Nature of Invention: Chemical molecule and synthesis route

Applicant: ChemEverse

Inventors: Sahil Kumar,Vibhanshu

Chemical Formula: CH2=C(R)-COO-(CH2CH2O)nR'

[R = H or CH_3 (from acrylic or methacrylic acid backbone)]

[R' = End group, often an alkyl or ether group]

Chemical Name : Polycarboxylate ether

Chemical synthesis routes:

(A) Synthesis routes:

Raw Materials and Chemicals needed:

a. Acrylic Acid **($C_3H_4O_2$)**
b. Methoxy Poly Ethylene Glycol (MPEG / PEGM 2000)**(C□H_2□$_{+1}$O□)**
c. Polyethylene Glycol (PEG-1000)**(H(OCH_2CH_2)□OH)**
d. Potassium Carbonate
e. Ethanol
f. Azobisisobutyronitrile (AIBN)**($C_8H_{12}N_4$)**
g. 3-Chloro-2-methyl-1-propene (CMP)**(CH_2=C(CH_3)-CH_2Cl)**
h. Methacrylic Acid**(CH_2=C(CH_3)-COOH)**
i. Sodium Hydroxide
j. Deionized Water

Reaction Steps :

1. **Synthesis of Main Chain (Copolymerization):**
   - Mix acrylic acid and CMP in a flask with AIBN as the initiator.
   - Perform the reaction at controlled temperatures (e.g., 50°C, 60°C, 70°C) for several hours (typically 6-8 hours) to form the main chain of the copolymer.
   - Monitor the reaction temperature carefully to avoid degradation of the polymer.

2. **Formation of Side Chains (Condensation Polymerization):**
    - Use the synthesized main chain and react it with MPEG or PEGM 2000 under condensation conditions to form the side chains of the polycarboxylate ether.
    - This step may involve esterification followed by neutralization reactions.

$$R-COOH+HO-(CH_2CH_2O)_n-CH_3 \rightarrow R-COO-(CH_2CH_2O)_n-CH_3+H_2O$$

3. **Neutralization and Finalization:**
    - Adjust the pH of the solution using sodium hydroxide to ensure the polycarboxylate groups are fully ionized.
    - This step is crucial for achieving the desired properties of the superplasticizer.

$$R-COOH+NaOH \rightarrow R-COO^-Na^+ + H_2O$$

Separation Steps :

1. **Filtration:**
    - Filter the reaction mixture to remove any insoluble impurities.
2. **Evaporation:**
    - Evaporate the solvent (e.g., ethanol) under reduced pressure to concentrate the PCE solution.
3. **Purification:**
    - Use techniques like dialysis or ultrafiltration to remove low molecular weight impurities and achieve higher purity.

Final Purity and Product Yield :

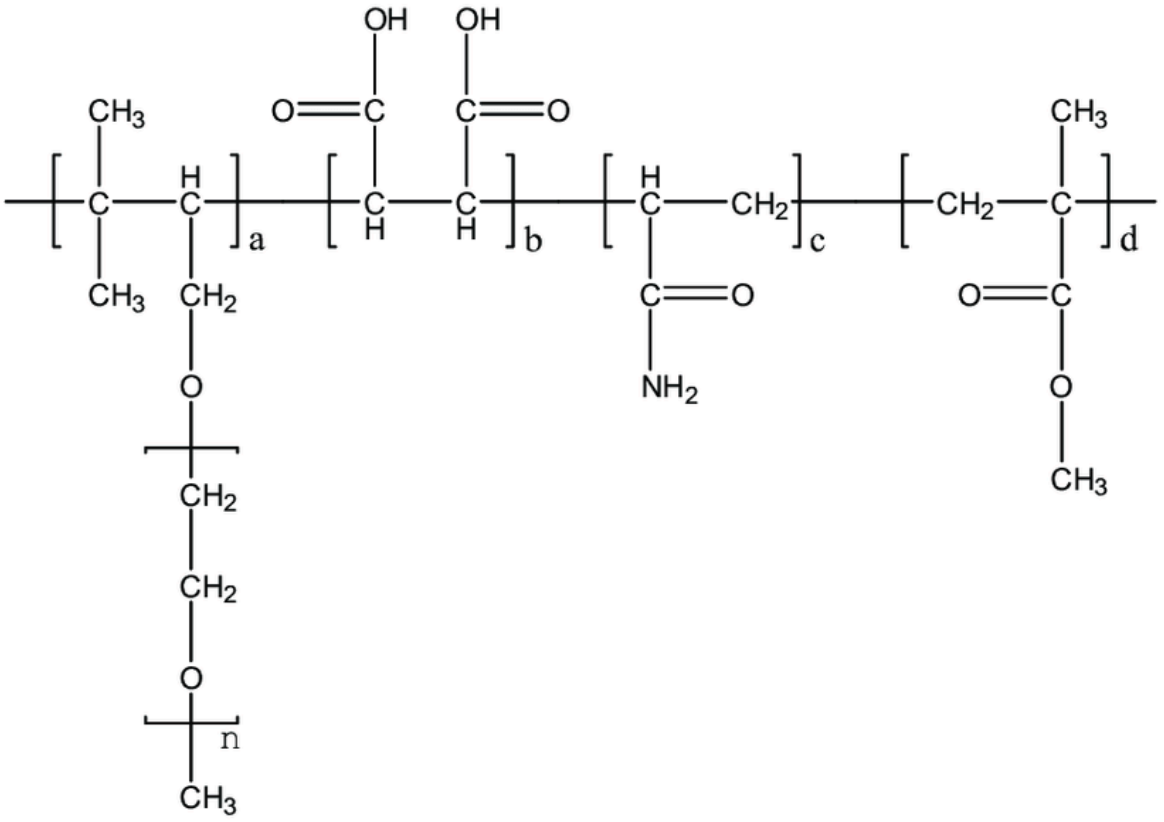
- **Final Purity:** The purity of the final product can be evaluated using techniques like Gel Permeation Chromatography (GPC) and Fourier Transform Infrared Spectroscopy (FTIR).
- **Product Yield:** The yield can vary based on the specific conditions and efficiency of the synthesis process. Typically, yields can range from 70% to 90% depending on the optimization of reaction conditions.

Reaction Conditions :

- **Temperature:** The synthesis temperature is critical, with optimal conditions typically around 70°C for the main chain formation.
- **Time:** Reaction times can vary from 6 to 8 hours depending on the specific step and conditions.
- **pH:** Adjust the pH to ensure complete ionization of the polycarboxylate groups, typically around pH 6-7.

References:

- Production of Modified Superplasticizer by Two-Step Synthesis of Polycarboxylate Ether, Advanced Journal of Chemistry, Section B, 2024.
- POLY CARBOXYL ETHER (PCE) PROJECT OF 18,000 T/Y, Environmental Clearance Document.
- Ether polycarboxylate superplasticizer and preparation method thereof, Patent CN104261720A.



synthesized formula of polycarboxylate superplasticizers (PCEs)

(B) Alternative Synthesis Routes:

Direct Polymerization Using Macromonomers :

- **Raw Materials:**
  - Polyglycol Methallyl Ethers (Polyglykol ML)
  - Acrylic Acid (AA)
  - Ammonium Persulfate (APS)
  - Water
- **Reaction Steps**:
  - Polymerization: Mix Polyglykol ML with AA in water and initiate polymerization using APS as the initiator. This method allows for direct incorporation of the polyether side chain into the main polymer chain without the need for esterification steps.
  - Neutralization: Adjust the pH of the solution using sodium hydroxide to ensure full ionization of the polycarboxylate groups.

- **Product Yield and Purity**: The yield can be high, typically above 80%, with purity depending on the efficiency of the separation steps.
- **Reaction Conditions**:
    - Temperature: Typically around 70°C to 80°C.
    - Time: Several hours, depending on the initiator concentration and temperature.

Polymer-Analogous Reactions :

- **Raw Materials**:
    1. Polyethylene Glycol Monomethyl Ether (MPEG)
    2. Methacrylic Acid (MAA)
    3. Toluene-p-sulfonic Acid
    4. p-Benzenediol
- **Reaction Steps**:
    1. Esterification: React MPEG with MAA in the presence of a catalyst like toluene-p-sulfonic acid at elevated temperatures (e.g., 80°C to 140°C) for several hours.
    2. Polymerization: Use the esterified product as a macromonomer for further polymerization with other monomers like AA.
    3. Neutralization: Adjust the pH using alkaline liquor.
- **Product Yield and Purity**: The yield can vary, but typically ranges from 70% to 90%. Purity depends on the separation efficiency.
- **Reaction Conditions**:
    1. Temperature: 80°C to 140°C for esterification.
    2. Time: Several hours for each step.

Bio-Based Modified Polyalkylene Oxide Derivatives :

- **Raw Materials**:
    - Isoprenyl Polyethylene Glycol Methyl Ether
    - Acrylic Acid (AA)
    - Sodium Persulfate
- **Reaction Steps**:

    - Polymerization: Use isoprenyl polyethylene glycol methyl ether as a monomer in a free-radical polymerization with AA, initiated by sodium persulfate.
    - Neutralization: Adjust the pH with sodium hydroxide.
- **Product Yield and Purity**: Yield can be high, typically above 80%, with purity depending on separation efficiency.
- **Reaction Conditions**:
    - Temperature: Typically around 70°C to 80°C.
    - Time: Several hours.

Separation Steps :

- **Filtration**: Remove insoluble impurities.
- **Evaporation**: Concentrate the solution under reduced pressure.
- **Dialysis or Ultrafiltration**: Remove low molecular weight impurities to achieve higher purity.

References:

- Synthesis and Properties of a Polycarboxylate Superplasticizer with a Jellyfish-Like Structure Comprising Hyperbranched Polyglycerols

- Synthesis and Modification of Polycarboxylate Superplasticizers—A Review

List the contributions of each author:

Sahil Kumar :

1. Focused on standard/ synthesis methods for PCE.
2. Investigated free radical polymerization of acrylic acid (AA) and 3-chloro-2-methyl-1-propene (CMP).
3. Used AIBN (Azo-bis-isobutyronitrile) as the initiator to drive polymerization.
4. Explored temperature variations (50°C, 60°C, 70°C) for optimal polymer yield.
5. Studied mechanisms for side-chain attachment using methoxy polyethylene glycol (MPEG) and PEGM 2000.

6. Examined esterification and neutralization reactions to improve superplasticizer performance.

Vibhanshu :

1. Focused on alternative pathways for PCE production.
2. Investigated different monomer combinations (using non-CMP-based copolymers).
3. Explored alternative initiators or catalysts for improved polymerization efficiency.
4. Considered green chemistry approaches (solvent-free methods and biodegradable catalysts).
5. Studied new esterification techniques and alternative PEG derivates for side-chain formation.
6. Evaluated the final product's efficiency compared to standard synthesis methods.

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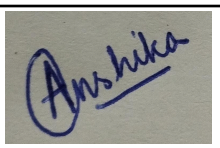
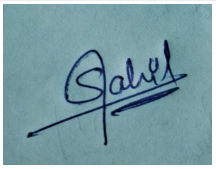
Anshika Agrawal	230160	
Sahil Kumar	230893	
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