ETHANOL DEHYDRATION OVER SHEVAROY BAUXITE

By G. S. Pant, N. Subrahmanyam, M Chanda and S. S. Ghosh

(Department of Chemical Engineering, Indian Institute of Science, Bangalore-12, India)

ABSTRACT

Vapour phase dehydration of ethanol to ethylene over Shevaroy bauxite has been studied in a fluidised-bed reactor. Factorial design of experiments has been carried out. A mathematical expression representing the dependence of conversion as a function of temperature and time factor has been proposed.

1. INTRODUCTION

Vapour phase dehydration of ethanol to ethylene using bauxite/alumina as catalysts has been a well studied reaction. However, most of the work reported in literature is for the reaction in fixed-bed reactors. Therefore, in the present study of alcohol dehydration over bauxite from Shevaroy (Tamilnadu State) a fluidised bed reactor was employed. Factorical design of experiments was carried out to determine the nature of dependence of conversion as a function of temperature and time factor.

2. CATALYST

Bauxite obtained from Shevaroy was first washed to remove clay material present and dried to remove free water. It was then activated to remove combined water and to increase its adsorptive power.

A few important characteristics of this bauxite are :

(1) Particle size $-65 + 80$ T.S.S.
(2) Bulk density   1.30 gms/cc.
(3) Surface area   68.34 sq. cm./gm.

The surface area of bauxite was determined by benzene adsorption method in a desiccator.[1]

3. EXPERIMENTAL

The equipment was of laboratory scale, as illustrated schematically in Figure 1. It consisted of feed system, vaporiser, reactor, condenser and
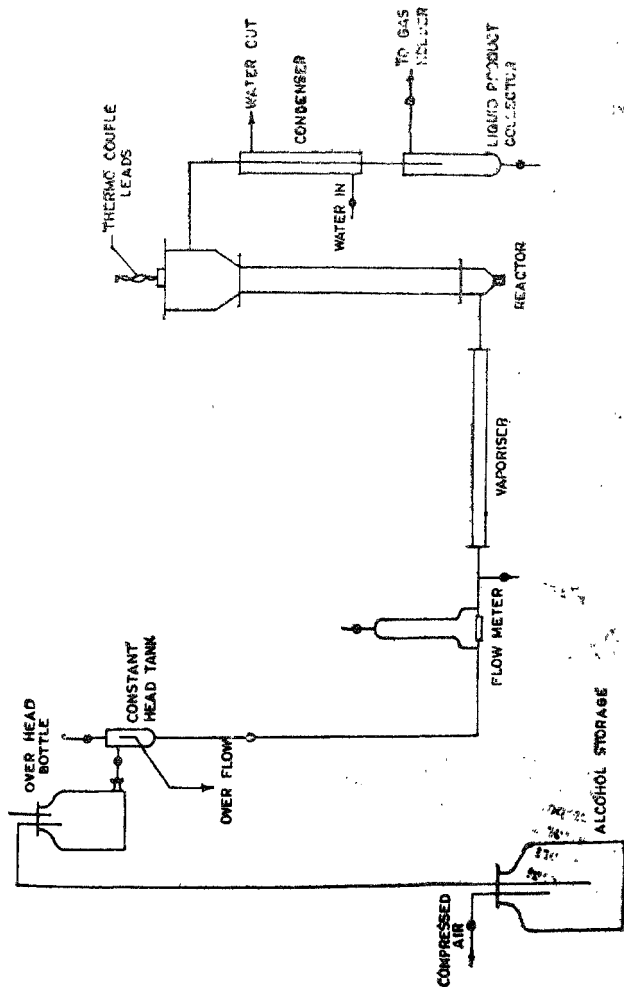
Fig. 1 FLOW DIAGRAM OF SET-UP.

FIG. 1

Flow diagram of Setup

gas collection system. Ethanol from storage carboy was pumped to the overhead by using compressed air, from where it was fed continuously to a vaporiser at constant rate. The vaporiser was 1/4″ × 3/8″ stainless steel tube of 24″ length, packed with porcelain beads and electrically heated. The temperature of the vaporiser was maintained at 125 ± 1°C. The reactor was a stainless steel tube 2″ dia. and 12″ long. A stainless steel wire mesh was used as the catalyst support and feed distributor. The reactor was heated by external resistance coils to the required temperature and controlled to within ± 5°C by a simmerstat. Hot gases leaving the reactor were passed through a double walled surface condenser to condense out the water produced by the dehydration of alcohol, as also any unconverted alcohol. The gas collection system consisted of a low pressure gas holder of 250 litres capacity. Suitable weights were used for the gas collection under positive pressure. A T-stop cock was placed midway between the liquid-product collector and the gas holder for tapping gas samples for analysis.

The vaporiser and the reactor were heated to the desired temperature and then alcohol was allowed to flow at a predetermined rate. The vapours then passed through the reactor containing a known weight of bauxite for a period of one hour. The condensate was collected in a flask and the gaseous product in the gas holder.

The gaseous product was analysed in a modified Orsat gas analysis set-up. The liquid product being mainly water and unreacted alcohol was not analysed even though very small amounts of acetic acid, ethyl acetate and acetaldehyde were present.

A design was used involving two factors namely temperature (A) and time factor (B), each at four levels with equal intervals. The experimental runs were carried out in a random order so as to improve the experimental efficiency. Care was taken to avoid batch to batch variation in the raw materials.

5. Results and Discussion

The responses obtained at various levels of temperature and time factor are presented in Table 1. The method of analysis employed is as suggested by Davies.[2] The first step is to calculate the linear, quadratic and cubic components of the effect of factor A for each level of factor B and that of factor B for each level of factor A. The interaction of components of the factors A and B are then calculated. The analysis of variance of Table 1 is presented in Table 2.

The conclusion that can be drawn within the range of experiment are therefore :

> (i) There is a significant linear increase in conversion with increase in temperature.

(ii) There is a significant linear decrease in conversion with increase in time factor.

(iii) The quadratic and cubic effects of temperature are small, so that a linear function provides an adequate representation.

(iv) The interactions of linear temperature with linear and quadratic time factor are significant which means that slope of the line is not the same for different levels of time factor.

All the components of the effect of temperature and the linear and cubic components of the effect of time-factor are significant which imply that there occurs a minimum or maximum conversion at some intermediate combination of temperature and time factor, or a point outside the range examined. Taking into consideration only those effects and interactions which are significant at 5% level (see Table 2) the conversion of ethanol to ethylene as a function of temperature and time factor could be represented as :

$$x = K_1 y + K_2 z + K_3 y^2 + K_4 z^2 + K_5 y^3 + K_6 yz + K_7 yz^2 \qquad [1]$$

TABLE 1

Responses obtained at various levels of temperature and time factor

Temperature (A) $A_1 = 380°C$

$A_2 = 420°C$

$A_3 = 460°C$

$A_4 = 500°C$

Time factor (B) $B_1 = 28.97$

$B_2 = 36.30$

$B_3 = 47.63$

$B_4 = 56.96$

Response :   Mole% conversion of ethanol to ethylene.

	B_1	B_2	B_3	B_4
A_1	34.78	38.25	28.63	26.54
A_2	46.22	55.03	43.10	39.64
A_3	80.70	92.37	71.70	47.63
A_4	78.30	89.43	75.20	51.28

Table II

Analysis of Variance of Table I

Source of variation	Sum of squares		Degrees of freedom		Mean square	Variance ratio
Main Effects						
A Linear	4597.00		1		4597.00	200.20*
A Quadratic	182.00	5096.10	1	3	182.00	7.93*
A Cubic	317.10		1		317.00	13.81*
B Linear	988.30		1		988.30	43.04*
B Quadratic	491.00	1590.70	1	3	491.00	21.38*
B Cubic	111.40		1		111.40	4.84
Interactions						
Linear A x Linear B	183.80		1		183.80	8.01*
Linear A x Quadratic B	156.30		1		156.30	6.81*
Quadratic A x Linear B	6.02	480.86	1	9	6.02	0.26
Remainder = Error	134.74		6		22.47	-----
Total		7167.66	15			

* Significant at 5% level of the F statistic, because with 1 and 6 degrees of freedom, 5% significance level requires a variance ratio of 5.99.

where x is the mole% conversion of ethanol to ethylene

y is the temperature

z is the time factor, and

$K's$ are constants.

By least square fitting their values are found to be :

$$K_1 = 1.09 \times 10^2$$
$$K_2 = -1.06 \times 10^3$$
$$K_3 = -3.28 \times 10^{-1}$$
$$K_4 = 1.21 \times 10$$
$$K_5 = 2.43 \times 10^{-4}$$
$$K_6 = 1.82$$
$$K_7 = 2.09 \times 10^{-2}$$

Given a temperature and time factor, within the range studied in the present investigation, Eq. [1] can be employed to calculate the conversion.
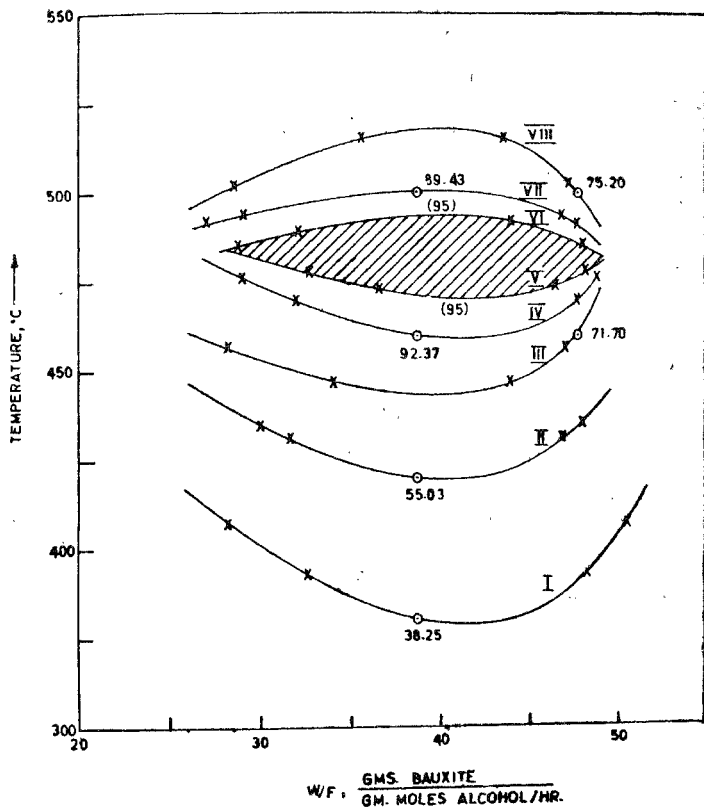
FIG. 2

Pilot of constant conversion contours.

6.   Optimum Conditions

The relationship between mole% conversion and the two independent variables viz., temperature and time factor, is represented as a contour diagram consisting of lines of constant mole% conversion as parameter, the coordinates being temperature and time factor.   Figure 2 shows such contours, on which the circles represent experimental points, while the crosses represent theoretical points calculated from Eq. [1].   In order to obtain theoretical points on any contour the corresponding value of the mole% conversion (x) is put in Eq. [1] and a value is assumed for temperature (y), so that the equation reduces to a quadratic equation by solving which one gets two values of time factor (z).   It is evident from the contour diagram that an optimum point of temperature and time factor lies inside the region bounded by the contours V and VI

7.   Conclusions

In the range of variables employed in the present factorial study viz., temperature, 380°C to 500°C and time factor, 28.97 to 56.96 gms. of bauxite per gm.mole alcohol per hour, the effects and interactions that are significant (at 5% level) are the linear, quadratic and cubic effects of temperature, the linear and quadratic effects of time factor and the interaction of linear temperature with linear-time factor and quadratic-time factor.

At all combinations of temperature and time factor in the shaded region of Fig- 2 the conversion is more than 95%.

References

1. Emmett, P. H.   . .   . .   Catalysis, Vol. 1, Reinhold, New York, 1964, pp. 1-26.

2. Davies, O. L.   . .   . .   The Design and Analysis of Indusrrial Experiments, Oliver & Boyd, 1956, pp. 247-327.