CHE261A Patent Application

Nature of Invention: Chemical molecule and synthesis route

Applicant: *GreenovateX*

Inventors: *Neeraj Kajala*

Chemical Formula: *C56H108O30*

Chemical Name: *Hydroxypropyl Methylcellulose (HPMC)*

Chemical synthesis routes:

1. ***Lab-Scale Preparation of Hydroxypropyl Methylcellulose (HPMC) via Alkylation and Etherification of Cellulose***

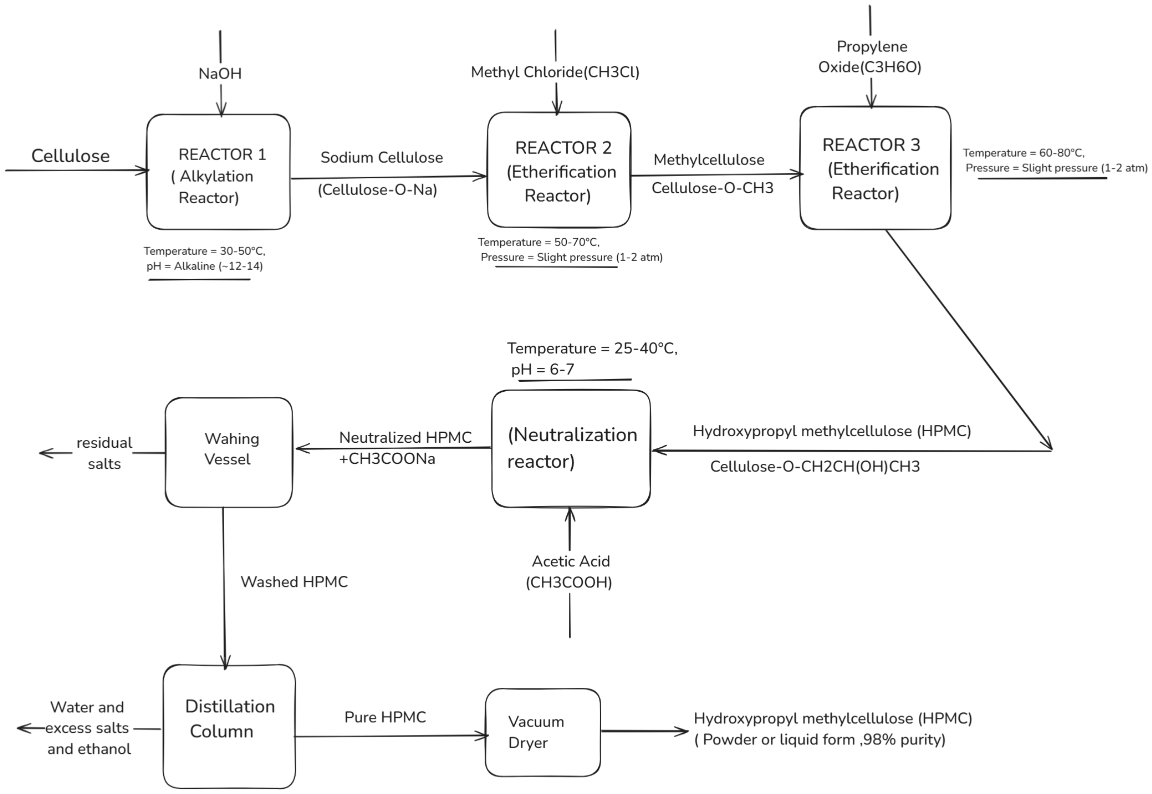
Chemicals required

- **Cellulose** (from wood pulp or cotton linters)
- **Sodium Hydroxide (NaOH)**
- **Methyl Chloride (CH3Cl)**
- **Propylene Oxide (C3H6O)**
- **Water (H2O)**
- **Acetic Acid (C3H3COOH)**
- **Isopropanol (C3H8O)**
- **Hydrogen Peroxide (H2O2)**

Vessels required

1. Alkylation Reactor (Stainless Steel or Glass-Lined Reactor)
2. Etherification Reactor (Stainless Steel or Glass-Lined Reactor)
3. Neutralization Tank
4. Washing Vessel
5. Filtration Unit (Vacuum Filter or Pressure Filter)
6. Drying Unit (Spray Dryer or Vacuum Dryer)
7. Product Storage Tank (HDPE or Stainless Steel)

Process analysis



Direct Etherification Method

Stage	Equipment Used	Output
Raw Material Prep	Mixing Vessel	Alkali Cellulose
Etherification	Reactor 1	Crude HPMC
Neutralization	Neutralization Tank	Neutralized HPMC
Filtration	Pressure Filter	Purified HPMC Solution
Drying	Spray Dryer	Pure HPMC Powder
Storage	Storage Tanks	Final HPMC Product

STEP 1: Alkylation of Cellulose with Sodium Hydroxide

Objective: **Convert cellulose into sodium cellulose for further reactions.**

Reaction :

Cellulose-OH + NaOH → Cellulose-O-Na + H2O

Reaction Conditions:

- **Temperature:** 30-50°C
- **Pressure:** Atmospheric
- **Reaction Time:** 1-2 hours
- **Catalyst:** Excess NaOH (~5-10% excess)
- **Continuous Stirring:** Ensures uniform reaction.

Process Flow:

- Cellulose is mixed with sodium hydroxide in a **Stirred Tank Reactor**.
- The mixture is heated to 30-50°C with continuous stirring.
- The resulting sodium cellulose is cooled and transferred to the next reactor.

Step 2: Etherification with Methyl Chloride

Objective: **Introduce methyl groups to the sodium cellulose to form methylcellulose.**

Reaction:

$$\text{Cellulose-O-Na} + CH_3Cl \rightarrow \text{Cellulose-O-}CH_3 + NaCl$$

Reaction Conditions:

- **Temperature:** 50-70°C
- **Pressure:** Slight pressure (1-2 atm)
- **Reaction Time:** 2-4 hours
- **Catalyst:** Methyl Chloride (CH_3Cl)

Process Flow:

- Sodium cellulose is transferred to **Reactor 1**, where it reacts with methyl chloride.
- The reaction is carried out under slight pressure to ensure complete methylation.
- The resulting methylcellulose is cooled and transferred to the next reactor.

Step 3: Etherification with Propylene Oxide

***Objective:* Introduce hydroxypropyl groups to the methylcellulose to form Hydroxypropyl Methylcellulose (HPMC)**

Reaction:

$$\text{Cellulose-O-Na} + C_3H_6O \rightarrow \text{Cellulose-O-}CH_2CH(OH)CH_3 + NaOH$$

Reaction Conditions:

- **Temperature:** 60-80°C
- **Pressure:** Slight pressure (1-2 atm)
- **Reaction Time:** 3-5 hours
- **Catalyst:** Propylene Oxide (C_3H_6O)

Process Flow:

- Methylcellulose is transferred to **Reactor 2**, where it reacts with propylene oxide.
- The reaction is carried out under slight pressure to ensure complete etherification.
- The resulting HPMC is cooled and transferred for neutralization.

Step 4: Neutralization and Washing

Objective: Neutralize the reaction mixture and remove impurities.

Reaction:

Cellulose-O-$CH_2CH(OH)CH_3$ +CH_3COOH → Cellulose-O-$CH_2CH(OH)CH_3$ + CH_3COONa

Reaction Conditions:

- **Temperature:** 25-40°C
- **Reaction Time:** 1-2 hours
- **pH:** Adjusted to 6-7 using acetic acid.

Process Flow:

- The crude HPMC is neutralized with acetic acid in a **Neutralization Tank**.
- The neutralized HPMC is washed with water to remove any residual salts.

Step 5: Filtration and Purification

Objective: **Remove impurities like NaCl, excess reactants, and solvents**.

Steps:

1. **Filtration:** Solid NaCl is removed via vacuum filtration.
2. **Solvent Extraction:** Ethanol or water is used to extract the surfactant phase.
3. **Distillation:** Excess solvent or water is evaporated

Process Flow:

- The reaction mixture is filtered to remove salts.
- The surfactant solution undergoes solvent extraction and is purified via distillation.

Step 6: Drying & Final Product Storage

Objective: Remove remaining moisture and store the final product.

Steps:

- Drying: The product is dried to achieve the desired consistency.
- Cooling & Storage: Final product is collected in tanks or packed into containers.

Process Flow:

- The purified product is dried under controlled conditions.
- The dried product is packed or stored as a liquid in holding tanks.

2 . Alternate Lab-Scale Preparation Using Sodium Cyanide

Chemicals required

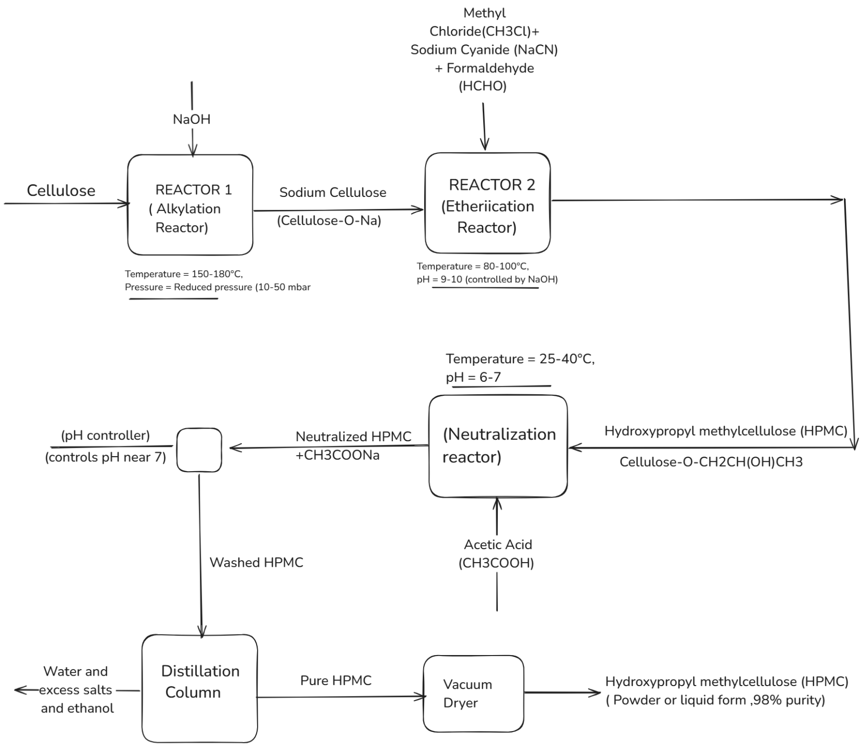
- **Cellulose** (from wood pulp or cotton linters)
- **Sodium Hydroxide (NaOH)**
- **Methyl Chloride (CH3Cl)**
- **Propylene Oxide (C3H6O)**
- **Sodium Cyanide (NaCN)**
- **Formaldehyde (HCHO)**
- **Hydrochloric Acid (HCl)**
- **Water (Deionized or Distilled)**
- **Ethanol or Isopropanol**
- **Hydrogen Peroxide (H2O2)**

Vessel required

- Storage Tanks

- Airtight Cyanide Storage Tank
- Jacketed Stirred Tank Reactor
- Vacuum System
- Heat Exchanger
- Nutsche Filter / Pressure Filter
- Thin Film Evaporator
- Liquid Extraction Column

Process analysis



Direct Etherification Method

Stage	Equipment Used	Output
Raw Material Prep	Mixing Vessel	Alkali Cellulose
Etherification	Reactor 1	Crude HPMC
Neutralization	Neutralization Tank	Neutralized HPMC
Filtration	Pressure Filter	Purified HPMC Solution
Drying	Spray Dryer	Pure HPMC Powder
Storage	Storage Tanks	Final HPMC Product

Step 1: Alkylation of Cellulose with Sodium Hydroxide

***Objective:* Convert cellulose into sodium cellulose for further reactions.**

Reaction:

Cellulose-OH + NaOH → Cellulose-O-Na + H_2O

Reaction Conditions:

- **Temperature:** 30-50°C
- **Pressure:** Atmospheric
- **Reaction Time:** 1-2 hours
- **Catalyst:** Excess NaOH (~5-10% excess)
- **Continuous Stirring:** Ensures uniform reaction.

Process Flow:

- Cellulose is mixed with sodium hydroxide in a **Stirred Tank Reactor**.
- The mixture is heated to 30-50°C with continuous stirring.
- The resulting sodium cellulose is cooled and transferred to the next reactor

Step 2: Etherification with Methyl Chloride and Sodium Cyanide

***Objective:* Introduce methyl and hydroxypropyl groups to the sodium cellulose using sodium cyanide and formaldehyde**.

Reaction:

Cellulose-O-Na + CH_3Cl + HCHO + NaCN → Cellulose-O-$CH_2CH(OH)CH_3$ + NaCl

Reaction Conditions:

- **Temperature:** 60-80°C
- **Pressure:** Slight pressure (1-2 atm)
- **Reaction Time:** 3-6 hours
- **Catalyst:** Sodium Cyanide (NaCN) and Formaldehyde (HCHO)

Process Flow:

- Sodium cellulose is transferred to **Reactor 2**, where it reacts with methyl chloride, formaldehyde, and sodium cyanide.
- The reaction is carried out under slight pressure to ensure complete etherification.
- The resulting HPMC is cooled and transferred for neutralization.

Step 3: Neutralization and Washing

***Objective*: Neutralize the reaction mixture and remove impurities.**

Reaction:

Cellulose-O-$CH_2CH(OH)CH_3$ + CH_3COOH → Cellulose-O-$CH_2CH(OH)CH_3$ + CH_3COONa

Reaction Conditions:

- **Temperature:** 25-40°C
- **Reaction Time:** 1-2 hours
- **pH:** Adjusted to 6-7 using acetic acid.

Process Flow:

- The crude HPMC is neutralized with acetic acid in a **Neutralization Tank**.
- The neutralized HPMC is washed with water to remove any residual salts.

Step 4: Filtration and Purification

Objective: **Remove impurities like NaCl, excess reactants, and solvents**.

Steps:

1. **Filtration:** Solid NaCl is removed via vacuum filtration.
2. **Solvent Extraction:** Ethanol or water is used to extract the surfactant phase.
3. **Distillation:** Excess solvent or water is evaporated

Process Flow:

- The reaction mixture is filtered to remove salts.
- The surfactant solution undergoes solvent extraction and is purified via distillation.

Step 5: Drying & Final Product Storage

Objective: Remove remaining moisture and store the final product.

Steps:

- Drying: The product is dried to achieve the desired consistency.
- Cooling & Storage: Final product is collected in tanks or packed into containers.

Process Flow:

- The purified product is dried under controlled conditions.
- The dried product is packed or stored as a liquid in holding tanks.

Comparision betwwen two methods:

- ***Alkylation and Etherification Method:***
  - *Higher yield (~95-98%)*
  - *Requires two separate reactors for alkylation and etherification.*

- *Longer reaction time (5-10 hours).*
- *More control over the degree of substitution.*

- ***Direct Etherification Method:***
  - *Simpler process with a single reactor.*
  - *Shorter reaction time (4-8 hours).*
  - *Lower yield (~90-93%).*
  - *Less control over the degree of substitution.*

Reasons Why the Sodium Cyanide Route is Not Preferred

Although the Sodium Cyanide (NaCN) route for HPMC production offers higher yield and lower raw material costs, it is less commonly used in industrial settings due to the following reasons:

Reason	Explanation
Toxicity	Sodium Cyanide (NaCN) is highly toxic, requiring airtight storage, full PPE, and strict handling protocols.
Hydrogen Cyanide (HCN) Risk	Cyanide can react with acids or moisture, releasing HCN gas, which requires scrubber systems to prevent gas leaks.
Cyanide Waste Disposal	Cyanide waste disposal is complex and requires neutralization with hydrogen peroxide (H_2O_2) or sodium hypochlorite (NaOCl) before safe discharge.
Effluent Treatment Costs	High costs due to the need for cyanide oxidation units in Effluent Treatment Plants (ETP).
Regulatory Compliance	Sodium cyanide and formaldehyde are restricted chemicals, requiring special handling permits and exposure monitoring.
High pH Sensitivity	The process requires continuous pH monitoring (pH 11-12) to avoid cyanide gas emissions.
Side Reactions	Cyanohydrin formation can lead to impurities, reducing final product purity and increasing purification steps.
Operational Costs	Although NaCN and formaldehyde are cheaper, the additional costs of safety systems, waste treatment, and regulatory compliance make the overall process more expensive.
Capital Expenditure (CAPEX)	Investment in gas scrubbers, emergency response systems, and effluent treatment increases CAPEX and OPEX.

References:

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https://www.sciencedirect.com/topics/chemistry/hydroxypropyl-methylcellulose https://en.wikipedia.org/wiki/Hydroxypropyl_methylcellulose

https://www.freepatentsonline.com/6232496.html

https://patents.google.com/patent/WO2016199944A1/en

List the contributions of author: Neeraj Kajala

- Designed and implemented a chemical synthesis route for the production of Hydroxypropyl Methylcellulose (HPMC), including raw material selection, process design, and optimization of key stages such as alkylation, etherification, purification, and drying.
- Investigated the alternate process using Sodium Cyanide and its flow diagram.
- Determined which process is more efficient and environmentally friendly for industrial production.

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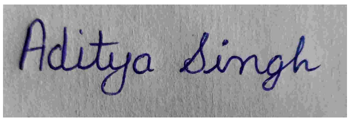
ADITYA SINGH	230079	
NEERAJ KAJALA	230691