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RESEARCH ARTICLE

DYNAMICS AND MODELLING OF HYDROLYSIS OF ACETIC ANHYDRIDE IN THREE UNEQUAL SIZE CSTRS IN SERIES

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ABSTRACT

After carrying out simulation for Packed bed reactor, we decided to do the same for Continuous stirred tank reactors in series (CSTRs in series) which is also referred as Back mix or Mixed flow rectors are widely used in chemical process industries for conductions single as well as multiphase reactions. These reactions have provisions for continuous inflow of reactants and outflow of product mixture. A certain pool of reaction mixture is always maintained in the reactor so that there is no variation of concentration, temperature or reaction rate throughout the reactor volume. CSTR is having much attention because of its simulation is simple due to absence of temperature and concentration gradient within the reacting fluids. Mathematical modeling and experimental verification of the steady state and dynamic behavior of CSTRs in series is useful for designing of a safe operation of reactor with suitable control. Here, hydrolysis reaction of acetic anhydride in the presence of acetic acid as solvents and sulphuric acid as catalyst was chosen as a reactions system. Experiments were carried out for heat loss for all the three reactors as model and then simulation was carried.

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INTRODUCTION

Continuous flow stirred tank reactors (continuous stirred tank reactor – CSTR) which is also referred as back mix or mixed flow reactors are widely used in chemical process industries for conducting single as well as multiphase reactions. These reactors have provisions for continuous inflow of reactants and outflow of the product mixture. A certain pool of reaction mixture is always maintained in the reactor. So, there is no variation of concentration, temperature or reaction rate throughout the reactor volume. CSTR is having much attention because of its simplicity of mathematical simulation due to the absence of temperature and concentration gradients within the reacting fluids. Mathematical modeling and experimental verification of the steady state and dynamic behaviour of CSTR is useful for designing of a safely operable CSTR with suitable control.

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CSTR in Series

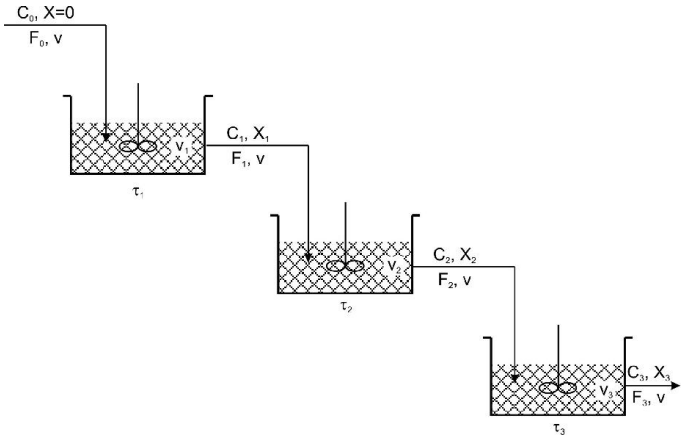
It is always advantageous to use CSTR in series as the total volume required to achieve specific conversion is less than the volume of a single CSTR.

In the present study three unequal size CSTRs in series were taken. Volumes of first, second and third reactors were taken 500, 300 and 100 CC respectively.

Residence Time Distribution (RTD) experiments were carried out by introducing negative step change and measuring concentration against time. Heat loss experiments were conducted for determining value of heat loss parameter. Experiments for parametric sensitivity also carried out.

The mass and energy balance equations for CSTRs in series were solved numerically by Range-Kutta method to give transient temperature, concentration with respect to time.

Unequal Size CSTRs in Series



Here,

F_0, F_1, F_2, F_3 = molar feed rate of a reactant in moles / time. Reactor – 1, 2, 3 & outlet of reactor-3 respectively.

C_0, C_1, C_2, C_3 = molar feed rate of a reactant in moles / volume to the reactor-1, 2 3 & outlet of reactor-3 respectively.

X_1, X_2, X_3 = conversion of a reactant achieved upto point 1, 2 & 3 respectively.

τ_1, τ_2, τ_3 = Time constant for reactor 1, 2 & 3 respectively
V_1, V_2, V_3 = volume of reactor 1, 2, 3.

v = volumetric feed rate in volume / time.

F = C.v.

$$\tau = \frac{V}{v} = \frac{VC}{F}$$

$(-r)_1$, $(-r)_2$, $(-r)_3$ = Rate of disappearance of a reactant in reactor 1, 2 & 3 respectively.

Relationship between conversion and molal flow rates for the system is given by the equations.

$F_1 = F_0 (1-X_1)$
$F_2 = F_0 (1-X_2)$
$F_3 = F_0 (1-X_3)$

Mole balance for a reaectant for reactor-2

In = Out + Disappearance
$F_1 = F_2 + (-r)_2 V_2$

$$V_2 = \frac{F_1 - F_2}{(-r)_2}$$

Putting values of F_1 & F_2

$$V_2 = \frac{F_0(1-X_1) - F_0(1-X_2)}{(-r)_2}$$

$$\therefore V_2 = \frac{F_0 - F_0X_1 - F_0 - F_0X_2}{(-r)_2}$$

$$\therefore \frac{V_2}{F_0} = \frac{X_1 - X_2}{(-r)_2}$$

We have, $\tau = \frac{VC}{F}$

$$\therefore \frac{\tau_2}{C_0} = \frac{V_2}{F_0} = \frac{X_2 - X_1}{(-r)_2}$$

Similarly, for the First reactor,

$$\frac{\tau_1}{C_0} = \frac{X_1 - X_2}{(-r)_1}$$, here $X_0 = 0$

$$\frac{\tau_1}{C_0} = \frac{X_1}{(-r)_1}$$

For third reactor,

$$\frac{\tau_3}{C_0} = \frac{X_3 - X_2}{(-r)_3}$$

Mole balance of a reactant for reactor-2 in terms of concentration is :

$vC_1 = vC_2 (-r_2) V_2$

$$\frac{V_2}{v} = \frac{C_1 - C_2}{(-r)_2}$$

$$\tau_2 = \frac{C_1 - C_2}{(-r)_2}$$

Similarly,

For CSTR-1, $\tau_1 = \frac{C_0 - C_1}{(-r)_1}$

For CSTR-3, $\tau_3 = \frac{C_2 - C_3}{(-r)_3}$

Selection of the reaction system

Hydrolysis reaction of acetic anhydride in the presence of acetic acid as solvent and sulfuric acid as catalyst was chosen as a reaction system because of known kinetics, significant heat of reaction, cost and availability of chemicals, availability of various physical, chemical and thermodynamics properties, existence of multiple steady states for a set of feed concentration, feed temperature and residence time which can be attained in laboratory without much difficulties.

Experimental Setup

Experimental setup is shown in Figure 1. The setup mainly consisted of three unequal size CSTRs in series. Volume of first, second and third reactor was 600, 300 and 100 CC respectively. Reactors were stainless steel cylindrical vessels of thickness around 1 mm with SS cover with holes for thermometers, inlet and outlet of cooling coils and vent holes. Magnetic stirrer was used in each reactor to stir the contents of the reactors. Each reactor was provided with inlet near the bottom and outlet near the top. The three reactors were attached through rubber tubes as shown in the figure. Reactants at a set flow rate were forced into the reactor from the overhead storage tanks to get constants flow rate. Levels of storage tanks of reactants were maintained to ensure constant flow rates. The height of each reactor was set in such a way to ensure smooth gravity flow from one reactor to the next one.

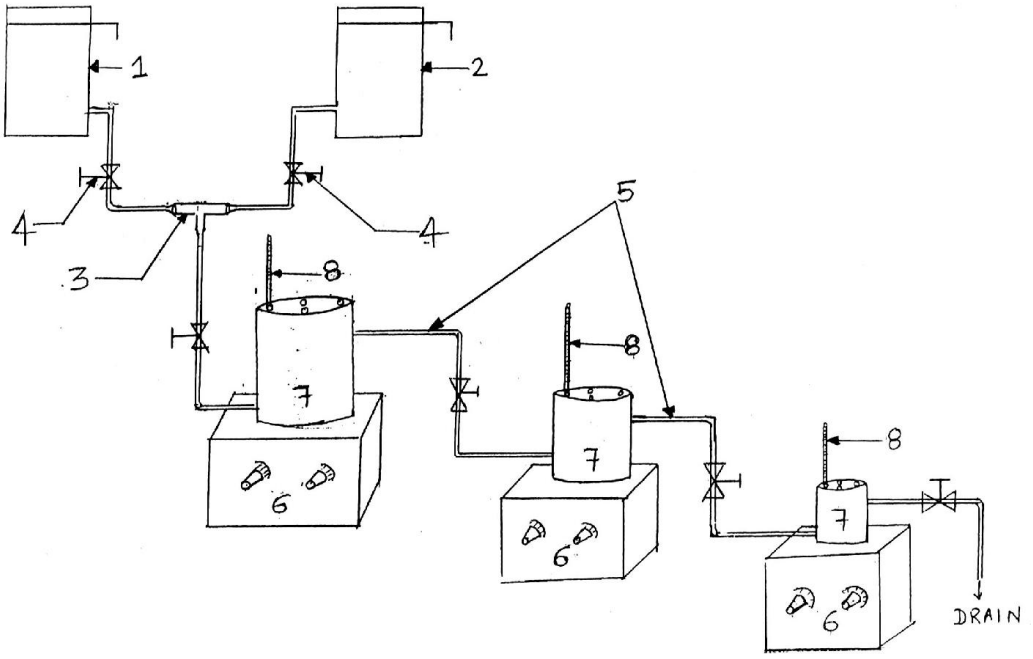


Figure 1. Experimental Setup

Nomenclature for Figure-1

1. Acetic Anhydride Storage Tank
2. Mixture (Acetic Acid + Water + H_2SO_4)
3. Glass – T
4. Pinch Cocks
5. Rubber Tubes
6. Magnetic Stirrers
7. Reactors of Different Volumes
8. Thermometers

PROCEDURE

1. For RTD Studies

RTD experiments were carried out to understand flow pattern in the CSTRs series. RTD experiments for verifying the three reactors were carried out by giving a step input into the individual reactors. Each reactor was first filled with 0.1 N NaOH solution. The magnetic stirrer was set under full speed. Pure water at different flow rate was allowed to flow into the each reactor individually. At definite time interval 5 CC samples from each reactor were collected and analysed for concentration of NaOH by titrating against Oxalic acid.

2. For Heat Loss Experiments

Reactors were made up of bare stainless steel without insulation. Therefore, heat loss was expected from the reactors to the atmosphere during the experimental run. Hence, to calibrate the nature of the heat loss taking place from the reactor, heat loss experiments were conduced in the manner described below : At first, the mixture of acetic acid and water was heated upto certain temperature. Then poured into the reactor and stop watch was started. Change in temperature of the mixture was noted with respect to time. Mathematical model was developed by using energy balance.

Rate of change of temperature of the reactor content = Rate of heat loss from the reactor to the atmosphere

$$V.\rho Cp\left(\frac{dT}{dt}\right) = \text{UA (T-T air)}$$

Where V = volume of reactor & ρ is density of liquid mixture. T is temp. of liquid in the reactor.

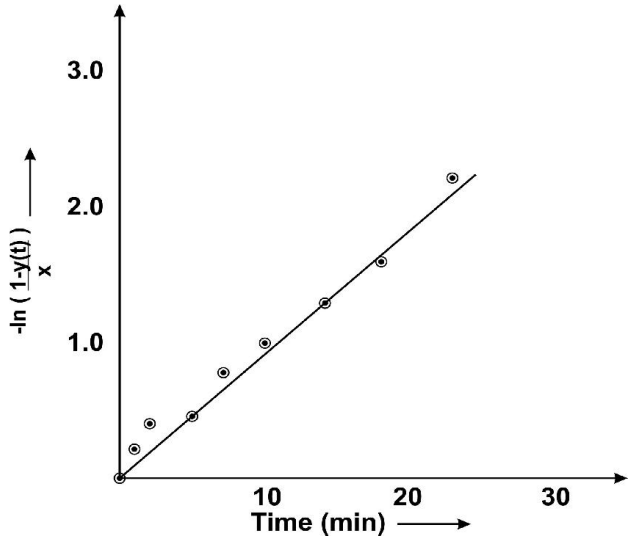


Figure 2. Results of RTD Studies for Reactor – 1

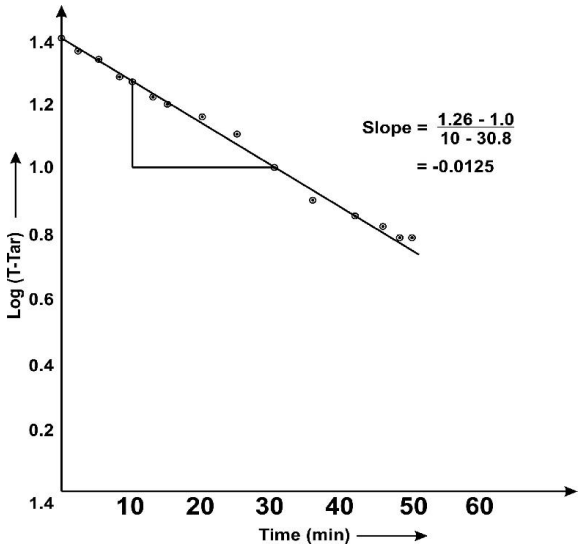


Figure 3. Results of Heat Loss Experiment for Reactor – 1

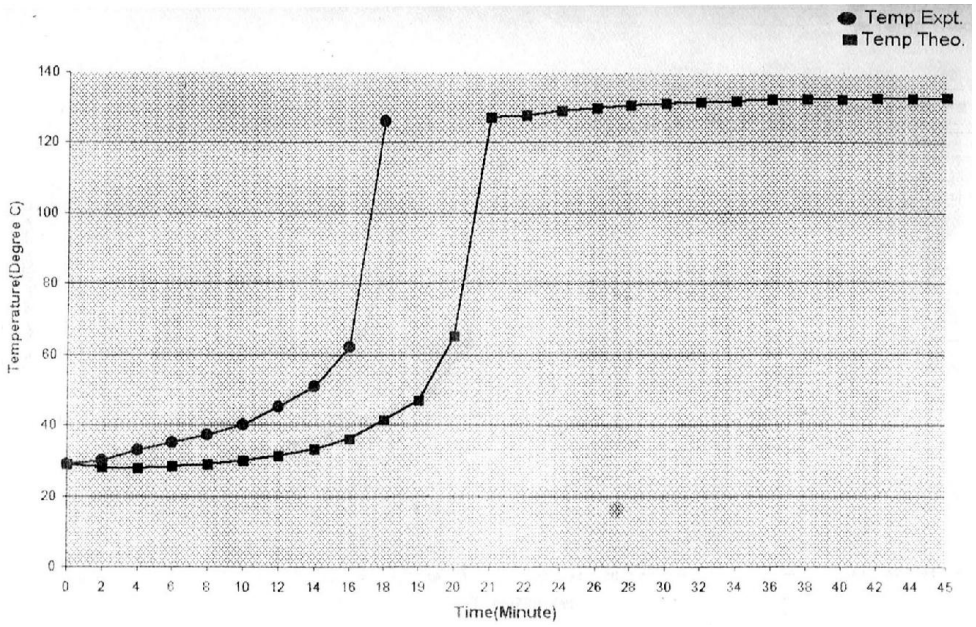


Figure 4. Results of Parametric Sensitivity for Reactor – 1

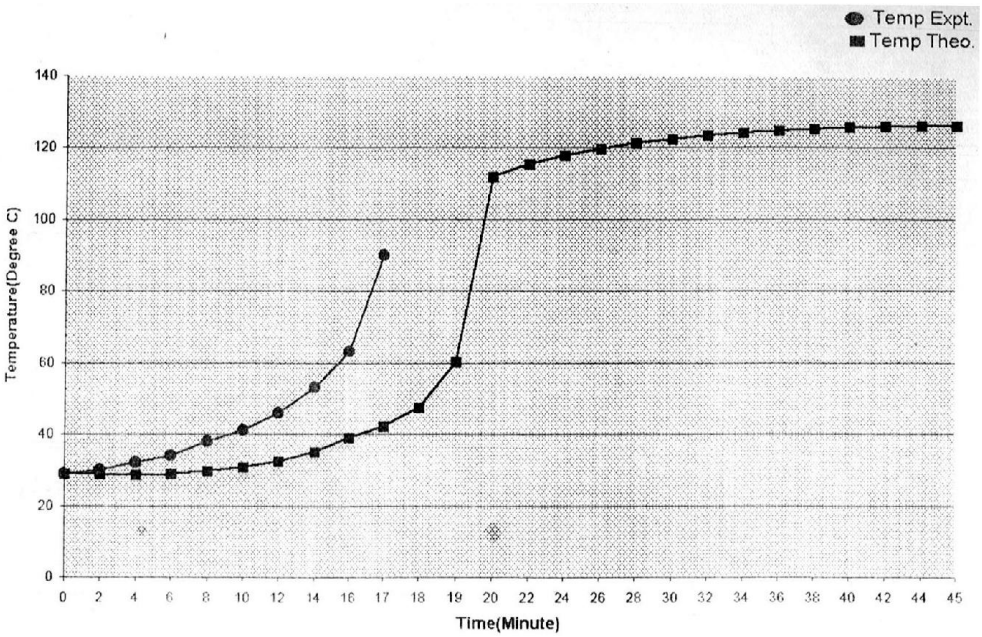


Figure 5. Results of Parametric Sensitivity for Reactor - 2

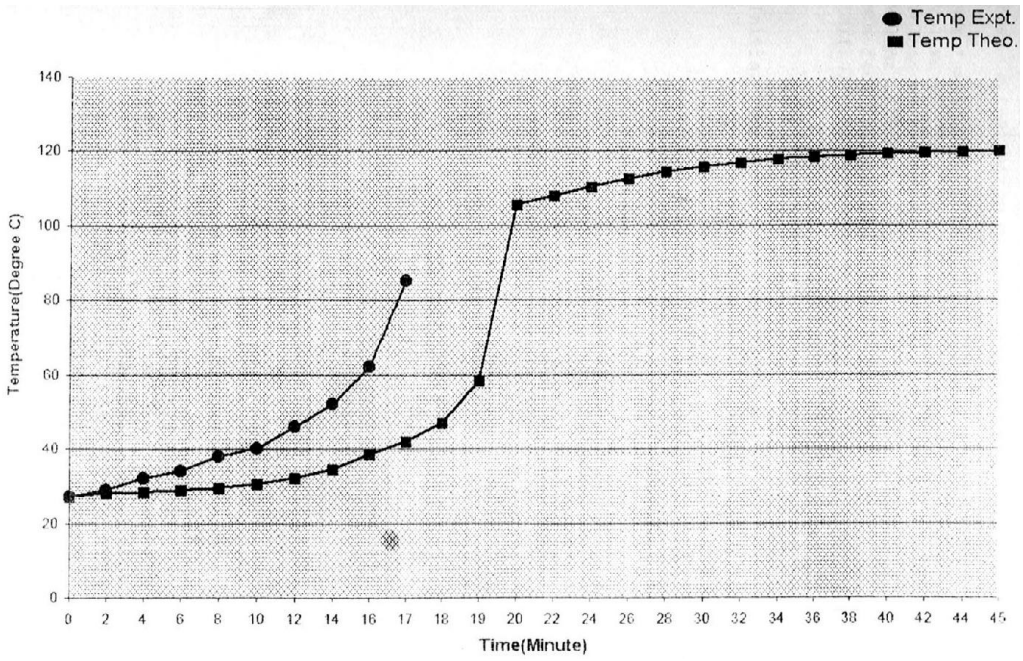


Figure 6. Results of Parametric Sensitivity for Reactor – 3

3. For Parametric Sensitivity

Flow rates & temperature of Acetic Anhydride from storage tank-1 and mixture of acetic acid, sulphuric acid and water from storage tank-2 were selected and obtained by screw pinch cock & thermostat. The drain tubes of the reactors were closed and were filled up to the outlet with acetic anhydride and / or the mixture of acetic acid, sulphuric acid and water having known composition and temperature. Liquid in the reactor starts overflowing almost instantaneously while the feed was continuously allowed to flow into the reactor at desired flow rates and temperatures. Stirring was started immediately after pouring the reactants into the reactor and stirrer speed was maintained constant to conform the ideality of the reactors. The experimental run was allowed to continue till the temperature of the reactors reaches a constant value i.e. until steady state.

RESULTS AND DISCUSSION

1. For RTD Studies

$\ln \{ 1 - y(t) / x \} = RTD - t / \tau$

where y(t) = concentration of sample at any time t for reactor
x = Final concentration for reactor

Graphs ln {1-y(t) / x} vs time were plotted for all three reactors. The linear plot with negative slop equal to ($1/\tau$) fits the experimental data for each reactor.

2. For Heat Loss Experiments

From heat loss experiments, graph of log (T-T air) vs time is shown in Figure 3 for reactor-1 which was obtained from mathematical model.

$$\frac{UA}{v\rho Cp} = 0.01217 \text{ (Slope of the graph)}$$

3. For Parametric Sensitivity

Results of parametric sensitivity for reactor 1, 2 & 3 are shown in Figure 4, 5 & 6 respectively for the given parameters. Flow rate of acetic anhydride from storage tank-1 :37 cc/min

Flow rate of mixture of acetic acid, sulphuric acid and water from storage tank-2 :58 cc/min

Feed Concentration of acetic anhydride :0.0067gmole/cc
Initial concentration of Acetic Anhydride in all reactors :0.00 gmole/cc Mix feed temperature :296.16 k Initial temperature in reactor-1, 2, 3 : 302.16, 302.16, 300.16 K respectively

It is clear from Fig 4, 5 & 6 that temperatures in all three reactors increases gradually and then becomes constant. Graphs of theoretical data obtained by modelling are also shown for comparison. It is clear from all graphs that constant temperatures are obtained little lower for all three experimental results. This happens due to heat loss through reactors during experiments because reactors are made of bare stainless steel without insulation. So, heat losses are expected from the reactors during the experimental run. Given reaction is exothermic by nature

Conclusion

The following conclusions are drawn for the present studies :

1. RTD experiments were conducted by giving a negative step change and concentration curve for transient concentration

data was found. The CSTR in series was thus found to be ideal for doing further reaction experiments.

2. Reaction experiments at different flow rates, concentrations, temperatures were conducted and transient temperature data were noted. A mathematical model was developed by writing energy & mass balance and solving numerically by 3rd order Range-Kutta method. Predicted temperatures by the model are in satisfactory agreement with transient experimental data.

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