Experiment-2

Rate Study in a Continuous Stirred Tank Reactor
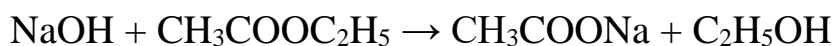
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Objectives

1. To determine the order of the reaction between sodium hydroxide and ethyl acetate using a CSTR.
2. To find the rate constant at a particular temperature.

Theory

Stoichiometric equation:

$NaOH + CH_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$

   (A)   (B)   (C)   (D)

Mole Balance: $\dfrac{V_R}{F_{A_0}} = \dfrac{X_A}{-r_A}$

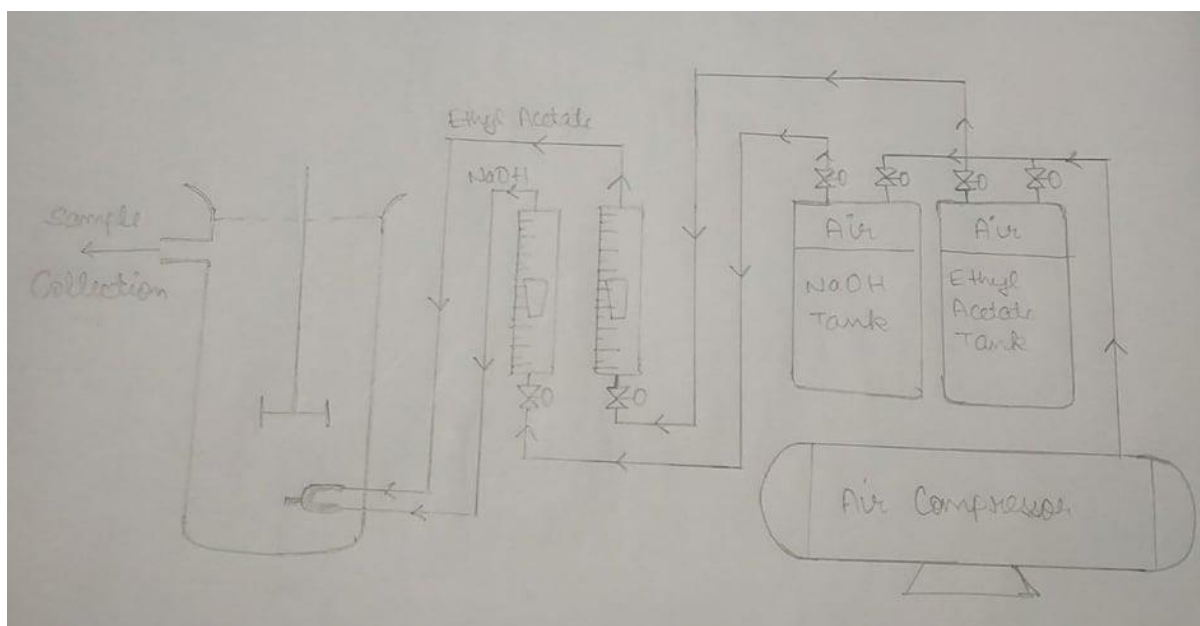Rate Equation: $-r_A = k_2 C_{A_0}^2 (1 - X_A)(M - X_A)$

Assuming 2nd order reaction.

Where $M = \dfrac{C_{B_0}}{C_{A_0}}$, X_A = conversion of A, k_2 = rate constant

From eqn (1) & (2), $\dfrac{V_R}{F_{A_0}} = \dfrac{V_R}{v_0 C_{A_0}} = \dfrac{\tau}{C_{A_0}} = \dfrac{X_A}{k_2^{\xi} C_{A_0} (1-X_A)(M-X_A)} = \dfrac{1}{k_2} f(X_A)$

Where, total volumetric flow rate, $v_0 = v_A + v_B$ and $\tau = \dfrac{V_R}{v_0}$

Schematic

Observations

Strength of Succinic Acid = N/50 = 0.02 N = 0.01 M (dibasic acid)
C_{A0} (before mixing) = 0.05 mol/ L
C_{A0} (after mixing) = 0.025 mol/ L
C_{B0} (before mixing) = 0.1 mol/ L
C_{B0} (after mixing) = 0.05 mol/ L
Volume of reactor = 2.815 L
M = 2

Flow Rate* (LPH)	τ (sec)	C_{A0} (mol/ L)	Titre Volume (ml)	X_A	Average X_A	f (X_A)
5.2	974.42		1.7	0.728	0.73	3406.2
			1.65	0.736		
			1.72	0.725		
9	563	0.025	2.1	0.664	0.664	2366.7
			2.2	0.648		
			2.0	0.68		
13.5	375.34		2.6	0.584	0.6	1714.3
			2.5	0.6		
			2.4	0.616		

Flow rates have been adjusted based on the calibration curve of the Rotameter

Sample Calculations

For flow rate = 9 LPH, titre volume = 2.0 mL

$N_1 V_1 = N_2 V_2$ (mea balance of NaOH and succinic acid).

$0.02 \times 2 = N_2 \times 5 \Rightarrow N_2 = 0.008 \, N = 0.008 \, mol/L$
(monoacidic base).

$X_A = \dfrac{0.025 - 0.008}{0.025} = 0.68$

similarly, for titre volume = 2.1 mL, $X_A = 0.664$

for titre volume = 2.2 mL, $X_A = 0.648$
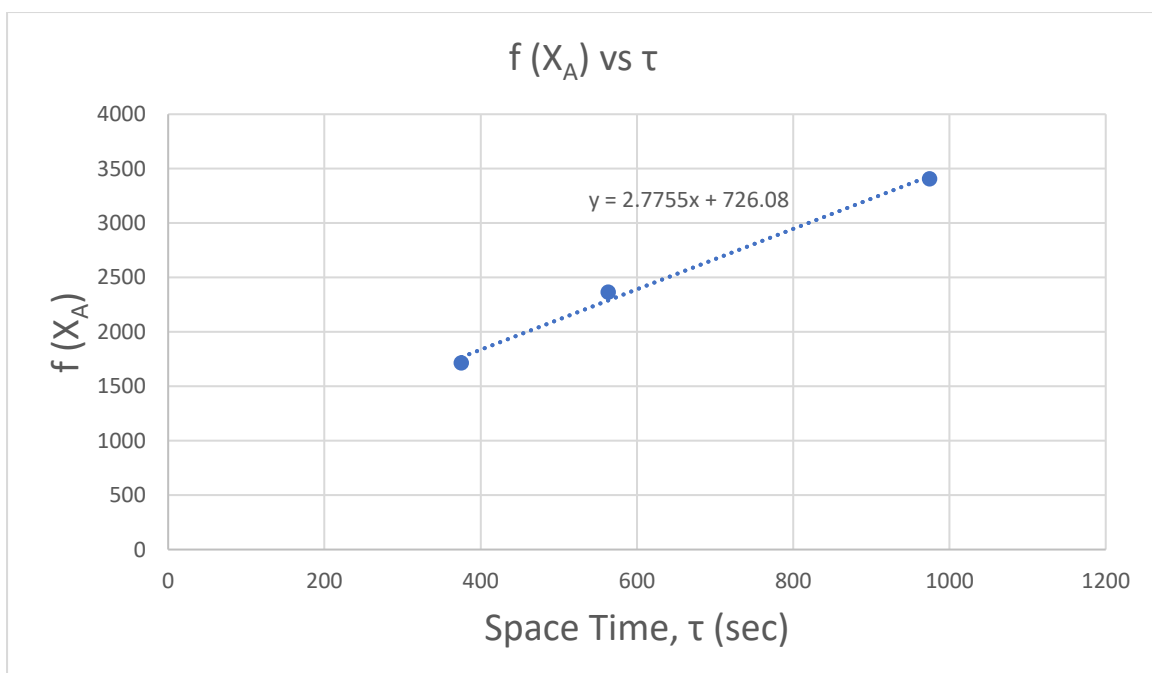
Average $X_A = \dfrac{0.68 + 0.664 + 0.648}{3} = 0.664$

$f(X_A) = \dfrac{X_A}{C_{A0}^2 (1-X_A)(M-X_A)}$, $M = \dfrac{C_{B0}}{C_{A0}} = \dfrac{0.05}{0.025}$

$= 2$

$f(X_A) = \dfrac{0.664}{0.025^2 (1-0.664)(2-0.664)} = 2366.7 \, l^2/mol^2$

$\tau = \dfrac{V_R}{v_0} = \dfrac{2.815}{9+9} \times 3600 = 563 \, sec.$

Plot

f (X_A) vs τ

y = 2.7755x + 726.08

f (X_A) — Space Time, τ (sec)

Results

Slope of above plot = 2.7755 l^2 mol^{-2} s^{-1}

Rate constant = slope × C_{A0} = 2.7755 × 0.025 = 0.069 l mol^{-1} s^{-1}

Therefore, the rate constant of the given reaction under existing temperature conditions comes out to be **0.069 l mol^{-1} s^{-1}**.

Discussion

- The rate constant value is drastically different from the one calculated using a PFR (more than 100% difference). This could be due to several underlying experimental sources of error.
- The water bath used in the experiment cannot exactly maintain a constant temperature because it also loses heat to the surrounding and is at non steady state thus, the reaction kinetics can be affected while the experiment is going on.
- Rotameters are usual sources of error because the markers inside are not in complete equilibrium thus, an approximate value is considered however, the calibration curve helps in attaining a bit of accuracy.
- Phenolphthalein must not be added in excess to the solution to be titrated.
- The colour change must be carefully noted during titration with the last drop to correctly identify the titre volume and a slight delay could result in faulty readings.
- Care must be taken that no drop of succinic acid is wasted while titrating as that can lead to error in the titre volume.
- The meniscus should be carefully observed in the titration to avoid any error in noting the titre volume due to parallax.
- The stirring rate in the CSTR must be maintained at a reasonable rate to facilitate proper mixing. In the calculations, the exit flow rate from the CSTR is not known therefore, the space time is calculated by assuming that the entry and exit flow is the same.

Conclusion

Therefore, through the obtained values of rate constant it can't be concluded which reactor is better for the rate study. Both methods carry their own pros and cons and the most one can do is to eliminate as many cons as possible.