Patent - 1 Isopropanol

Applicant: ProChem Innovations

Inventors: 1. Akshat Srivastava

2. Shrimi Aggarwal

3. Jatin Madan

4. Manas Sontakke

Chemical Formula: C_3H_8O

Chemical Name: Isopropanol

Chemical synthesis routes:

a. Raw Materials:
   i. Acetone
   ii. Bimetallic Cu-Al mixed oxide
   iii. Hydrogen Stream

b. Chemical Reactions:

Hydrogenation of acetone to IPA is a reversible reaction. The forward reaction is exothermic and the backward dehydrogenation reaction is endothermic. So, the reaction can be used as a potential chemical heat pump system. But, to achieve this, a high selectivity of IPA is required.

The other reactions that occur along with direct hydrogenation cause the formation of diacetone alcohol (DAA), mesityl oxide (MO) and methyl isobutyl ketone (MIBK). The reactions are:

Direct hydrogenation of acetone to IPA

$$(CH_3)_2CO + H_2 \rightarrow (CH_3)_2CHOH \Delta H = -55 kJ/mol$$

Condensation of acetone to diacetone alcohol

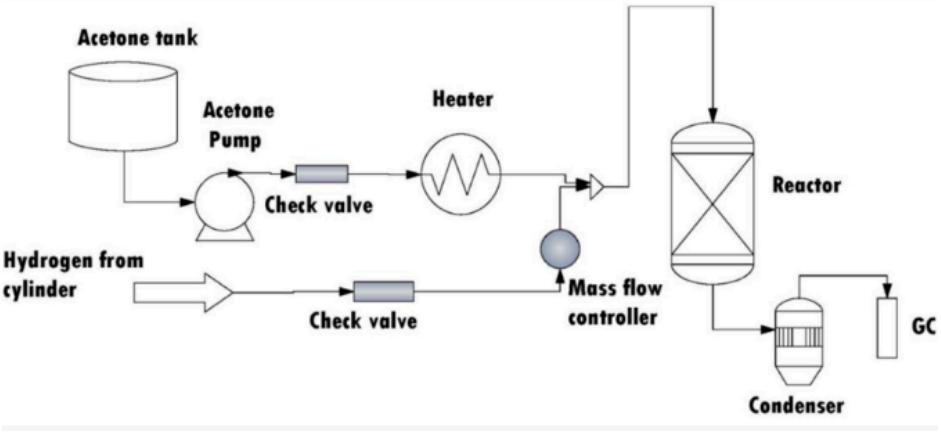
$$2(CH_3)_2CO \rightarrow (CH_3)C(O)CH_2C(OH)(CH_3)_2 \Delta H = -23 kJ/mol$$

Dehydration of diacetone alcohol to mesityl oxide

$$CH_3C(O)CH_2C(OH)(CH_3)_2 \rightarrow (CH_3)C(O)CH = C(CH_3)_2 \Delta H = 21 kJ/mol$$

Hydrogenation of mesityl oxide to methyl isobutyl ketone

$$CH_3C(O)CH = C(CH_3)_2 + H_2 \rightarrow (CH_3)C(O)CH_2CH(CH_3)_2 \Delta H = -21 kJ/mol$$



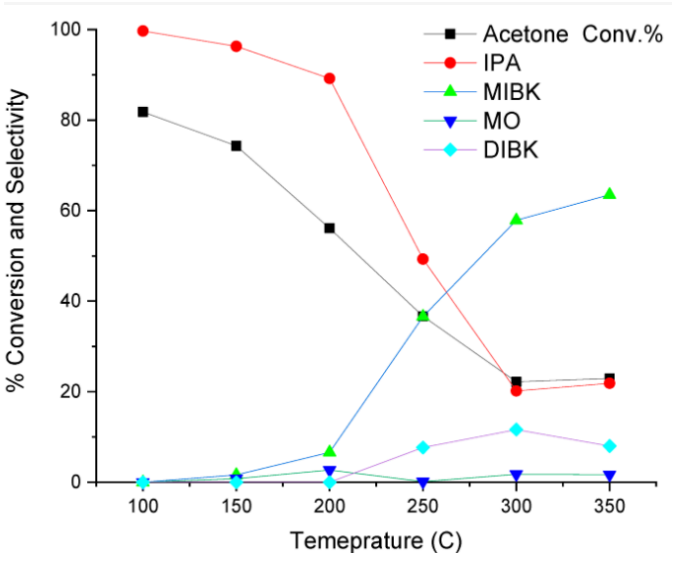
c. Experimental Analysis:

i. The performance of the synthesized catalysts was evaluated in a continuous packed bed reactor by varying parameters such as temperature (150 to 225 °C), H2/acetone mole ratio (0.5 to 1.25) and space-velocity (0.09 to 0.145 kmol acetone/kg cat. h).

ii. The optimum temperature at which the highest selectivity of IPA was obtained was 175 °C. The catalyst showed maximum selectivity for IPA at H2/Acetone mole ratio of unity. Beyond this mole ratio, the IPA selectivity declined.

iii. The study of variation in space velocity suggested that a high space velocity favors IPA selectivity. The best activity was obtained over a Cu: Al ratio of 0.5.

iv. The kinetic study was conducted with the best catalyst. The rate equations based on LHHW kinetic models were validated. The activation energy was estimated to be 44.3 kJ/mol.



d. Selection of Catalyst:

i. The catalysts that showed high IPA selectivity are Raney nickel, nickel-copper alloys, supported noble metals (Pt, Pd, Ru, Rh, Ir) and copper chromite

ii. There are certain demerits of the preferred catalysts such as the pyrophoric nature of Raney nickel (causing handling problems), expensive noble metals and the toxic nature of chromites (environmental hazard).

e. Other Methods of production:
   i. The commercial IPA manufacturing process comprises indirect and direct hydration of propylene.
   ii. The indirect hydration process causes separation and corrosion problems as it uses sulphuric acid as the catalyst.
   iii. The direct process is less corrosive but it requires pure propylene unlike the indirect process, which uses dilute propylene from the refinery stream.

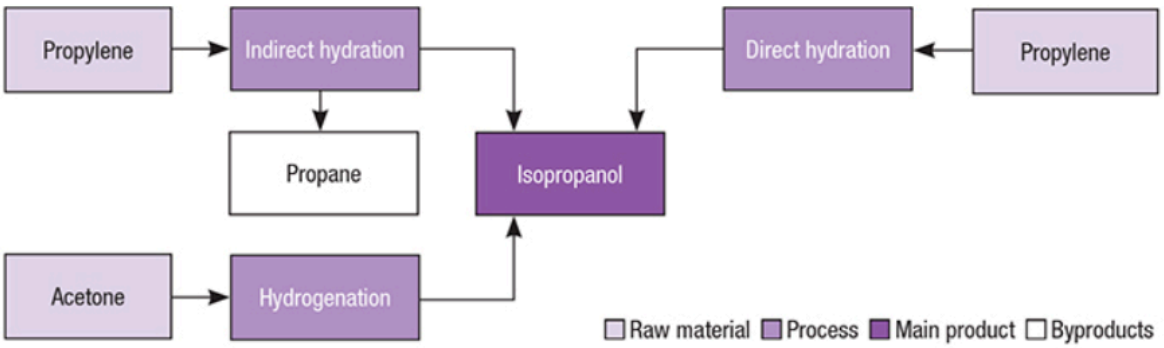


Figure 2. Isopropanol production can occur via different pathways, as shown here

Therefore, catalytic hydrogenation of acetone over solid catalysts serves as a better option for synthesis of IPA.

f. Separation and Purification:
   i. A method for recovering acetone from a mixture consisting of acetone, isopropanol and water which comprises distilling said mixture consisting of acetone, isopropanol and water in the presence of an extractive distillation agent such as dimethylsulfoxide.

   ii. Recovering the acetone as an overhead product and obtaining the isopropanol, water and the extractive distillation agent as bottoms product .

References:

a. Kinetics of acetone hydrogenation for synthesis of isopropyl alcohol over Cu-Al mixed oxide catalysts - ScienceDirect
b. US5081321A - Preparation of isopropanol - Google Patents
c. Technology Profile: Isopropyl Alcohol Production from Acetone - Chemical Engineering | Page 1 (chemengonline.com)

List the contributions of each author:

- Author 1,2 worked on selection of the chemical.
- Author 1,3,4 worked on the selection of the manufacturing process.
- Author 1,2,3 had carried out the literature search and found the reaction steps, and product yield and found necessary separation steps to achieve desired product purity.

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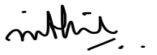
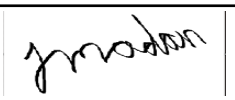
Nikhil Gupta	220708	
Akshat Srivastava	220104	
Shrimi Aggarwal	221036	
Jatin Madan	220475	
Manas Sontakke	220609	

Patent - 2 Ethoxylated β-sitosterol

Applicant: ProChem Innovations

Inventors: 1. Jatin Madan

2. Akshat Srivastava

3. Shrimi Aggarwal

4. Manas Sontakke

Chemical Formula: $C_{29}H_{49}$ - $[OCH_2CH_2]_nOH$

Chemical Name: Ethoxylated β-sitosterol

Chemical synthesis routes:

a. Raw Materials:
   i. β-Sitosterol (98% purity or higher)
   ii. Ethylene oxide (99% purity or higher)
   iii. Potassium hydroxide (KOH) pellets or solution (catalyst)
   iv. Ethanol (solvent for KOH catalyst solution)
   v. Hydrochloric acid (for neutralization)
   vi. Organic solvents (e.g., hexane, ethyl acetate) for extraction and purification

b. Chemical Reactions:
   Ethoxylation Reaction:
   β-Sitosterol + n(CH_2CH_2O) → $C_{29}H_{49}$ - $[OCH_2CH_2]_nOH$
   (Ethylene oxide) (Ethoxylated β-sitosterol)

c. Experimental Analysis:
   Reaction Steps:
   Weigh and add β-sitosterol to a round-bottom flask with a magnetic stir bar and condenser.
   Prepare a 0.3% w/w KOH solution in ethanol and add it to the flask.
   Purge the flask with nitrogen to create an oxygen-free environment.
   Slowly add ethylene oxide at a molar ratio of 5:1 relative to β-sitosterol.
   Maintain the temperature at 160°C and continue stirring for 4-6 hours.
   Typical yield: 88-92%
   Final purity: 95-97%

Reaction Conditions:

Temperature: 160°C

Pressure: Atmospheric pressure (or slightly elevated, e.g., 2-3 bar)

Molar ratio of ethylene oxide to β-sitosterol: 5:1

Reaction time: 4-6 hours

Inert atmosphere: Nitrogen or other inert gas

Safety Considerations:

Ethylene oxide is highly reactive, flammable, and toxic. Handle with caution in a well-ventilated fume hood.

Use appropriate personal protective equipment (PPE) and follow proper safety protocols.

Dispose of waste materials according to established procedures and regulations.

d. Selection of Catalyst:

Catalyst: Potassium hydroxide (KOH), 0.3% w/w relative to β-sitosterol. Using KOH has some advantages like being an inexpensive and readily available catalyst. However, it requires neutralization and byproduct removal steps. Other catalysts like acids (H2SO4) or heterogeneous solid bases can also be used.

e. Other Methods of production:

i. Transesterification Route: β-Sitosterol is reacted with polyethylene glycol (PEG) in an organic solvent like tert-butanol or acetone using a lipase enzyme catalyst at 60-80°C for 24-48 hours to produce ethoxylated β-sitosterol with a typical yield of 70-80%.

ii. Williamson Ether Synthesis: β-Sitosterol is reacted with ethylene carbonate in an organic solvent like tetrahydrofuran or dimethylformamide using a base catalyst like sodium hydride (NaH) or potassium hydroxide (KOH) at 60-100°C for 6-12 hours to produce ethoxylated β-sitosterol with a typical yield of 60-75%.

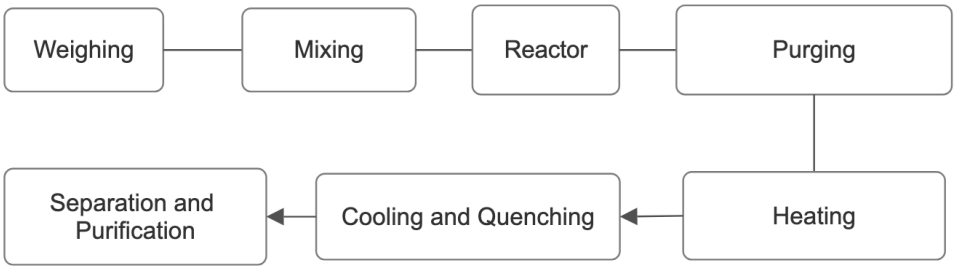
f. Separation and Purification:

Neutralization: Cool the reaction mixture and neutralize the KOH catalyst by adding hydrochloric acid until slightly acidic.

Extraction: Extract the crude product using an organic solvent (e.g., hexane or ethyl acetate) in a separatory funnel.

Concentration: Concentrate the organic layer using a rotary evaporator to obtain the crude ethoxylated β-sitosterol.

Purification: Purify the crude product by recrystallization or column chromatography to remove impurities



References:

a. https://link.springer.com/article/10.1007/BF01145563
b. https://patents.google.com/patent/US4153622A/en

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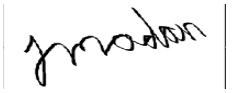
Jatin Madan	220475	
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Shrimi Aggarwal	221036	
Manas Sontakke	220609	

Patent - 3 Cinnamic Acid

Applicant: ProChem Innovations

Inventors: 1. Shrimi Aggarwal

2. Akshat Srivastava

3. Jatin Madan

4. Manas Sontakke

Chemical Formula: $C_9H_8O_2$

Chemical Name: (E)-3-phenylprop-2-enoic acid

Chemical synthesis routes:

Raw Materials:

Benzaldehyde (C_6H_5CHO)
Acetic anhydride ($(CH_3CO)_2O$)
Base catalyst (e.g., sodium acetate or pyridine)
Solvent (e.g., toluene or xylene)

Synthesis Process:

Preparation of Reaction Mixture:

- Mix benzaldehyde, acetic anhydride, and a base catalyst in a suitable reaction vessel. The molar ratio of benzaldehyde to acetic anhydride is typically 1:1, and the base catalyst is used in catalytic amounts.

Perkin Reaction:

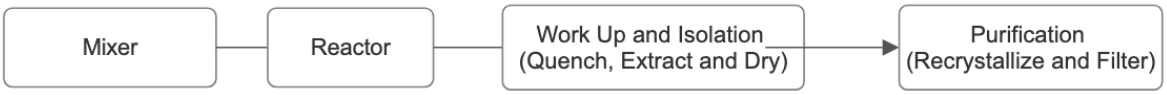
- The Perkin reaction involves the condensation of benzaldehyde with acetic anhydride in the presence of a base catalyst. The reaction proceeds as follows:

$$C_6H_5CHO + (CH_3CO)_2O + CH_3COONa \longrightarrow C_9H_8O_2 + CH_3COOH$$

- The benzaldehyde reacts with acetic anhydride in the presence of the base catalyst to form an acyloin intermediate.
- The acyloin intermediate undergoes intramolecular dehydration to form the α,β-unsaturated carboxylic acid, which is cinnamic acid.

Work-Up and Isolation:

- After the reaction, the mixture is typically cooled and quenched with water to hydrolyze excess acetic anhydride and decompose the base catalyst.
- The crude reaction mixture is then extracted with a suitable solvent such as ethyl acetate or dichloromethane to separate cinnamic acid from the reaction by-products.
- The organic layer containing cinnamic acid is separated, dried over anhydrous sodium sulphate, and concentrated under reduced pressure to obtain crude cinnamic acid.



Purification:

- The crude cinnamic acid can be purified through recrystallization from a suitable solvent such as ethanol or ethyl acetate.
- The recrystallized product is filtered, washed with cold solvent, and dried to obtain pure cinnamic acid.

References:

- https://www.ncbi.nlm.nih.gov/pmc/articles/PMC6147640/#:~:text=Cinnamic%20acids%20have%20been%20prepared,(180%2D190%C2%B0C)
- https://pubchem.ncbi.nlm.nih.gov/compound/Cinnamic-Acid

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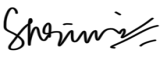
Shrimi Aggarwal	221036	
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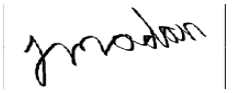
Jatin Madan	220475	
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