

## Quadrant II – Transcript and Related Materials

Programme: Bachelor of Science Second Year (S. Y. B. Sc.)

Subject: Industrial Chemistry

Paper Code: CHC153

Paper Title: General Industrial Chemistry

Unit: IX - Sulphonation

Module Name: Introduction, Sulphonating Agents and Chemical and Physical Factors in Sulphonation

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### Notes

#### Introduction:

- *Sulphonation* : Defined as any chemical process by which sulphonic acid group ( $-\text{SO}_3\text{H}/-\text{SO}_2\text{OH}$ ) or the corresponding salt of sulphonyl halide group ( $-\text{SO}_2\text{Cl}$ ) can be introduced into an organic compound.
- If the groups are attached to N atom, then they are known as N-Sulphonates/ sulphamates. E.g.  $\text{RNHSO}_2\text{ONa}$

#### Types of Sulphonation / Sulfonation

- |                                      |                      |
|--------------------------------------|----------------------|
| 1. Sulphochlorination                | 2. Halosulphonation  |
| 3. Sulphoxidation<br>Sulphoarylation | 4. Sulphoalkylation/ |

#### Classification of Sulphonates

- |                           |                                 |
|---------------------------|---------------------------------|
| 1. Aliphatic or Alicyclic | 2. Aromatic                     |
| 3. Heterocyclic           | 4. N-Sulphonates / Sulphamates. |

#### Uses and Applications:

1. Most are employed in their acid/salt form in various applications.

E.g. Methane/ Toluene Sulphonic acid                      Catalyst.



3) Batch/Residence time in relation to temperature and reagent strength

4) Catalyst

5) Solvents: Excess acid, Chlorinated and aqueous solvents.

- Presence of –OH, –NH<sub>2</sub> and alkyl (EDG) groups in the aromatic nucleus, increases the ease of Sulphonation
- Presence of Halogen, Nitrogen, Carbonyl and Sulphonyl (EWG) group makes Sulphonation more difficult.
- p-xylene is more difficult to sulphonate as compared to m or o xylene because of the bulky size of the entering sulphonyl group.
- Hg catalyst facilitates trisulphonation of benzene.
- In one case, in presence of catalyst; only α – sulphonate is formed in the Sulphonation of anthraquinone.

1) Concentration of Sulphonating agent:

- SO<sub>3</sub> concentration to minimum – leads to complete sulphonation.
- As the concentration of H<sub>2</sub>O increases the rate of sulphonation decreases.
- Excess acid is used to achieve maximum completion of sulphonation of alcohols.
- Removal of H<sub>2</sub>O by chemical method can be done by using SOCl<sub>2</sub>.

2) Chemical structure of the substrate:

- Aromatic compounds can be easily sulphonated than the other compounds

3) Batch/ Residence time in relation to temperature and reagent strength:

- To obtain the maximum efficiency, the reaction must be reduced to minimum compatibility with product quality and yield which may be accomplished by Using strong Sulphonating agents by raising the reaction temperature or by using larger proportion of the reagent.

#### **4) Catalysis:**

Adding of catalysis in small amounts can have marked effect on some sulphonation in various ways

- i) Changing the orientation
- ii) Facilitating the reaction
- iii) Inhibiting side reactions

#### **5) Sulfonation solvents:**

i) Excess acid:  $\text{H}_2\text{SO}_4$  – inexpensive, low viscosity solvent

ii) Chlorinated solvents – e.g. o-Dichlorobenzene is used in the modified form of 'Baking process' for sulphonating the aromatic amines. E.g.  $\text{CH}_2\text{Cl}_2$ ,  $\text{CHCl}_3$ ,  $\text{CCl}_4$ ,  $\text{Cl-CH=CH-Cl}$ .

iii) Aqueous solvents: Used for oxidative chlorination of thiol derivatives to the sulphonyl chlorides.

- $\text{H}_2\text{O}$  participates in the reaction providing oxygen, as well as a reaction medium.
- Addition of acetic acid increases the solubility of the organic compounds.

iv) Miscellaneous solvents:

- Dry Nitrobenzene is employed for converting 2-naphthol to 2-naphthol-1-sulfonic acid using Chlorosulphonic acid at  $0^\circ\text{C}$ .
- Carbohydrates (Cellulose and Alginic acid) can be sulfated with Conc. Acid, using an aliphatic alcohol (Butanol) as suspending medium.
- Pyridine  $-\text{SO}_3$  : reaction medium for preparation of Leuco Vat dyes, Alginic Acid can be sulphated by this method.

**Solvent Stability:** Reactive solvents can be employed with little loss if

- Organic compound is much more easily sulphonated than the solvent.
- Sulphonating agent is being added to the mixture of solvent and compound being sulphonated.

- **Temperature of Sulphonation can be maintained below the point at which the attack of the solvent becomes exclusive.**