Imam Muhammad Bin Saud Islamic University King Abdulaziz City for Science & Technology

College of science

Department of Chemistry

Production of Methyl Isobutyl Ketone from Acetone – Self Condensation

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List of Abbreviation

1- GC: Gas Chromatography.

2- MIBK: Methyl Isobutyl Ketone.

3- MO: Mesityl oxide.

4- DIBK: Dissobutyl ketone.

5- IPA: Isopropyl alcohol

6- DA: Diacetone alcohol

7- PH: Phorone

8- IPH: Isophorone

Objectives

To produce MIBK in the gas-phase are multifunctional nitrogenous catalyst.

Introduction:

Methyl Isobutyl Ketone is a stable, colorless liquid that is both a medium-boiling and medium-evaporating solvent. Methyl-isobutyl-ketone is a chemical products of industrial Interest from acetone self-condensation under hydrogen atmosphere.

It is used as a solvent for polishes and dyes or for obtaining other chemical products (stabilizers, etc.).

Due to it is volatility, which is lower than of acetone, MIBK is used a solvent with higher performance.

The conventional technology of MIBK synthesis from acetone consists of a sequence of three steps and it has been the only one used until late 1960s.

Sulphuric acid, and during the third stage MO is hydrogened to MIBK.

Recently, an one-stage process for manufacturing MIBK has been developed, a process which uses bifunctional catalysts containing a metal (Pd usually) deposited on a solid acid support. The process presuppose the reversible condensation of acetone to MO on the acid centers and, in succession, the hydration of MO to MIBK on the metallic centers.

Advantages:

- Slightly soluble in water and miscible with most organic solvents
- High solvency power
- Low viscosity and a viscosity reducer
- Prevents gelling in vinyl resin-based coatings

Used Equipments:

Experimental measurement of the unit:

A- Gas Chromatography (GC).

B- HPLC pump.

C- Flow-meter for hydrogen gas.

D- Furnace.

E- Tubular, micro glass reactor.

F- Condenser.

G- Stainless steel tubes.

Preparation of support:

A) Preparation of Mg(OH)2. . $\frac{1}{2}$ H_2O in Ethanol:

1- Weighted 7.6923g of magnesium nitrate hydrate was dissolved in 60ml of ethanol at room temperature in 100ml beaker, under continuous magnetic stirring.
2- In another 100ml beaker, an amount of (5.95ml) of cyclothexylamine was dissolved in 20ml of ethanol at room temperature.
3- Cyclohexylamine solution was poured into magnesium solution (white precipitate appeared), upon magnetic stirring.
4- 100ml Ethanol was added to the reaction mixture, left stirring seven days.
5- The precipitate was filtered off through M-size fritted filter, and then was washed with 100ml Ethanol.
6- The precipitate was dried under vacuum for one day.
7- (After drying) the precipitate was mixed with 200ml Ethanol and was magnetically stirred for one day for the removal of any impurity.
8- The precipitate was filtered off and was dried.

B) Preparation of MgO (solid-base support):

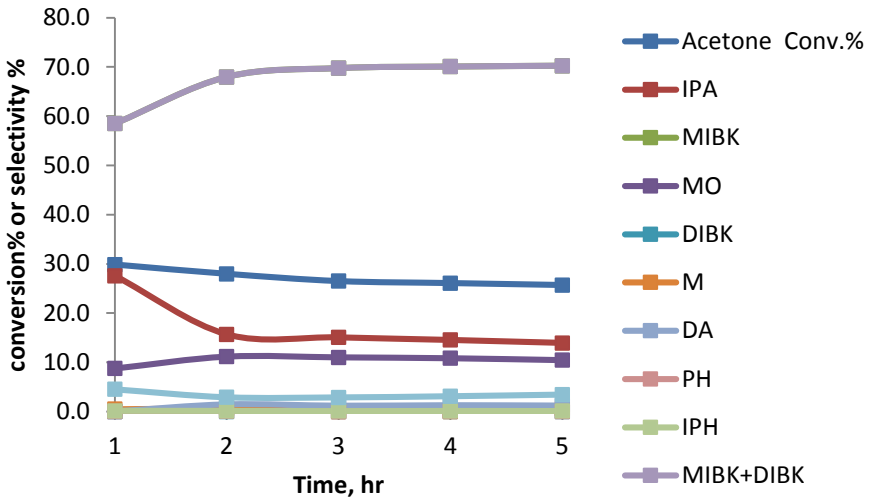
Calcine the obtained $Mg(OH)_2$. $\frac{1}{2}$ (H_2O) at 500 C for three hours.

Preparing the Reactor

1- Removal of oxygen from acetone by bubbling Argon gas.
2- After preparation of the catalyst, take 0.25g and mixed with porcelain with the same amount, and put it in the middle of the reactor.
3- Activate the catalyst, {50-60ml/min of H_2} at 250C over night.

With the same steps but change amount of H_2 to 10ml/min and 15ml/min.

The reaction was performed at 150°C where either hydrogen or acetone flow was changed to study the effect of H2; acetone mole reaction on the conversion of acetone and selectivity towards MIBK. Hydrogen flow was fixed at 5.0ml/min while acetone flow was 0.01, 0.015, 0.02, or 0.025ml/min. Similar study was carried out when using fixed flow rate of 10ml/min of hydrogen and varied flow rates of acetone. Five samples were analyzed by GC, at every reaction condition to investigate the stability of the catalyst.



At 150 C, 0.010 ml/min of acetone, and 5 ml/min of H_2:

The products :

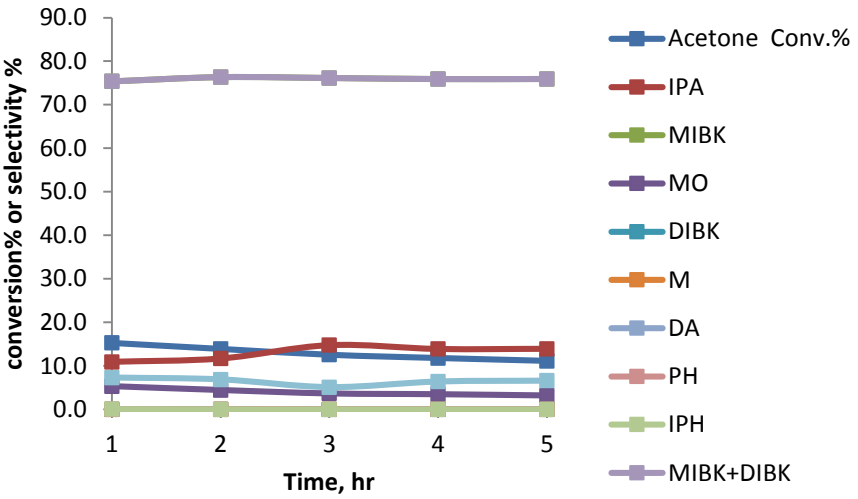
The conversion of acetone is 30%

Isopropyl alcohol (IPA) is 28%

Methyl isobutyl ketone (MIBK) is 59%

Mesityl Oxide (MO) is 9%

Other compound between 0-5 %



At 150 C, 0.015 ml/min of acetone, and 5 ml/min of H_2:

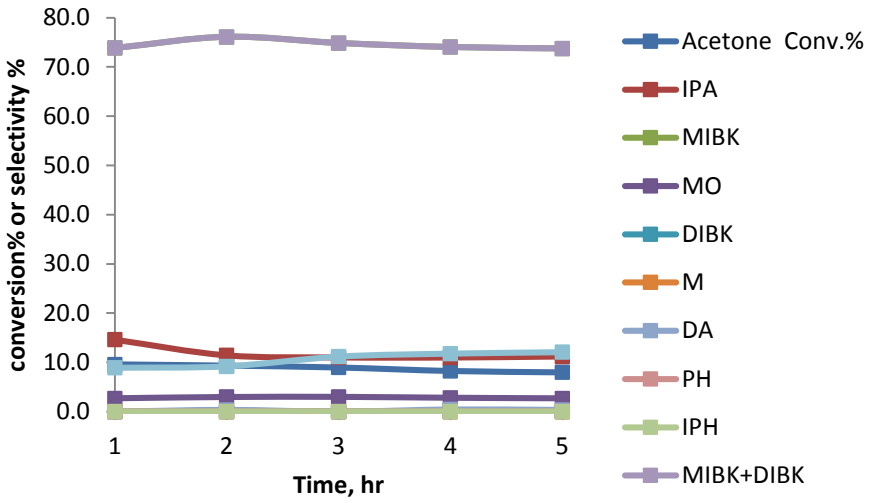
The products :

The conversion of acetone is 15 %

Isopropyl alcohol (IPA) is 11 %

Methyl isobutyl ketone (MIBK) is 75 %

Other compounds between 0-7 %



At 150 C, 0.020 ml/min of acetone, and 5 ml/min of H_2:

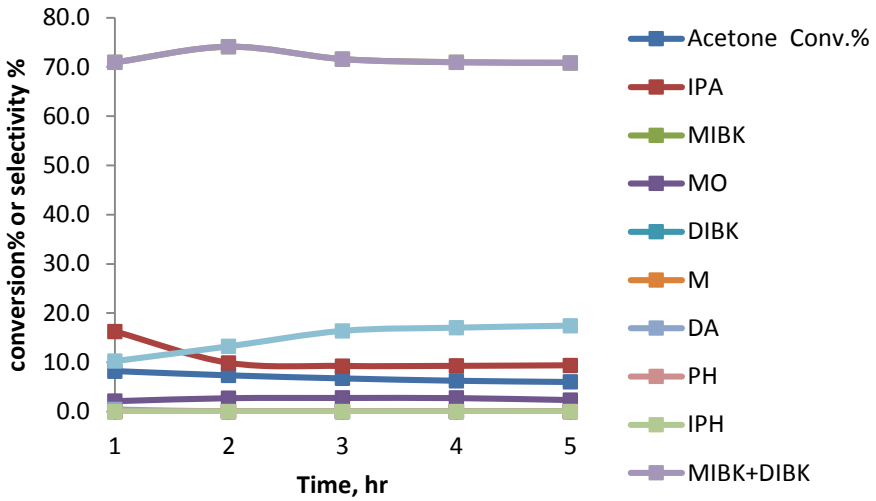
The products :

The conversion of acetone is 10 %

Isopropyl alcohol (IPA) is 15 %

Methyl isobutyl ketone (MIBK) is 74 %

Other compounds between 0-10 %



At 150 C, 0.025 ml/min of acetone, and 5 ml/min of H_2:

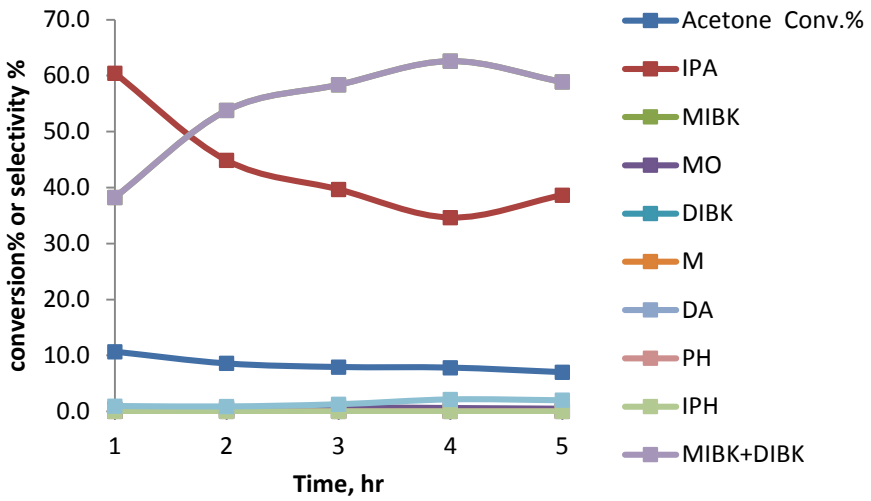
The products :

The conversion of acetone is 9 %

Isopropyl alcohol (IPA) is 18 %

Methyl isobutyl ketone (MIBK) is 71 %

Other compounds between 0-10 %



At 150 C, 0.010 ml/min of acetone, and 10 ml/min of H_2:

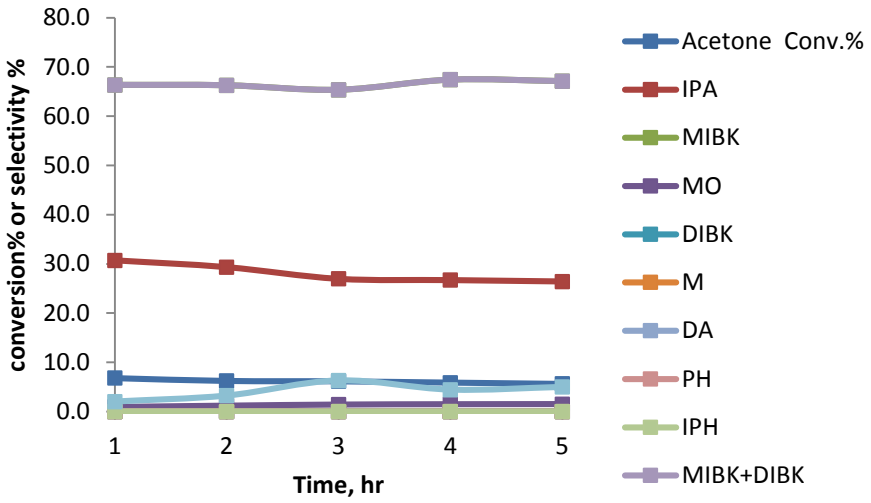
The products :

The conversion of acetone is 11 %

Isopropyl alcohol (IPA) is 61 %

Methyl isobutyl ketone (MIBK) is 39 %

Other compounds are 0 %



At 150 C, 0.015 ml/min of acetone, and 10 ml/min of H_2:

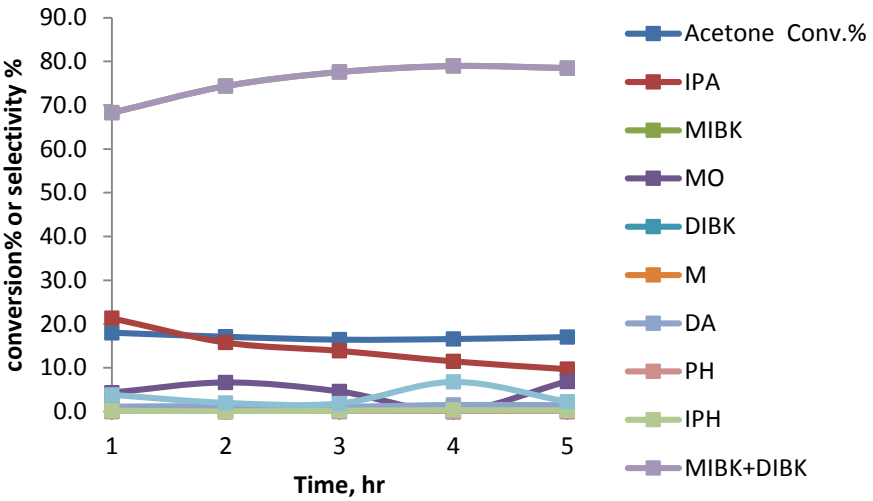
The products :

The conversion of acetone is 8 %

Isopropyl alcohol (IPA) is 31 %

Methyl isobutyl ketone (MIBK) is 67 %

Other compounds between 0-2 %



At 150 C, 0.020 ml/min of acetone, and 10 ml/min of H_2:

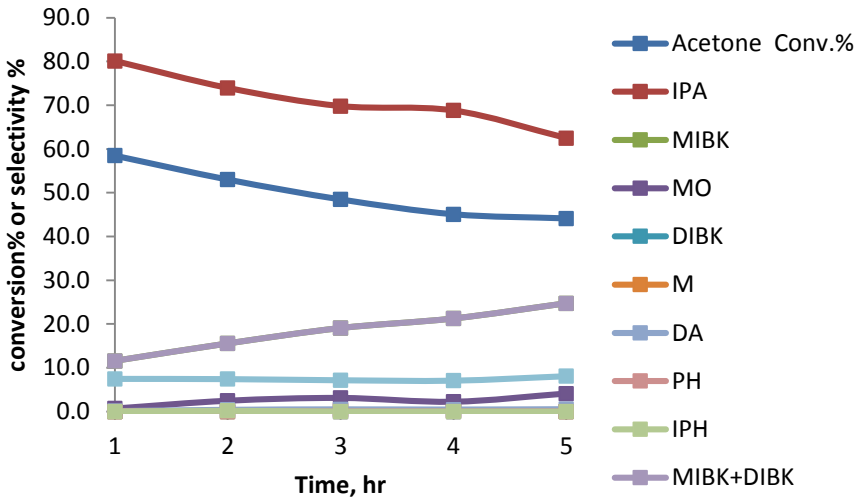
The products :

The conversion of acetone is 19 %

Isopropyl alcohol (IPA) is 21 %

Methyl isobutyl ketone (MIBK) is 69 %

Other compounds between 0-5 %



At 150 C, 0.025 ml/min of acetone, and 10 ml/min of H_2:

The products :

The conversion of acetone is 59 %

Isopropyl alcohol (IPA) is 80 %

Methyl isobutyl ketone (MIBK) is 11 %

Other compounds between 0-8 %

Conclusion:

The best results for the production of Methyl isobutyl ketone is 75 %

it was at 150 C, 0.015 ml/min acetone, and 5 ml/min H_2

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