I J R B A T, Issue (IX), Vol. II, May 2021: 23-27
A Double-Blind Peer Reviewed & Refereed Journal
OPEN ACCESS
e-ISSN 2347 – 517X
Original Article
INTERNATIONAL JOURNAL OF RESEARCHES IN BIOSCIENCES, AGRICULTURE AND TECHNOLOGY

VAPOUR PHASE DEHYDROGENATION OF CYCLOHEXANOL USING COPPER BASED CATALYST- OPTIMISING TEMPRATURE ANF BED HEIGHTS

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Communicated: 16.02.21 | Revision :12.03.21 & 24.04.2021 Accepted: 05.05.2021 | Published: 30.05.2021

ABSTRACT:
This work examines the dehydrogenation of cyclohexanol in vapour phase over copper based catalyst. The effect of support of promoter's e.g. rare earth oxides of metals like Thorium, Cerium, Aluminum and Uranium and other heavy metals were studied. The impregnation method was preferred for the preparation of these catalyst on pumice stone. The effect of reaction temperature, bed heights, feed flow rate were considered to maximize conversion and minimize side reactions. The reaction was best carried out at 225°c to 300°c.

Keywords: Vapour phase, dehydrogenation, heavy earth metals, impregnation, flow rate and bed heights.

INTRODUCTION:

Cyclohexanone is a colourlesss mobile liquid with an odour suggestive of peppermint and acetone; chiefly used as an intermediate also as a solvent for resins, laquers and dyes. Bolveault, first prepared cyclohexanone by the dehydrogenation method. Further, dehydrogenation of cyclohexanone to phenol has also been reported. To prevent further side reaction, catalyst composition, temperature was optimized and side products were practically prevented and eliminated.

Fixed bed of catalyst were proposed and preferred over fluidized bed to insure regular flow pattern with favorable velocities. Various types of catalysts were prepared by impregnation method on pumice stones, silica gel, having different mesh pore sizes. The activity and selectivity of all the catalysts were studied and the catalyst finally selected copper-ceria oxide, catalyst having – 40 + 60 mesh pore size on pumice stone, with a bed height of L/D = 2 obtaining maximum conversion of cyclohexanol to cyclohexanone as the product; with temperature ranging from 225°c - 300°c.

MATERIAL & METHODS:

1. Procedure :

A flow diagram of the apparatus is shown in fig. 1, The reactor consisted of a 30 mm inside diameter. Pyrex glass tube heated to the reaction temperature by means of kanthal wire, well insulated from the outside to prevent heat loss. The pre heater was connect to the bottom of the reactor through the standard glass joint. In between the reactor and preheater there is a distributor pack with porcelain pieces of – 40 + 60 mesh pore sizes. The catalyst supported by a stainless steel wire gauge which was fixed to the top of the standard glass joint. On the top of the reactor, there is separator which is connected to the series of condensers as shown in fig 1. through the condensers, ice cold water was circulated. The receivers in which the product

(cyclohexanone) was collected, was kept in ice - salt mixture. Untrapped hydrogen gas was absorbed in the bubblers containing 0.5 N hydroxylamine hydrochloride solution.

The feed to the reactor is 95% cyclohexenol, the feed to the reactor was through the calibrated orifice meter of the preheater, of required feed rate. A stream of Nitrogen gas was also probed through the reactors when heated. The product was collected in the sample flask. This sample were a analyzed for the cyclohexanone. The flow rate of the alcohol was checked by collecting a sample of alcohol for known time and weighting it. Each catalyst was studied separately at a temperature 225°c - 300°c, with an interval of 25^0 c; so that small variation in temperature should not interfere the conversion between the two temperature; and can be easily differentiated. Once the study of catalyst is complete, the glass apparatus was compactly cleaned and dried. The glass bead packing material inside ,the reactor, was also washed with acetone and dried.

Preparation of catalyst.

Preparation of copper – ceria – oxide deposited on pumice stone. -40+60 mesh pore size.

This was prepared by dissolving 270 gm of copper nitrate, and 0.9 gm of cerium nitrate were heated to 600 ◦c. Then 100gm of pumice stone of desired size
(- 40 + 60, -) were added to the solution and stirred from time to time. the mixture was kept for 24 hrs. So that the solution enters the pores of the pumice Stones. Then it was dried at 110◦ c in an air oven after drying, impregnated pumice stone was heated at 400◦c for six hours and cooled. The catalyst was their reduced by passing a slow stream of hydrogen gas at 275 ◦ c, till constant weight is obtained. This gives the composition of the catalyst as 94% pumice stone carrier, with 6% impregnated material.

3 Analytical procedure:

Estimation of cyclohexanone

In a 1000 ml volumetric flask, 18 g m of hydroxyl amine hydrochloride was dissolved in 500 ml of water, to which 10 ml of the bromo-phenol blue indicator solution was added and then diluted to volume with 95% ethanol. (V/V) The solution was then heated on a water bath for 30 minutes; cooled and adjusted to blue – green color by adding either 1N HCL or 1N Na OH and stoppered. later on 100 ml of this reagent was transferred into each of the two 250ml conical flasks. Weight of the sample was transferred to one of the flask, by means of a weighing pipette. both the flasks were allowed to stand at room temperature for 15 minutes. One blank titration was run, to use as a color standard with each set of determinations.

The flask with the sample was them titrated against 1N Sodium hydroxide until, the color matched with the color of the blank, after the later has been diluted with volume of water equal to the volume of 1N Sodium hydroxide, used in is direct measure titration.

The number of moles of Sodium hydroxide used in direct measure of cyclohexanone in the sample as shown by the equation.

$$\text{Cyclohexanone \%} = \frac{9.814 \times V \times N}{W}$$

V = Vol. of the NaOH solution used for titration in ml.
N = Normality of NaOH solution.
W = Weight of the Sample in gm.

Sample Collection :
Wt. of the Sample 0.9901 gm.

Vol. of 1N NaOH consumed = 3.3 ml.

Cyclohexanone % = 9.814 x 1 x 3.3 / 0.9901

= 32.71 %

Wt. of condensed cyclohexanone = 7.02 gm

Molecular wt. of cyclohexanone = 98.16 gm

Gram mole of Cyclohexanone = 0.3271 x 7.02 / 98.16 = 0.02339 g m /mole

Gram mole of cyclohexanone feed = 7.02 / 100.16

= 0.70089

Conversion % = 0.02339 / 0.070089 x 100

= 33.41 %

RESULT & DISCUSSION:

1. The effect of time factor plays a significant role in producing maximum conversion of cyclohexanol to cyclohexanone.

Time Factor = W/F, where W = wt of the selected catalyst, and F is feed flow rate. As shown in the above graph, at time factor increases, conversion also increases.

2. In the present investigation, it also shows that selectivity of the catalyst were higher with temperature. Temperature increases, conversion also increases.
3. The bed height employed for the experiment were L/D = 1,2,3, the effect of bed height on conversion was studied, indicating that conversion was maximum for L / D = 2.

CONCLUSION:

The aquatic macrophytes vary in different habitats based on nutritional components present in water bodies across the globe. The Konsari Lake in Chamorshi tehsil of Gadchiroli district is a serene water body harbouring different macrophytes and therich biodiversity of weeds support a large number of fauna too in all the seasons.

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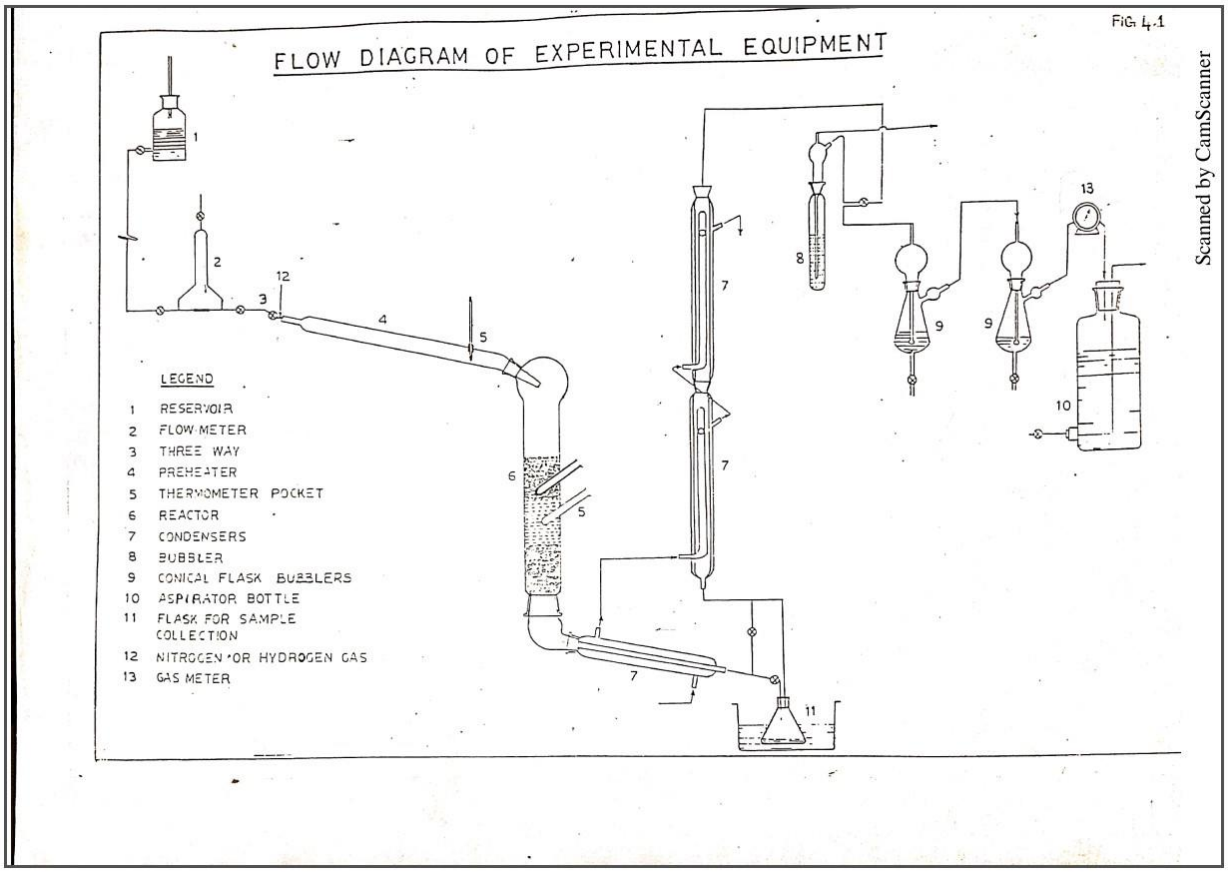
FIG. 4.1
FLOW DIAGRAM OF EXPERIMENTAL EQUIPMENT
LEGEND
1 RESERVOIR
2 FLOW METER
3 THREE WAY
4 PREHEATER
5 THERMOMETER POCKET
6 REACTOR
7 CONDENSERS
8 BUBBLER
9 CONICAL FLASK BUBBLERS
10 ASPIRATOR BOTTLE
11 FLASK FOR SAMPLE COLLECTION
12 NITROGEN OR HYDROGEN GAS
13 GAS METER
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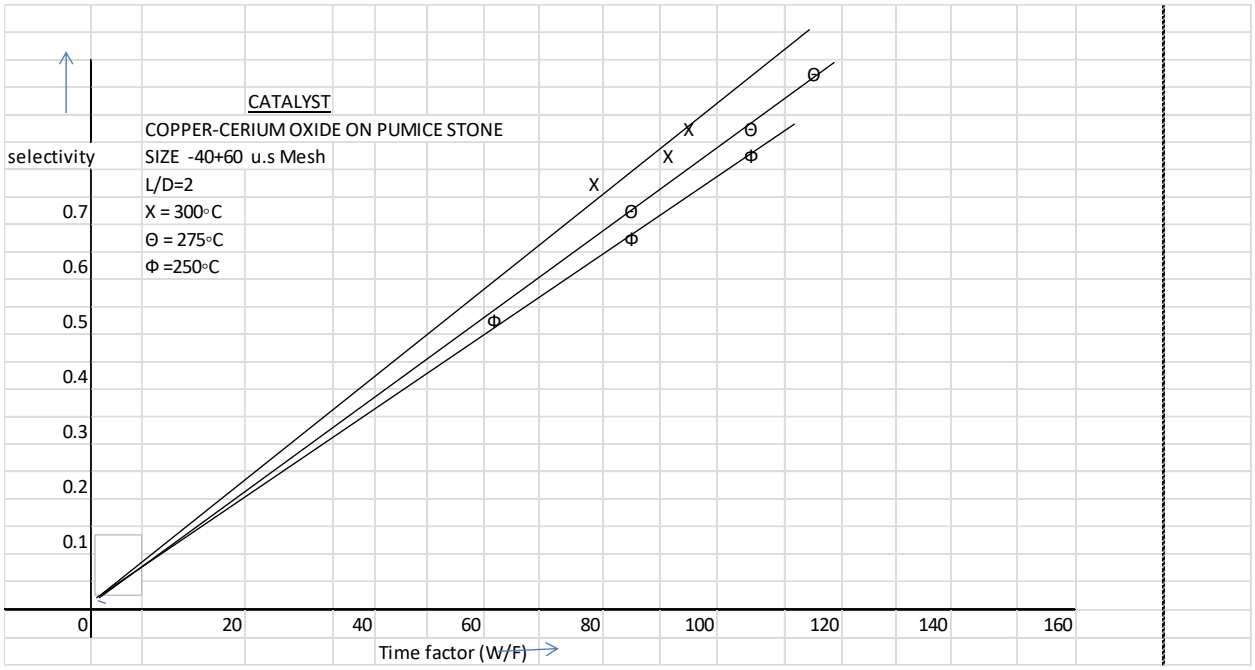
CATALYST
COPPER-CERIUM OXIDE ON PUMICE STONE
SIZE -40+60 u.s Mesh
L/D=2
X = 300°C
Θ = 275°C
Φ =250°C
selectivity
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
20
40
60
80
100
120
140
160
Time factor (W/F)

Temperature	**Feed Rate**	**W / F**	**Conversion**		**Gm / mole of feed**		**Hydrogen**	**Overall conversion (cyclohexanone)**
			alc	Ket	Alc	Ket		
300	38.46	153.0	29.99	70.01	0.2999	0.7134	0.7040	0.7040
	49.59	118.8	38.50	61.50	0.3844	0.6266	0.6198	0.6198
	69.69	84.4	48.90	51.10	0.4882	0.5207	0.5160	0.5161
275	40.21	146.4	37.42	62.58	0.3736	0.6377	0.6306	0.6306
	46.15	127.5	42.50	57.50	0.4243	0.5859	0.5799	0.5800
	68.10	86.4	52.86	47.14	0.5278	0.4803	0.4764	0.4764
250	42.86	137.3	46.97	53.03	0.4690	0.5404	0.5354	0.5354
	52.54	112.0	53.89	46.11	0.5381	0.4698	0.4661	0.4661
	69.77	84.4	61.00	39.00	0.6091	0.3974	0.3948	0.3948

(Temp. 250^{0}c – 300^{0}c)

- Table ∝ graph : Cat : Copper - cerium oxide on pumice stone size – 40 + 60 mesh pore size. W= 60 gm Bed height L/D =2