Nature of Invention: Chemical molecule and synthesis route

Applicant: QuantiVEX

Inventors: Shubham Singh, Pulkit

Chemical Formula: **($R\text{-}C_6H_4\text{-}SO_3Na$)**

Chemical Name: Linear alkyl benzene sulphonate

Chemical synthesis routes:

a. **Sulfonation of linear alkyl benzene**

Raw materials

1.Linear alkyl benzene($R\text{-}C_6H_4$) ,

2.SO_3 (sulphonating agent)

3. Hydrogen peroxide (a protic reagent and oxidising agent)

4. A Neutralizing agent

Sulfonation:

Reaction:

linear alkyl benzene + So_3(sulfonating agent) → linear alkyl benzene sulfonic acid

Mechanism:

Sulfonation of linear alkyl benzene (LAB) is an **electrophilic aromatic substitution (EAS)** reaction. The sulfonating agent, sulfur trioxide (SO_3), acts as an electrophile and attaches to the benzene ring of LAB.

1. **Activation of Sulfur Trioxide (SO_3):**
   a. SO_3 is a strong electrophile and reacts with benzene directly.
   b. It forms a highly reactive intermediate due to the resonance stabilization of the benzene ring.
2. **Formation of the Arenium Ion (σ-complex):**
   a. The benzene ring donates electrons to SO_3, forming an unstable carbocation (arenium ion).
   b. This intermediate is stabilized via resonance.
3. **Proton Transfer and Formation of the Product:**
   a. A base (often water or trace H_2SO_4) removes a proton from the arenium ion.
   b. This restores aromaticity, leading to the formation of **linear alkyl benzene sulfonic acid**.

Reaction Conditions:

- **Temperature:** 0 - 90°C (typically controlled to prevent over-sulfonation), 25 – 50°C more preferable.
- **Pressure:** Up to 100 psi or more.
- **Solvent:** Can be carried out in a gas phase or liquid phase; sulfuric acid may be used as a solvent.
- **Reaction Type:** Highly **exothermic**, requiring efficient cooling.

Process Efficiency:

- Yield: 96~99%
- Purity: 96~98%

Sulfonation Process: Detailed Mechanism and Kinetics

The **sulfonation of Linear Alkyl Benzene (LAB) follows first-order kinetics**, with the reaction rate expressed as:

$$r = k[LAB][H_2SO_4]^2[H_2O]^{-1}$$

where excess sulfuric acid increases reaction speed, but excess water formation slows it down. The activation energy for the reaction is **18.75 kcal/mol**, as determined from Arrhenius modeling .

To ensure complete conversion (>98%), the molar ratio of **LAB:H_2SO_4 should be maintained at 1:5**. The **semi-batch reactor configuration** with **controlled acid addition** helps manage exothermic heat release. A **cooling jacket** is required to maintain **optimal reaction temperature (60°C)**.

Bleaching:

Reaction:

linear alkyl benzene sulfonic acid + hydrogen peroxide →linear alkyl benzene sulfonic acid

(Dark coloured) (Light coloured)

Mechanism:

1. Oxidation of Impurities:
   a. Impurities (e.g., polymeric species, oxidized byproducts) contain conjugated double bonds, leading to colour.
   b. H_2O_2 oxidizes these compounds, breaking their extended conjugation and rendering them colourless.

2. Radical Formation:
    a. Under suitable conditions, H_2O_2 decomposes to form hydroxyl radicals (·OH).
    b. These radicals oxidize organic impurities to CO_2 and water.

Reaction Conditions:

- **Temperature:** 0 - 50°C
    - Higher temperatures accelerate oxidation but may degrade the product.
- **Pressure:** Atmospheric pressure
- **Catalyst:** Sometimes metal catalysts (e.g., sodium tungstate) are used to enhance bleaching efficiency.
- **Solvent:** The reaction is typically carried out in an aqueous medium.

Process Efficiency:

- Yield: 98~99%
- Purity: 96~98%

Neutralization:

Reaction:

$$\textit{Linear alkyl benzene sulfonic acid} \xrightarrow[\text{(Neutralizing agent)}]{} \textit{linear alkyl benzene sulfonate}$$

Mechanism:

1. **Acid-Base Reaction:**
    a. The sulfonic acid ($-SO_3H$) group reacts with a base, donating a proton (H^+).
    b. This forms the corresponding **sulfonate salt ($-SO_3^-$ Na^+)**.
2. **Salt Formation and Stabilization:**
    a. The product (linear alkyl benzene sulfonate, or **LAS**) is **water-soluble** and **stable**.
    b. This reaction prevents further oxidation and degradation.

Reaction Conditions:

- **Temperature:** Room temperature to 50°C
- **Pressure:** Atmospheric pressure
- **pH control:** The final solution must be **mildly alkaline (pH ~7-8)** to ensure complete neutralization.
- **Agitation:** Stirring is essential to ensure uniform mixing.

Process Efficiency:

- Yield: 98~99%
- Purity: greater than 99

Purification Processes:

After neutralization, the LABS solution still contains impurities such as **residual salts, excess alkali, unreacted LAB, sulfones, sulfates, excess neutralizing agents and side products**. These must be removed for high product quality.

a) Removal of Unreacted LAB

- **Liquid-Liquid Extraction:** Using a solvent such as **methanol or ethanol**, LAB can be selectively extracted.
- **Vacuum Distillation:** Since LAB has a lower boiling point than LABS, distillation can separate it effectively.
- **Adsorption:** Activated carbon or silica gel can absorb residual LAB.
- **Vacuum Stripping:** unreacted LAB is separated using **vacuum distillation** or **thin-film evaporation**. This ensures a **high-purity LABS product** with minimal contamination.

b) Removal of Sulfones and Sulfates

- **Washing with Water or Brine:** Dissolves and removes **sulfates** and some polar impurities.
- **Solvent Purification:** Methanol or ethanol can help separate sulfones from LABS.
- **Filtration through Activated Carbon:** Removes sulfones and organic residues.

c) Filtration and Drying

- Once LABS is purified, it is filtered to remove any remaining solid impurities.
- **Common filtration techniques:**
  - **Vacuum Filtration:** Speeds up separation of solids.
  - **Membrane Filtration:** Removes ultra-fine particles.
- **Drying Methods:**
  - **Spray Drying:** Converts LABS solution into dry powder form.The liquid LABS is sprayed into a hot air chamber, where water evaporates, leaving behind dry LABS powder.
  - **Vacuum Drying:** Used when LABS must be dried without exposure to high heat.
- Activated carbon treatment may be applied to remove any **color impurities**.

d) Crystallization (for High Purity LABS)

- Dissolve LABS in a solvent (e.g., ethanol).
- Slowly cool the solution to allow pure LAS crystals to form.
- Filter and dry the crystals.

Purity Testing and Final Product Evaluation

After synthesis, the **quality of LAS** is determined using:

1. **FTIR/NMR spectroscopy**: Confirms sulfonate ($-SO_3^-$) formation.
2. **Titration of residual acid**: Ensures neutralization is complete.
3. **High-Performance Liquid Chromatography (HPLC)**: Detects byproducts to maintain commercial-grade purity.

Scaling Up: Industrial Considerations

For **large-scale production**, Falling Film Reactors (FFR) are used to improve **reaction efficiency and heat management**. The **final product is spray-dried** to produce LAS in **powdered form**, making it ideal for detergent formulations.

References:

1)https://patents.google.com/patent/US2827484A/en

2)https://pmc.ncbi.nlm.nih.gov/articles/PMC8867685/

3)https://hithaldia.in/eccn/vol6_1a/J5_6.pdf

4)https://patentimages.storage.googleapis.com/1b/67/78/712514f5f0fc50/WO1997014676A1.pdf

List the contributions of each author:

1. **Shubham Singh:** worked on selection of the chemical and selection of the manufacturing processes and and contributed to the manufacturing analysis.

2. **Pulkit:** worked on studying the process, doing its manufacturing analysis.

Contributors:

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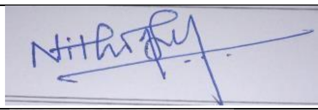
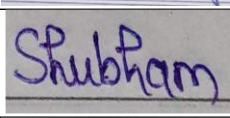
CEO Name	Nithin TM	
First author Name	Shubham Singh	
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