



Selective synthesis of cumene by isopropylation of benzene using catalytic membrane reactor

V.V. Bokade^{a,*}, U.K. Kharul^b

^a Catalysis Division, National Chemical Laboratory, Pune 411008, India

^b Chemical Engineering and Process Development Division, National Chemical Laboratory, Pune 411008, India

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ABSTRACT

Selective isopropylation of benzene to cumene is demonstrated using catalytic membrane reactor (CMR), to our knowledge for the first time and the performance was evaluated against conventional plug flow reactor (PFR). Almost complete elimination of byproducts like polyalkylated benzene and higher aromatics could be made possible by using CMR. These are present in significant amount when reaction is carried out with PFR. The cumene selectivity increased up to a maximum of 97.25% by CMR as against 90.05% with conventional PFR. The effect of reaction parameters that can greatly influence the process economics, such as liquid hourly space velocity (LHSV), reactant mole ratio and catalyst/reactor volume ratio were investigated. Changes in these parameters made for CMR are industrially advantageous. The process by CMR is more economical and ecofriendly than PFR as far as milder process parameters (higher LHSV, lower reactant feed mole ratio, less catalyst require, etc.) and process intensification (reaction and separation in single reactor) are concerned. The study provides new insights for benzene isopropylation reaction.

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1. Introduction

Cumene is generally prepared by alkylation of benzene with isopropylene or isopropanol by Friedel–Craft alkylation route [1]. It is widely used as a precursor for the manufacture of phenol, acetone, α -methyl styrene, etc. Various processes are demonstrated in the literature for the preparation of cumene. Friedel–Craft alkylation reaction is catalyzed by either protonic acids (e.g., H_3PO_4) or Lewis acids (e.g., BF_3) on various supports like amorphous or crystalline aluminosilicates. The reaction proceeds through dehydration of an alcohol to alkene then react via a carbonium ion mechanism with gas-phase benzene to produce cumene. Isopropylation of benzene can be a liquid-phase catalytic reaction [2].

Two processes most widely used on industrial scale are UOP's Cumox process [3] and Monsanto–Lummus Cumene process [4].

The Cumox process uses mixture of propylene and excess benzene reacted in the presence of solid phosphoric acid as a catalyst. The process offers 99.3% (by weight) conversion of propylene with 92.5% selectivity to cumene. In Monsanto–Lummus process, dry benzene and propylene are mixed in the alkylation reactor with $AlCl_3$ –HCl catalyst. The main feature of this process is low benzene recycle ratio. A commercial process using zeolite catalyst is also demonstrated [5–8] that uses isopropanol, benzene and a heterogeneous catalyst–zeolite β .

Processes documented in the literature have several drawbacks like high catalyst volume, high reaction temperature, high feed mole ratio (high benzene in the feed than required), lower space velocity, lower yields, byproduct formation leading to higher capital and operational costs corrosion problems, etc.

In the present work, we have demonstrated a novel process for the preparation of cumene that uses the concept of catalytic membrane reactor (CMR). The main objective behind using CMR was to reduce the drawbacks in current processes. The reaction between benzene and isopropanol was investigated and the performance of CMR was evaluated vis-à-vis conventional plug flow reactor (PFR) using zeolite β as the catalyst. The most significant features of other processes that are being practiced on industrial scale (Monsanto and Cumox) are also compared with CMR.

Abbreviations: PFR, plug flow reactor; CMR, catalytic membrane reactor; IPA, isopropyl benzene; DIPB, diisopropyl benzene; HBF, high boiling fractions; LHSV, liquid hourly space velocity h^{-1} ; GC, gas chromatography; FID, flame ionization detector; L, length of catalyst bed; D, diameter of reactor.

* Corresponding author. Tel.: +91 20 25902458; fax: +91 20 25902634.

E-mail address: vv.bokade@ncl.res.in (V.V. Bokade).

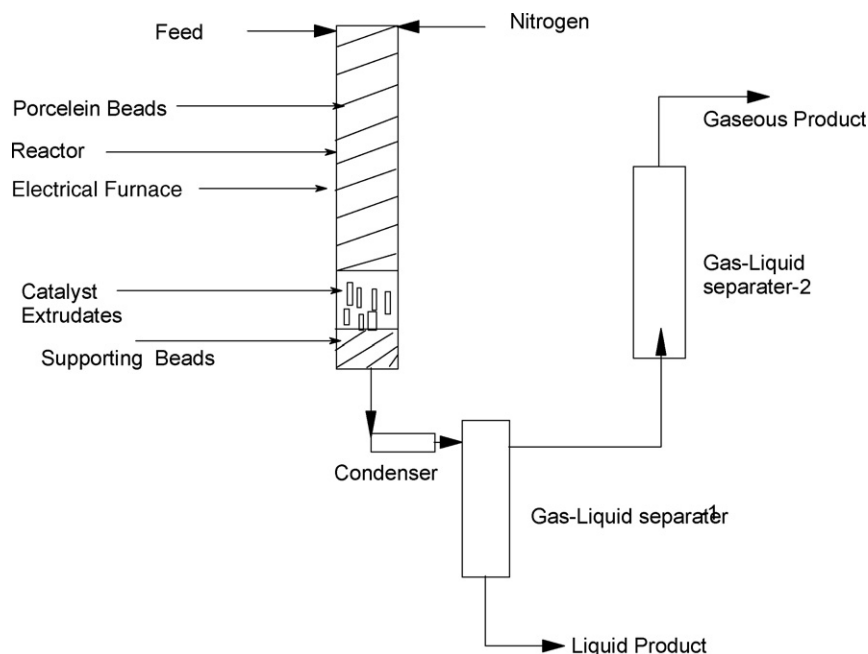


Fig. 1. Experimental conventional plug flow reactor (PFR) set up.

2. Experimental

2.1. Chemicals

Isopropanol, benzene, cumene and diisopropyl benzene (DIPB) (all >99% pure) were procured from s.d. Fine Chemicals Ltd., Mumbai and used without further purification.

2.2. Catalyst

Silica to alumina molar ratio of zeolite β is generally between 10 and 200. In this work, the large pore-12 embarked ring zeolite β having BET surface area of $250 \text{ m}^2/\text{g}$ and Si/Al ratio in the range of 26–30 was used.

2.3. Process for cumene preparation by PFR and CMR

The experiments using PFR (Fig. 1) as well as CMR (Fig. 2) were carried out at similar temperature (210°C) and time (5 h) in order to be able to better compare the performance of both methods. A SS316 fixed bed cocurrent down flow reactor is used in the present investigation. This reactor also has a special arrangement to equip the membrane, which is made of proprietary material. The zeolite β as a catalyst in powder form was coated on the membrane having $70\text{-}\mu\text{m}$ thickness and same catalyst layered membrane was rested on SS316 circular plate having 0.5-mm holes across the cross-sectional area. The same SS316 plate having membrane with coated catalyst powder was sealed with flanges at the bottom of the reactor. The small size porcelain beads were kept at the top portion of the reactor, which is the preheating zone

of the reactor. The complete assembly is enveloped in electrical heating shell. Each shell is connected by the K-type thermocouple to measure catalytic membrane bed temperature. The outlet stream of the reactor was connected to the isothermal gas-liquid separator in order to separate the product cumene from benzene (Figs. 1 and 2).

In the beginning, catalyst was activated at 220°C in presence of air for 5–6 h and then under running nitrogen for half an hour. The desired quantity of feed (isopropyl alcohol and benzene) was pumped to the reactor by using ISCO syringe pump. Care was taken to preheat reactants at 210°C and get completely mixed prior to reaction on the catalyst surface. Samples were withdrawn from the reactor at defined time intervals and were analyzed using gas chromatography (Shimadzu model, FID detector with hydrogen as a carrier gas, using xylene master column). The sets of conditions used for carrying out cumene synthesis by PFR and CMR routes are given in Table 1.

3. Results and discussion

Initially, the reaction using PFR was carried out as per the reported procedure [5–8]. The reaction conditions were chosen based on maximum possible IPA conversion and cumene selectivity. The results obtained in the present work by PFR route were in agreement with the reported ones and are summarized in Table 2.

In PFR, the catalyst is always in some shape (extrudate, tablet) and size with some binder, which creates the diffusional resistance and increase catalyst loading. In present work, catalyst used is in extrudate form of $2 \text{ mm} \times 3 \text{ mm}$ irregular size with alumina as a binder having catalyst:binder ratio of 80:20.

Table 1
Reaction conditions for PFR and CMR

Parameter	Conventional PFR	Vertical CMR (I)	Vertical CMR (II)	Vertical CMR (III)
Mole ratio (IPA:benzene)	1:6.5	1:2	1:3	1:3
Catalyst/ 100 cm^3 (reactor volume)	4.4	3.0	2.6	2.2
LHSV (h^{-1})	2.5	3.6	3.6	2.4
Reaction temperature ($^\circ\text{C}$)	210	210	210	210

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