15CH210 Chemical Process Technology

Unit 4 - Natural Products

S. Balasubramanian
Chemical Engineering

- Edible and essential oils Industries
- Manufacture of soaps, detergents and glycerine
- Introduction and Manufacture of Kraft Pulp, Paper
- Soda recovery process
- Manufacture of starch and its derivatives
- Manufacture of sugar

- Manufacture of edible oil
- **Hydrogenation of Edible oil**

R	No. of Double Bonds	Melting point	Reactivity
Stearic	0	69	Nil
Oleic	1	14	Fair
Linoleic	2	- 5	Rapid
Linolenic	3	- 11	Extremely rapid

Hydrogenation

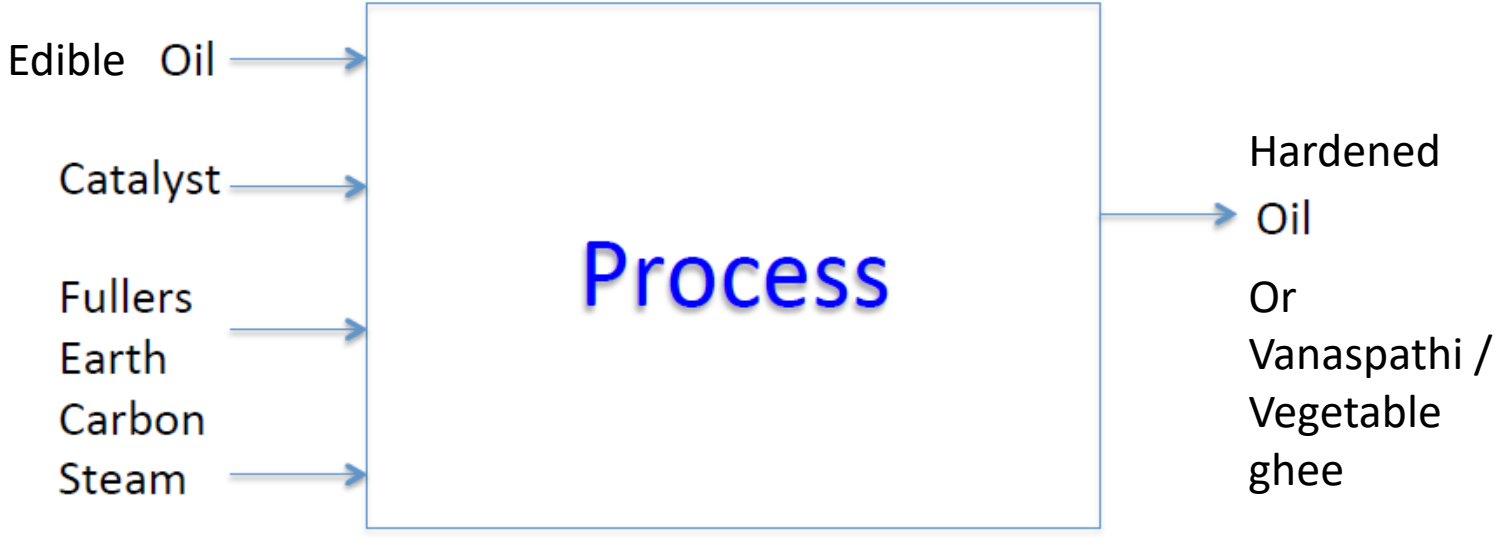
Hydrogenation is a unit process which is used in the fat and oil industry to remove the double bonds, raise melting point of the fat, and improve its resistance to rancid oxidation.

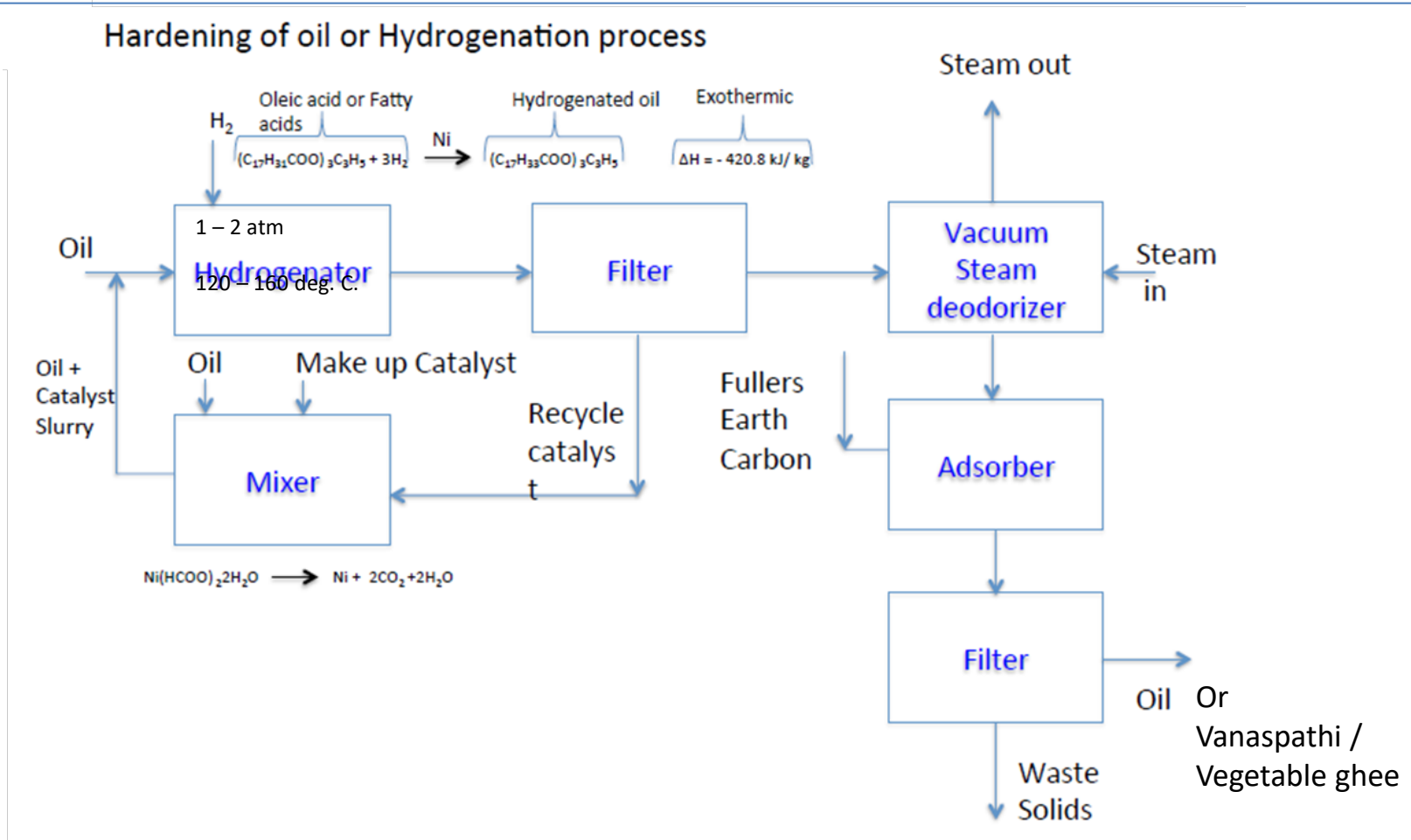
Hardening of oils

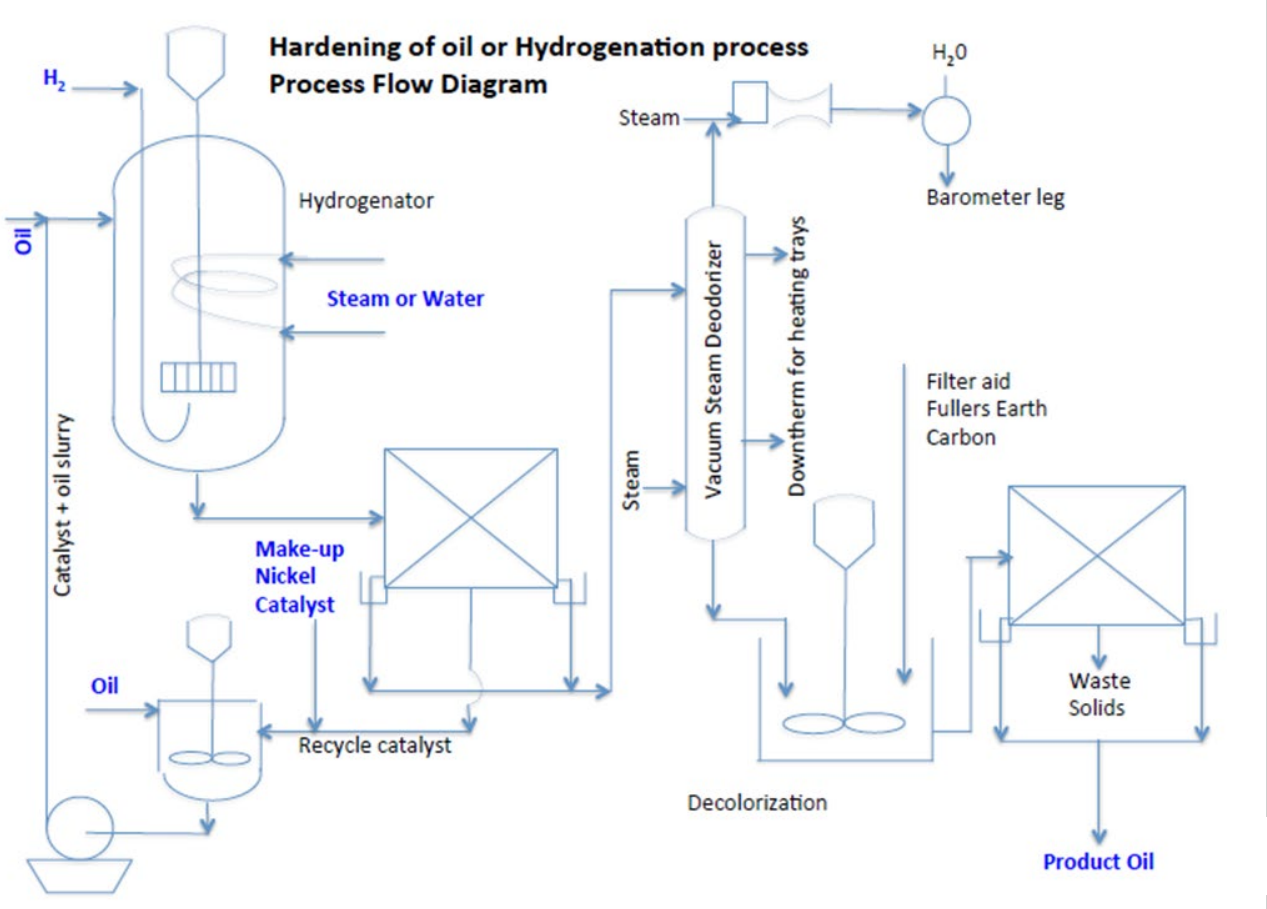
Glycerides of unsaturated acids are liquid at room temperature and so are unsuitable for edible fats. By converting the unsaturated acids into saturated acids, oils are changed into fats by introduction of hydrogen.

This introduction of hydrogen is known as *hardening of oils*. The oil is heated and hydrogen is passed under pressure, in the presence of finely divided nickel catalyst.

The major end product in India is Vanaspathi, a solidified household oil for cooking. Other products are vegetable ghee, hardened industrial oil, and so on.


Edible Oil
Catalyst
Fullers Earth
Carbon
Steam
Process
Hardened Oil
Or
Vanaspathi / Vegetable ghee


Hardening of oil or Hydrogenation process
Steam out
Oleic acid or Fatty acids
Hydrogenated oil
Exothermic
H_2
Ni
$(C_{17}H_{31}COO)_3C_3H_5 + 3H_2$
$(C_{17}H_{33}COO)_3C_3H_5$
$\Delta H = -420.8$ kJ/ kg
1 – 2 atm
Oil
Hydrogenator
120 – 160 deg. C.
Filter
Vacuum Steam deodorizer
Steam in
Oil + Catalyst Slurry
Oil
Make up Catalyst
Recycle catalyst
Fullers Earth Carbon
Mixer
Adsorber
$Ni(HCOO)_2 2H_2O \rightarrow Ni + 2CO_2 + 2H_2O$
Filter
Oil
Or Vanaspathi / Vegetable ghee
Waste Solids



DOWTHERM™ A is a eutectic mixture of two very stable compounds, biphenyl - $C_{12}H_{10}$. diphenyl oxide - $C1_2H_{10}O$.

S. No	Process	Equipment	Unit operations	Unit process	Objective	Operating conditions
1.	Hydrogenation (Batch/ Continuous)	(a) Hydrogenator	———	Hydrogenation	Remove the double bond to improve the resistance to rancidity	1 – 2 atm 120 -160 deg. C
		(b) Filter	Solid-liquid separation	———	Separation and recycle of oil and oil slurry with catalyst	———
		(c) Vacuum steam deodorizer	Liquid-gas separation	———	Remove volatile odorous materials by using steam	———
		(d) Adsorber	Solid-liquid Separation by pressing	———	Removal of color components using adsorbents	———
		(e) Filter	Solid-liquid separation	———	Separation of oil and solid waste	———
		(f) Catalyst mixer	Solid-Liquid mixing	———	Mixing of catalyst With oil	

Uses

Edible oils	Essential oils
Food - Hydrogenated oil (Vanaspathi)	Cosmetics
Soaps and Detergents	Perfumes
Cosmetics	Soaps
Paints and Varnishes	Medicines

If an oil is hydrolyzed and or saponified with alkali soaps are obtained.

Any metallic salts of fatty acid is soap, but the term soap is applied to water soluble salts.

Soaps comprises of the sodium or potassium salts of various fatty acids but chiefly of oleic, stearic, palmitic, lauric and mystiric acids.

The saturated fat gives hard soaps, whereas unsaturated fat gives soft soaps on saponification

Soaps comprises of the sodium or potassium salts of various fatty acids but chiefly of oleic, stearic, palmitic, lauric and mystiric acids. It also comprises of salts of sodium and zinc oxide catalyst.

Detergents differ from soap in their action in hard water.

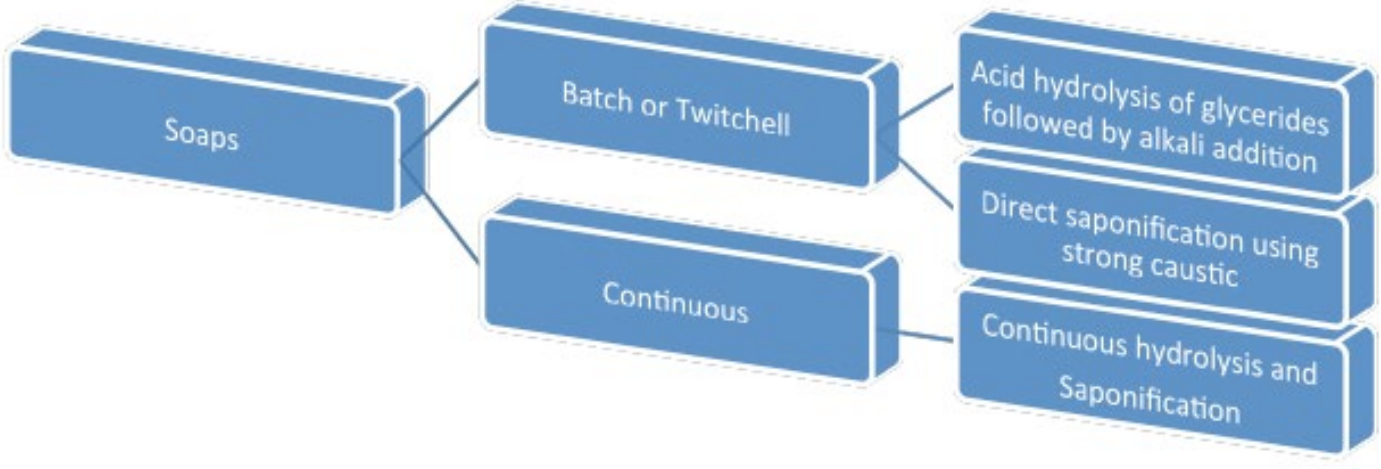
Detergents may react with hard water ions, but the resulting products are either soluble or remain colloidally dispersed in water.

Scientifically, the term detergent covers both soap and synthetic detergents or "Syndents" but it is widely used to indicate synthetic cleaning compound.

S. No	Soaps	Detergents
01.	Soaps from insoluble compounds with the calcium and magnesium ions present in hard water	Detergents may react with hard water ions, but the resulting products are either soluble or remain colloidally dispersed in water.
		Synthetic cleaning compound
02.	To make soap Tallow /or rice bran oil + Hydrolysis → (Splitting fats) Tallow fatty acid or fa	Alkylbenzene sulfonate + oleum → Disulfonate and Sulfuric acid
03.	Tallow /or rice bran oil + NaOH → (Saponification) Sodium salt	Tallow fatty alcohol + oleum → Fatty alcohol sulfate
04.	Salt of fatty acid + builders, etc → Soap	Sulfonate + Sulfate + NaOH → Sodium salts
		Sodium salt + Builders → detergents

Glycerin is a clear, nearly colorless liquid having a sweet taste but no odor.

The term glycerin is chosen for the technical product containing the pure trihydroxy alcohol *"glycerol"*.


Soaps
Batch or Twitchell
Continuous
Acid hydrolysis of glycerides followed by alkali addition
Direct saponification using strong caustic
Continuous hydrolysis and Saponification

	Batch (Twitchell) Process	Continuous process
Temperature, deg. C	150 – 175	230 - 250
Pressure, mPa (g)	5.2 – 10	4.1 – 4.9 40 – 45 atm
Catalyst	Alkyl aryl sulfonic acids, Oxides of calcium, zinc and magnesium i.e. CaO, ZnO, MgO.	Same catalyst or optional
Acid used	Sulfuric acid	_____
Time, h	12 – 48	2 – 3
Operation equipment	Batch	Continuous
Hydrolysis	85 – 98 %	97 – 99%
Glycerol obtained	5 – 15%	10 – 25%
Advantages	Low temperature, adaptable to small scale	Small floor space, uniform product quality, high yield of acids, high glycerin concentration, automatic control
Disadvantages	Catalyst handling; ling reaction time, need more than one stage for good yield	High temperature and pressure, High cost and greater operating skill

Continuous hydrolysis and Saponification

Hydrolysis (Fat – Splitting)

$(R . COO)_3 . C_3H_5 + 3H_2O \longrightarrow 3R . COO . H + C_3H_5(OH)_3$

(Triglyceride) + (Water) ⟶ Fatty acid + Glycerin

Fatty acid radical representing oleic, stearic, palmitic, lauric and mystric acids

$(C_{17}H_{35}C00)_3\ C_3H_5 + 3H_2O \longrightarrow 3C_{17}H_{35}COOH + H_2O$

Glyceryl stearate + Water ⟶ Fatty acid (Stearic Acid) + Glycerin

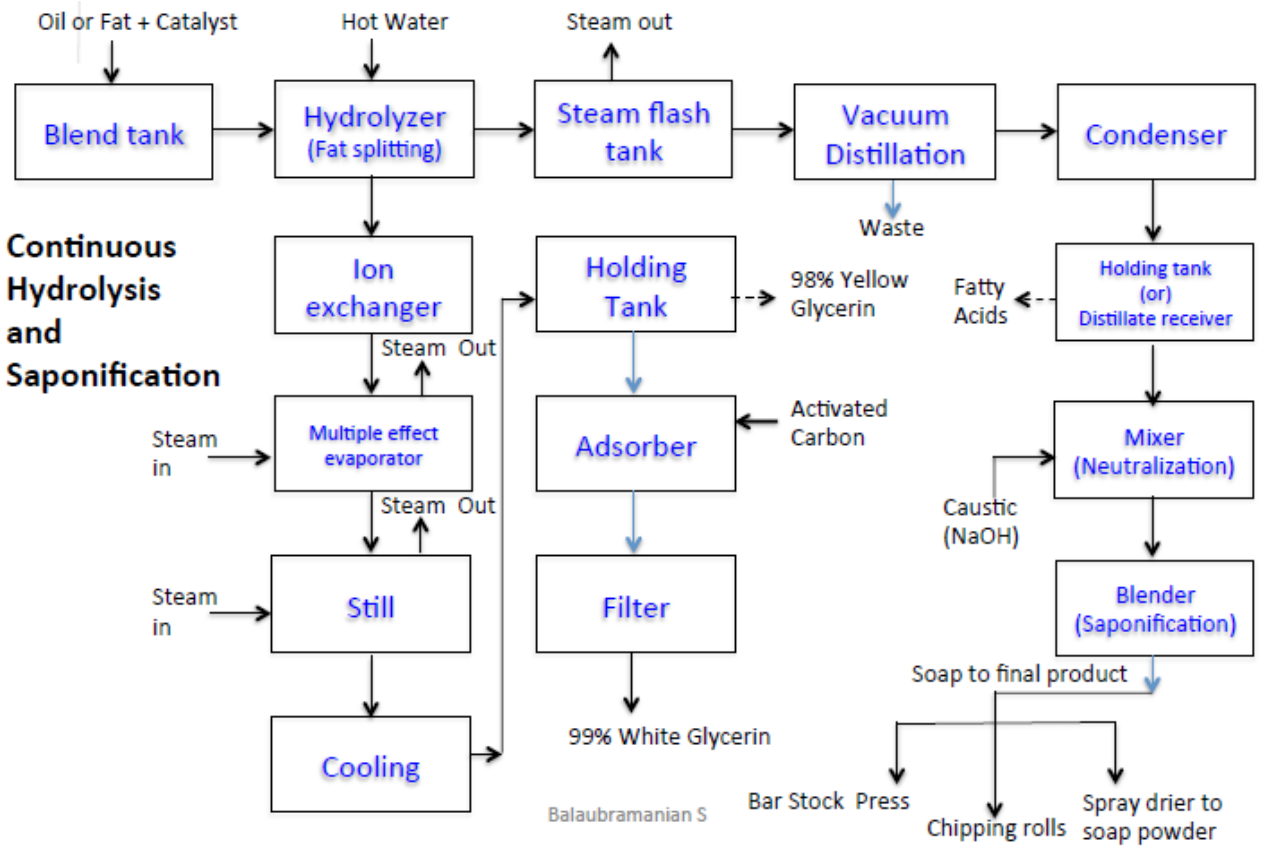
Saponification (Caustic Addition)

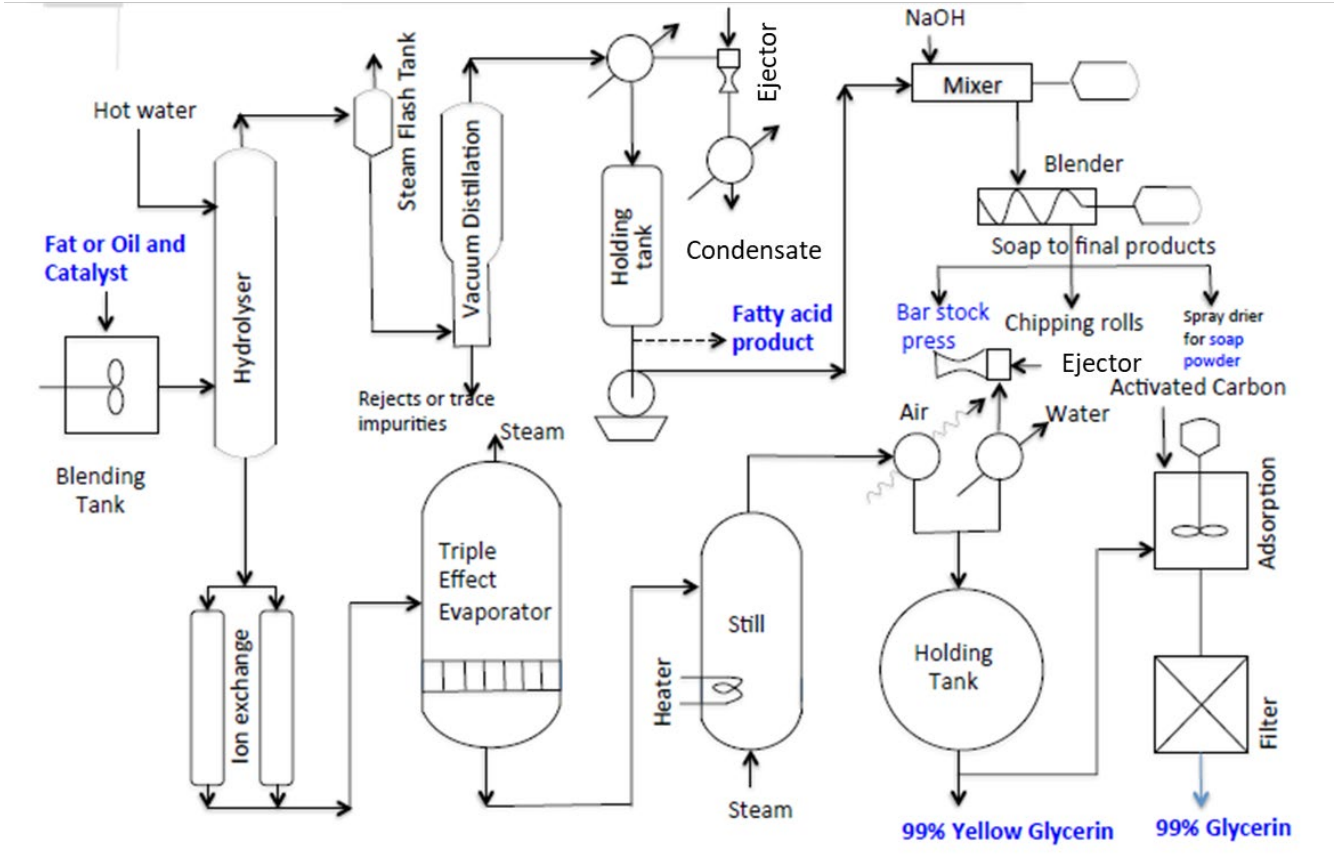
$R . COOH + M . OH \longrightarrow R . COO . M + H_2O$

Alkali element such as Na or K

$C_{17}H_{35}COOH + NaOH \longrightarrow C_{17}H_{35}COONa + H_20$

(Stearic acid) + (Caustic) ⟶ Sodium stearate + water


Oil or Fat + Catalyst
Hot Water
Steam out
Blend tank
Hydrolyzer (Fat splitting)
Steam flash tank
Vacuum Distillation
Condenser
Waste
Continuous Hydrolysis and Saponification
Ion exchanger
Holding Tank
98% Yellow Glycerin
Fatty Acids
Holding tank (or) Distillate receiver
Steam Out
Steam in
Multiple effect evaporator
Adsorber
Activated Carbon
Mixer (Neutralization)
Steam Out
Caustic (NaOH)
Steam in
Still
Filter
Blender (Saponification)
Soap to final product
Cooling
99% White Glycerin
Bar Stock Press
Chipping rolls
Spray drier to soap powder
Balaubramanian S

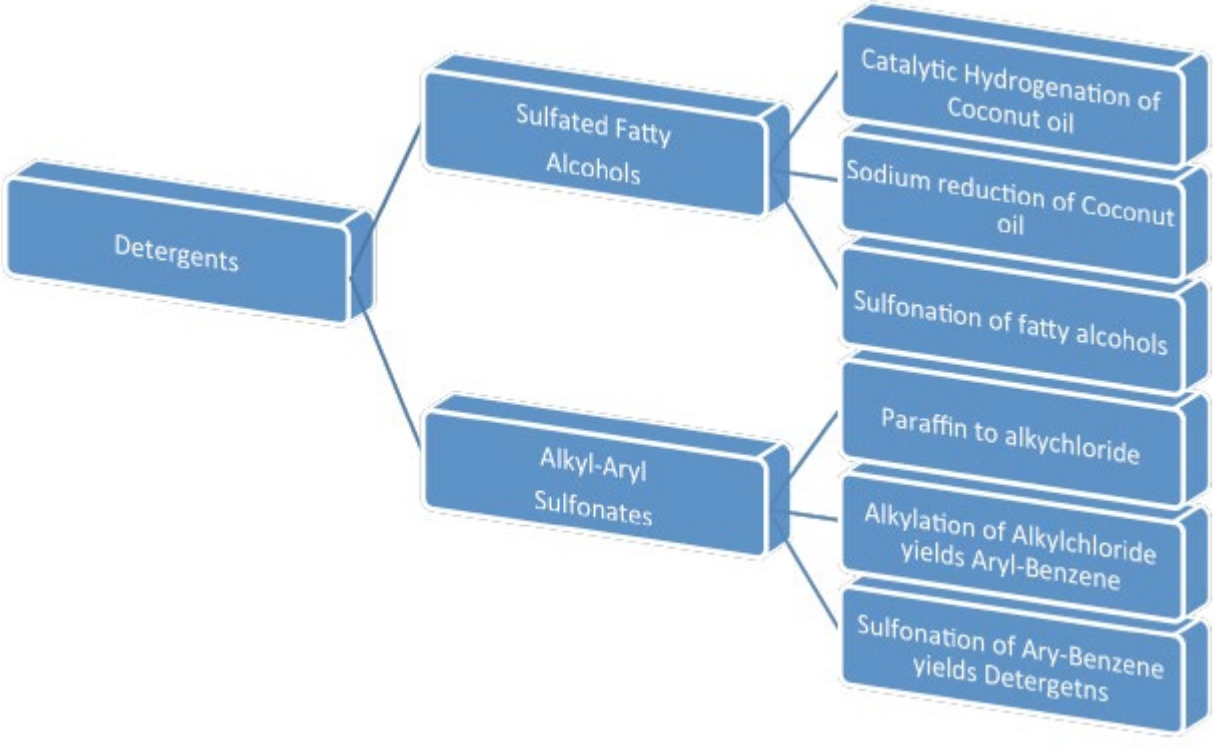

Hot water
Fat or Oil and Catalyst
Blending Tank
Hydrolyser
Steam Flash Tank
Vacuum Distillation
Rejects or trace impurities
Holding tank
Ejector
Condensate
Fatty acid product
NaOH
Mixer
Blender
Soap to final products
Bar stock press
Chipping rolls
Spray drier for soap powder
Ejector
Activated Carbon
Air
Water
Adsorption
Filter
Steam
Triple Effect Evaporator
Ion exchange
Heater
Still
Steam
Holding Tank
99% Yellow Glycerin
99% Glycerin

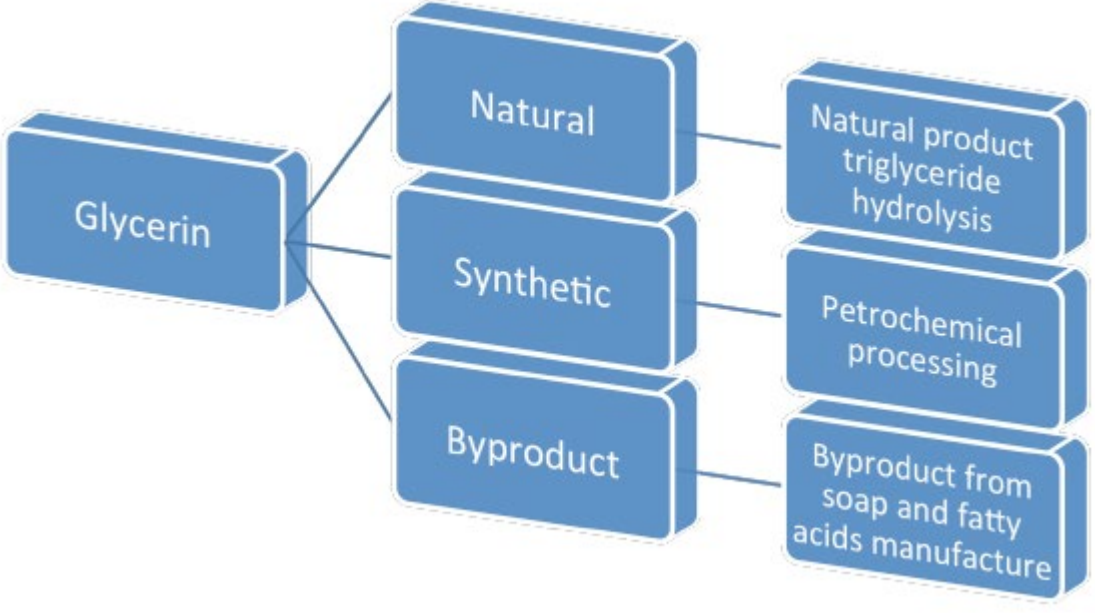
S. No	Process	Equipment	Unit operations	Unit process	Objective	Operating conditions
1.	Continuous Hydrolysis and Saponification (Continuous Process)	(a) Blend tank	Physical blending	_____	Blending of fat or oil and catalyst	_____
		(b) Hydrolyzer	_____	Hydrolysis	Splitting of fats	230 - 250 deg. C 40 – 45 atm
		(c) Ion exchanger	Ion exchange	_____	Separation of dissolved salts and glycerides collected from the hydrolyzer as the bottom product	_____
		(d) Evaporator	Evaporation	_____	Separation of water and glycerides (or) Concentration of glycerides	_____

S. No	Process	Equipment	Unit operations	Unit process	Objective	Operating conditions
		(e) Distillation still	Distillation	_____	Glycerides obtained from the evaporator is purified to produce 99% Yellow Glycerin	_____
		(f) Cooler (or) Heat exchanger	Cooling or Heat transfer	_____	Cool the glycerides obtained from the still	_____
		(g) Holding tank	Storage	_____	Cooled glycerides Stored	_____
		(h) Adsorber	Adsorption	_____	Removal of color constituents using activated carbon as the adsorbent	_____

S. No	Process	Equipment	Unit operations	Unit process	Objective	Operating conditions
		(i) Filter	Filtration	_____	Final separation of glycerides	_____
		(j) Steam flash tank	Steam separation	_____	Separation of steam from fatty acid	_____
		(k) Vacuum distillation column	Distillation	_____	Separation of fatty acids and trace impurities	_____
		(l) Condenser	Condensation	_____	Vapors of fatty acids converted to liquid	_____
		(m) Holding tank (or) Distillate receiver	Storage	_____	The distillate from the condenser is stored	_____

S. No	Process	Equipment	Unit operations	Unit process	Objective	Operating conditions
		(n) Mixer	Mixing	_____	Neutralization of fatty acids	_____
		(o) Blender	Blending	Saponification	Saponification of neutralized fatty acids	_____
		(p) Spray drier	Drying	_____	Drying of soaps into powder after saponification.	_____


Detergents
Sulfated Fatty Alcohols
Alkyl-Aryl Sulfonates
Catalytic Hydrogenation of Coconut oil
Sodium reduction of Coconut oil
Sulfonation of fatty alcohols
Paraffin to alkychloride
Alkylation of Alkylchloride yields Aryl-Benzene
Sulfonation of Ary-Benzene yields Detergetns


Glycerin
Natural
Synthetic
Byproduct
Natural product triglyceride hydrolysis
Petrochemical processing
Byproduct from soap and fatty acids manufacture

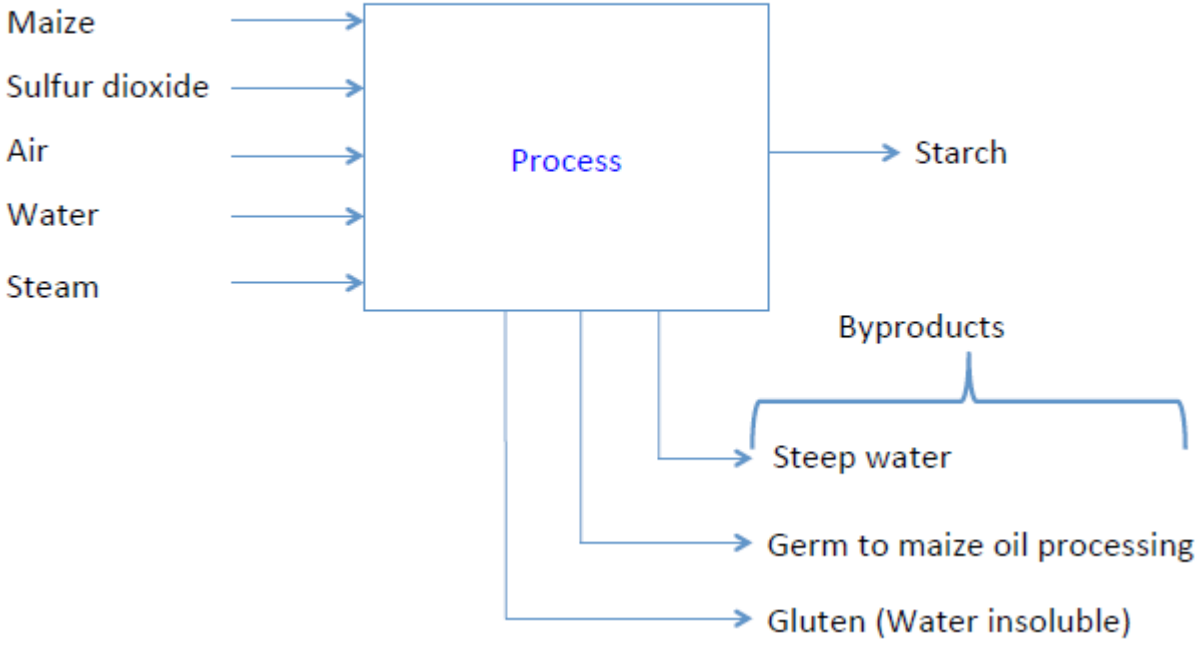
A partial list of soaps and detergents; glycerin is given to show the diversity of application

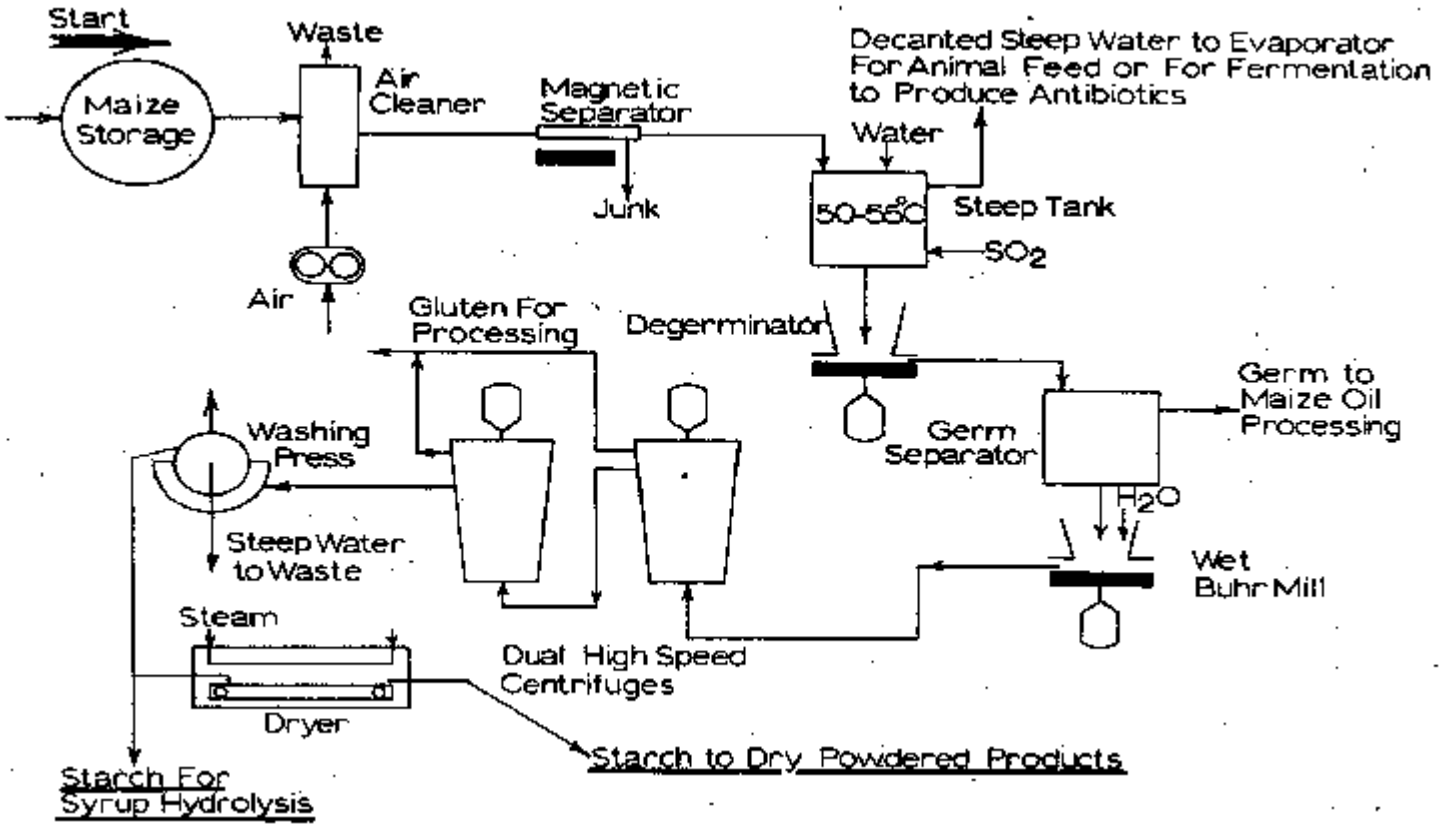
Soaps and detergents

Textile manufacture
Sanitation
Food processing
Shaving soaps
Synthetic rubber and plastics emulsion polymerization
Paints - water emulsion formulations
Paper - Application of sizing

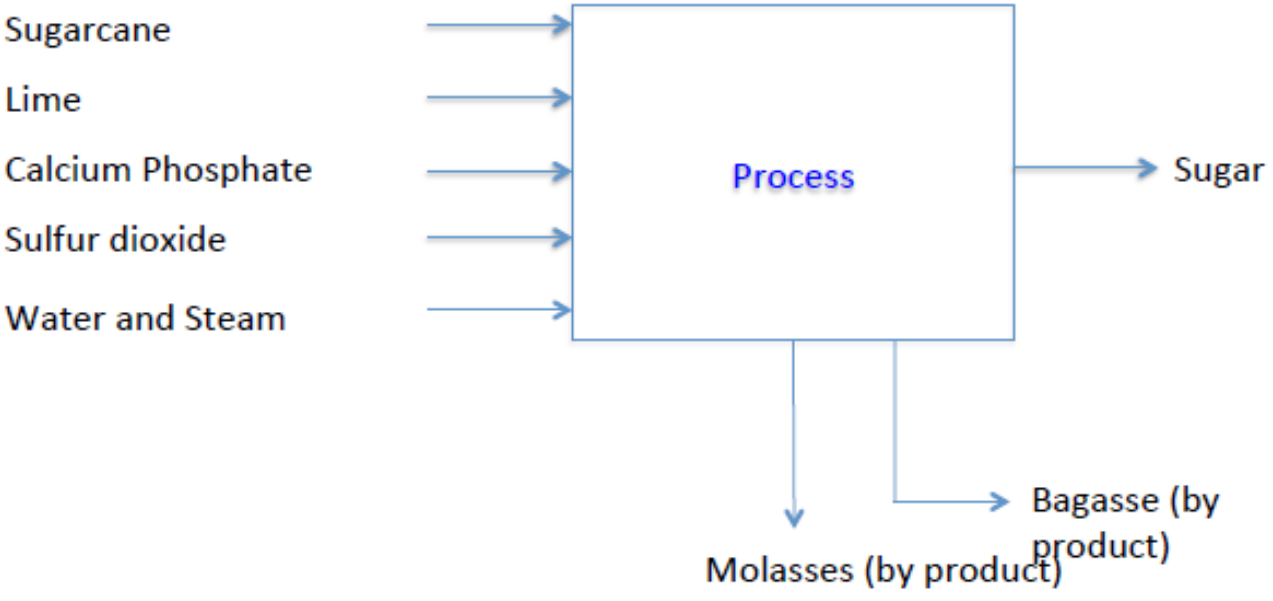
Glycerin

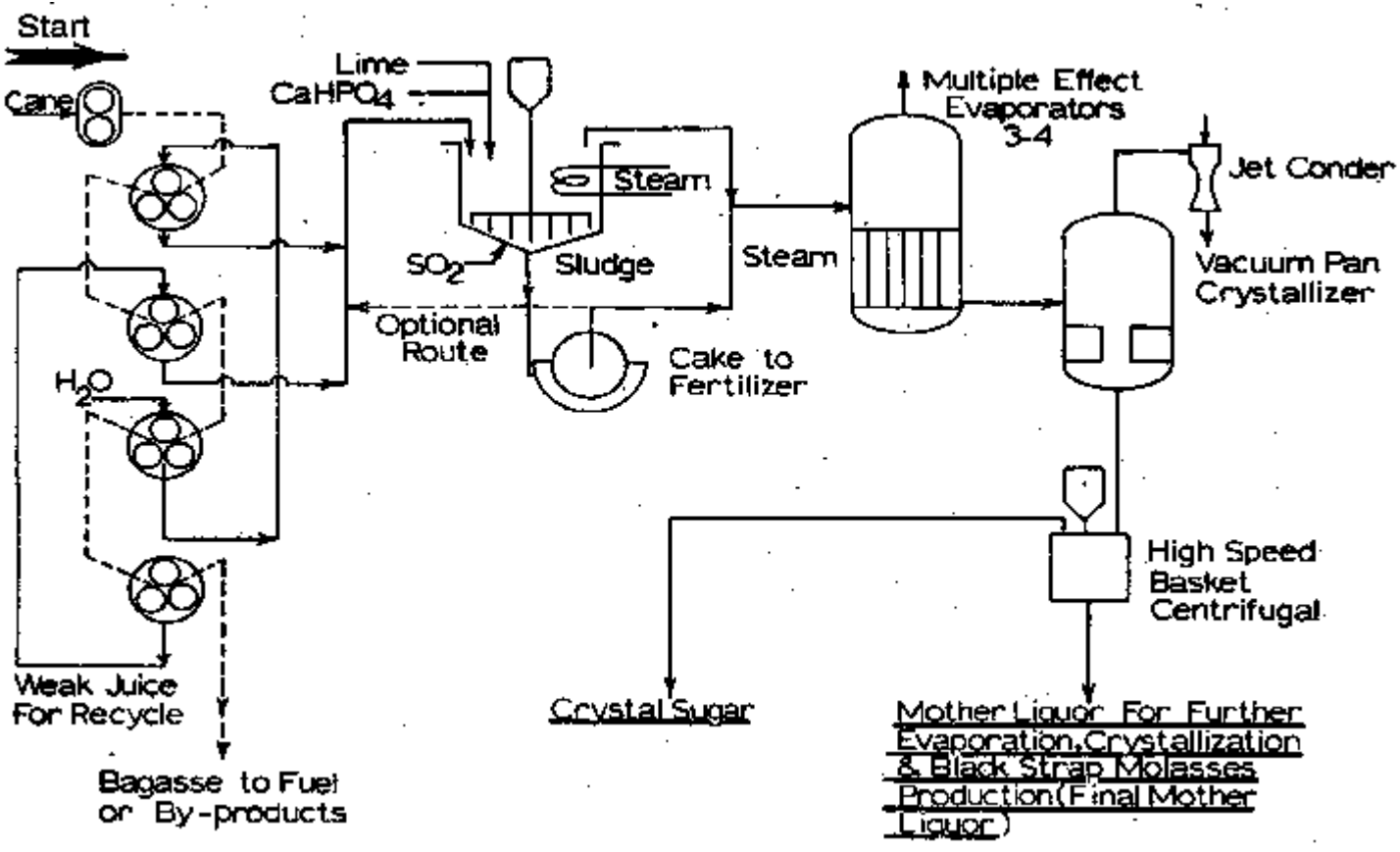
Alkyl resin and Plastics
Tobacco humidification
Cellulose Plasticizer
Explosives
Food and Pharmaceuticals


Maize
Sulfur dioxide
Air
Water
Steam
Process
Starch
Byproducts
Steep water
Germ to maize oil processing
Gluten (Water insoluble)


Start
Maize Storage
Waste
Air Cleaner
Magnetic Separator
Junk
Air
Decanted Steep Water to Evaporator For Animal Feed or For Fermentation to Produce Antibiotics
Water
50-55°C
Steep Tank
SO_2
Gluten For Processing
Degerminator
Germ to Maize Oil Processing
Germ Separator
H_2O
Wet Buhr Mill
Washing Press
Steep Water to Waste
Steam
Dryer
Dual High Speed Centrifuges
Starch to Dry Powdered Products
Starch For Syrup Hydrolysis

Process	Equipment	Unit Operation	Unit Process	Objective	Operating Conditions
Continuous	Maize Storage	Storage	-------	To store the maize	-------
Continuous	Air cleaner	Cleaning	-------	Remove the dust and fines	-------
Continuous	Magnetic Separator	Particle Separation	-------	Separate metal particles	-------
Continuous	Steep tank	Decantation	-------	Add water to facilitate the germination and Removal of steep water by decantation. SO_2 is added as bacteriostatic.	50 – 55 deg. C
Continuous	Degerminator	Grinding	-------	To rupture the cells to remove germs	-------
Continuous	Germ separator	Particle Separation	-------	To separate the germs and send it to maize Oil processing	-------
Continuous	Wet Bhur Mill	Size reduction and separation	-------	Crush the fiber and removes the undesired hulls	-------
Continuous	Dual High Speed Centrifuge	Solid-Liquid Separation	-------	To separate the gluten a water insoluble protein and starch	-------


Sugarcane
Lime
Calcium Phosphate
Sulfur dioxide
Water and Steam
Process
Sugar
Bagasse (by product)
Molasses (by product)


Start
Cane
Lime
$CaHPO_4$
Steam
SO_2
Sludge
Optional Route
Cake to Fertilizer
Steam
Multiple Effect Evaporators 3-4
Jet Conder
Vacuum Pan Crystallizer
H_2O
High Speed Basket Centrifugal
Weak Juice For Recycle
Bagasse to Fuel or By-products
Crystal Sugar
Mother Liquor For Further Evaporation, Crystallization & Black Strap Molasses Production (Final Mother Liquor)

S. No	Process	Equipment	Unit operations	Unit process	Objective	Operating conditions
1.	Continuous	(a) Primary Crusher	Crushing	_____	To facilitate initial crushing and Squeezing of juice from sugarcane	_____
	Continuous	(b) Secondary Crushers	Solid-liquid separation (Crushing)	_____	Extracting the maxing amount of available juice from sugarcane . Expel the crushed cane (Bagasse) .	_____
	Continuous	(c) Thickener/ Clarifier/ Mixer-Settler	Solid-liquid separation (Flocculation)	_____	To separate the colloidal impurities in juice by adding lime and calcium phosphate. pH is also adjusted by adding sulfur dioxide.	_____

S. No	Process	Equipment	Unit operations	Unit process	Objective	Operating conditions
	Continuous	(d) Rotary Drier	Solid-Liquid Separation (Filtration)	_____	To separate the cake and liquid juice obtained from the underflow of thickener/ clarifier.	_____
	Continuous	(e) Evaporator	Liquid-Liquid Separation (Evaporation)	_____	To separate the solvent water from the juice. To concentrate the solute	_____
	Continuous	(f) Vacuum Pan Crystallizer	Solid – Liquid Separation (Crystallization)	_____	To produce sugar crystals and mother liquor or black strap molasses.	____
	Continuous	(g) Basket Centrifuge	Solid – liquid Separation	_____	Separation of Sugar crystals from mother liquor.	

Cellulose

Cellulose is a polysaccharide used in chemical industries in the form known as *chemical cellulose* for preparation of fibers, paper, and plastics.

Pulp

Pulp is commercial and or chemical cellulose derived from bamboo, bagasse, and wood by mechanical or chemical methods.

Paper

Paper is defined as matted or felted sheets of fibers, usually cellulosic and generally formed on a fine wire screen from a fiber-water suspension.


Nylon fabrics


Nylon Fibers

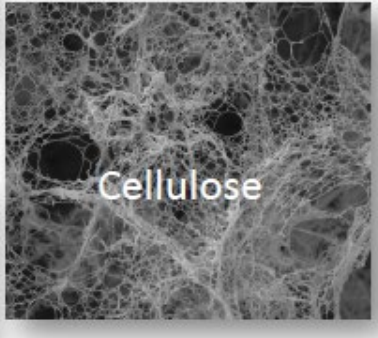

Cellulose


IMAGE
COPIER


TENITE
cellulosic plastics
Cellulose
Plastics
From Trees to Plastic

Ballarpur Industries Limited(BILT)

ITC Bhadrachalam Mill is located at Sarapaka, Andhra Pradesh

Tamil Nadu Newsprint and Papers Ltd. Kagithapuram in Karur.

Pudumjee Pulp & Paper Mills Ltd. Pune (Maharashtra, India)

The West Coast Paper Mills Ltd. Karnataka

The Andhra Pradesh Paper Mills Ltd. Rajahmundry, AP

JK Paper Jaykaypur, Orissa.

Star Paper Saharanpur, Uttar Pradesh

Titagarh Paper Mills Company Ltd. Titagarh and Kakinara

Sirpur Paper Mills. Kaghaznagar, Andhra Pradesh

Sri Krishna Paper Mills and Industries Ltd. Delhi

Vikarabad Pulp and Paper Mills Pvt. Ltd. Andhra Pradesh

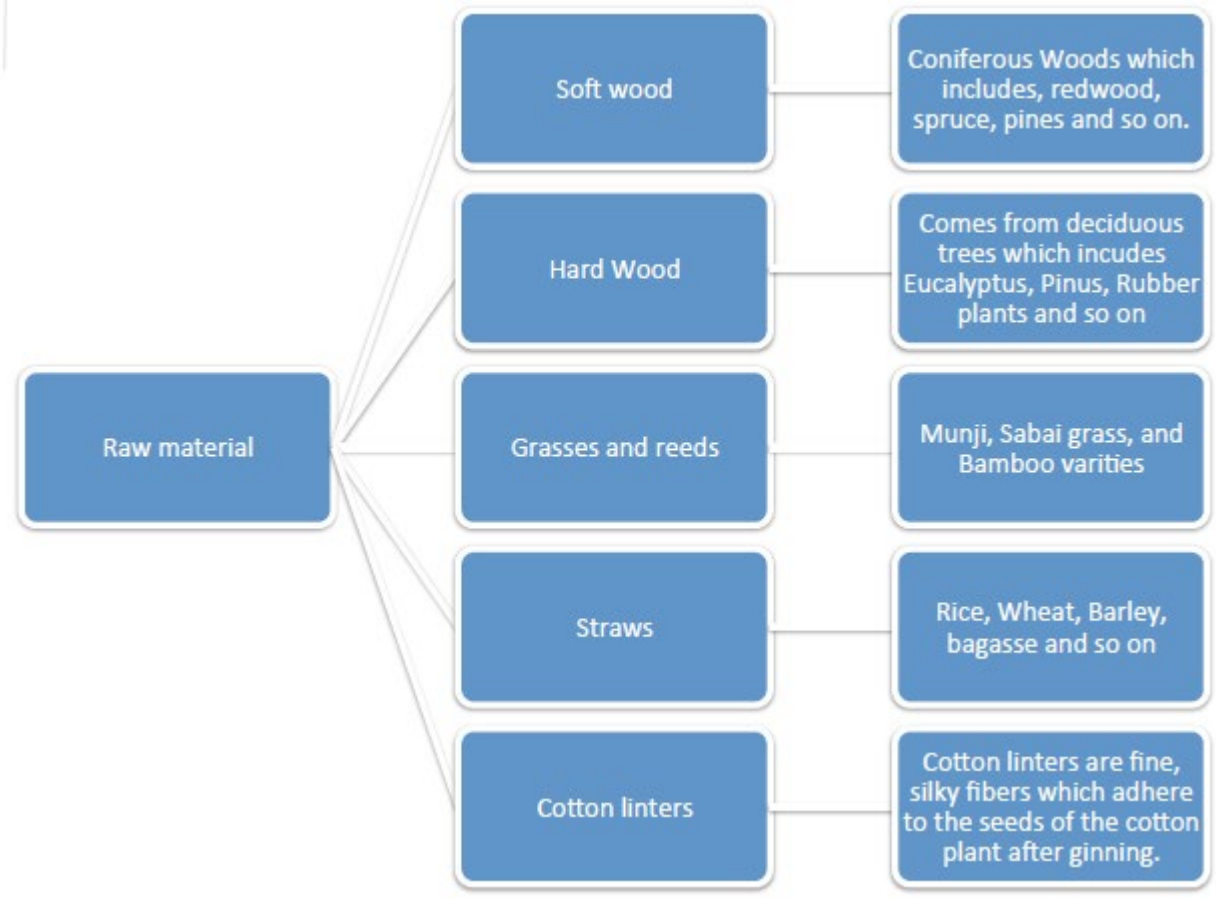
http://business.mapsofindia.com/paper-and-pulp-industry/paper-and-pulp-industry.jpg

Pulping

Paper production requires a disintegration of the bulky fibrous material to individual or small agglomerate fibers. This is called *Pulping*.

The requirement of a good raw material for pulp and paper production,

1. The ideal fiber for high grade paper should be long i.e. fiber must be long
2. High in cellulose content
3. Low in lignin content


Raw material
Soft wood
Coniferous Woods which includes, redwood, spruce, pines and so on.
Hard Wood
Comes from deciduous trees which incudes Eucalyptus, Pinus, Rubber plants and so on
Grasses and reeds
Munji, Sabai grass, and Bamboo varities
Straws
Rice, Wheat, Barley, bagasse and so on
Cotton linters
Cotton linters are fine, silky fibers which adhere to the seeds of the cotton plant after ginning.

Soft wood

Coniferous Woods which includes, redwood, spruce, pines and so on.

Hard Wood

Comes from deciduous trees which incudes Eucalyptus, Rubber plants and so on

Grasses and reeds

Munji, Sabai grass, and Bamboo varities

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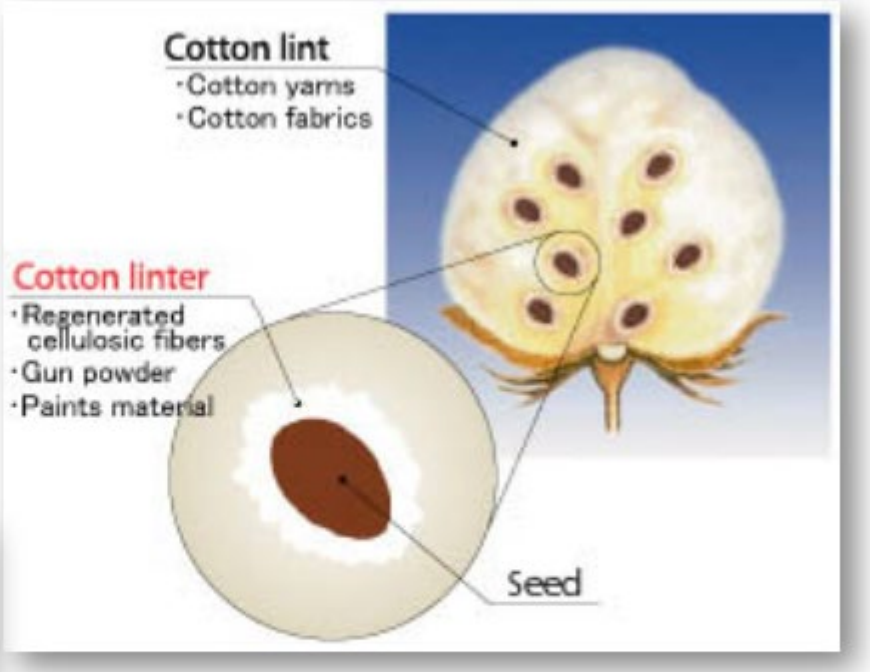
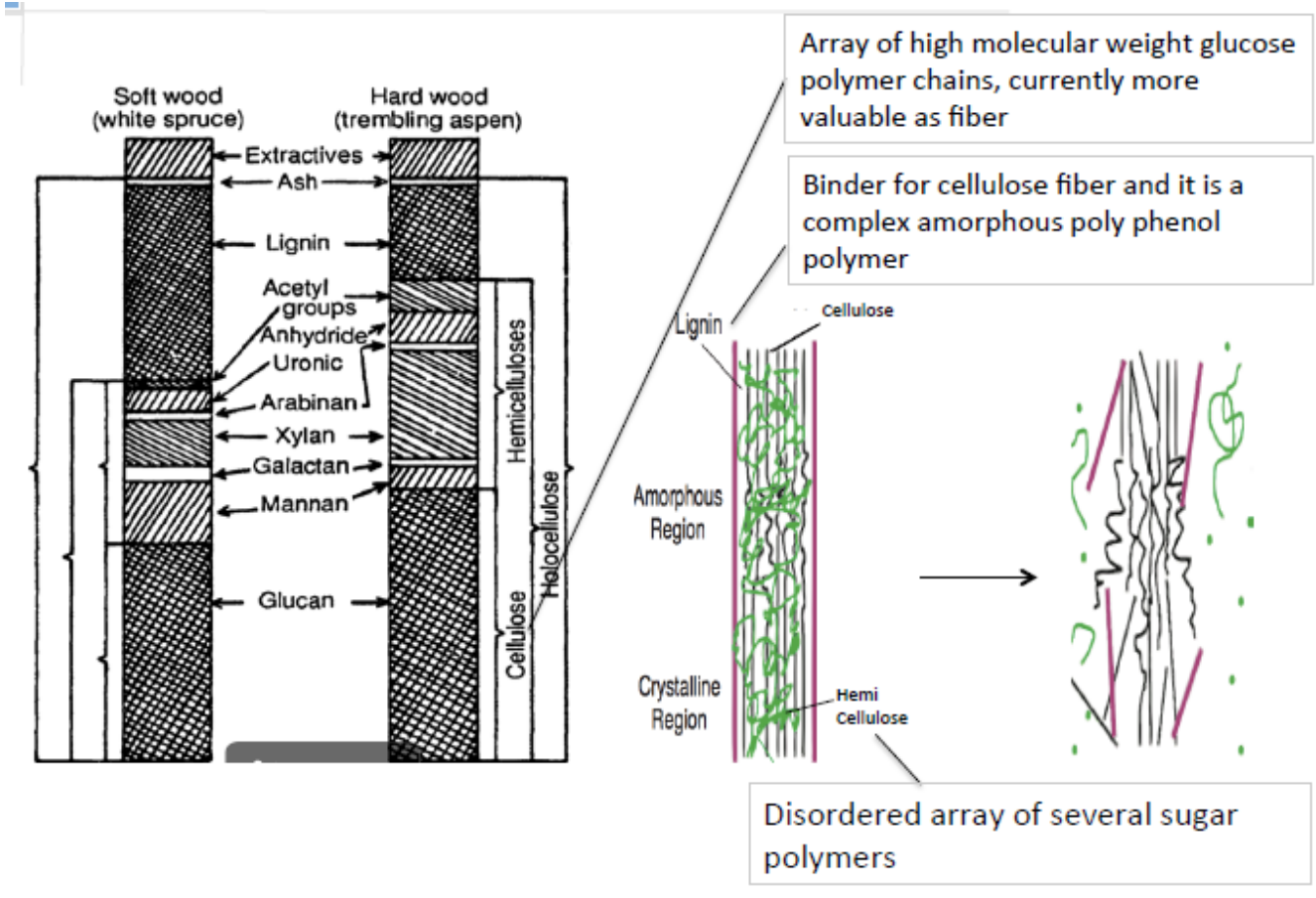
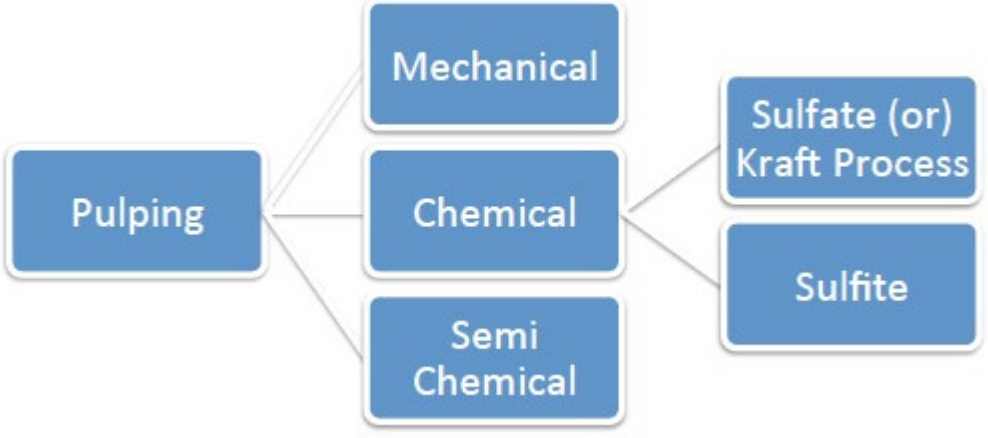
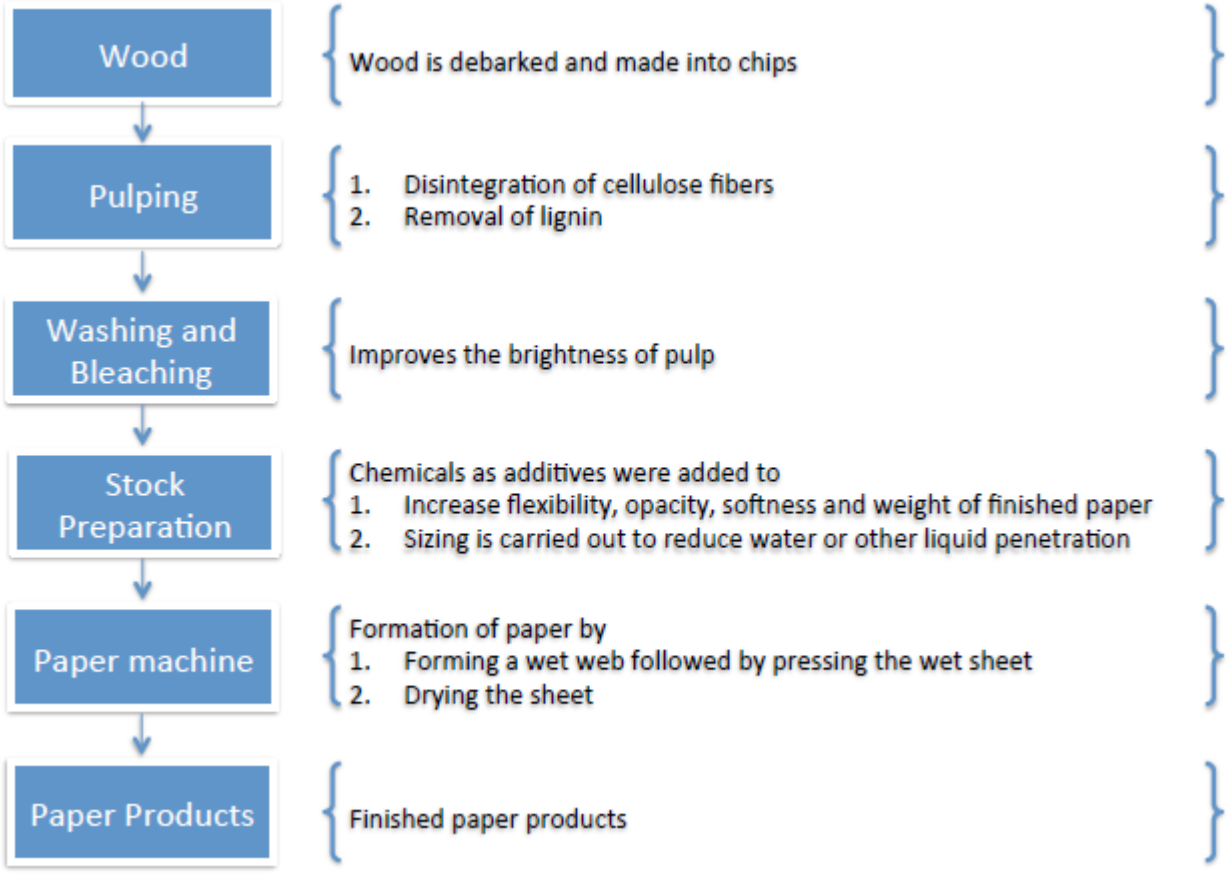
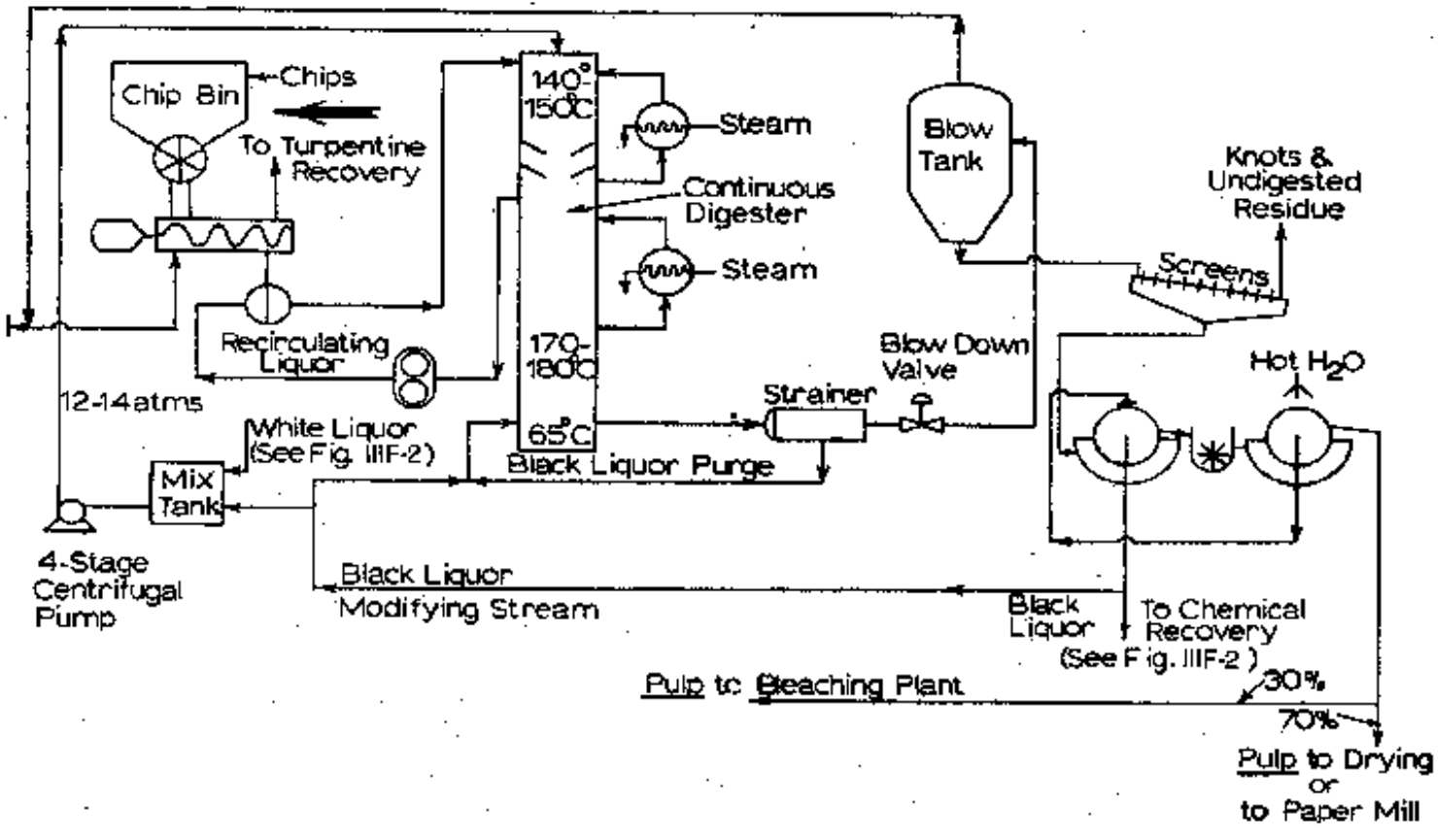


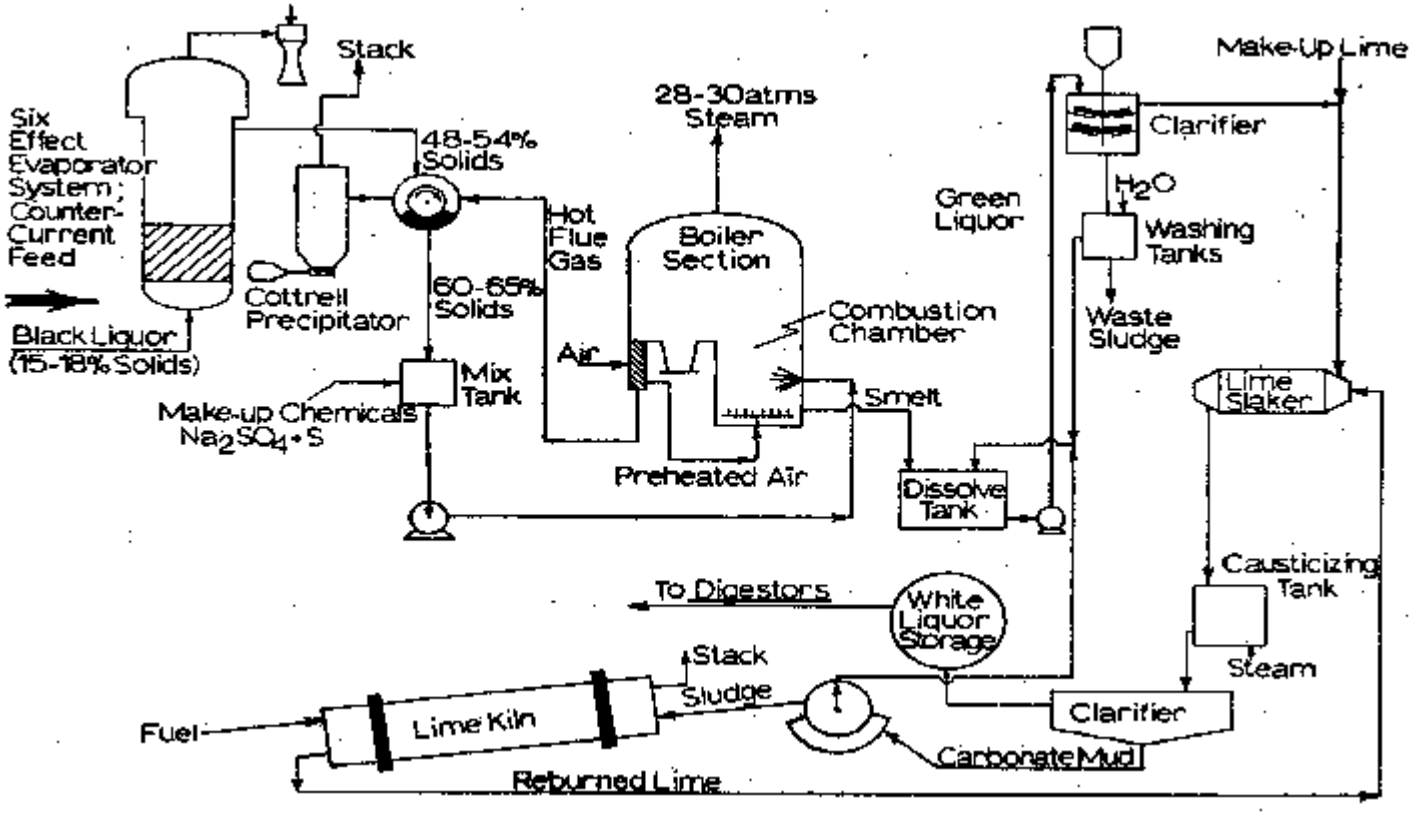

Soft wood
(white spruce)
Hard wood
(trembling aspen)
Extractives
Ash
Lignin
Acetyl groups
Anhydride
Uronic
Arabinan
Xylan
Galactan
Mannan
Glucan
Hemicelluloses
Holocellulose
Cellulose
Array of high molecular weight glucose polymer chains, currently more valuable as fiber
Binder for cellulose fiber and it is a complex amorphous poly phenol polymer
Lignin
Cellulose
Amorphous Region
Crystalline Region
Hemi Cellulose
Disordered array of several sugar polymers


Pulping
Mechanical
Chemical
Semi Chemical
Sulfate (or) Kraft Process
Sulfite


Wood
Wood is debarked and made into chips
Pulping
1. Disintegration of cellulose fibers
2. Removal of lignin
Washing and Bleaching
Improves the brightness of pulp
Stock Preparation
Chemicals as additives were added to
1. Increase flexibility, opacity, softness and weight of finished paper
2. Sizing is carried out to reduce water or other liquid penetration
Paper machine
Formation of paper by
1. Forming a wet web followed by pressing the wet sheet
2. Drying the sheet
Paper Products
Finished paper products


Chip Bin
Chips
To Turpentine Recovery
140°-150°C
Steam
Continuous Digester
Steam
Blow Tank
Knots & Undigested Residue
Screens
Recirculating Liquor
170-180°C
Blow Down Valve
Hot H_2O
12-14 atms
Strainer
White Liquor (See Fig. IIIF-2)
65°C
Black Liquor Purge
Mix Tank
4-Stage Centrifugal Pump
Black Liquor Modifying Stream
Black Liquor
To Chemical Recovery (See Fig. IIIF-2)
Pulp to Bleaching Plant
30%
70%
Pulp to Drying or to Paper Mill

Process	Equipment	Unit Operation	Unit Process	Objective	Operating Conditions
Continuous	Chip bin	Storage	--------	Receive and store the chips from the chipper	--------
Continuous	Digester		Hydrolysis	Solubilizing the lignin components by adding white liquor	65 – 180 deg. C 12 atm presssure
Continuous	Strainer	Solid-Liquid Separation	--------	Removal of black liquor and solubilized lignin	--------
Continuous	Blow down tank	Storage and Transportation	--------	Cool and transport the digested pulp to further processes. Separate the turpentine as the top product and reuse it to preheat the chips entering into the chip bin.	--------
Continuous	Screen	Size separation	--------	To remove the wood knots and other undigested residue	--------
Continuous	Washing	Solid-Liquid Separation	--------	Recover the black liquor by washing the digested wood cellulose. Prepare the cellulose for bleaching	--------

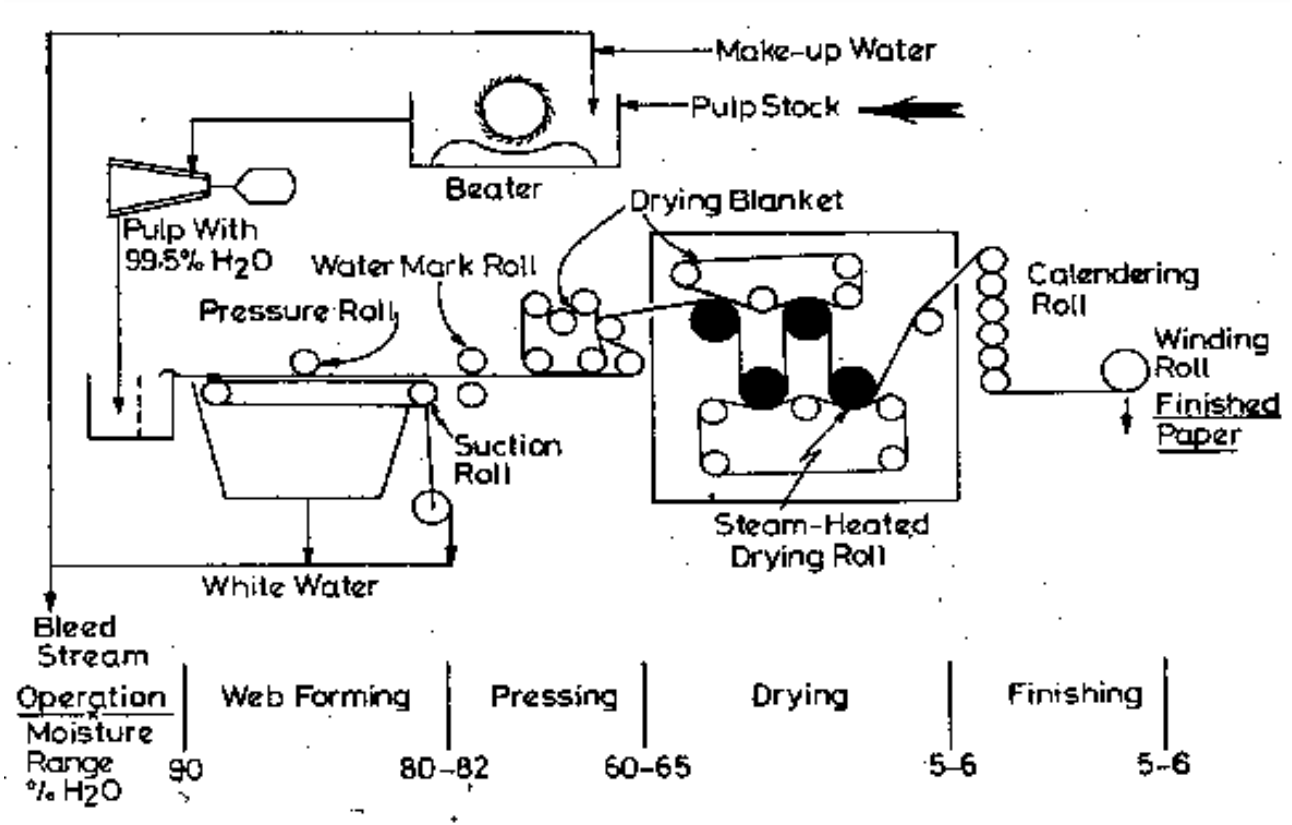

Stack
Six Effect Evaporator System; Counter-Current Feed
48-54% Solids
28-30atms Steam
Hot Flue Gas
Boiler Section
Green Liquor
Clarifier
Make-Up Lime
H_2O
Washing Tanks
Waste Sludge
Cottrell Precipitator
60-65% Solids
Combustion Chamber
Air
Black Liquor (15-18% Solids)
Mix Tank
Make-up Chemicals $Na_2SO_4 + S$
Smelt
Lime Slaker
Preheated Air
Dissolve Tank
To Digestors
White Liquor Storage
Causticizing Tank
Steam
Stack
Sludge
Fuel
Lime Kiln
Clarifier
Carbonate Mud
Returned Lime

Process	Equipment	Unit Operation	Unit Process	Objective	Operating Conditions
Continuous	Multiple Effect Evaporator	Solid-Liquid separation	--------	To concentrate the black liquor	5 – 6 stages
Continuous	Smelting furnace	--------	Oxidation	Burn the organic carbon to produce an inorganic Molten slag. CO_2 is liberated. To facilitate the process alkali is supplied via Na_2SO_4 $2NaR$ (Lignin) + Air → $Na_2CO_3 + CO_2$ $Na_2SO_4 + 2C$ from R → $Na_2S + 2CO_2$	--------
Continuous	Dissolve tank	Physical Separation	--------	Bring contact between slag and water to yield Green liquor	--------
Continuous	Clarifier	Solid-Liquid Separation	--------	To separate insoluble impurities such as unburned carbon and also to obtained a clear liquor by adding lime	--------
Continuous	Washing tank	Solid-Liquid Separation	--------	To separate and cool the clarifier sludge	--------
Continuous	Rotary drum Filter	Solid-Liquid separation	--------	Thickening sludge obtained from the clarifier	--------
Continuous	Lime kiln	Drying	--------	Remove the moisture and recovers the lime to reuse as causticizing agent.	--------
Continuous	Lime Slacker	Mixing		Mixing quick lime with water to prepare white liquor	

Process	Equipment	Unit Operation	Unit Process	Objective	Operating Conditions
Continuous	Causticizing Tank	--------	Causticizing	To prepare white liquor for digestion Na_2CO_3 (aq) + $Ca(OH)_2$ (s) → 2NaOH (aq) + $CaCO_3$ (s) $CaCO_3 \rightarrow CaO + CO_2$ $CaO + Water \rightarrow Ca(OH)_2$	--------
Continuous	Clarifier	Solid – Liquid Separation	-------	Separate white liquor and solid impurities	--------
Continuous	Storage tank	Storage	-------	Store the white liquor	-------

Primary process involved in the paper sheet formation:

- Random arrangement of fibers into a wet web
- Removal of free water from wet web by wet pressing
- Progressive removal of additional water by heated rolls


Make-up Water
Pulp Stock
Beater
Pulp With
99.5% H_2O
Drying Blanket
Water Mark Roll
Pressure Roll
Calendering Roll
Winding Roll
Finished Paper
Suction Roll
Steam-Heated Drying Roll
White Water
Bleed Stream
Operation
Moisture Range % H_2O
Web Forming
Pressing
Drying
Finishing
90
80–82
60–65
5–6
5–6

Process	Equipment	Unit Operation	Unit Process	Objective	Operating Conditions
Continuous	Beater	Mechanical Separation	-------	Disintegrate the fibers	-------
Continuous	Press rolls	Solid-Liquid Separation	-------	Random arrangement of fibers into a web form. Removal of free water (White water) from wet web by pressing and compaction	80% of water removal
Continuous	Suction rolls	Solid-Liquid Separation	-------	Water removal	60–65% of water removal
Continuous	Drying Blanket	Solid-Liquid separation	-------	Removal of residual moisture	5–6% of remaining water removal
Continuous	Calenderia rolls	Physical Rolling	-------	Rolling of sheet of paper	-------
Continuous	Winding rolls	Physical Rolling	-------	Rolling finished paper	-------

Uses

Writing-printing papers
News print papers
Coated printing papers
Packaging papers
Tissue papers
Corrugated boards

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3. Finar IL, Organic Chemistry Vol. 1 6th Edition Pearson Education 2009 pp.116-117