(A) Soap Industry.

* Introduction:

The present soap was gradually developed from the crude mixtures of alkaline and fatty materials. The old process is known as batch process, in which a bulk of fatty acid is treated with caustic alkali in reaction kettles. The continuous saponification process was developed by Lever brothers in 1945.

* Importance of soap :→

Soaps are the Na or K salts of long chain monocarboxylic acids like oleic, stearic, palmic, lauric and myristic acids. Na salts are called hard soaps and K salts are called soft soaps.

* Raw Materials for soaps :→

Triglycerides are the basic raw material for the manufacture of soap utilizing a variety of processes.

(1) **Oils and fats:** Principal raw materials. The main sorces of slow lathering hard oils are tallow, palm, whale, fish oils and greases etc. Quick lathering hard oils include coconut oil, unedible olive oil etc.

(2) **Caustic soda: (NaOH):** Available in the form of flakes, blocks and sticks as well as 70° (90.32%), 72° (92.9%) and 74° (94.98%). Special type soft soaps and shaving creams use caustic potash (KOH) of 18.5 to 20%.

(3) **Resin:** It is a plant exudation product mainly contains abietic acid. Colourless variety of resin is used in the manufacture of laundry soaps and dark variety is used in the manufacture of coloured soaps.

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* Resin makes lather formation faster.
* It increases the cleaning action of soap and
* It is used in softening of hard soaps.

④ Common salt or Sodium Chloride :→ Used for salting out of soap. About 12.5 parts of NaCl salt per 100 parts of oil to be used for saponification.

⑤ Binding materials: It improves soap texture, correct the alkalinity of the solution and prevent the formation of precipitates in hard water. e.g. Na-silicate, soda ash, tri sodium Phosphate and borax.

⑥ Fillers:→ Wt of soap increases by adding certain fillers e.g. talc, starch, glauber salt, Pearl ash etc.

⑦ Colouring Matter :→ Organic dyes and inorganic pigments are generally used for bar and flake soap products. Following colouring maters are generally used.

	Shade of soap	Colouring matter added
(a)	violet	Methyl violet.
(b)	Brown	Bismark brown
(c)	Red	Rhodamine or saffranine.
(d)	White	Zinc oxide.
(e)	Green	Chrome green.
(f)	Yellow	Cadmium Yellow or Metanil yellow.
(g)	Blue	Methylene blue, or Ultra marine
(h)	Pink	Eosin.
(i)	Rose	Vermilon.

⑧ Perfumes and Perfume fixatives :→ The essential oils known as perfumes impart fragrance to soap. They may be Natural or synthetic.

⑨ Some other additions in soaps :→ Super fatting agents, Disinfectants and Germicides are other addition in soaps.

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* Manufacture of soap :→

Laundry, toilet or bath soaps are manufacture by hot process. Transparant and other special types of soaps are produced by cold process. Glycerol is recovered as a by product of soap industry. The hot process is of two types - the batch process and the continuous process. Computer control allowed an automated plant for continuous saponification by NaOH, oils and fats to produce large amount of soap in a short time.

Soap making from oils and fats involves two important steps.

(A) Processing of oils and fats and the blending in required proportion.

(B) Preparation of neat soap by saponification.

(A) Processing of oils and fats :→ The quality of soap depends upon the qualities and properties of oil and fat stock used for its preparation. The oils and fats are improved by refining and bleaching and by hydrogenation.

Fat from animal or vegetable sources are blended with catalyst like ZnO and heated with steam in a blend tank. The mixture of hot molten fat and catalyst is introduced at the bottom of hydrolysing tower which breaks fat into droplets. The temp. inside hydrolyzer is 250°C at 40-45 atm pressure. From top of hydrolyser hot H_2O is introduced so that it extracts glycerine dissolved in fatty phase and gets collected at the bottom due to high density. The faty acids are

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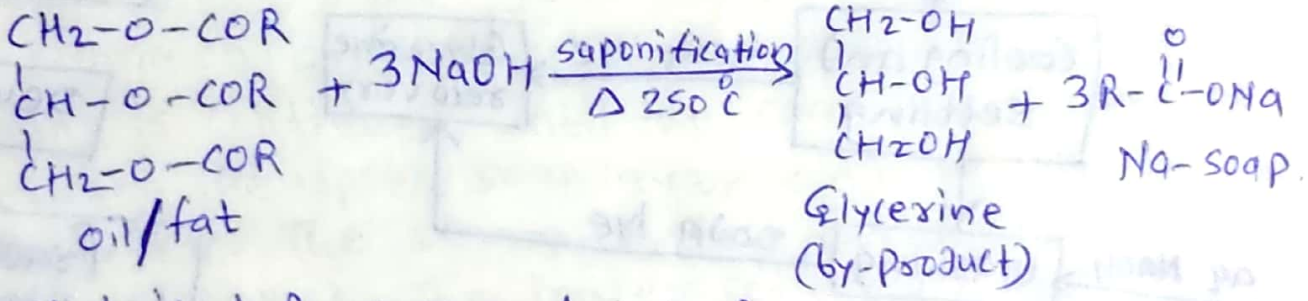
discharged from the top of hydrolyser to a decanter, where H_2O is completely separated. For separation of different fatty acids a vaccum distillation is used. The high boiling long chain fatty acids are pumped to a neutralizer.

(B) Preparation of Neat Soap by saponification of oils and fats by continuous process :→

There are three important processes of saponification used in soap making. (a) Full-boiled process (b) Half boiled process and (c) Cold process. Out of these 3 processes full-boiled process is commonly used for soap making.

*Reaction :→



* Full boiled Process or batch Process or continuous Process

(I) Charging of Raw Materials :→

In the continuous process the raw oils and fats and the catalyst usually ZnO are blended and fed into a hydrolyzer or splitting tower filled with steam coils through which steam is passed for heating the charge. The splitting of fat takes place continuously in a counter current manner at about 250°C and 40 atmospheric pressure. The aqueous layer is withdrawn and used for the recovery of glycerol.

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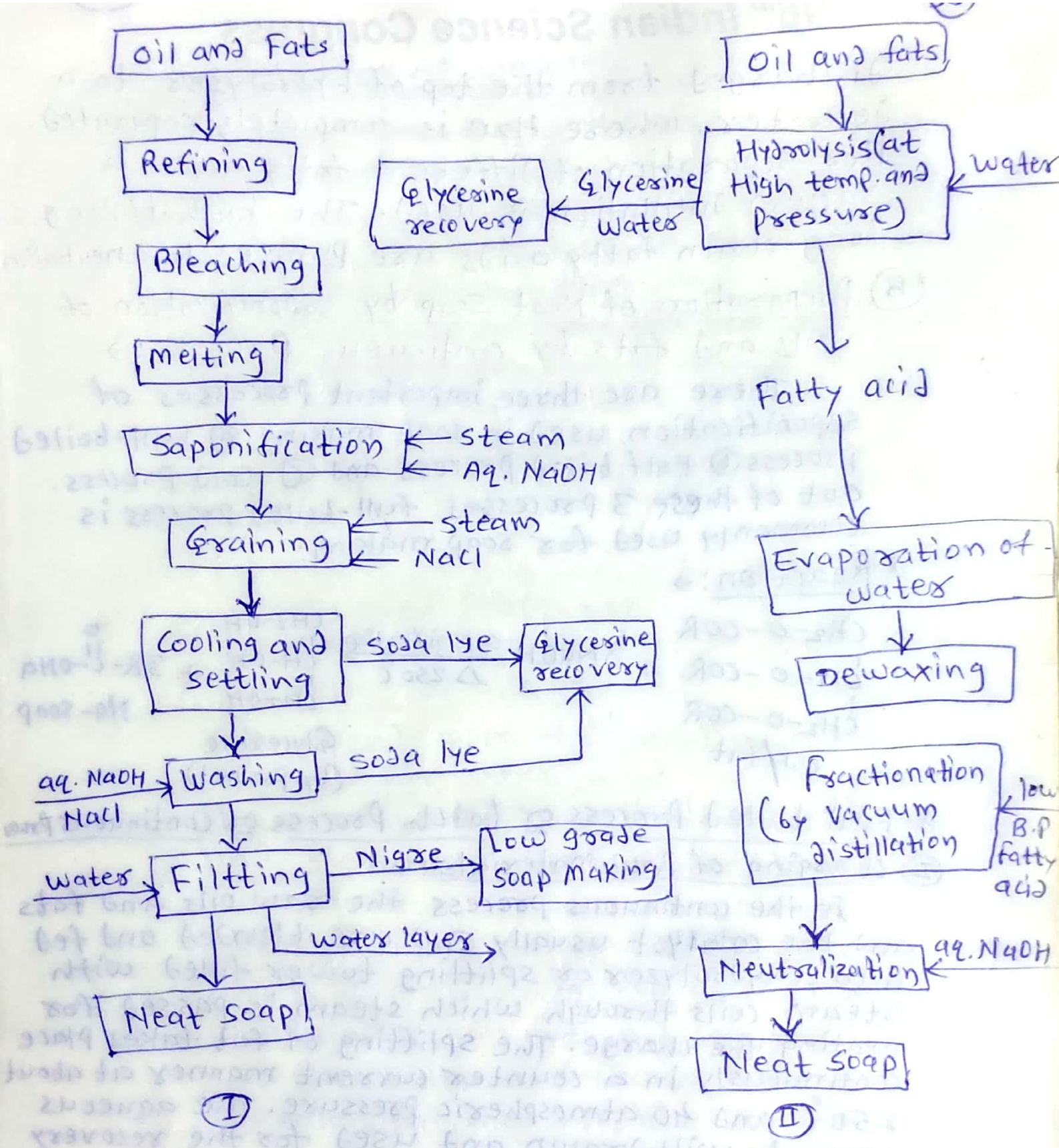

* Fig: Flow sheet for Manufacture of neat soap:

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(b) Saponification :→ The purified fatty acids are pumped to a neutraliser which is fitted with high speed mixer. The fat-aqueous alkali mixture is heated to boiling by steam. small amount of caustic soda (NaOH) is added time to time, as long as the oils and fats or fatty acids will get neutralised. The boiling is continued until saponification is essentially complete. The saponification is completed in 3 to 4 hours giving a smooth and creamy paste, which is then passed to a granulator.

(c) Graining :→ The graining is effected by addition of common salt (NaCl) to the saponified mass. The boiling is continued until the soap has separated, forming the upper soap layer and the lower soda lye layer. The steam is then turned off and the kettle settled. The lower soda lye containing glycerine and water together with dissolved small quantities of soap, caustic soda and other organic impurities are drawn off at the bottom. The degree of graining is a critical factor determining the volume of the lye and the amount of soap left in the lye. This whole operation is known as saponification change and requires about 8 hours. Depending upon the requirements, Preservatives, colours, germicides, perfumes and builders are added to blend the soap.

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④ Finishing :→ In this process the pressure on the blended neat soap is raised to about 35 atmosphere and soap is heated to about 200°C in high pressure steam exchanger. This heated soap is released to a flash tank at atmospheric pressure, where a partial drying takes place.

The continuous process takes about a day to complete the process as against several days required by the batch process. The solid soap thus obtained contains about 75% soap, 30% water and 0 to 20% NaCl.

For laundry soap, fillers or builders, such as sodium silicate, sodium phosphate, sand, soda-ash etc are added in various proportions.

* Special Soap products :→

① Toilet soap :→ The toilet soaps are prepared from purest mixture of tallow and coconut oil in the ratios 80:20 or 90:10 respectively. It contains no fillers. It has about 10% moisture. Caustic soda (NaOH) is used, which makes a hard and stable soap. Caustic potash (KOH) produces a soft soap. The good quality toilet soaps have the following important characteristics.

1. Easy solubility in water with profuse lathering.
2. Persistence of the soap fragrance.
3. Smooth, glossy appearance without any cracks.
4. The colour of the soap should not be transferred to fabric or skin.

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② Super fatted soap :→ These soaps are made by the addition of 1 to 6% of unsaponified oil or fatty acid or anhydrous lanolin or soft paraffin jelly in toilet soap base during the milling process, which produces a soft cold cream effect by leaving a residual film on the skin after washing.

③ Transparent soap :→ These soaps are usually made by semi-boiled method. Addition of alcohol, sugar, glycerin or castor oil to the hot soap inhibits the crystal growth during frame cooling and yields a glassy or transparent soap. e.g. Pears, savlon, etc.

④ Medicated soap :→ These toilet soaps contain small quantities of antibacterial or disinfecting agents like phenol, cresol, halogenated carbanilides, polybrominated salicylanilides. These agents are added during milling process. Antibacterial agents are effective in suppressing the growth of gram-positive skin bacteria responsible for body odour.

⑤ Shaving Soaps and shaving creams :→

Shaving soaps are formulated by saponifying a mixture of good quality tallow and coconut oil, with small quantities of castor oil, lard oil and lanolin with caustic potash and soda using full boiled saponification process. During milling process small amounts of glycerine and white

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paraffin are added to improve smoothing property. This soap is perfectly neutral in order to prevent irritation. It produces lather that persists for long time.

Shaving creams are generally formulates by saponifying a mixture of coconut oil and stearic acid with mixed solution of caustic potash and soda. About 5-10% glycerol is added and 4-8% of stearic acid is left neutralized to have creamy and lasting lather.

(6) Floating soap :→ These soaps are made by bubbling air through the melted neat soap until the amount of air incorporated increases its volume to such an extent that the solid soap becomes lighter than water. It contains about 30% water e.g. Lecancy soap.

(7) Cleaning Powder and shampoos :→

(a) Cleaning Powder :→ These soaps products are manufactured in bar, paste or powder forms and contain insoluble materials like talc, quartz sound, pumice, china clay, feldspar, etc in addition to filling agents like sodium silicate, sodium carbonate, sodium borate etc. Generally these soap products contain very low percentage of soap. (5-10%). It is used for domestic and industrial cleaning operations.

(b) shampoos :→ Raw materials used for manufacture of shampoos:

(1) A synthetic detergent such as sodium or potassium lauryl sulphate, alkyl benzene, polyoxy ethyl sulphonates etc.

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(ii) A solubilising agent such as alcohol, Ureq, sodium toluene sulphonate.
(iii) Amine oxides as foam stabilizers.
(iv) Disodium salt of EDTA as cleaning agents.
(v) Formaldehyde as preservative.
(vi) Lanolin as hair softner and
(vii) Lemon juice is used as scum remover in case of soap shampoos.

* Qualities of good shampoos :→

(1) It must not irritate the skin and the eyes and it should act as hair dresser in addition to hair cleaner.
(2) After drying, the shampoos makes the hairs easily combable, quick setting, lustrous and attractive.
(3) It may be coloured or colourless and may be available as solution emulsion, paste or even powder.
(4) It must posses the quality of thoroughly cleaning the hair, removing all dirt, dust and oiliness.
(5) It should posses a very pleasant and agreeable odour.

(B) Detergents Industry:

Synthetic detergents (syndets) are soap substitutes and were initially developed to avoid the short supply of soaps due to the shortage of edible oils and fats. It contain surface active agents (surfactants) along with detergent additive. It have better wetting and cleaning properties compared to soap

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the cleaning activity of synthetic detergent is not affected by hardness of water.

* Advantages of Detergents:→

① Detergents can be used in hard water and in textile processing industry.
② Detergents are made from the petroleum products, the oils and fats could be saved.
③ It can be used for washing delicate fibres like knitted wool and silk.
④ It is more active than soap in comparatively low concentration.
⑤ It is excellent foaming agent.
⑥ It has germicidal and bactericidal properties.

* Disadvantages of Detergents:→

① Most of the detergents are not easily biodegradable as compare to soap and hence cause water pollution.
② Manufacturing process of surfactants also cause air pollution problems.
③ Detergents are more toxic than soap.
④ Detergents requires the addition of soil-suspending agents while soap does not require it.
⑤ Detergents recovery is very difficult as compared to that of soap, when used in large amount.
⑥ Antibacterial agents are not very effective when used in synthetic detergents.

* Meaning of the terms:→

① Detergents and Surfaltant:→

Detergents may be regarded as a chemical formulation, which essential consists of surface active agents and subsidary constituents such as fillers, builders, boosters etc. Detergents may be solid, liquid, paste or powder.

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Surfactants are nothing but the surface active agents. When dissolved in water or dispersed in a liquid it cleans the surface by removing oil, in which dust particles are dispersed. The cleaning action of a surfactant depends upon its surface activity. It is a property which decrease the surface tension at the boundary surface between two phases. Thus those substances that lower the surface tension of water are called surfactants. Surfactants are the organic compounds in which two dissimilar groups water soluble and water insoluble are present within the molecule. e.g. sodium lauryl sulphate, sodium stearyl sulphate, sodium aryl sulphonates, linear alkyl benzene sulphonate (LAS), amide sulphonates etc.

(2) Emulsion and Emulsifying agents:→

Emulsion refers to any dispersion of one liquid in another. When two immiscible liquids e.g. water and oil are shaked together, the oily liquid is dispersed in fine droplets, but the emulsion is not stable.

Emulsifying agents or emulsifiers are the agents that lower the interfacial surface tension so that the emulsion is easily formed and also stabilise the emulsion by protecting the dispersed fine droplets of oil by forming a layer around them. The layer is formed by solubilizing the non polar part of the emulsifying agent in oil drop, while the polar group remains in water.

(3) Wetting and Non-wetting:→

Wetting means the spreading of liquid on

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* Types of surfactants :→

On the basis of their hydrophilic or solubilising groups present in the molecule the surfactants are classified into four types.

(I) Anionic (II) Nonionic (III) Cationic and (IV) Ampholytic

(I) Anionic Surfactants :→

The hydrophilic group in anionic surfactant is polar and negatively charged in aqueous solution or dispersions. e.g. (a) Sulphates and sulphated products e.g. Fatty alcohol sulphates, ethylene oxide adduct sulphates.

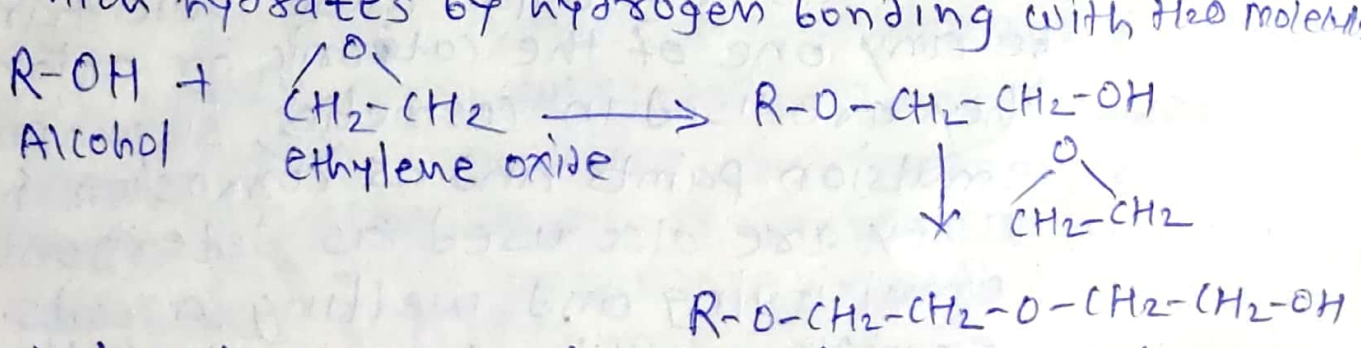
(b) Sulphonates :→ e.g. alkyl benzene sulphonates, Linear alkyl benzene sulphonates, Petroleum sulphonates, dialkyl sulphonates, olefin sulphonates.

(c) Carboxylates :→ e.g. soaps and amino carboxylates.

(d) Phosphate esters :- e.g. Na or K-alkyl Phosphates.

(II) Nonionic surfactants :→

These surfactant bear no charge when dissolved or dispersed in aqueous medium. The hydrophilic tendency in a non-ionic surfactant is due to presence of oxygen molecule (Phenol, alcohol) which hydrates by hydrogen bonding with H_2O molecule



Hydroxyl groups and ether linkages are the strongest hydrophilic groups in non-ionic surfactants.

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a surface with ease and this is attributed to a very small contact angle i.e. zero or close to zero between the liquid and the solid surface, so that the liquid spreads over the solid surface easily.

In case of non-wetting the contact angle between the liquid and the solid surface is greater than 90° and hence the liquid tends to ball up and run off the solid surface.

④ **Hydrophobic and Hydrophillic:→**

Hydrophobic means water hating or water disliking or water repelling substances, which are insoluble in water and hence repel H_2O. Hydrophilic means "water loving" or water liking substances.

⑤ **Amphipathic structre:→**

If a molecule contains two dissimilar structural groups e.g. water suluble and water insoluble, such a molecule is known to have amphipathic structure.

$CH_3-CH_2-(CH_2)_8-CH_2-C(=O)-ONa$

Hydrophobic (H_2O insoluble) — Hydrophillic (H_2O soluble)

⑥ **Micelles:→**

Micelles are the aggregates of many small molecules or groups of atoms which are held together by secondary valencies.

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$$R\text{-}OH + \underset{\text{Ethylene oxide}}{\overset{O}{\diagup\!\diagdown}\!CH_2\text{-}CH_2} \longrightarrow R\text{-}O\text{-}CH_2\text{-}CH_2\text{-}OH$$

Alcohol

$$R\text{-}O\text{-}CH_2\text{-}CH_2\text{-}O\text{-}CH_2\text{-}CH_2\text{-}OH$$

Hydroxyl groups and ether linkages are the strongest hydrophilic groups in non-ionic surfactants.

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Non-ionic surfactants are more effective than anionic surfactants in removing soil at the lower temperatures necessarily for laundering synthetic fibres. They are also more effective for removing body oils. e.g. ① Ethylene oxide adducts e.g. Polyoxy ethylene surfactants, ethoxylated alkyl Phenols and aliphatic alcohols etc.
② Polymeric nonionics
③ Alkylol amide and sorbitol compounds.
④ Carboxylic esters and amides.

(III) Cationic surfactants :→

The hydrophilic groups in cationic surfactants are amino or quarternary nitrogen. These amino groups or quarternary nitrogen bear a positive charge when dissolved in an aqueous medium. They acts as wetting agents rather than detergents. They are more expensive. They are also used as softeners for textile and paper. They can also be used as antibacterial algicidal agents eg. ① Amines containing oxygen amine oxides, poly oxyethylene, alkylamines.
② Amines having amide linkage and quarternary ammonium salt. ③ Amines not containing oxygen.

(IV) Ampholytic surfactants :→

These surfactants contain both anionic and cationic groups. These ionic functions may be any one of the cationic or anionic groups. They are used in cosmetics, shampoos, water emulsion paints and as corrosion inhibitors. They are also used as detergents sanitizers emulsifying and wetting agents. A typical example of this class is N-fatty-β-amino propionic esters.

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* Raw materials for Detergents :→

The important raw materials required for the manufacture of detergents are

ⓐ straight chain alkyl benzene.
ⓑ Fatty acids and alcohols
ⓒ Detergent builders and
ⓓ Additives.

ⓐ Straight chain alkyl benzenes:

Detergents are made from phenyl substituted n-alkanes containing 11 to 14 carbon atoms. The straight chain paraffins or olefins needed are produced from petroleum or from ethylene.

ⓑ Fatty acids and alcohols :→

Saturated fatty acids such as stearic acid as well as unsaturated fatty acids like oleic acids have been used in many industries as free acids or their salts. Fatty alcohols are Prepared by two important methods: Ziegler catalytic process and methyl ester hydrogenation process starting from α-olefins.

ⓒ Detergent Builders :→

These include inorganic salts such as Na-sulphate, Na-Silicate, Na-carbonate, Na-Phosphate. They are called detergent builders because they increases the cleansing or washing activity of surfactants and thus make the detergent more effective. Addition of these builders into synthetic detergents lowers the cost of the resulting Product without affecting its wetting and detergents activities.

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(d) Additives :→

These are the substances added to detergent for several purposes.

(i) Bleaching agents: such as sodium hydrosulphite or sodium hypochloride incorporated in some of the surfactants for removing colouring matter.

(ii) Corrosion inhibitors :→ Na-silicate, benzotriazole etc are added in order to protect the metals, utensils, dishes and other materials from the action of detergent and H_2O.

(iii) Opacifying agents :→ These are water soluble polymeric compounds used in the formulation of self stable clear or creamy liquid syndet.

(iv) Optical brighteners :→ These are the derivatives of coumarin or stilbene. These are colourless fluorescent dyes which are absorbed by textile fibres from solution and are not removed on rinsing. Their function is to convert ultraviolet radiation into visible blue light, thus making the grayness on the washed garment. The mount of optical brighteners added to the detergents varies from 0.001 to 0.1 %.

(v) Soil redeposition preventing agents :→

Na-carboxy methyl cellulose is extensively used in detergents to prevent soil redeposition and is more effective with cotton. For synthetic fibres, Polymers such as Polyvinyl Pyrrolidone and Polyvinyl alcohol are used for preventing soil redeposition.

(vi) Hydrotropes :→ These are added to liquid detergents. Their function is to 'drive' the detergents and builders into solution to

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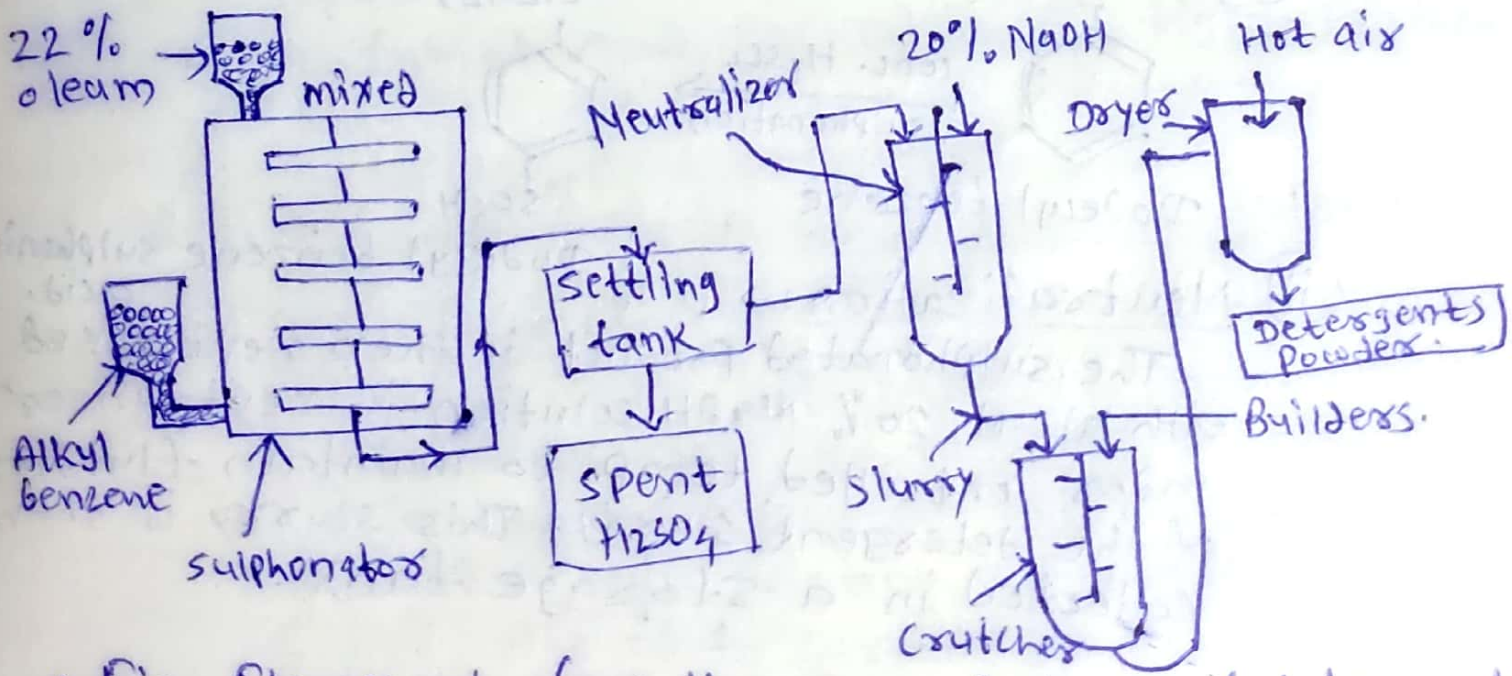
effect the solubilising action. e.g. Na or K salts of benzene, toluene, cumene and xylene sulphonates are most commonly used hydrotropes.

(VII) Enzymes :→ Recently certain enzymes are used in detergents for removing stains. These include proteolytic and amylolytic enzyme detergent additives. They are used in about 1000 to 1300 units per litre in the wash. Enzymes used for this purposes are α-amylase, alkaline protease, mexatase etc.

* Manufacture of Detergents :→

The alkyl benzene sulphonate (ABS) is the important raw material used in the manufacture of detergents. Alkyl benzenes are produced by condensing olefins with benzene in the presence of anhydrous $AlCl_3$. This reaction is known as Friedel-Crafts Alkylation reaction. (FCA).



* Fig: Flowsheet for the manufacture of detergents.

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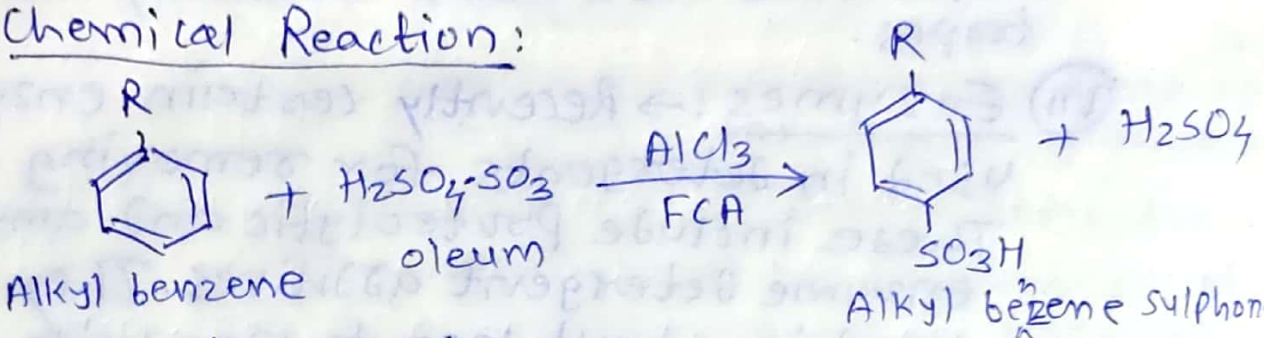
(a) **Raw materials:** → Alkyl benzene sulphonate (ABS) anhydrous $AlCl_3$ catalyst, oleum, sodium hydroxide additives etc.

(b) **Chemical Reaction:**



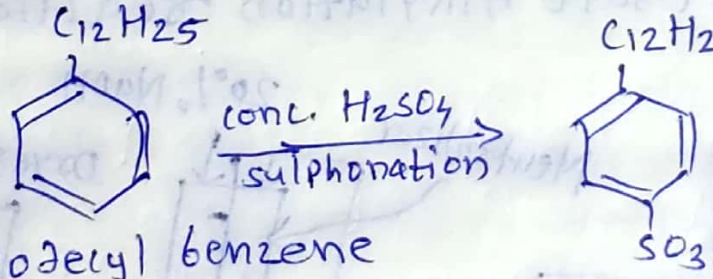
Alkyl benzene

Alkyl benzene sulphonate

(c) **Steps involved in the manufacture of detergent**

(i) **Sulphonation:** → Alkyl benzene is fed continuously into the sulphonator with oleum. Sulphonator is a vessel fitted with a mixer. Here alkyl benzene is converted into alkyl benzene sulphonate. The temp. is maintained between 32–45°C for about 2 hours. Then the reaction mixture is passed to the settling tank. The reaction is allowed to settle in the settling tank for 4 hours and lower spent H_2SO_4 layer is removed. The upper portion is sent to the neutraliser.

e.g.



Dodecyl benzene

Dodecyl benzene sulphonic acid.

(ii) **Neutralization:** →

The sulphonated product is then neutralised with about 20% NaOH solution in neutraliser under controlled temp. to maintain fluidity of the detergent slurry. This slurry is then collected in a storage tank.

PTO

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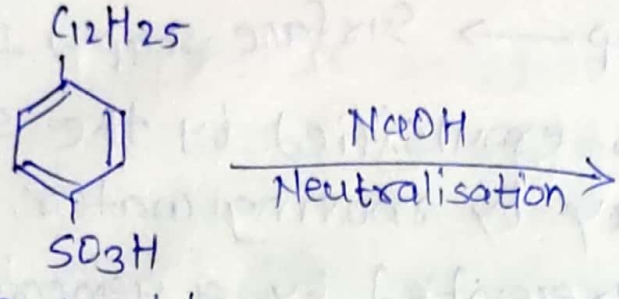

Dodecyl benzene sulphonic acid

Dodecyl benzene sodium sulphonate.

(iii) Mixing :→ The surfactant slurry, sodium triphosphate and most of the miscellaneous additives are added into crusher. During mixing water is removed and paste is thickened by tripolyphosphate due to hydration reaction.

(iv) Granulation :→ The above thickened paste is then passed through spray tower, where it is sprayed under high pressure. Hot air is passed through furnace. Dried granules of desired shape and sizes are formed.

(v) Packaging :→ The dried granules are transfered to storage tank. These granules are then separated in a cyclone, then screened, perfumed and packed in air tight Polythene bags.

* Washing Action of Soaps and Detergents :→

(a) Washing action of soap :→ When a soap is dissolved in water, it has hydrophobic character of the hydrocarbon like tails, due to formation of oriented monolayers at the surface with the hydrocarbon tails pointing outwards. The removal of dirt adhered to surface by soap solution proceeds according to general equation -

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Surface-Dirt + Soap ⟶ Surface-soap + Dirt-soap.

The dirt itself is emulsified by the soap and carried away by moving water.

Soap is represented by a general formula $R-COO^- Na^+$ where as anionic detergents can be represented as $R-O-SO_3^- Na^+$. In both cases R represents a long chain alkyl radical. Thus both molecules contain two parts.

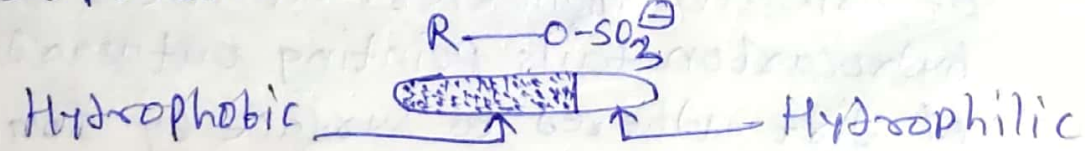
$CH_3-CH_2-(CH_2)_6-CH_2-COONa$ (Na-soap)

Hydrophobic (water insoluble) — Hydrophilic (water soluble)

Hydrophobic means water hating or water repelling substance. Hydrophilic means water loving or water liking or water attracting substances.

(6) Washing action of Detergents:→

The washing or cleaning action of detergent consists of ① Thoroughly wetting the dirt and the surface of the article being washed.
② Removing the dirt from the surface and
③ Maintaining the dirt in a stable solution or suspension.



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When washing is done with detergent, it increases the wetting ability of the water so that it can easily penetrate the fabric and get to the location of the soil. The soil is removed by the process of wetting, emulsifying dispersing or solubilizing the soil by the cleaning agent. Detergent molecule can aggregate in water into spherical clusters called micelles. The hydrocarbon part (R) of the molecules gather together on the inside of the micelle and the Polar groups ($-OSO_3^-$) are on the outside. Oil soluble, water insoluble compounds such as dyes are often dissolved into the centre of the micelle attracted by the hydrocarbon groups. This process is known as solubilization. In this way during soil removal, hydrophobic (water hating) ends of the molecules are attracted to a soil particle and then the soil particle is surrounded by the hydrophobic ends. At the same time hydrophilic ends are pulling the soil particles away from the fabric and then washed away. This action enables a soap or detergent to remove soil, suspend it and keep it away from redeposition on clothes.

* Comparison of Soaps and Detergents.

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Soaps.

(i) Soaps are the salts of long chain mono-carboxylic acids. i.e. Na or K salts of higher fatty acids.

(ii) Soap making involves the use of oils and fats which have potential food values.

(iii) The cleaning action of soap is reduced in hard water.

(iv) It is poor foaming agent.

(v) It cannot be used in acidic solution, due to formation of sticky ppt getting adhered to textile fibres.

(vi) It is less active and requires more concentration.

(vii) It has no germicidal and bactericidal properties.

(viii) Its recovery is possible, when used in large amounts.

(ix) It is biodegradable and hence not cause water pollution.

Detergents.

(i) Detergents are chemical formulations that consists of surface active agents and subsidiary constituents such as filler, booster, builders, etc.

(ii) The surfactants required for detergents are made from the petroleum products.

(iii) The cleaning activity of detergents is not affected by hardness of water.

(iv) It is excellent foaming agent.

(v) It can be used in acidic solution and also for washing delicate fiber like wool and silk.

(vi) It is more active and require low conc".

(vii) It has germicidal and bactericidal properties.

(viii) Its recovery is very difficult as compared to soap when used in large amounts.

(ix) It is not easily biodegradable and hence causes water pollution.

—X—

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