



## A Comparative Study of Saponification Reaction in a PFR and CSTR

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### Abstract

A Comparative study of Saponification reaction has been conducted in a Continuous stirred tank reactor (CSTR) and Plug flow reactor (PFR). The reaction chosen for investigation was hydrolysis of ethyl acetate with sodium hydroxide. The objectives here are to examine the effect of process conditions on steady state conversion and rate constant in a PFR and CSTR. The variables examined for comparative study are feed rate, temperature and residence time. Data were collected at constant concentration of 0.1 M of both the reactants and at a constant pressure of 1 atmosphere. A reaction conversion of 72.8% and specific rate constant of 1.27 L/mol.sec were obtained for PFR as compared with a decreased value of 48.6% and 0.0478 L/mol.sec for baffled CSTR under steady state conditions. Conversion increases with increase of temperature both in case of PFR and CSTR under studied range of temperature. Maximum conversion and rate constant of 68.4% and 0.688 L/mol.sec respectively were obtained for tubular reactor as compared with reaction conversion and rate constant of 63.4% and 0.127 L/mol.sec respectively for CSTR as temperature varies. For PFR with a reactor volume of 0.40 liter, reaction conversion varies from 50.4 % to 39.2 % corresponding to flow rates of 50 ml/min to 80 ml/min respectively. In case of CSTR with adjusted reactor volume of 1.5 liter, reaction conversion varies from 46.2% to 44.8% corresponding to feed rate values from 50 ml/min to 80 ml/min. In both cases of PFR and CSTR, residence time decreases with increased feed rate and this leads to decreased values of reaction conversion for both types of reactors. The results obtained in this investigation may be useful in maximizing the industrial level production of desired product and to predict the type of flow reactor more suitable for ethyl acetate saponification reaction.

**Keywords:** PFR, CSTR, conversion, temperature.

### Introduction

A Chemical reactor is one of the primary components of chemical process industry used to carry out either endothermic or exothermic reactions. A chemical reactor can be classified into two categories, batch and flow reactors based on the empirical information. A batch reactor operates under unsteady-state conditions and it holds the reaction mixture during the reaction. In the investigation of kinetics of heterogeneous reactions<sup>1</sup>, steady state- flow reactors are mainly used. Two main categories of ideal reactors used in chemical industries operate under steady-state conditions are -plug flow reactors and continuous stirred tank reactors.

PFRs are widely familiar as continuous tubular reactor play a key role in chemical industries. In case of requirement of continuous operation, tubular reactors are frequently used but without back-blending of reactants and products. The plug flow type continuous reactors offer large volumetric reaction conversion due to lower yield of undesired product. The advantages of plug flow reactors include high volumetric unit conversion and capability of running for longer period without maintenance.

PFRs volume will usually be lower than CSTR volume for same conversion and reaction conditions for isothermal reactions

greater than zero order<sup>2</sup>. From the industrial production point of view, PFR can be arranged as a single tube or in the form of a coil relying on the process application. PFR is extensively employed in the chemical process industry for liquid and gas phase reactions.

CSTRs offer wide applications in the areas of food, chemical and the pharmaceuticals due to good blending and scale-up attribute<sup>3</sup>. CSTRs – as a single reactor or, constantly, configured as reactors in series or parallel are practically always operated under steady state conditions. It is used primarily for liquid phase reactions and is assumed to be perfectly mixed. The temperature and concentration (consequently, the rate of reaction) are independent of time and position inside the CSTR.

The outlet streams from a CSTR have the same concentrations as the reaction mixture within the reactor. Agitator vessels are generally used throughout the chemical process industries for various applications such as storing, blending and reacting materials. In addition to this, most agitated vessels use baffles, and the design of baffles system must satisfy process objectives economically. On the other hand, presence of baffles in the reactor system has some other consequences- like abolishing the formation of vortex, increase the power input, and to enhance the mechanical strength<sup>4</sup>. The existence of baffles in the reactor system yields axial type flow.

Oil hydrolysis under antacid conditions is known as saponification<sup>5</sup>. Sodium acetate ( $\text{CH}_3\text{COONa}$ ) and ethanol ( $\text{C}_2\text{H}_5\text{OH}$ ) are produced by hydrolysis of ethyl acetate by sodium hydroxide. Numerous investigations were conducted on the advancement for this hydrolysis reaction. Second order saponification of ethyl acetate with sodium hydroxide is a well-known reaction in the literature dealing with chemical kinetics<sup>6</sup>. Ethyl acetate saponification reaction was studied with distinct behavior in semi batch and batch reactors for comparative purpose by the investigator<sup>7</sup>. In this work, a good compliance with the simulated outcomes has been obtained making it feasible to verify the mathematical models developed.

Several measurement techniques<sup>8-9</sup> at different temperatures have been used to study the second order saponification reaction. The other technique depends on the conductometric measurement to determine reactor performance was investigated<sup>10</sup> and this technique is based on the conductivity measurement of product sample, prevents periodic removal of samples for analysis.

## Material and Methods

**Chemicals:** Analytical reagent (AR) - grade chemicals were utilized to conduct the experiments using PFR and CSTR. Ethyl acetate with 99.5% purity and sodium hydroxide with 98.0-100% purity were used to carry out the saponification reaction. High purity distilled water was used to prepare the stock solutions of NaOH (0.1M) and  $\text{CH}_3\text{COOC}_2\text{H}_5$  (0.1 M)

**Experimental Setup:** A CSTR (Armfield, U.K.) with internal diameter of 0.153 meter and maximum volume of 2.0 liter is used to perform the experiments. Temperature of the reaction mixture is controlled by a stainless steel coil inside the reactor. A turbine agitator in conjunction with four vertical baffles provides efficient mixing and heat transfer.

A tubular reactor (Armfield, U.K.) of length 20.9 meter, internal diameter ~ 5.0 mm and total volume of 0.4 liter has been used for collecting the data for analysis. The tubular reactor in which the reaction under consideration takes place is a pliable coil. To maintain a fixed temperature of reaction mixture, the coil is immersed in circling water which is necessarily kept at a pre-decided fixed temperature.

**Experimental Procedure:** Solutions of sodium hydroxide (0.1M) and ethyl acetate (0.1M) were prepared using high-purity distilled water to conduct the experiment at different operating conditions. Data were collected for a PFR and a CSTR for comparing their performances. After reaching the required process conditions in both types of reactors, actual-time conductivity values were noted accordingly. The steady state conversion and the rate constant were calculated for both reactors using conductivity data.

## Results and Discussion

**Steady State Condition:** Data at steady state conditions were collected for CSTR and PFR at a fixed temperature equal to 30°C and at a concentration ~ 0.1 M of both reactants. In case of CSTR, feed flow rate was adjusted to 60 ml/min of reactants, reactor volume adjusted to 1.5 liter and agitation rate kept at 130 rpm. Feed rates of both the reactant and reactor volume were selected as 80 ml/min and 0.41 liter respectively for tubular reactor.

Real-time conductivity was monitored at 5-minute intervals up to steady-state situation. Steady- state of desired operating conditions was achieved after a period of 35 minutes as shown in figure-1. NaOH and  $\text{CH}_3\text{COOC}_2\text{H}_5$  impart conductance to the reactor content as reaction progresses. Sodium hydroxide concentration decreases as reaction proceeds leading to decrease in conductivity values. A reaction conversion of 72.8% and rate constant ~ 1.27 L/mol.sec were obtained for PFR as compared with a lower value of 48.6% and 0.0478 L/mol.sec for baffled CSTR under specified operating conditions at steady state.

**Temperature:** Experiments were conducted under a fixed feed rate of 60 ml/min for PFR and CSTR. The effects of temperature on conversion and rate constant for tubular reactor and CSTR are shown in figure- 2. Maximum conversion and a rate constant of 68.4% and 0.688 L/mol.sec were obtained at a temperature of 40°C for PFR when volume of reactor was adjusted to 1.5 liter and agitator rate to 130 rpm.

Under the constant operating conditions of feed flow rates and reactant concentration, reaction conversion and specific rate constant of 63.4% and 0.127 L/mol.sec respectively were obtained at highest temperature studied (40°C) with a reactor volume of 0.41 liter. Overall, tubular reactor performed better under constant operating conditions as compared with CSTR.

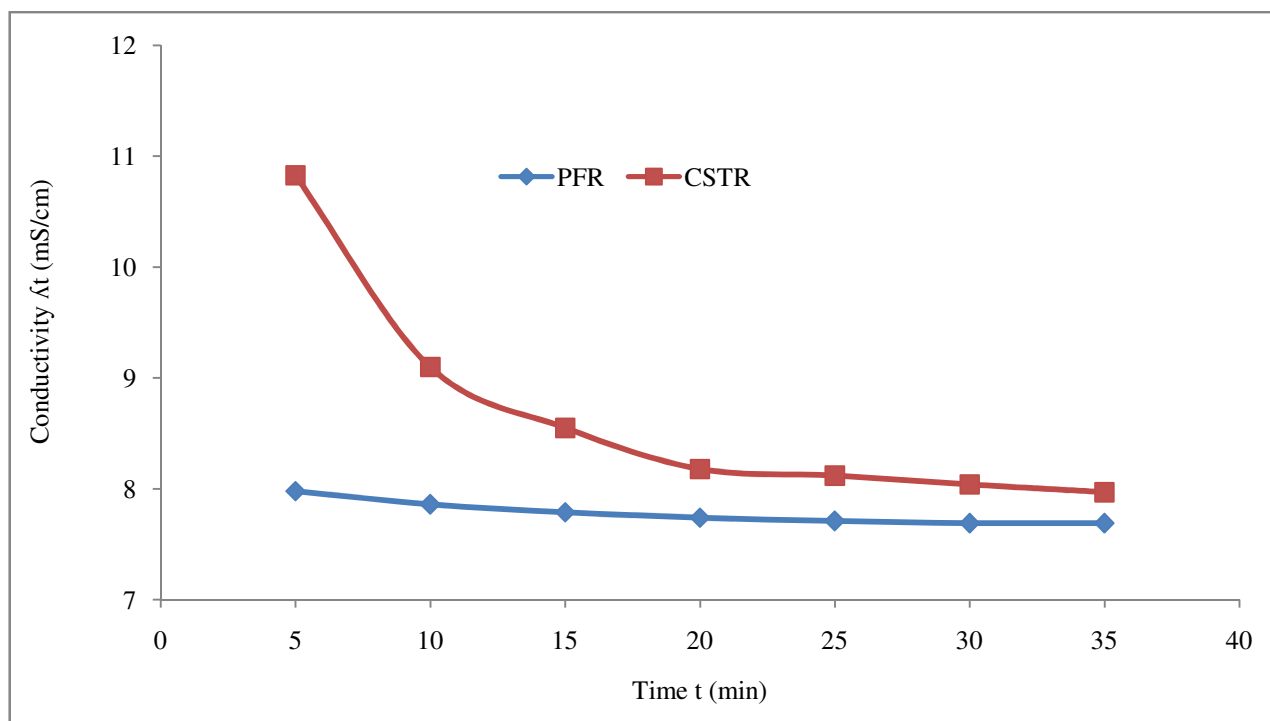
**Flow rate:** Data were collected for PFR and CSTR at constant temperature of 30°C and concentration of 0.1 M of both the reactants. In general, reaction conversion decreases as feed rate is increased for both types of reactors. With increased feed rate, residence time decreases and this leads to decreased conversion values. For PFR with reactor volume of 0.40 liter, reaction conversion varies from 50.4 % to 39.2 % as feed rate varies from 50 ml/min to 80 ml/min respectively as shown in figure-3.

In case of CSTR with adjusted reactor volume of 1.5 liter, reaction conversion varies from 46.2% to 44.8% corresponding to feed rate values from 50 ml/min to 80 ml/min respectively. Rate constant of 0.0354 L/mol.sec was obtained at fixed feed rate of 50 ml/min as compared with a rate constant of 0.0834 L/mol.sec at a feed rate of 80 ml/min in case of CSTR.

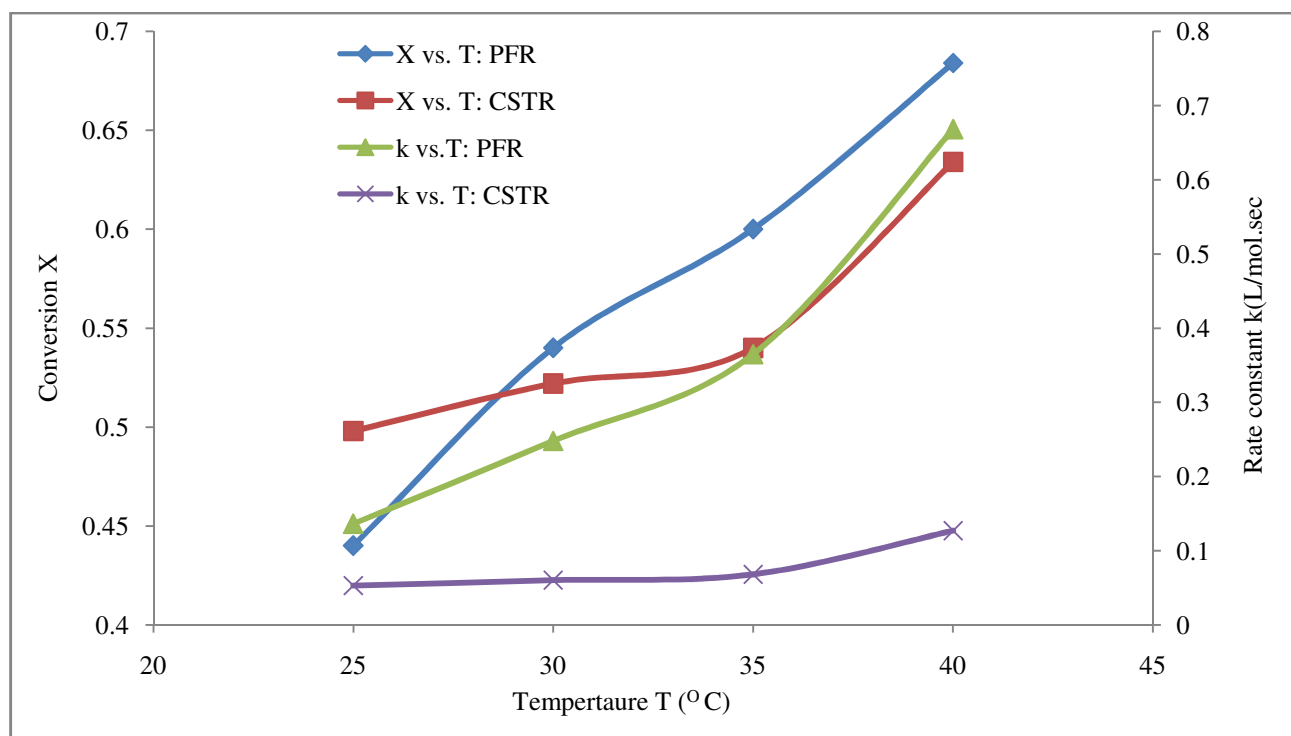
Figure-4 shows the variation of residence time with feed rate in case of PFR and CSTR. In both cases, residence time decreases with increased feed rate and this leads to decreased values of

reaction conversion for both types of reactors. At the same feed rate under certain constant operating conditions, residence time of CSTR is more than that of PFR i.e. at a fixed feed rate of 60

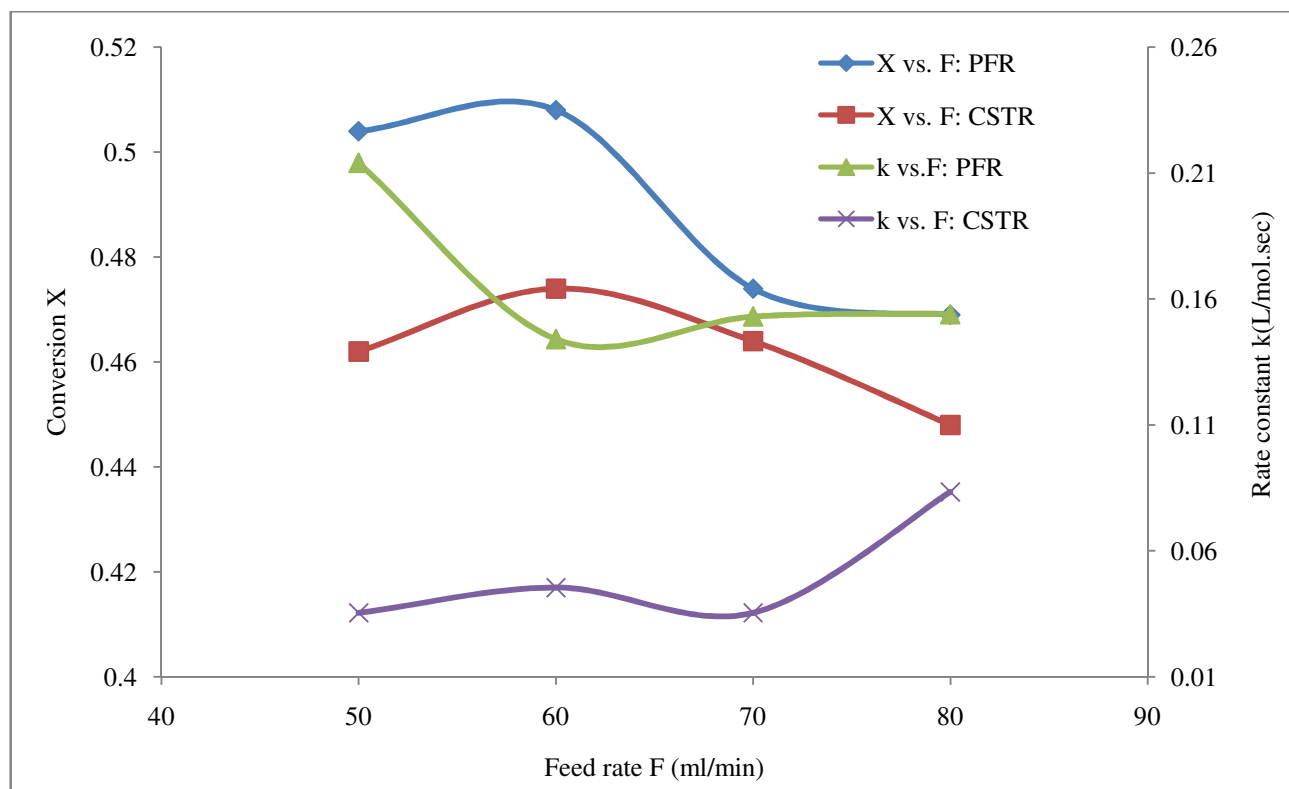
ml/min; residence time of CSTR is 750 second, compared with a residence time of 205 second for PFR.



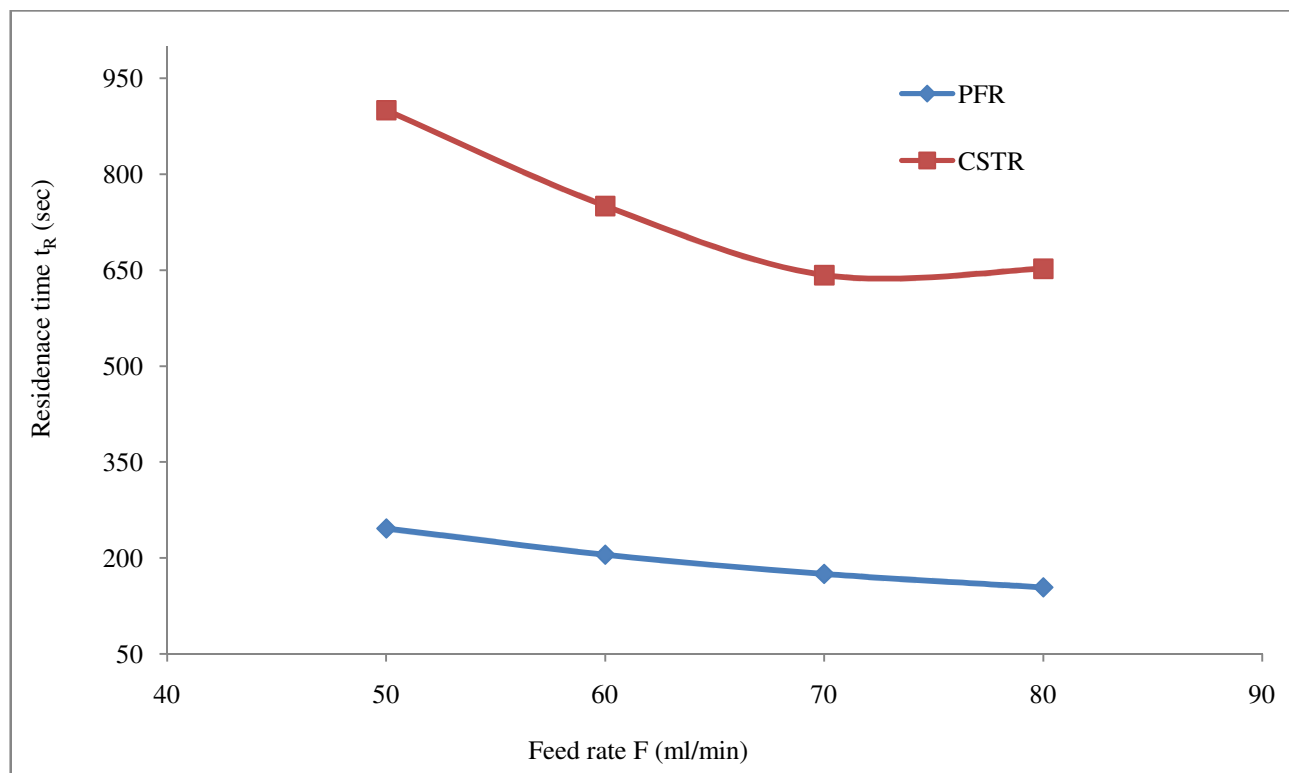
**Figure-1**  
Conductivity versus time curves for PFR and CSTR



**Figure-2**  
Conversion and rate constant versus temperature curves



**Figure-3**  
Conversion and rate constant versus feed rate curves



**Figure-4**  
Residence time versus feed rate curves for PFR and CSTR

## Conclusion

During this research,  $\text{CH}_3\text{COOC}_2\text{H}_5$  hydrolysis with NaOH was examined, and effects of process conditions on steady-state reaction conversion and rate constant were analyzed for PFR and CSTR. Reaction progression was tracked by actual-time conductivity under various process conditions. The results obtained for PFR and CSTR were analyzed and compared.

A decrease of actual-time conductivity values with time specifies the progression of hydrolysis reaction in case of PFR and CSTR. A reaction conversion of 72.8% and rate constant  $\sim 1.27$  L/mol.sec were obtained for PFR as compared with a lower value of 48.6% and 0.0478 L/mol.sec for baffled CSTR under steady state conditions. Conversion increases with increased temperature both in case of PFR and CSTR under studied range of temperature. Maximum conversion and rate constant of 68.4% and 0.688 L/mol.sec respectively were obtained for tubular reactor as compared with reaction conversion and rate constant of 63.4% and 0.127 L/mol.sec respectively for CSTR as temperature varies. For PFR with reactor volume of 0.40 liter, reaction conversion varies from 50.4 % to 39.2 % corresponding to flow rates of 50 ml/min to 80 ml/min respectively. In case of CSTR with adjusted reactor volume of 1.5 liter, reaction conversion varies from 46.2% to 44.8% as feed rate varies from 50 ml/min to 80 ml/min. In both case of PFR and CSTR, residence time decreases with increased reactants flow rate and this leads to decreased values of reaction conversion for both types of reactors.

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