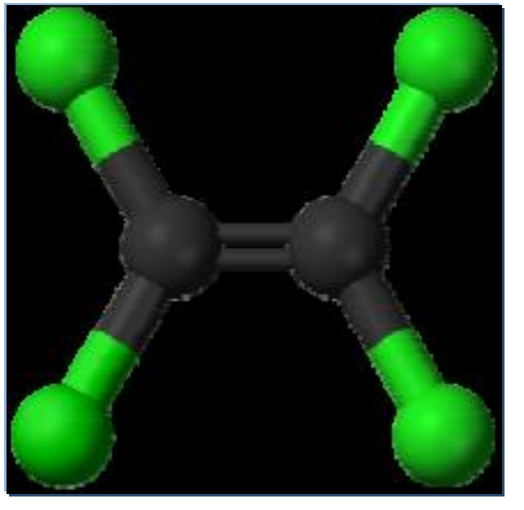
- Removal of impurities by solvent dissolution is called Extraction

Trichloro ethylene
(B.P. : 87^{o}C, Non-flammable)



Trichloroethylene (TCE)

Perchloro ethylene
(B.P. : 121^{o}C, Non-flammable)



Perchloroethylene (PCE)

Solvent Scouring

Advantages:

- Good solvents for cotton wax
- Removal of wax at room temperature
- Removal of solvent from fabric more favorable due to low specific heat of solvent
- Chlorinated solvents are non flammable

Disadvantages:

- The economy of the process depends on the recovery of solvent
- Very low amount of wax is removed with the help of large amount solvent

Hence ***solvent assisted scouring was developed***

Solvent Assisted Scouring

The system has following components:

- Solvent
- Wetting agent (Pine oil)
- Emulsifier

✓ Transparent mixture of all three produced with same HLB values (HLB Value: 13 - 13.5 for all the components)

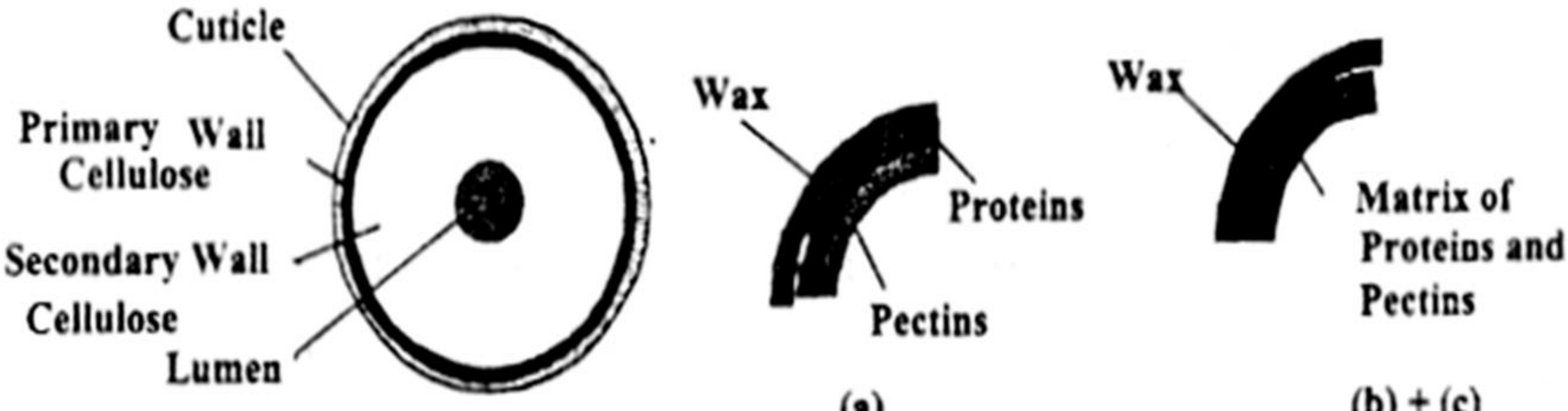
✓ 4% concentration of the above recipe used for scouring

Enzymatic Scouring

Greek word ***enzymos, which means*** **'in the cell or ferments'.**

- ❖ Enzymes are very large, complex, protein molecules consisting of amino acid
- ❖ Bio-catalysts, not consumed in the reaction
- ❖ Substrate specific, acts under narrow range of conditions of temperature, pH and agitation

Possible Structures of Cotton Surface

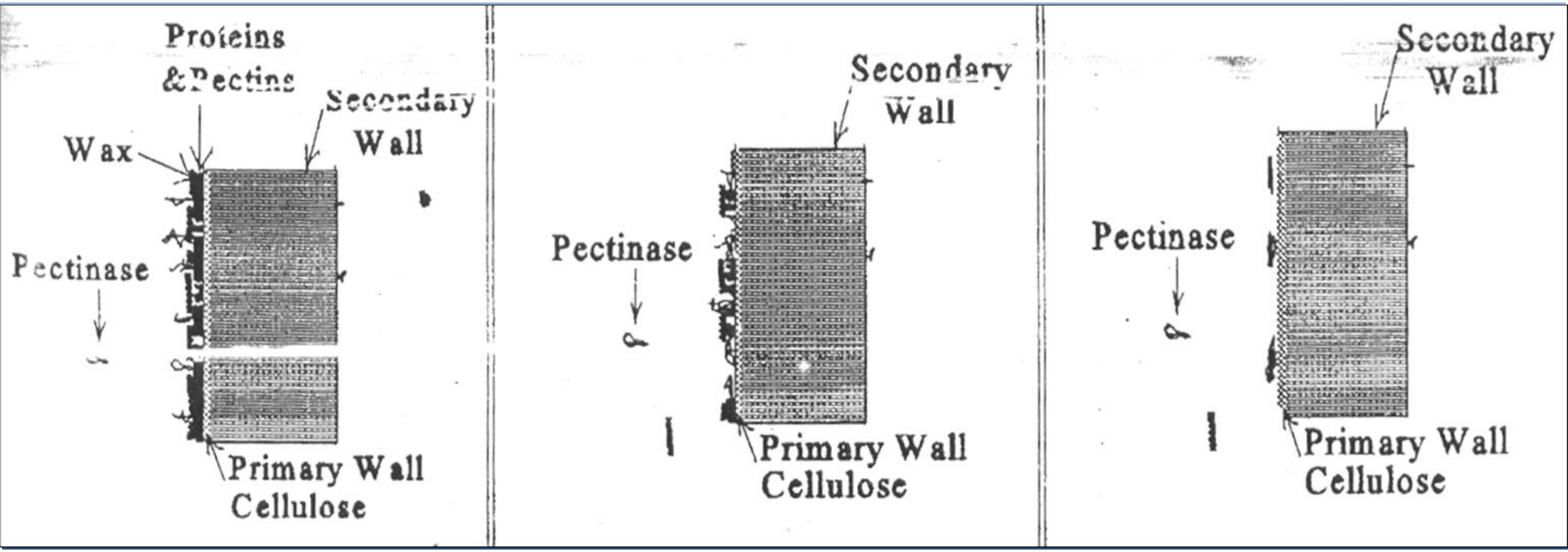


Raw cotton fiber in its swollen state

Three possibilities for the structure of the cuticle
a) layered b) matrix
c) matrix with micropores in the wax layer

- ❖ The proteins, pectins and waxes are present as distinct layers in the cuticle
- ❖ The matrix of pectins and proteins is covered by a microporous thin layer of waxes

Mechanism of Pectinases on Cotton surfaces



- Pectinases penetrate the cotton cuticle through cracks or micro pores
- Pectic substances are hydrolyzed with the aid of pectinases
- Link between the cuticle and the cellulose body breaks, absorbent fibres formed

Commercial product (Scourzyme® L)

Alkaline pectinase: The removal of non-cellulosic components from fabric is done (scouring).

Enzyme product	Temp. (°C)	pH	Continuous	Pad-roll	Jet, jig, winch
Scourzyme® L	50-65	7.5-9.0	+/-	+++	+++

Excellent: +++
Good: +
Possible under certain conditions: +/-

Machinary

Conventionally the following machines have been used for scouring:

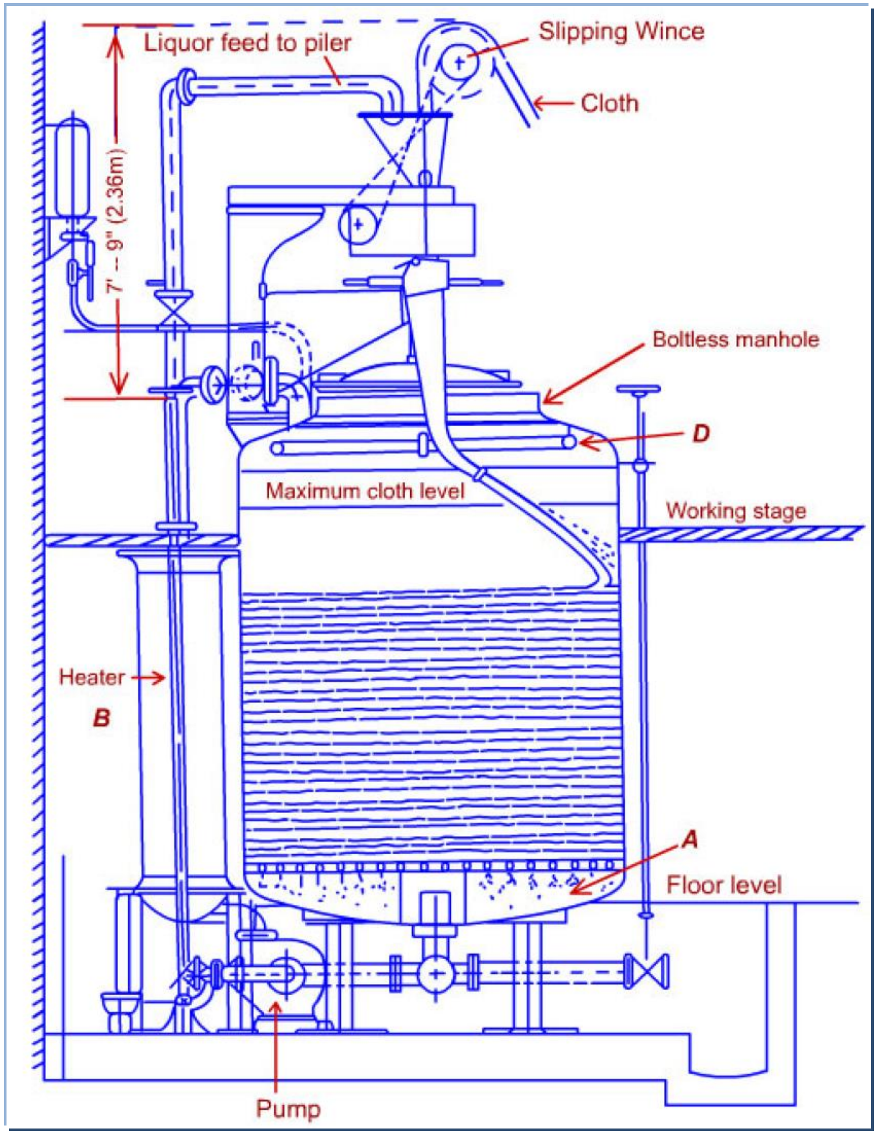
- ❑ **Pressure kier (batch system)**
- ❑ **J-BOX (continuous system)**
- ❑ **Vaporloc System (continuous)**

Pressure kier (batch system)

Kier, closed vessel in which the desized fabric is heated at high temperature for prolonged time.

Conditions	Range
M:L Ratio	1:3
NaOH Concentration	10 gpl
Temperature	130ºC
Time	6–10 hr

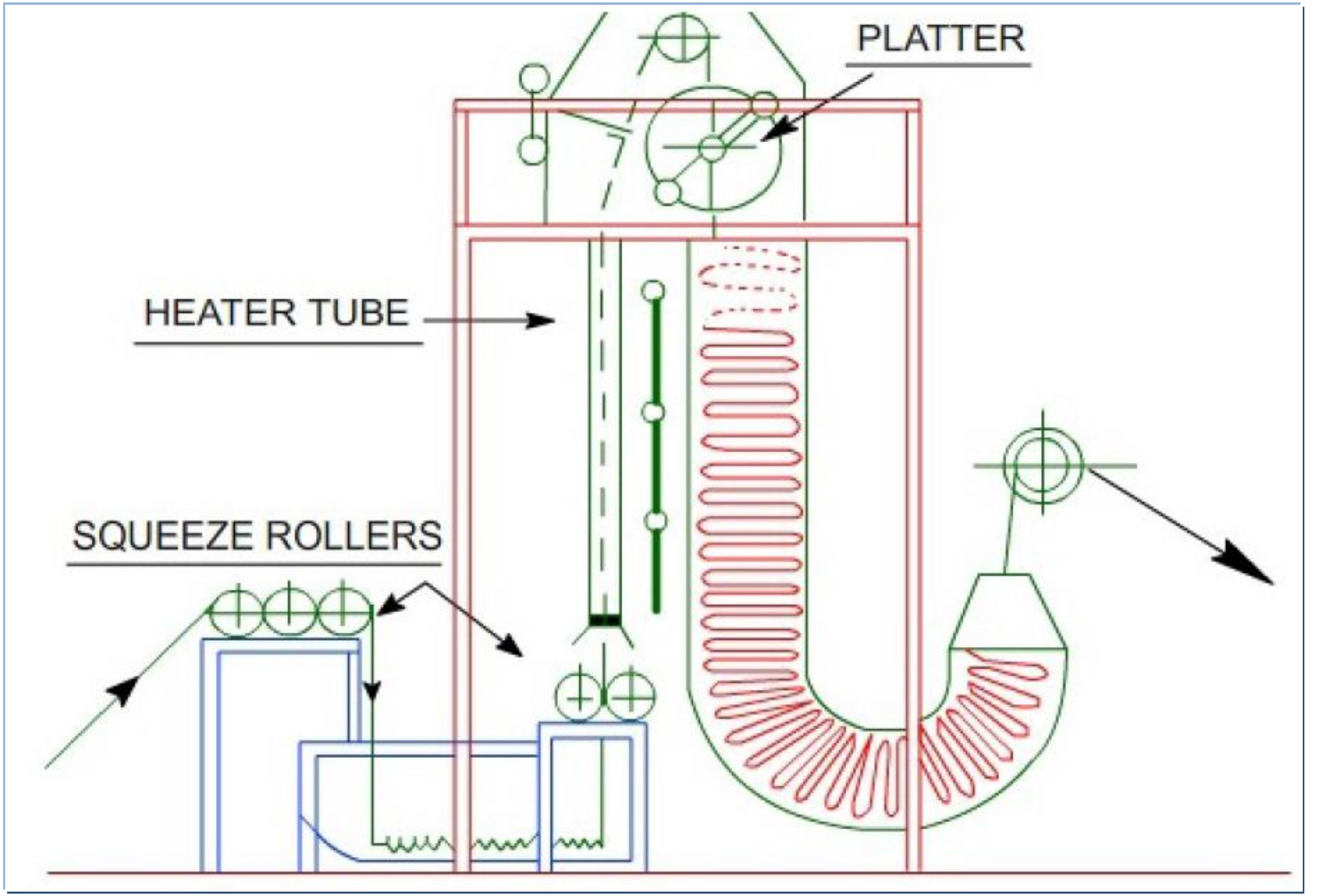
Pressure kier (batch system)



J-Box (Continuous System)

- It is a continuous system
- Fabric is fed from one end and the scoured fabric comes out from the other end
- The capacity is such that sufficient residence time, allowed inside the machine for degradation and removal of impurities
- Inside temperature: 100 ^{o}C
- Saturation of fabric with recipe before treatment in the J-Box
- Time: 40 – 60 min

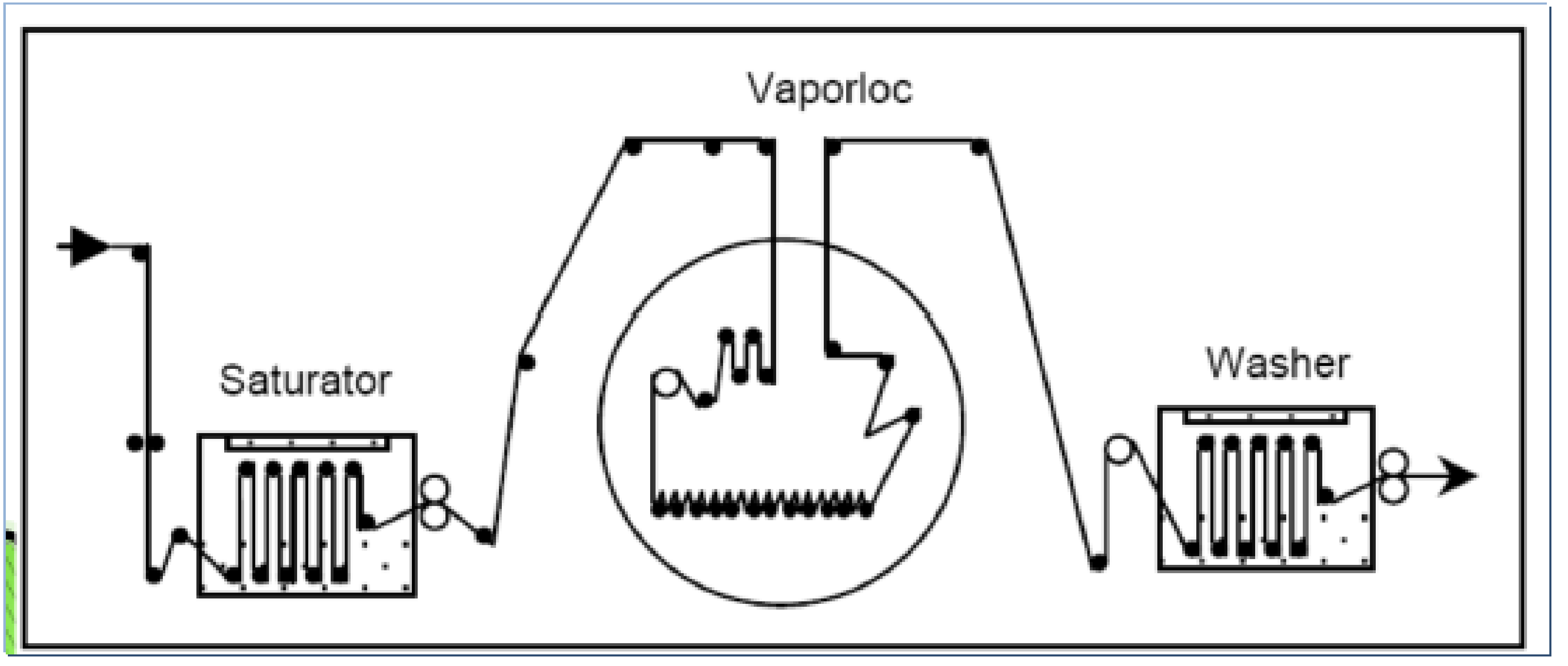
J-Box (Continuous System)



Vapor lock system (Continuous System)

- A continuous system, pressure maintained inside the system
- Shorter treatment times at higher processing speeds
- Saturation of fabric with NaOH + Wetting agent solution
- Pressure inside the chamber: 30 lb/in^2
- Temperature: 134 ^oC
- Time: 90 – 120 sec

Vapor lock system (Continuous System)



Assessment of scouring efficiency

Assessment of scouring efficiency

- Practical tests of absorbency

- Measurements of:

- ✓ Weight loss
- ✓ Protein content
- ✓ Residual wax content
- ✓ Methylene blue absorption (removal of pectic substances)

Test of Absorbency

Drop test:

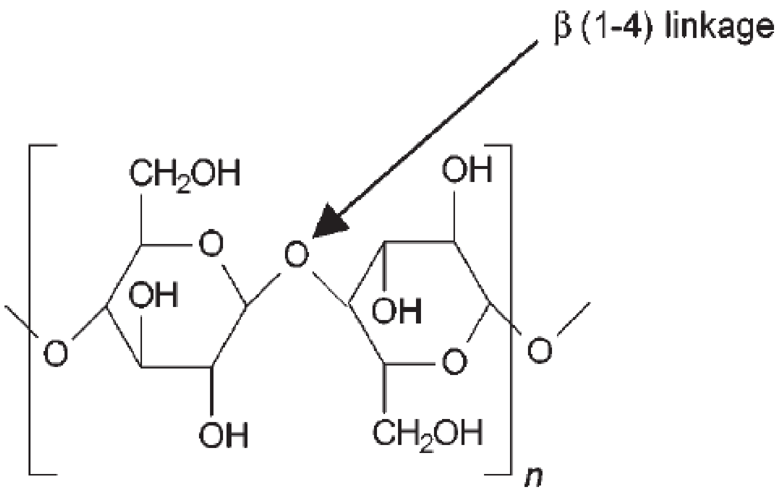
- Water drops are allowed **to fall by gravity from a burette placed at a certain height from the fabric** surface

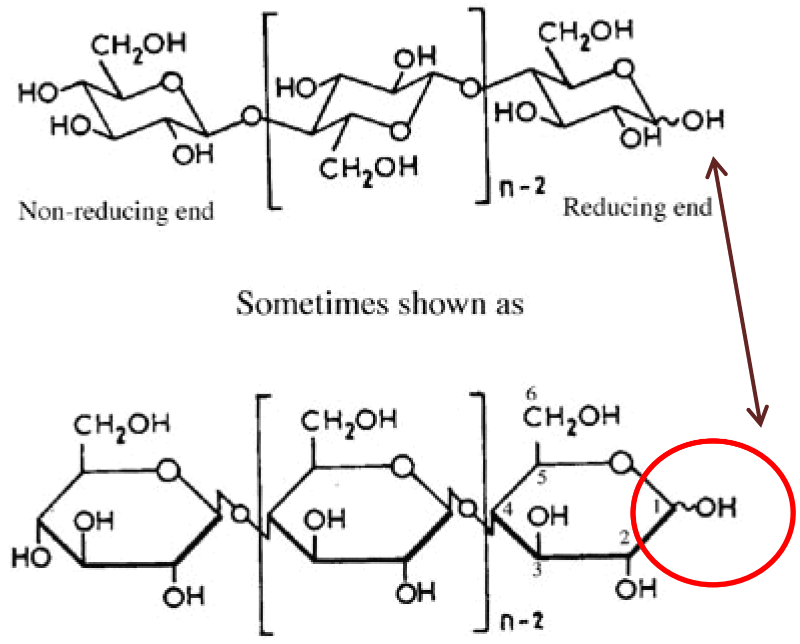
- The fabric is placed straight on a table **without any creases**

- The time required for the drops to collapse is noted as **wetting time**

Degradation of Cotton

Cotton Structure:



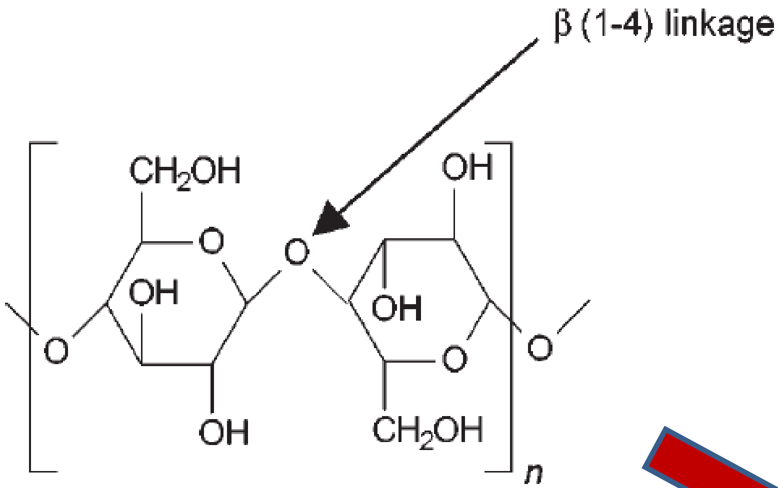


Cellulose

Cellulose molecule contains three different kinds of units:

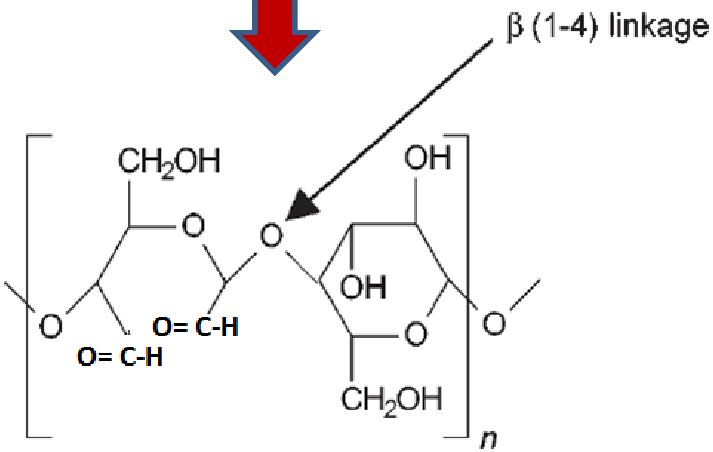
- Reducing end with a free hemi-acetal (or aldehyde) group at C-1,
- Non-reducing end with a free hydroxyl at C-4,
- Internal rings joined at C-1 and C-4.

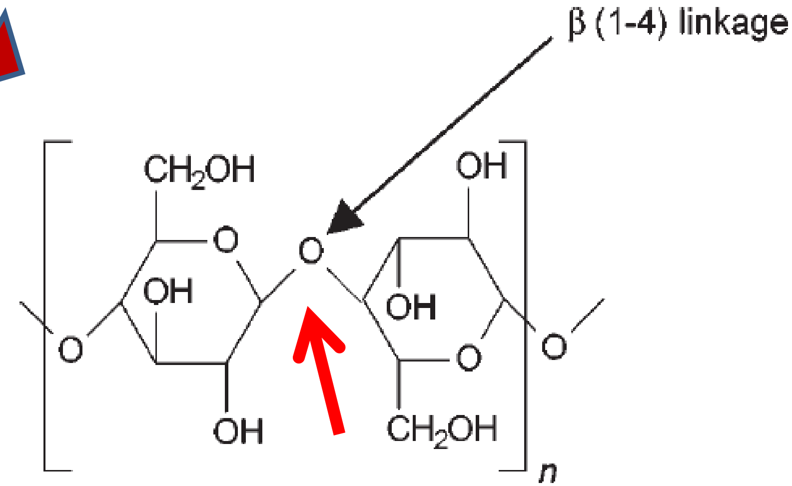
Way of degradation of cotton



Degradation results in creation of:

- aldehyde groups (-CHO) /
- acidic group (COOH)





Assessment of degradation of cotton

- ***Copper number,*** *a measure of the reducing groups present in cellulose*

- **Cuprammonium fluidity,** which is a measure of molecular chain length of cellulose.

- **Tensile strength** of the cotton material before and after scouring.

Copper Number

Copper Number, defined as grams of ***cupric copper reduced to*** **cuprous oxide** by 100 g of cellulose under standard conditions of boiling in alkaline medium.

- ✓ The formed **cuprous oxide** is dissolved in a solution of **iron alum and sulphuric acid** for reducing an equivalent amount of **iron to the ferrous state**.

- ✓The **reduced iron** is then determined by titration with a standard solution of **ceric ammonium sulphate**.

  (Ortho ferrous phenathroline is used as an indicator)

Copper Number

$$\text{Copper number} = \frac{63.5 \times V \times N \times 100}{W \times 100}$$

- V is the ml of ceric ammonium sulphate solution consumed after deducting blank reading
- N is the normality of ceric ammonium sulphate solution
- W is the weight of the bone dry cellulose sample

Copper Number

Substate	Copper number
Pure cellulose	~ 0.05
Raw cotton	~ 0.9
Well-scoured and bleached cotton	> 0.3
Regenerated fibre	> 1.2

Cuprammonium Fluidity

Cuprammonium fluidity is a measure of molecular chain length of cellulose.

(By measuring the fluidity of cotton material dissolved in cuprammonium hydroxide solution)

- ❑ The degradation, in terms of reduction of the degree of polymerization, can be assessed by measuring fluidity.

- ❑ The DP of a polymer is directly proportional to the viscosity of its solution.

Substrates	Fluidity
Unscoured / Unbleached Cotton	2
Scoured / Bleached Cotton	5 or less
Unbleached viscose	10

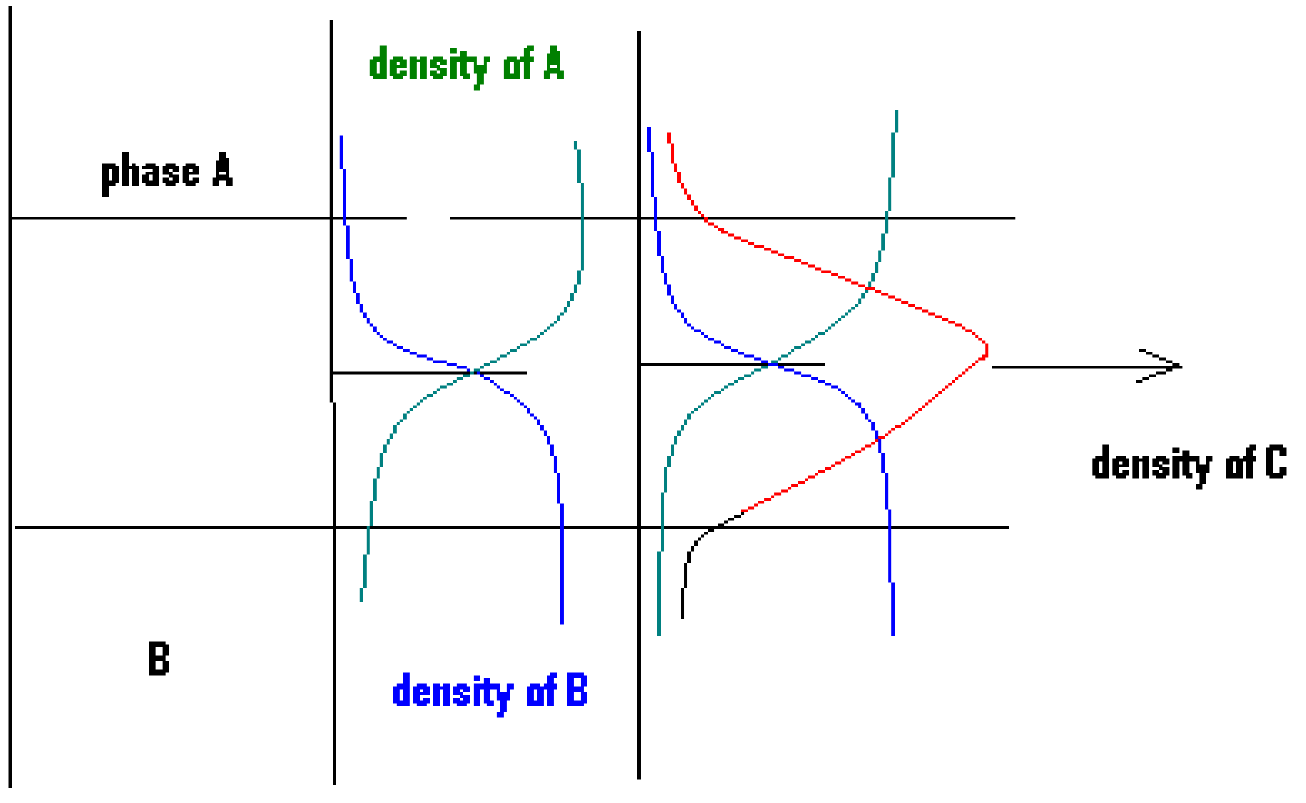
Surfactants

Basic concept of Surfactants

- Surfactants are organic molecules with amphiphilic nature.

- Have a **hydrophobic portion** (generally a long fatty chain) and a **hydrophilic head** (an anion or a cation and sometimes an ethylene oxide chain).

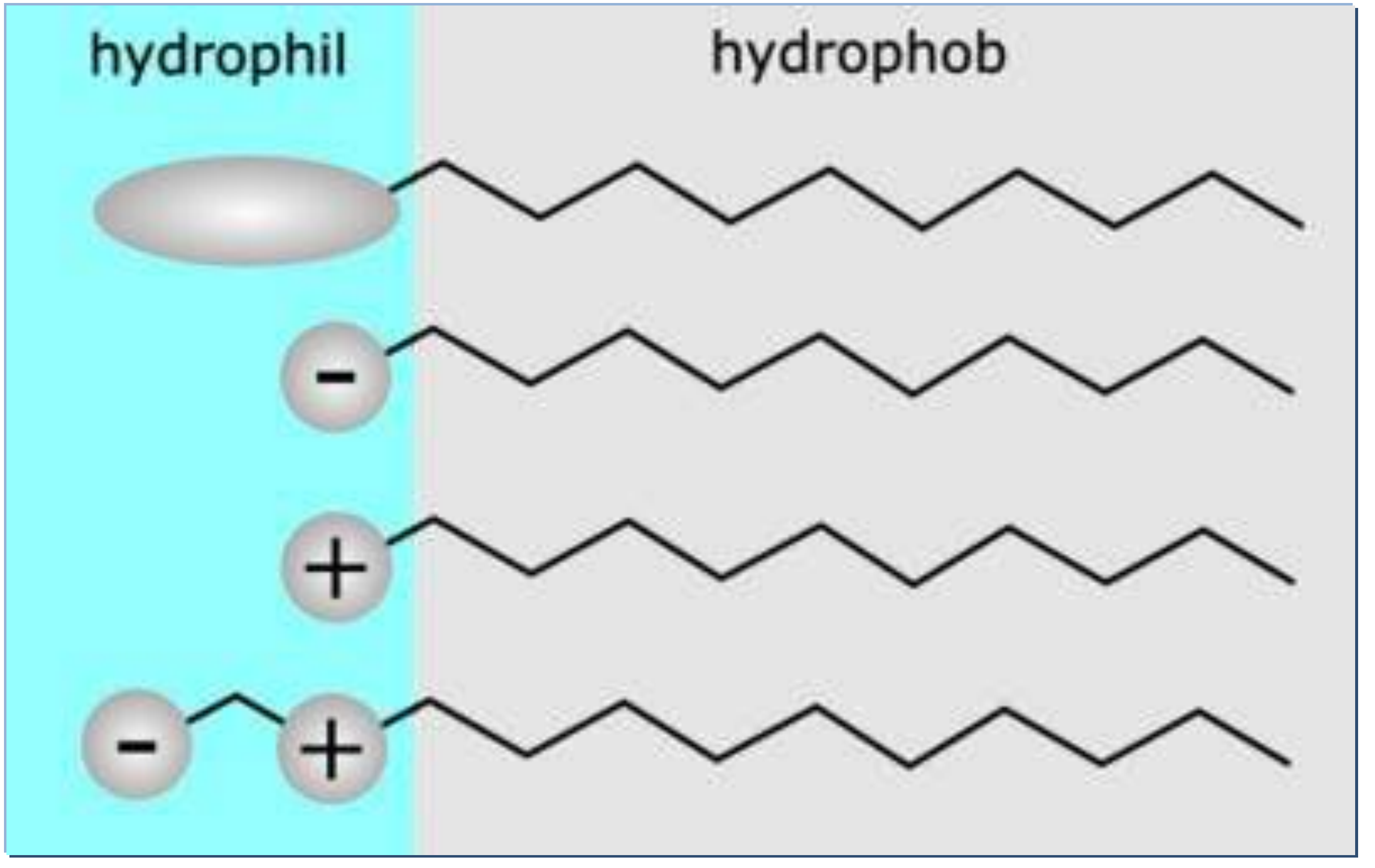
- Due to its dual nature a surfactant tries to occupy space in the boundary of a hydrophilic & a hydrophobic medium.



Relative densities of materials at the interface between two bulk liquids is shown by green and blue curves. ‘C’ is interfacially active component.

A surfactant (surface active agent) lowers the equilibrium interfacial tension between the medium in which it is dissolved and any other contacting fluid.

Structure of Surfactant



Hydrophobic tail is compatible with a hydrophobic medium while the head is compatible with a hydrophilic medium

One of the most important tasks a surfactant performs is <u>reducing the surface tension of the medium in which it is dissolved.</u>

✓ This helps the treatment liquor to wet the surface to be treated in a more effective way.

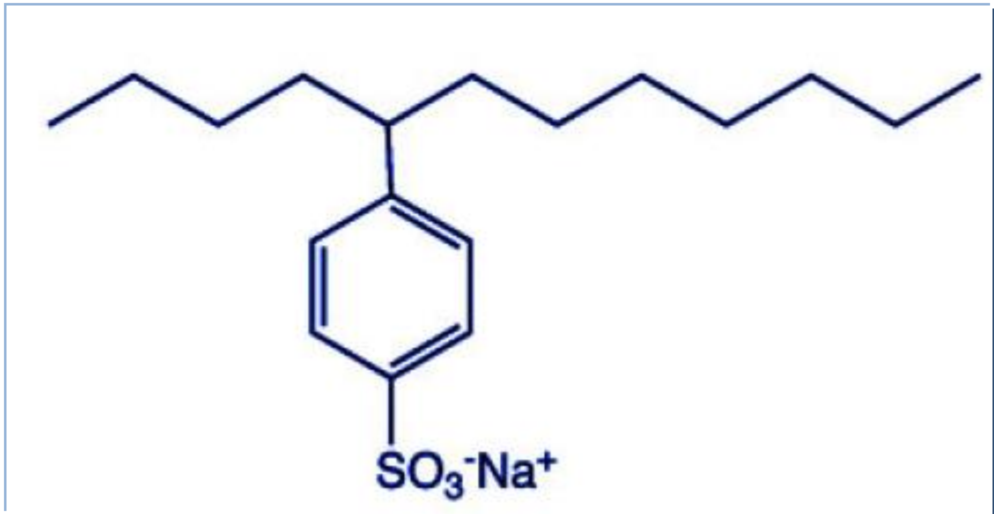
- Desized cotton fabric is hydrophobic and repels water. If one tries to scour it with a caustic solution, the wetting is not good

- Unless the scouring liquor wets the fabric properly, scouring will not be efficient

- However, if a wetting agent is added to the scouring liquor, it is able to wet the fabric suitably

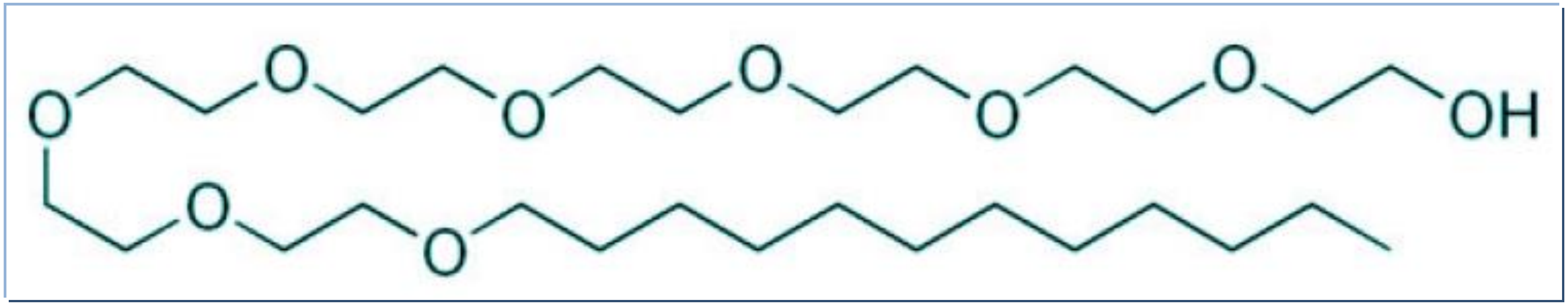
Surfactants: Example

Cetyl (C16), the most common component of most soap, anionic surfactant



4-(5-Dodecyl) benzenesulfonate, a common anionic surfactant

Non-ionic Surfactant

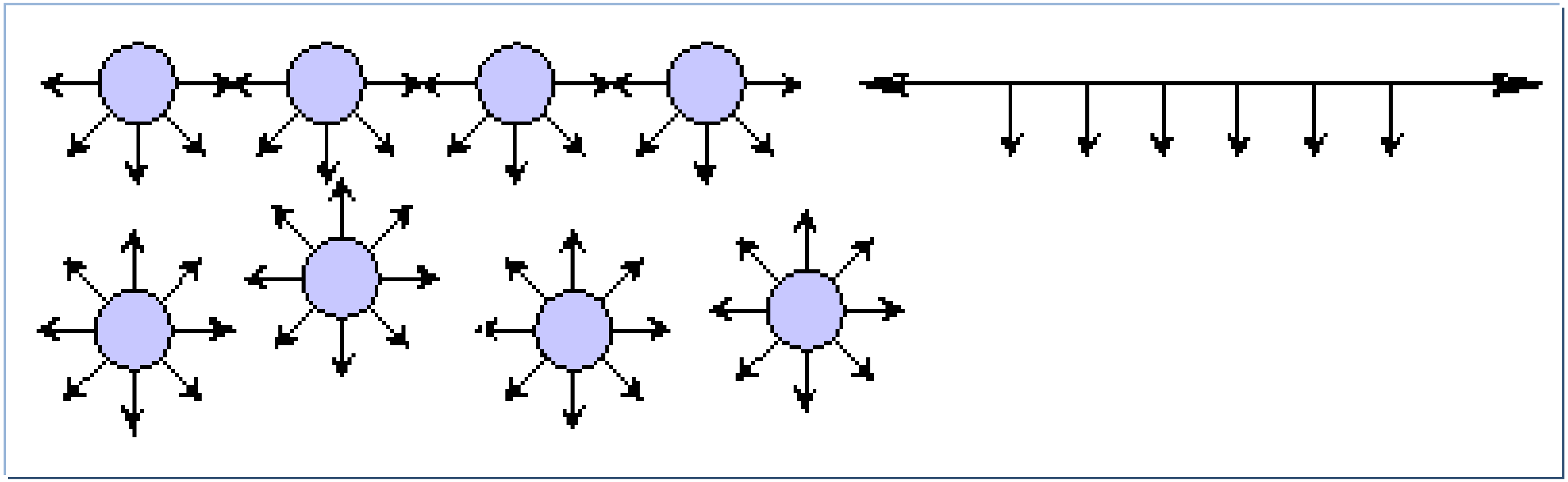


Octaethylene glycol monododecyl ether ($C_{12}E_8$)

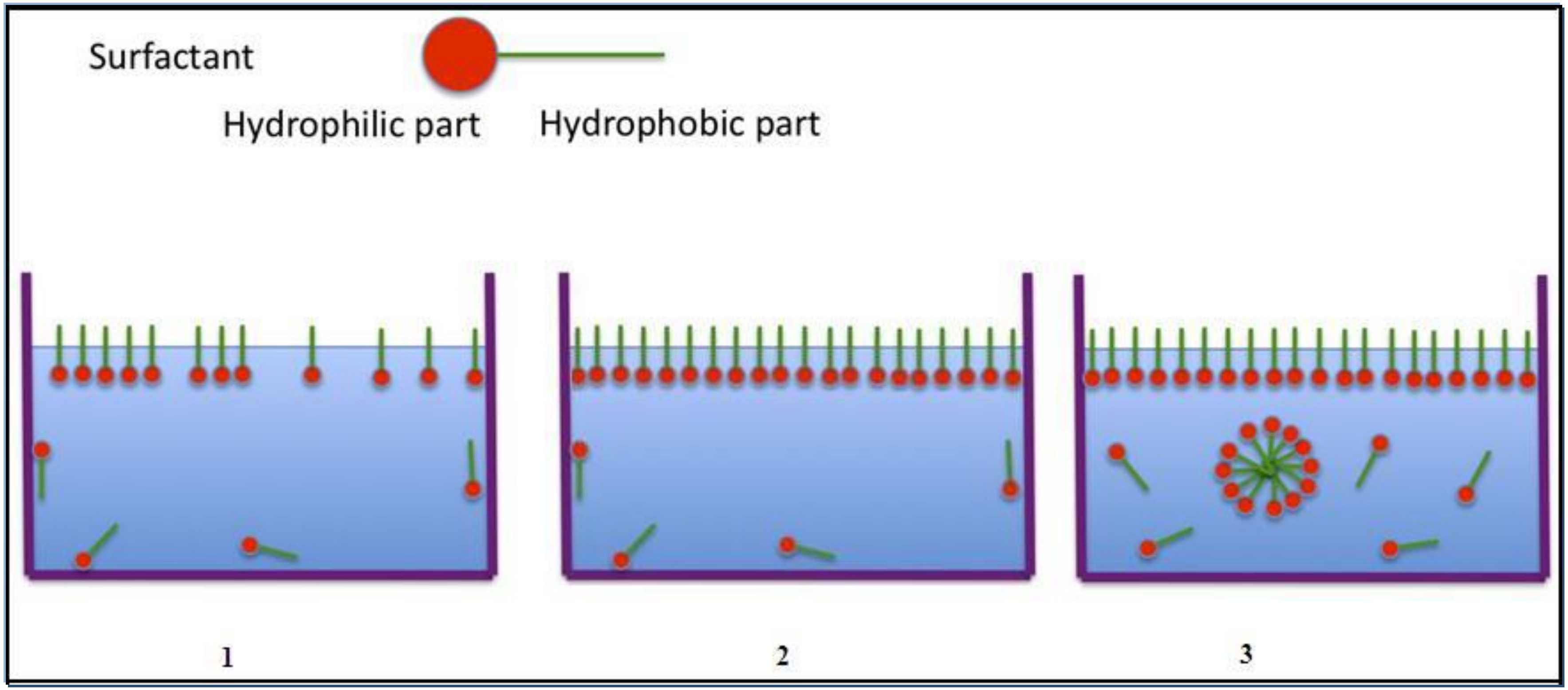
Surface tension reduction in presence of Surfactant

When a surfactant is added to the water, the surfactant molecule invariably goes to the air-water interface to replace the water molecules.

✓ This reduces the net pull on top layer of the water molecules & hence surface tension of water goes down.



Surfactant Aggregation in Solution



How are the impurities removed ?

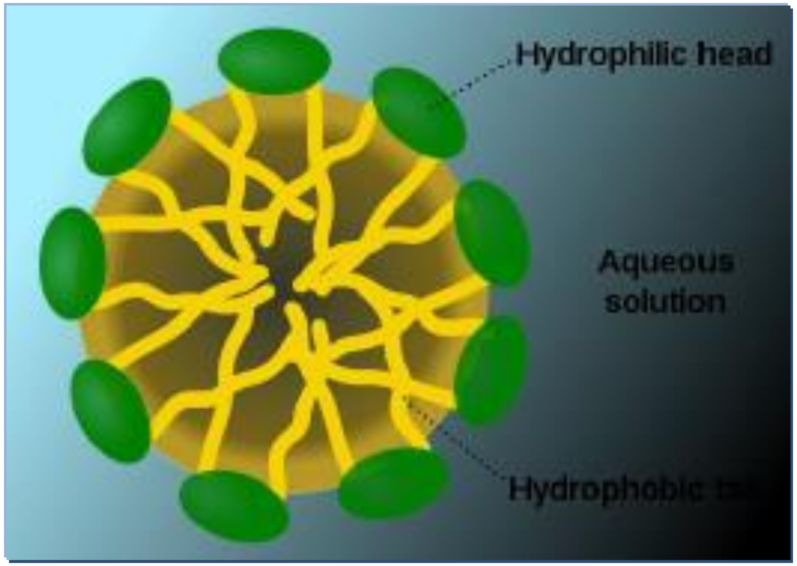
If surfactant is dissolved in water, its molecule tend to come to **air-water interface in such a way that hydrophobic fatty chain is oriented outwards in the air(1), while the hydrophilic head remains submerged in water.**

- As more surfactant is added in water, it keeps moving to the surface (2) up to saturation.

- The extra surfactant molecules move in the bulk and form **microstructures to shield the hydrophobic fatty chain from water (3).**

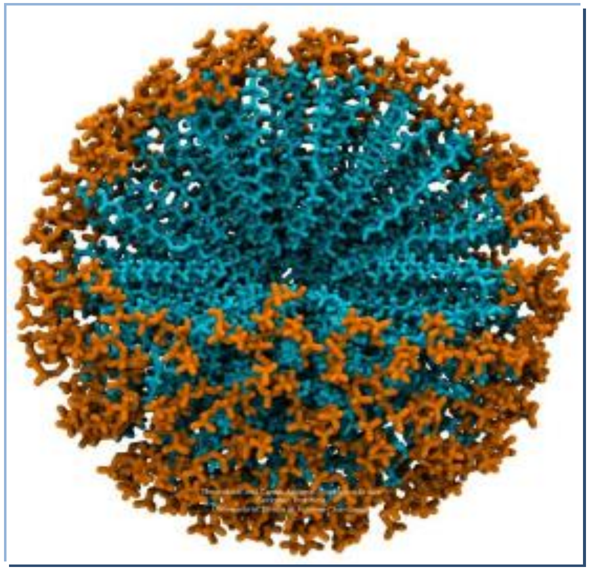
CMC:

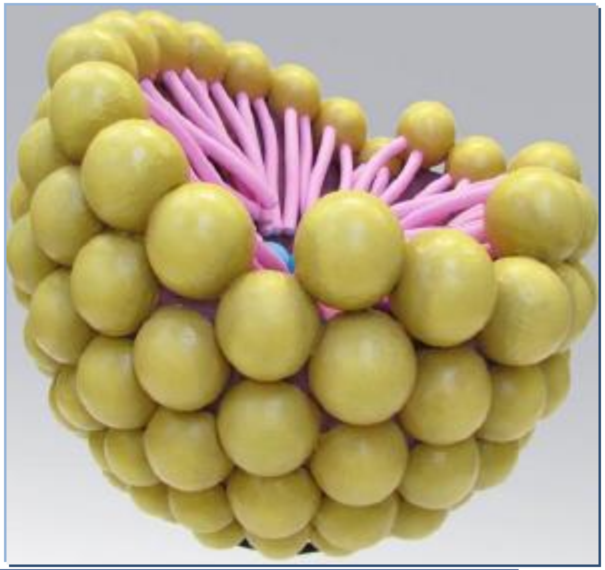
The concentration at which the micelles or aggregates start forming is known as critical micelle concentration (CMC).



- Large no. of monomers (50-100) assemble to form a closed aggregate.

- Non-polar parts shielded from water.



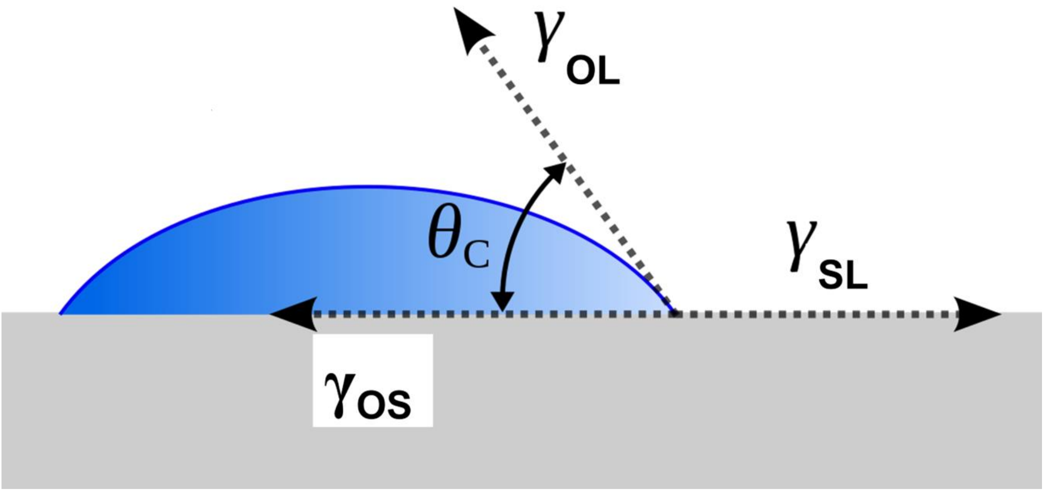


Mechanism of Impurities Removal

- Roll-up
- Emulsification
- Solubilization

MECHANISM OF IMPURITIES REMOVAL

(1). Roll-up:



A drop of oil on a substrate is put in an liquid medium.

❖ The forces acting on the droplets are:

Υ_{OS} : (surface tension at oil solid interface)
Υ_{SL} : (surface tension at solid liquid interface)
Υ_{OL} : (surface tension at oil liquid interface)

At equilibrium these forces are balanced as: $\boxed{\Upsilon_{SL} = \Upsilon_{OS} + \Upsilon_{OL}\cos\theta}$

Roll-up: Mechanism of impurities removal………

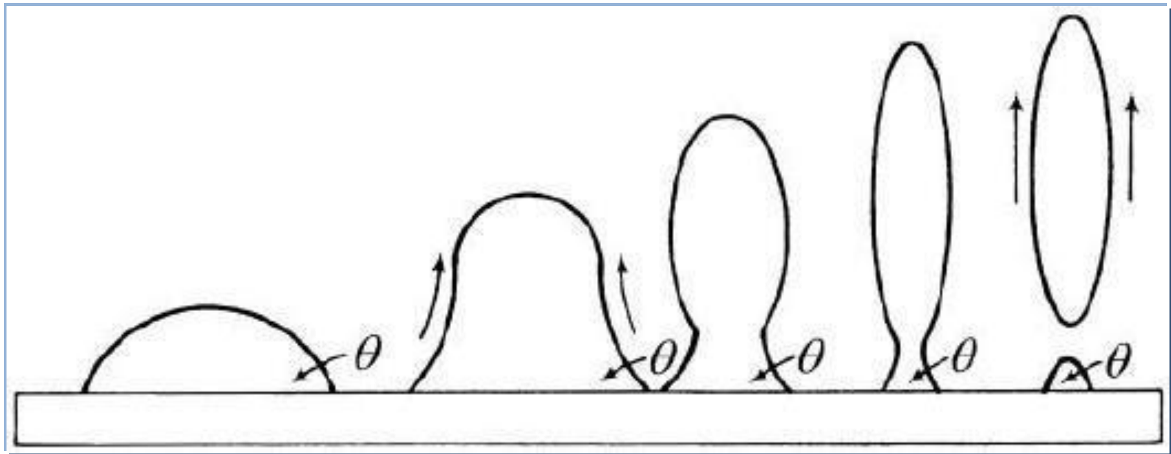
- When a surfactant is added, γ_{SL} goes down which causes θ to increase.

- The net result is the tendency of the drop to become rounder by roll-up.

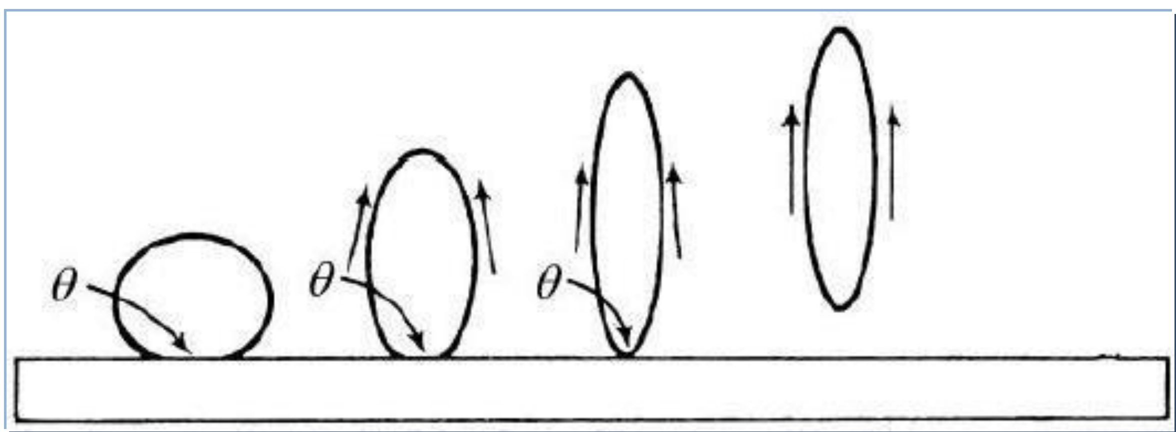
- With sufficient mechanical agitation, the droplet may become spherical and get detached from the original drop, leaving behind some residual oil.

- It is important to keep the drop in suspension to prevent it from re-deposition.

Roll-up: Mechanism of impurities removal……



With mechanical agitation only (θ –constant)



Due to surfactant-(θ keeps increasing)

(2). Emulsification: Mechanism of impurities removal

- ❖ An emulsion is a dispersion of a liquid in another liquid in which it is not soluble.

- ❖ When two immiscible liquids are mixed & shaken vigorously, one of them breaks into small droplets and gets dispersed in another.

- ❖ However, such a dispersion is thermodynamically unstable hence the liquids again form two distinct continuous phases.

The way of dispersion of one liquid phase in another becomes stable, which is known as emulsion

Emulsification: Mechanism of impurities removal……

- An emulsifier is a surfactant whose molecules cover the surface of two droplets.

- One portion of the surfactant molecule becomes compatible with one phase and the other part with second phase.

- This way of dispersion of one liquid phase in another becomes stable, which is known as emulsion.

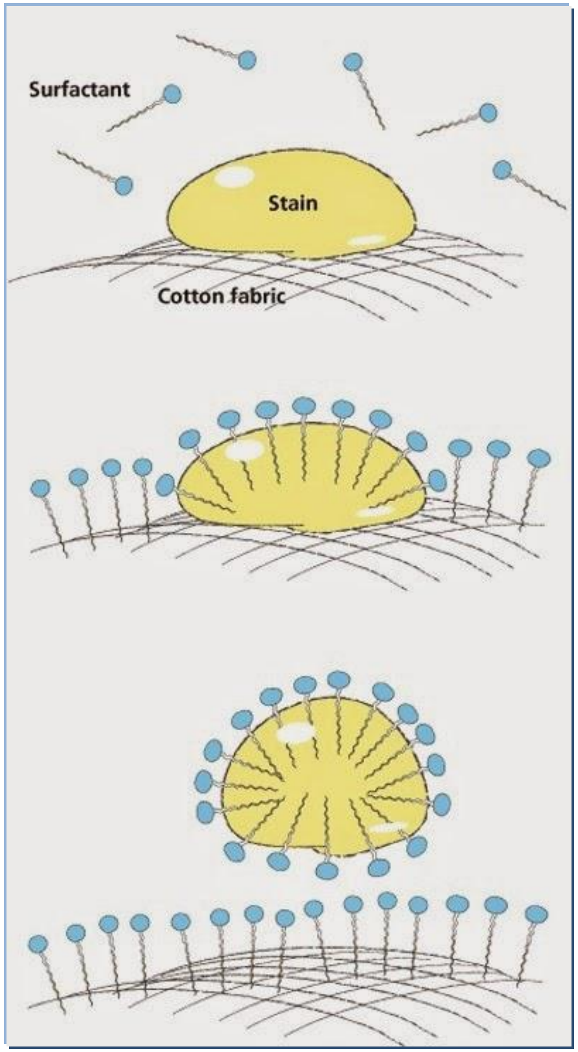
(**Milk is a common example of emulsion of fat in water**)

Emulsification: Mechanism of impurities removal……

- In an emulsion, the size of dispersed droplets is not defined and may vary to a large extent.

- Removal of hydrophobic impurities from textiles using surfactant in an aqueous medium by emulsification is a common mode.

- Agitation helps and is provided either by movement of goods, liquor or both.



(3). Solubilization: Mechanism of impurities removal

The hydrophobic impurity may be removed by solubilization also.

Two Ways:

(a) Using a surfactant (above CMC)

(b) Solubilization using a solvent

(3a). Using a surfactant above CMC

❖ When the micelle traps the hydrophobic soil in its central cavity, it is removed by solubilization.

❖ It is similar to emulsification; however, the disposed droplet size is almost constant and is almost equal to the cavity inside micelle. Such system can be treated as one phase system.

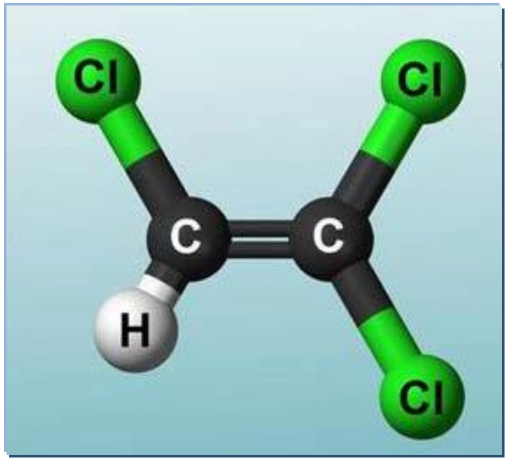
(3b). Solubilization using a solvent

Two approach:

Using a solvent to dissolve away the hydrophobic soil. For example, common dry cleaning uses organic solvents to remove hydrophobic greasy or oily soils from textile materials. At industrial scales, this has safety & logistical problems related to handling of large amount of organic solvents.

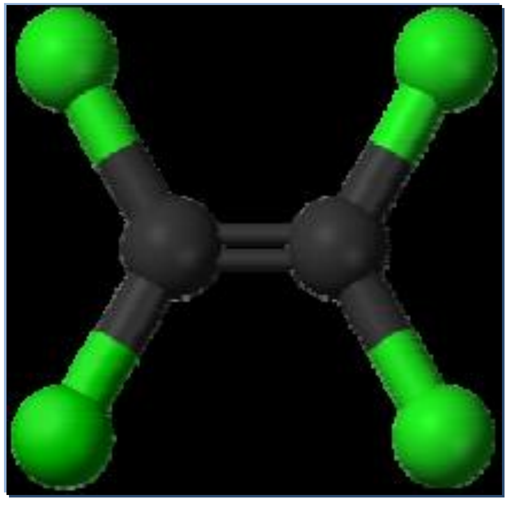
A solvent may be emulsified in an aqueous medium and the resultant emulsion, which may contain only a small amount of solvent may be used to remove the oily soil.

Trichloro ethylene
(B.P. : 87 ^{o}C, Non-flammable)



Trichloroethylene (TCE)

Perchloro ethylene
(B.P. : 121^{o}C, Non-flammable)



Perchloroethylene (PCE)

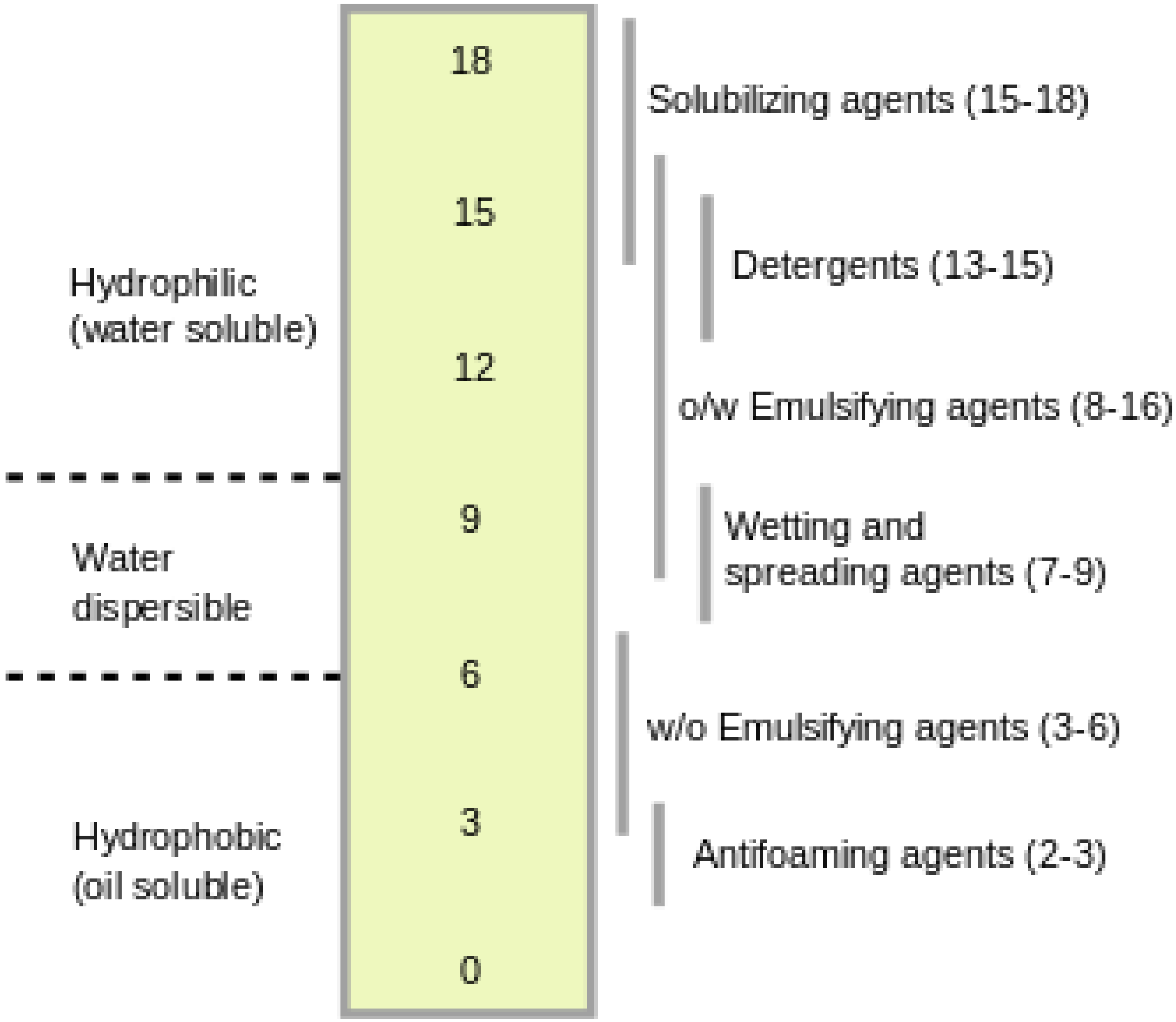
Concept of HLB value

For surfactants & oils/fats/waxes, etc.

HLB stands for **hydrophile lipophile balance**. It indicates relative fraction of hydrophilic and hydrophobic components in a surfactant, oil, solvent, etc.

HLB Value	Characteristic
1-4	Not miscible in water
4-8	Dispersible in water
8-12	Milky emulsion in water
>12	Soluble in water

Concept of HLB value



HLB value………..

- Silicon oil, HLB –9
- Kerosene oil, HLB –12

❖ For emulsification of a particular impurity, a surfactant of similar HLB value is selected.

- Either single component.
- Combination of two different HLB surfactants.

For scouring 13 - 13.5% HLB value surfactants are used.

Determination of the Required HLB values
and
Blending of Surfactants

Oils used in the formulation of emulsions require a certain HLB value to be formulated as:

Either, **w/o emulsion** or **o/w emulsion**.

For the same oil, the required HLB value for O/W emulsion is ***higher*** *than the required HLB value for W/O emulsion.*

Oil	O/W emulsion	W/O emulsion
Stearic acid	15	6
Cetyl alcohol	15	-------------
Stearyl alcohol	14	-------------
Lanolin, anhydrous	12	8
Mineral oil, light	12	4
Liquid paraffin	10.5	4
Castor oil	14	-------------
Beeswax	9	5
Petrolatum	7-8	4
Wool fat	10	8

Calculation of the required HLB for a mixture of oils, fats or waxes.

- **Multiply the required HLB of each ingredient by its fraction from the total oily phase.**

- **Add the obtained values to get the total required HLB for the whole oily phase.**

Example:

Problem

Liquid paraffin:	**35%**	HLB (liquid paraffin): 10.5
Wool fat:	**1 %**	HLB (wool fat): 10.0
Cetyl alcohol:	**1%**	HLB (cetyl alcohol): 15.0
Emulsifier system :	**7%**	
Water to	**100%**	

What is the total required HLB no.?

The total percentage of the oily phase is 37.

The proportion of each is:

- Liquid paraffin: 35/37 x 100 = 94.6%
- Wool fat: 1/37 x 100 = 2.7%
- Cetyl alcohol: 1/37 x 100 = 2.7%

The total required HLB number is obtained as follows:

Liquid paraffin (HLB 10.5) 94.6/100 X 10.5 = 9.93
Wool fat (HLB 10) 2.7/100 x 10 = 0.3
Cetyl alcohol (HLB 15) 2.7/100 X 15 = 0.4

Total required HLB = **10.63**

Calculation of ratio of emulsifier to produce a particular required HLB value

One of the most important aspects of the HLB system is that HLB values are additive if the amount of each in a blend is taken into account.

Thus, blends of high and low HLB surfactants can be used to obtain the required HLB of an oil.

The HLB of the mixture of surfactants, consisting of fraction x of A and (1-x) of B is assumed to be the algebraic mean of the two HLB numbers,

i.e.:

$$\textbf{HLB mixture} = x\,\text{HLB}_A + (1-x)\,\text{HLB}_B$$

Rearrangement of the above equation in percent (%) form will be:

$$A = 100\,(X-\text{HLB}_B) / (\text{HLB}_A - \text{HLB}_B)$$

$$B = 100 - A$$

Where, X is the required HLB of the surfactant (oil) mixture

PROBLEM

A formulator is required to formulate an o/w emulsion of the basic formula:

Liquid paraffin:	50 g
Emulsifying agents (**required HLB 10.5**):	5 g
Water to:	100 g

Calculate the fraction of Tween 80 (HLB of 15) and Span 80 (HLB of 4.3) used to produce a ***physically stable liquid paraffin emulsion.***

Solution

Assume that Tween 80 is A and Span 80 is B.

$A = 100\ (X - HLB_B)\ /\ (HLB_A - HLB_B)$

$= 100\ (10.5 - 4.3)\ /\ (15 - 4.3)$

$= 57.9\%$

$B = 100 – A$

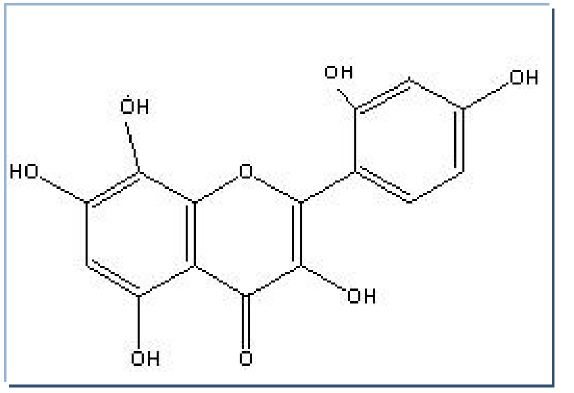
$= 100 - 57.9 = 42.1\ \%$

$A = (57.9 \times 5) \times 100 = 2.89$ g

$B = 5 – 2.89 = 2.11$ g

Bleaching

Introduction to bleaching



The source of the colour:

- ❖ Flavone pigment in case of cotton
- ❖ Soil and dirt acquired from atmosphere
- ❖ Contact with plant parts and seeds, etc.
- ❖ Colour acquired during mechanical processing e.g. grease, oil, etc.

Objectives

To destroy the natural or acquired coloring materials to bring the textiles in a white state.

❑ **This may be necessary when:**

- The fabric has to be supplied in white colour
- It needs to be dyed in pastel shades
- It needs to be printed

For dyeing in dark shades –it is an optional process

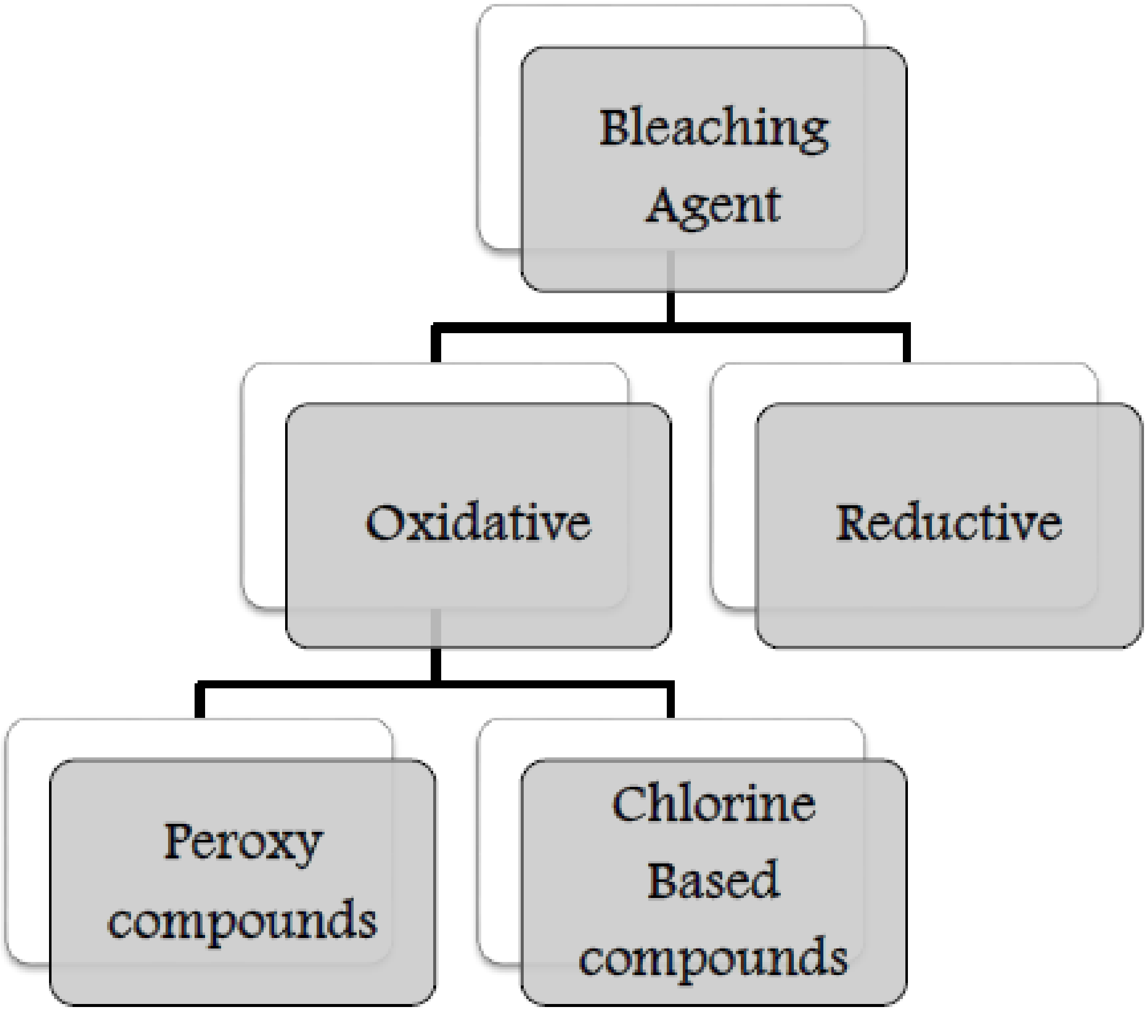
❑ **Additionally:**

❖ To destroy the motes or the seed coat fragments

❖ To remove residual impurities left by other pretreatment processes like desizing, scouring, etc.

❑ **Increased absorbency**

Classification of Bleaching Agents



Different Bleaching Agents

Peroxy Oxidative Bleaching Agents	Chlorine based Oxidative Bleaching Agents	Reductive Bleaching Agents
Hydrogen peroxide	Bleaching powder	Sulphur dioxide
Potassium permanganate	Sodium hypochlorite	Sodium hydrosulphite
Peracetic acid	Lithium hypochlorite	Acidic sodium sulphite
	Sodium chlorite	Sulphoxylates

Main Bleaching Agents

- ✓ **Sodium hypochlorite (NaOCl)**
- ✓ **Sodium chlorite ($NaClO_2$)**
- ✓ **Hydrogen peroxide (H_2O_2)**

Sodium Hypochlorite (NaOCl)

- NaOCl is sodium salt of Hypochlorus acid (HOCl) having redox potential of 1400-1550 mV.

- Strongest oxidizing agent

❑ NaOCl solution in water undergoes the following reaction:

$NaOCl + H_2O \longrightarrow NaOH + HOCl$ (**Weak acid)**

(pH of 11-11.5)

❖ Bleaching occurs due to the following reaction:

$HOCl \longrightarrow HCl + [O]$

Sodium Hypochlorite (NaOCl)

•What happens in acidic pH?

In acidic medium the consumption of hypochlorite is very fast and can lead to cellulose degradation by oxidation. The reaction is given below:

$$NaOCl + HCl \longrightarrow NaCl + HOCl$$

$$HOCl + HCl \longrightarrow H_2O + Cl_2$$

(Excess acid)

Process Parameters:

- pH
- Temperature
- Time

In practice, cotton is bleached with NaOCl solution containing 1 - 3 gpl available chlorine at room temp. in the pH range 9.5 - 11.

Sodium Hypochlorite (NaOCl)

Active Chlorine

- Active chlorine is a unit of concentration used for hypochlorite bleaching

- Active chlorine is determined by adding excess potassium iodide to sample of bleach solution

- Titration of iodine with standard sodium thiosulfate solution

Sodium Hypochlorite (NaOCl): Active Chlorine

ClO^-	+	$2I^-$	+	$2H^+$	$\longrightarrow$	I_2	+	H_2O	+	Cl^-
(Hypochlorite ion)		(Potassium iodide)		(Acidic solution)		(Iodine)				(Common salt)

Similarly,

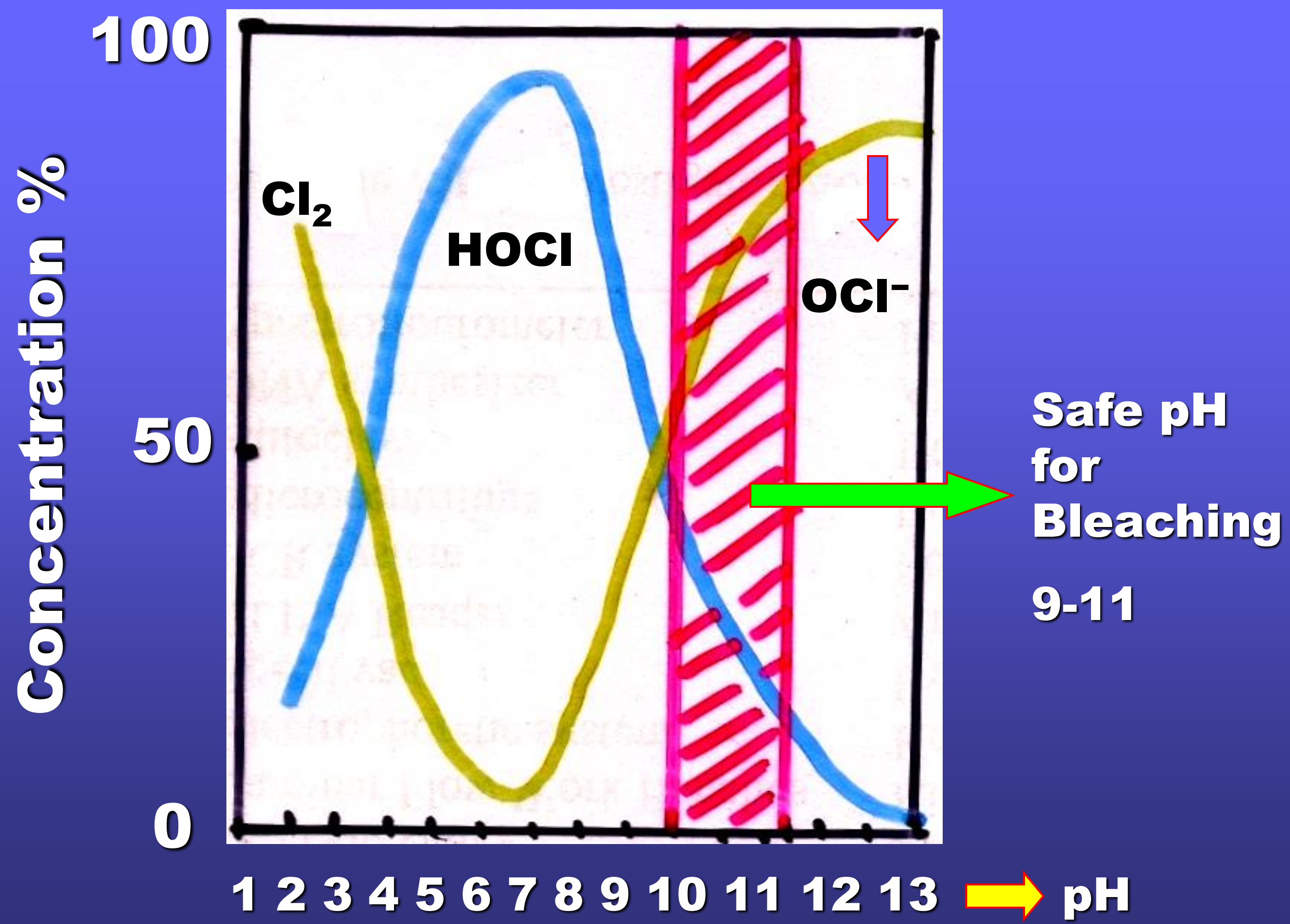
$$Cl_2 + 2I^- \longrightarrow I_2 + 2Cl^-$$

Determination of liberated I_2 by titration:

$$2S_2O_3^{2-} + I_2 \longrightarrow S_4O_6^{2-} + 2I^-$$

2 moles of thiosulfate are equivalent to 70.9 grams (1 mole) of active chlorine or 51.5 g of hypochlorite ions. In other words a 51.5 gpl solution of OCl− (or 75 gpl NaOCl) is equv. to 70.9 gpl solution of available Cl_2.

Bleaching with Sodium Hypochlorite (NaOCl): pH effect



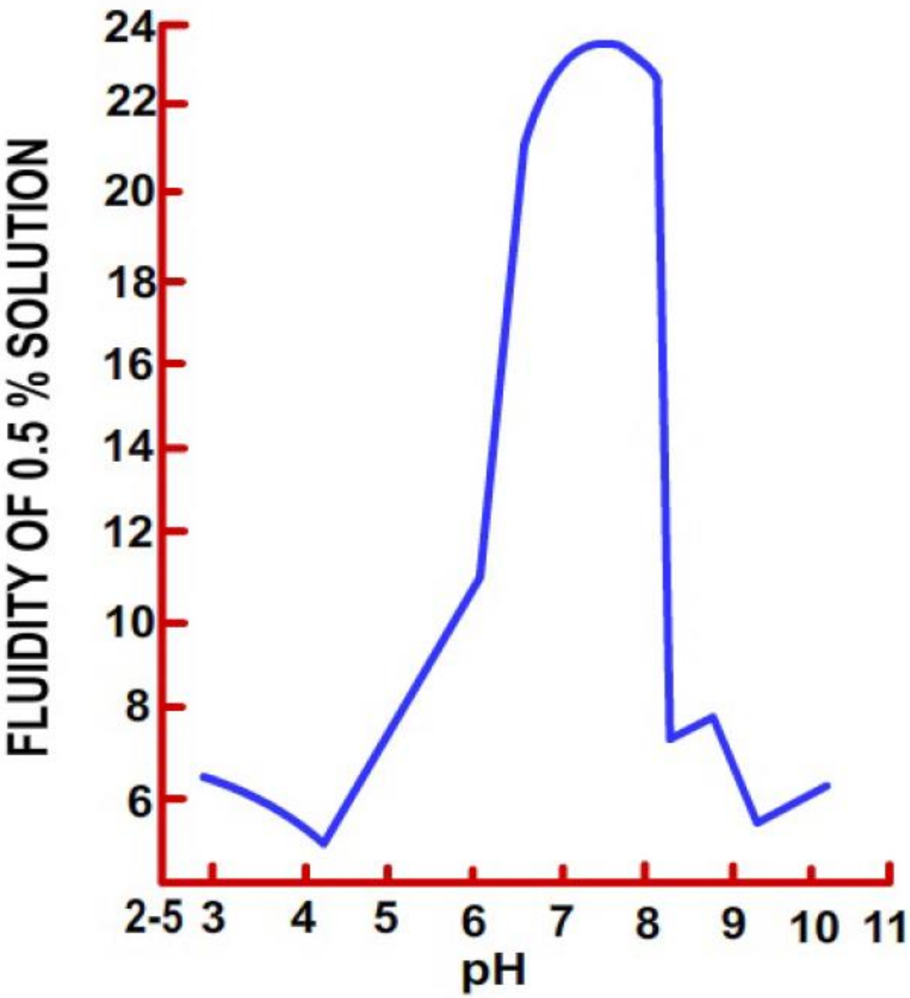
Bleaching with Sodium Hypochlorite (NaOCl): pH effect

- Fraction of HOCl with change in pH
- Between pH 5 to 10, drastic change in concentration of free acid

pH	Fraction of hypochlorite as HOCl
10.0	0.003
9.0	0.03
8.0	0.21
7.43	0.50
7.0	0.73
6.5	0.91
6.0	0.96
5.0	0.997

Free acid (HOCl) with change of pH

Bleaching with Sodium Hypochlorite (NaOCl): pH effect



✓ The degradation of cotton measured by Cupra-ammonium Fluidity test

✓ Maximum degradation is at near pH 7 region

Practical Bleaching Conditions

Parameters	Conditions
pH: 9.0	Time to bleach: 45 min
pH: 11.0	Time approximately: 4 hrs
Temperature	Room temperature
Concentration	2-3 gpl available chlorine

Sodium Hypochlorite Bleaching

Advantages & Disadvantages

Advantages:

- Economical
- Room temperature process

Disadvantages:

- Fibre damage
- Formation of AOX
- Yellowing on storage
- May degrade many dyes and FBAs
- Not used for bleaching of synthetic or protein fibres
- Not very satisfactory white is produced
- Unused NaOCl can form chloroform and affect the sea-life

Antichlor Treatment

Residual hypochlorite is removed by treatment with Reducing agent:

- ❑ 2 - 2.5% of a reducing agent
- ❑ Time: 15 min
- ❑ Temperature: 40 ^{o}C

✓ **Reducing agent:**

Sulphites, bisulphites, hydrosulphites, thiosulphates, etc.

Sodium Chlorite Bleaching

Sodium Chlorite Bleaching

- Fibre-gentle Bleaching Agent
- Redox Potential : 1040 – 1200 mV

$NaClO_2 + H_2O = NaOH + HClO_2$ (pH 8.5)

$HClO_2 = HCl + 2$ <O>

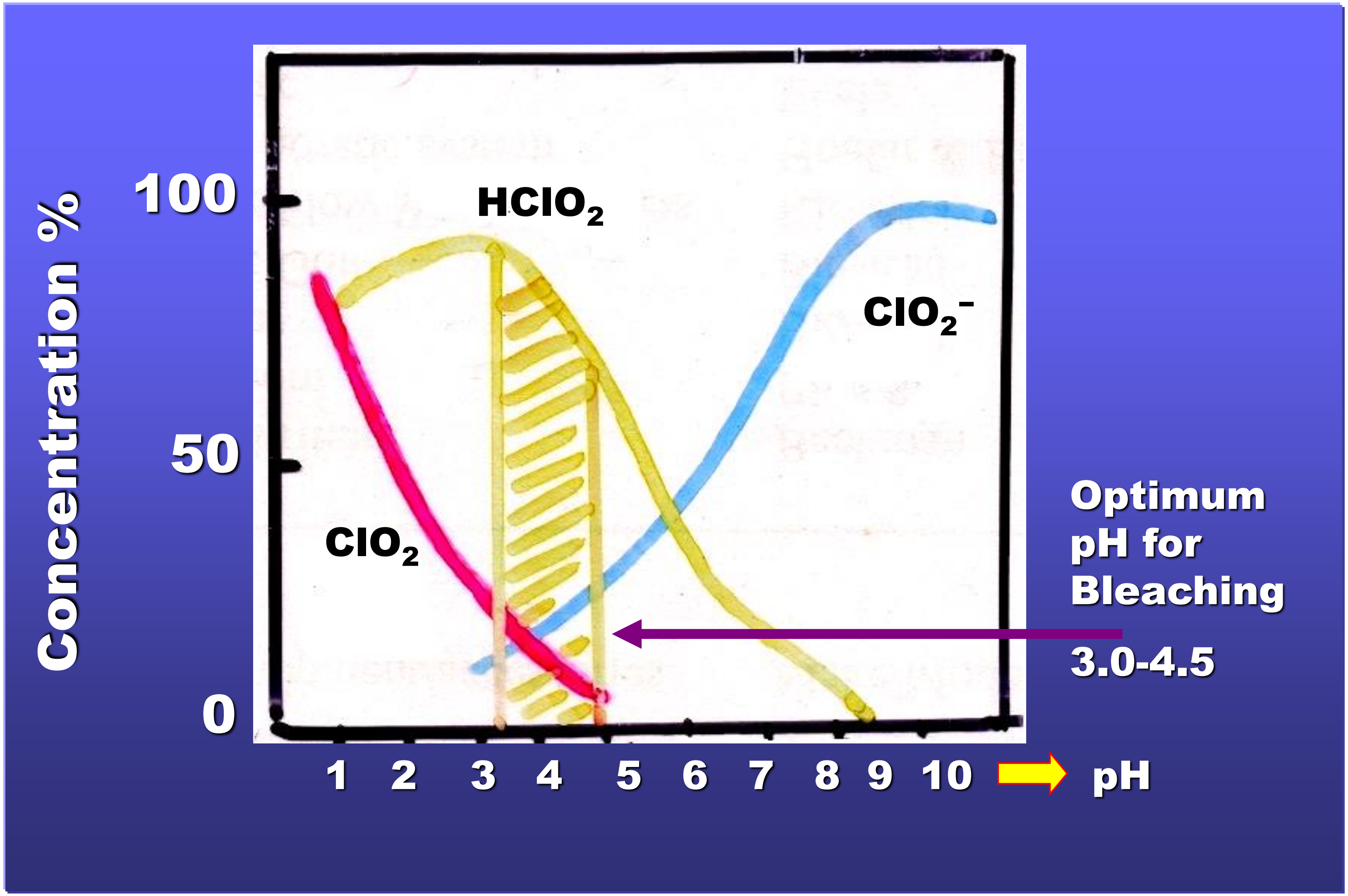
$NaClO_2 + HCl = NaCl + HClO_2$

(pH 2.5 – 3.0)

$$\left.\begin{array}{l} 5\ HClO_2 = 4ClO_2 + HCL + 2H_2O \\ 3\ HClO_2 = 2HClO_3 + HCl \end{array}\right\}$$

(@ Lower pH)

Sodium Chlorite Bleaching



Sodium Chlorite Bleaching

Process Parameters: pH, Temperature, Time

- ❖ Stabilized at higher pH
- ❖ At pH 1-2, evaporation of ClO_2
- ❖ Balanced pH around 3.5 - 4

- ✓ Bleaches at high temperature (80 – 90 ^{0}C)
- ✓ At boil, the bleaching time is a few hours (1- 4)
- ✓ The rate of bleaching doubles for every 10 ^{o}C rise in the process.

Sodium Chlorite Bleaching: Corrosion Problem

ClO_2 is a toxic corrosive gas

- **Corrosion resistant materials**: Stainless Steel with 2.5% Molybdenum, Titanium / Ceramic lining

- **ClO_2 scavenging chemicals**: $(NH_4)H_2PO_4$, sodium and ammonium nitrate, nitric acid, melamine, urea, etc.

- **Chlorite stable surfactants**: foam of the surfactant can trap the ClO_2 gas formed.

Sodium Chlorite Bleaching

Advantages & Disadvantages

Advantages:

- Both cotton and synthetic fibres can be bleached.
- Hardness of water does not impair the process (unlike H_2O_2)
- For knitted fabrics, soft feel is retained
- Good white color with excellent mote removal
- Little or no cellulose degradation

Disadvantages:

- Expensive than NaOCl or H_2O_2
- No silk and wool (yellowish pink color) bleaching possible
- Corrodes the metals
- ClO_2 is a toxic gas
- Wax removal is not very satisfactory

Hydrogen Peroxide Bleaching

Hydrogen Peroxide Bleaching

- An eco-friendly bleaching agent: by-products only water and oxygen
- Redox potential: 810 – 840 mV
- Universal bleaching agent–most of the fibres can be bleached
- Provides permanent bleaching action

H-O-O-H

Hydrogen Peroxide Bleaching

$$H_2O_2 \longrightarrow H^+ + HO_2^- \text{ (pH: ~ 3)}$$

$$2HO_2^- \longrightarrow 2HO^- + O_2$$

Bleaching species: **Perhydroxyl ion (HO_2^-)**

In alkaline medium H_2O_2 decomposes as:

$$H_2O_2 + OH^- \longrightarrow OOH^- + H_2O$$

Above pH 10.8, formation of HO_2^- ions is rapid.

- Stable in acidic pH
- Decomposes in presence of alkalis or UV light.

Hydrogen Peroxide Bleaching: Auxiliaries

Activators:

✓ Stabilized at acidic pH
✓ Unstable in basic pH

• Sodium hydroxide (NaOH) is commercially used as an activator

Stabilizers:

✓ Regulate the formation of perhydroxyl ions formation
✓ Prevent rapid decomposition of bleach bath
✓ Prevent fibre damage

Hydrogen Peroxide Bleaching: Stabilizers

Act by-

- ✓ Buffering action (pH regulation)
- ✓ Sequestering of heavy transition metal ions
- ✓ Complexing with perhydroxy ions

Commonly used Stabilizer: Sodium Silicate

- ✓ Easily available, economical and effective
- ✓ $Na_2O:SiO_2$ ratio (1:1)
- ✓ Stabilizing action of silicates is improved by Ca or Mg ions
- ✓ Water of hardness should be controlled

Hydrogen Peroxide Bleaching: Sequestering Agents

- ❖ Metals or UV light causes **fission of H_2O_2**
- ❖ The rate may be so high that cellulose is oxidized
- ❖ Oxycellulose has poor mechanical properties

To prevent harmful effects of metal cations:

Sequestering agents are often used to minimize the harmful effects of metal cations.

Example:

- Poly phosphonates
- Poly hydroxyl carboxylic acids
- Amino polycarboxylic acids
- Phosphonic acids
- Polyacrylic acids

Hydrogen Peroxide Bleaching: Sequestering Agents

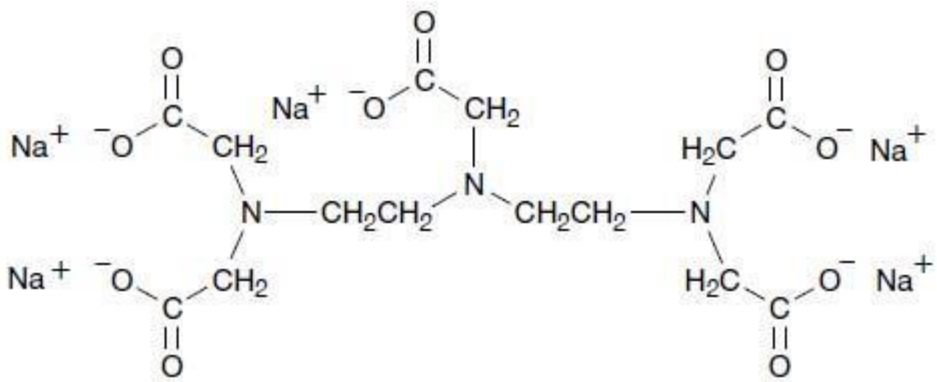
Common sequestering agents:

- Di-ethylene tri-amine penta acetic acid (DTPA)
- Ethylene di-amine tetra acetic acid (EDTA) as sodium or magnesium salts

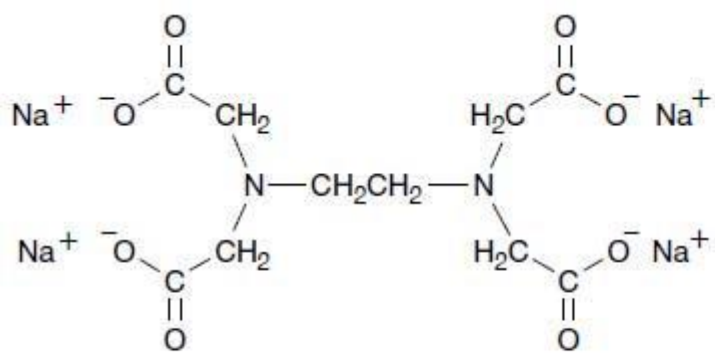
These compounds can form Chelates or metal complexes with metal ions

The complexes formed may have complicated 3-D structures.

Hydrogen Peroxide Bleaching: Sequestering Agents



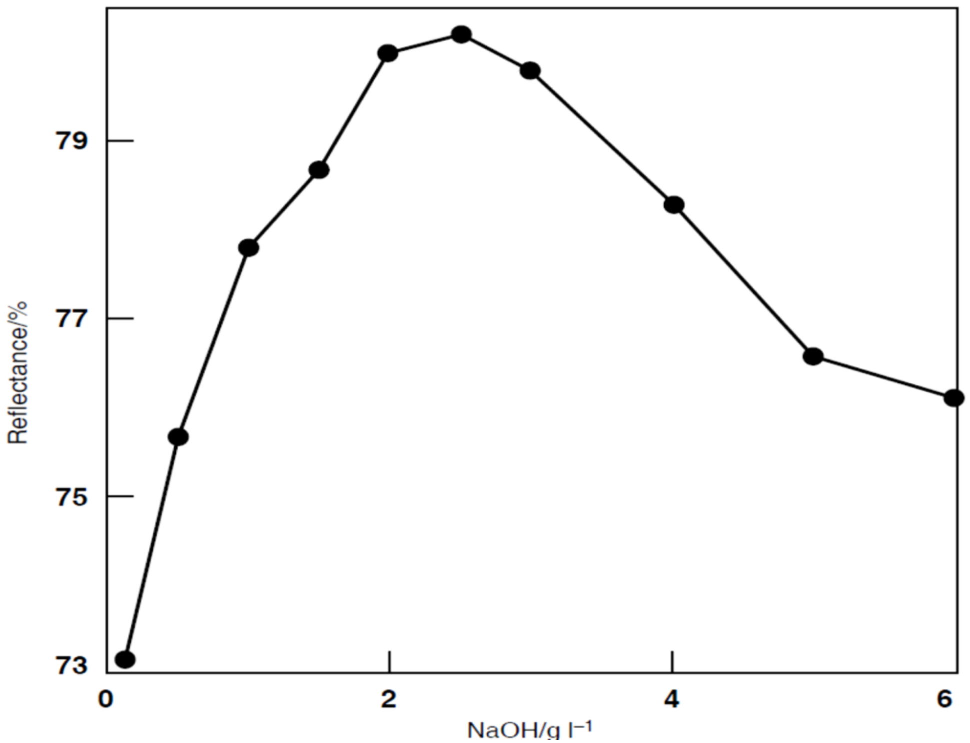
DTPA (Di-Ethylene Tri-Amine Penta Acetic Acid)



EDTA (Ethylene Di-Amine Tetra Acetic Acid)

Hydrogen Peroxide Bleaching: pH effect

In acidic pH, H_2O_2 is stable, so alkaline pH is used for bleaching



Desired pH: 10.5 (cotton)

Effect of concentration of alkali on whiteness

Hydrogen Peroxide Bleaching: Temperature effect

- Bleaching of cotton is carried out at boil (90-100 ^{o}C)
- Rapid bleaching in pressurized equipment at 120 ^{o}C
- Bleaching at low temperature (~80^{o}C) increase process time

Flexibility: from 30 ^{o}C – 130 ^{0}C

Hydrogen Peroxide Bleaching: Recipe for cotton

- H_2O_2 (50%) : 1 – 2%
- Activator (TSP): 2 – 3% (to adjust the pH 10.5)
- Stabilizer (Sodium silicate): 1 – 2%
- Non-ionic surfactant: 3 gpl
- Metal chelating agent (EDTA): 0.01%
- Temp: 1-2 hrs.(@ 95 ^{0}C)

Activated Bleaching Process

Bleaching with Hypochlorite followed by Peroxide bleaching

- NaOCl: 3 gpl
- Temperature: 30 ^{0}C
- Time: 30 – 45 min

1st Bleaching J-Box

H_2O_2 (50%) : 0.5 – 0.8%

Activator (TSP): pH (10.5)

Stabilizer (Sodium silicate)

Metal chelating agent (EDTA)

Temp: 2 hrs.(@ 95 ^{0}C)

2nd Bleaching J-Box

Hydrogen Peroxide Bleaching

Advantages & Disadvantages

Advantages:

- Universal bleaching agent.
- Combine scouring and bleaching
- No antichlor treatment
- Non-corrosive and no unpleasant odour
- No AOX problem
- Improved absorbing and TEWEGA rating

Disadvantages:

❖ Silicates as stabilizers

❖ Catalytic damage to cotton

❖ Not effective for synthetic fibres