

## Investigation the Effect of Agitation and Flow Rate in Three-Stage Continuous Stirred Tank Reactors Connected In Series for Ethyl Acetatesaponification Reaction

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**Abstract**-Investigation study of three-stage continuous stirred tank reactor for ethyl acetate hydrolysis reaction has been accompanied. Sodium hydroxide and Ethyl acetate were selected for reaction evaluation. The parameters evaluated for saponification reaction were flowrate (40ml/min, 70 ml/min, and 100 ml/min), agitation rate (minimum, medium, and maximum), and temperature of non-isothermal conditions. Nine experimental performances were investigated and for all the experiments initial concentration of ethyl acetate and sodium hydroxide were constant. The NaOH(4%) and CH<sub>3</sub>COONa (6%) were used for the data recorded. The minimum conductivity values, the maximum temperatures, and conversion values of the reaction were 45.2%, 66.34% at a temperature of 24.9 °C for R<sub>1</sub>, 41.1%, 69.53% at a temperature of 24.5 °C for R<sub>2</sub>, and 40.7%, 69.76 % at the temperature of 24 °C for R<sub>3</sub> for 40 ml/min flowrate and minimum agitation rate individually. Based on the medium agitation rate and medium flow rate (70ml/min) the minimum value of conductivity and maximum value of conversion was 38.3% and 71.59% obtained at a temperature of 27.1 °C of R<sub>1</sub>, 30.5%, and 77.53% at a temperature of 27.5 °C of R<sub>2</sub>, 27.8% and 79.59% at a temperature of 26.8 °C of R<sub>3</sub> respectively. Compared to others the reactions operated at a maximum flow rate (100ml/min) and maximum agitation rate of the conductivity and conversion values were 43.1% and 67.94% of R<sub>1</sub> at the temperature of 27.8 °C, 34.4 % and 74.56% R<sub>2</sub>, 34.1% and 74.79 % of R<sub>3</sub> at the temperature of 28.3 °C. The three-stage CSTRs connected in series value of conductivity decreased accelerable, but the value of conversion increased progressively. The results gained in this evaluation may help optimize the production of desired products at a large level and predict the advantage of multiple-stage reactors connected in series extra appropriate for the saponification reaction of ethyl acetate.

**Keywords**- three-stage continuous stir tank reactors; saponification; conductivity; flow rate; conversion; agitation rate

### I. INTRODUCTION

Chemical reaction carryout in chemical process industries is performed inside chemical reactors. according to a mode of operation for either endothermic or exothermic reaction chemical vessel equipment's classified into a batch and flow type. Unsteady state mode of operation and it holds homogenous or heterogenous reactant components for a specific time of the reaction inside the batch reactor [1]. Whereas the flow type reactors operated in steady-state and open system mode of reaction conditions [2]. Among them, continuously stirred tank reactors are used for homogenous reaction systems and operating at the steady-state condition. Well, mixing and easy to scale up in the sector of food production, pharmaceuticals and chemical industry areas. Single continuous stirred tank reactor of multiple-stage CSTRs offer wide applications [3]. Widely CSTRs are used for liquid homogenous reaction with uniform and perfectly mixing consideration. The arrangement of CSTRs is a large single reactor tank or small reactors connected in parallel or in series that always operating under steady-state. Inside the CSTR the reaction

rate depends on the agitation rate, temperature, flow rate, and residence time, not on the concentration and temperature of the reaction. Under steady-state, the concentration of the reacting components inside the reactor has the same as exit streams and the reaction mixture makes uniform concentration and temperature by mixing with the aid of an agitator. The existence of baffles used to reduce vortex, enhance mechanical strength and increase power input to satisfy the processing system axial flow type and economically [4].

A saponification reaction is called the Break down of oil material under acid conditions [5]. The output products from the oil hydrolysis are CH<sub>3</sub>COONa and C<sub>2</sub>H<sub>5</sub>OH. Different experimental evaluation was carried out for the ethyl acetate hydrolysis reaction, the second order of reaction and its kinetics is a well-known in the literature [6, 7, 8, 9]. The reactor of batch and semi batch operation was examined for the saponification reaction of ethyl acetate [10, 11, 12] and mathematical models was developed. For measurement of the second order a lot of techniques with variant temperatures have been used to investigation [13,

14]. Conductivity based techniques was also evaluated for performance of the reaction rate[15]. So, for evaluations and analysis of this reaction system used the conductivity measurement techniques.

## II. MATERIAL AND METHODS

**Chemicals:** laboratory solutions- were used for mien the lab workemployinga three-stage continuous stirred tank reactor. With 99.5% and 98-100%,the purityof Ethyl acetate and sodium hydroxide were utilized to perform the hydrolysis reaction. The stock solution was prepared with distilled waterto contain high purityof 6%  $\text{CH}_3\text{COOC}_2\text{H}_5$  and 4 % NaOH.

**Investigational Setup:** A three-stage CSTRs (Gunt, Germany) tank volume of 1.2 liters and length of delay section 2.8 m and delay section volume 0.2 liter and approximate weight of the reactor tank is 32 kg is used for investigating the experiments.for collecting the data, a three-stage CSTR (Gunt, Germany) has been usedfor analysis and the reaction conditions are non-isothermal and mixture temperature is not used heat coil inside the three-stage reactions.Mixture solutions are mixed via agitator added by turbine for well and efficient mixing and making uniform temperature.The reaction carried out inside the three-stage reactor is aided by a mixer and the reaction takes place inside the tank.

**Working Procedure:** to experiment first stage reagent of 6% ethyl acetate and 4% sodium hydroxide solutions were prepared with purity to distill waterat different operating conditions. The experiments were evaluated at process conditions minimum, medium and maximum and 40 %, 60% and 80% and 30, 60, 90, 120, and 180 seconds,mixer of the reactor,flow rate and residence time respectively.Reactor training boards were used for collecting and evaluated the saponification reaction data inside the three-stage CSTRs. The conductivity values and reaction temperatures were noted when the reaction mixture and the required process parameters were set and reached the three-stage CSTRs using the conductivity meter and reactor board. Concentration and conversion values were recorded and calculated at steady-state conditions for all the three reactors connected in series.



Figure 1. Experimental setup of three-stage continuous stir tank reactors connected in series

## III. RESULTS AND DISCUSSION

**Agitation rate:** the agitation rate of the saponification reaction of ethel acetate strongly affects the ration rate. It operated at minimum, medium, and maximum agitation rate at 40, 70, and 100 flow rates measured in ml/min. The maximum values of the conversion (figure 2) at minimum agitation rate and residence of 180 sec were 66.88 % of  $R_1$ , 69.53 % of  $R_2$ , 69.76 % of  $R_3$ , and at medium agitation, the rate was 56.82 % of  $R_1$ , 59.79 %  $R_2$ , 60.54 % of  $R_3$  at a residence time of 180 sec according to figure 3. Also, from the maximum agitation rate at 40 ml/min flow rate in the three-stage CSTR connected in series, the yield of conversion presented in figure 4 was 66.03 %, 70.37%, 72.43% of  $R_1$ ,  $R_2$ , and  $R_3$  correspondingly obtained at 180 sec of residence time. At 70 ml/min fixed flow rate the actual conversion values were 69.69%, 74.18%, 75.48% at minimum (figure 5), 71.59 %, 77.53 %, 79.59 % at medium (figure 6), 69.23 %, 74.64 %, 76% of  $R_1$ ,  $R_2$  and  $R_3$  at maximum (figure 7) agitation rate. According to the maximum flow rate operated at fixed 100ml/min the reaction rate of the Sodium hydroxide with ethyl acetate gave a conversion of 69.92 %, 77.3 %, 77.84 % at minimum (figure 8), 68.85 %, 76.54 % at 180 sec of residence time, 76.31 % at 90 residence time at medium (figure 9), 67.94 %, 74.56 %, 74.79 % at maximum agitation rate elaborated in figure 10 with the residence time of rate generation 180 sec of  $R_1$ ,  $R_2$ , and  $R_3$  respectively. The conductance value of the reaction mixture (NaOH and  $\text{CH}_3\text{COOC}_2\text{H}_5$ ) decreases with the progress of the reaction inside the three-stage continuous stirring tank reactors connected in series.

**Flowrate(ml/min):** the flow rate evaluated in the three-stage CSTRs connected in series were 40, 70, and 100 ml/min at minimum, medium, and agitation rate of the mixer. The value of conductivity decreases intensely and conversion increases gradually. When minimum, medium and maximum agitation rate operated for 40 ml/l flow rate conversion values were attained 66.34%, 56.74 %, 66.03 % of reactor one( $R_1$ ), 69.38 %, 60.09%, 70.37 % of reactor two( $R_2$ ), 69.76 %, 60.85 %, 72.43 % of reactor three ( $R_3$ ) presented in figure 2, figure 3 and figure 4 individually. Additionally, for medium flowrate (70 ml/min) evaluated at minimum, medium, and maximum agitation rate the maximum conversion values of 69.69%, 71.59%, 68.39% at 27.1 °C of  $R_1$ , 73.95%, and 73.95 % at a temperature of 27.3 °C, 77.53% at a temperature of 27.5 °C, 76% at a temperature of 32.5 °C of  $R_3$  were investigated in figure 5, figure 6 and figure 7 respectively. Further, the conversion values for maximum flow rate(100ml/min) adjusted at mixing rate of minimum, medium and maximum were 69.92% at a temperature of 28 °C, 68.85% at a temperature of 27.7 °C, 67.94% at a temperature of 27.8 °C of  $R_1$ , 77.3% at a temperature of 28.5 °C, 76.54%, 74.56% at a temperature of 28.3 °C of  $R_2$ , 77.84% at a temperature of 30.1 °C, 76.31% at a temperature of 32.1 °C, 74.79% at a temperature of 34.1 °C of  $R_3$  in figure 8, figure 9 and figure 10 generated correspondingly. So according to figure(11), the effect of the flowrates on the reduction of the

concentration of the mixtures during the progress of the reaction inside the three-stage CSTRs connected in series was nearly constant.

**Temperature:** the experimental reaction system for three-stage continuous stirring tank reactors connected in series were operated at non-isothermal conditions. The saponification of ethyl acetate ( $\text{CH}_3\text{COOC}_2\text{H}_5$ ) and sodium hydroxide ( $\text{NaOH}$ ) is exothermic. With a flow rate of 40 ml/min, 70 ml/min, and 100 ml/min, as well as the mixing rate of the agitation, were affected strongly. At minimum agitation rate and 40 ml/min flow rate, the average temperature was 24 °C according to figure 2. On average the value of the reaction temperature was 27 °C justified in figure 5, figure 6, and figure 7 at medium and 70 ml/min flowrate of the reactants. Finally, at maximum agitation rate and maximum flow rate (100 ml/min), the temperature also reached a maximum (34-36.5 °C), because the ions of the mixture inside the reactor were high. Summerly, the temperature of the reaction mixture increases with increasing flowrate and agitation rate of saponification reaction inside the three-stage CSTRs connected in series.

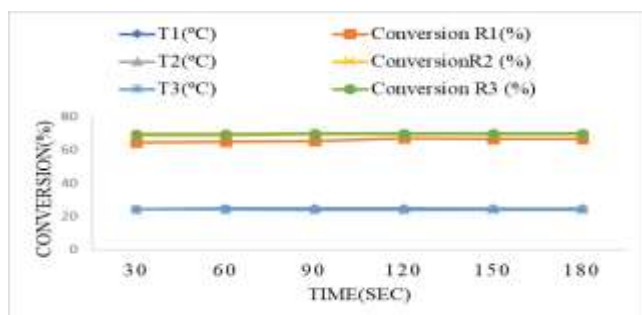


Figure 2. The conversion value of reactors at minimum agitation rate and 40 ml/min flow rate

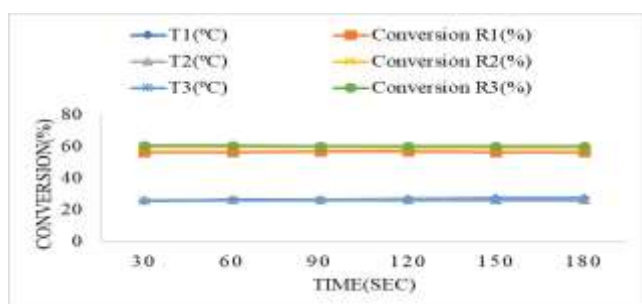


Figure 3. The conversion value of reactors at medium agitation rate and 40 ml/min flow rate

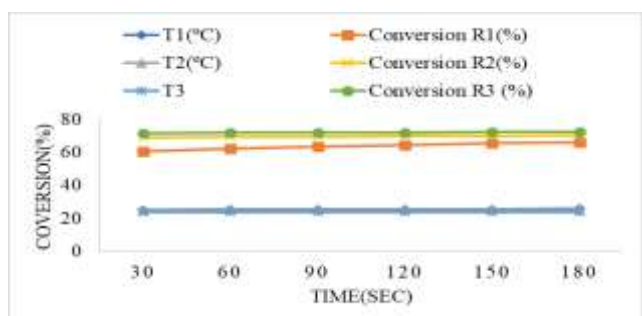


Figure 4. conversion value of reactors at maximum agitation rate and 40 ml/min flowrate

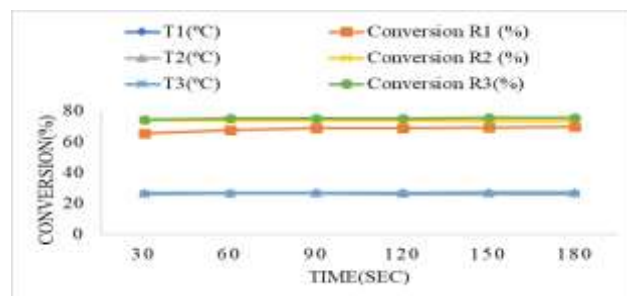


Figure 5. conversion value of reactors at a minimum agitation rate and 70 ml/min flowrate

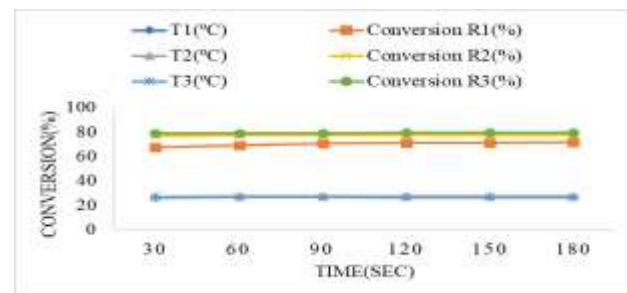


Figure 6. The conversion value of reactors at 70 ml/min flowrate and agitation rate

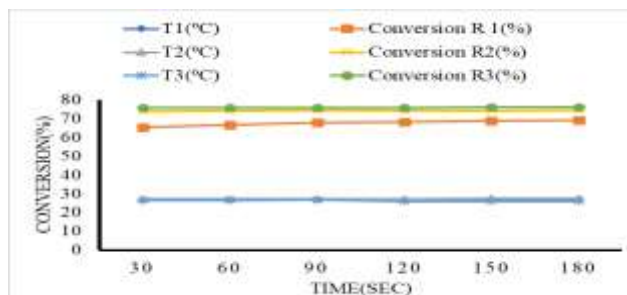


Figure 7. Conversion values of reactors at 70 ml/min flow rate and maximum agitation rate

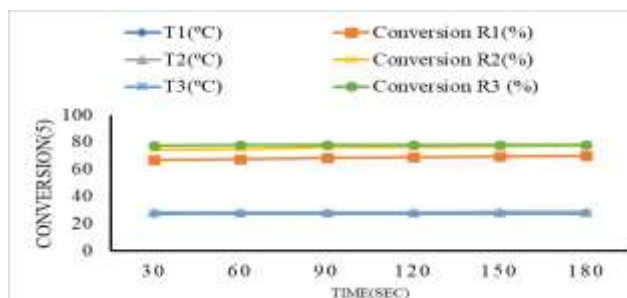


Figure 8. The conversion value of reactors at 100 ml/min flow rate and minimum agitation rate

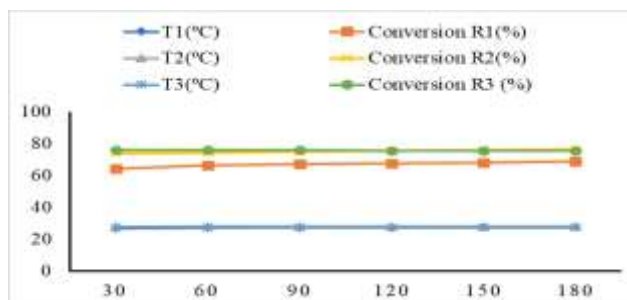


Figure 9. Conversion values of reactors with 100 ml/min flowrate and medium agitation rate



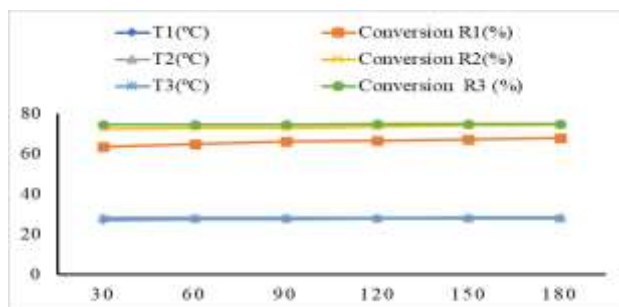


Figure 10. The conversion value of reactors at 100 ml/min flowrate and maximum agitation rate

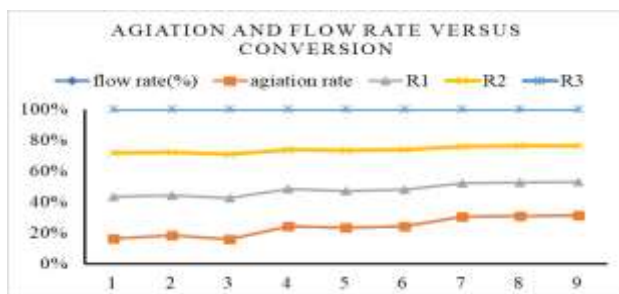


Figure 11. Effect of agitation rate and flow rate on the conversion of reactors connected in series

#### IV. CONCLUSION

The saponification reaction of ethyl acetate was evaluated during this experiment, and process parameters (agitation rate, flow rate, and residence time) at SteadyState condition were examined for a three-stage continuous stirrer tank reactor connected in series. The actual value of conductivity and conversion of the reaction system was noted and justified. The residence reaction of the system increases the real value of conductivity decrease in the three-stage reactor. At maximum agitation rate of the reaction system at flow rate of 40ml/min, the conversion values were 66.03%, 70.37% and 72.43 % of  $R_1$ ,  $R_2$  and  $R_3$ , which is greater than minimum (66.88%, 69.53%, and 69.76%) and medium (57.04%, 60.09%, and 60.85%) agitation rates respectively. Compared saponification reaction operated at 70 ml/min flow rate and medium agitation rate the values of conversion were 71.59 %, 77.53%, 79.59 % for  $R_1$ ,  $R_2$  and  $R_3$ , which is greater than operated at minimum (9.69%, 74.18%, and 75.48%) and maximum (69.23%, 74.64%, and 76%) agitation rate of the three three-CSTR in series. When the SteadyState reaction conditions adjusted at 100 ml/min flow rate, the maximum conversion yields were 69.92%, 77.35%, 77.84 % of  $R_1$ ,  $R_2$ , and  $R_3$  at minimum agitation rate compared to medium (68.85 %, 76.56%, and 75.63%) and maximum agitation rate (67.94 %, 74.56 %, and 74.79 %) separately. In the three-stage reactors connected in series the value of conversion increase when the flow rate increase and the agitation rate decrease dramatically.

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#### REFERENCES

- [1] Levenspiel O. Elements of Chemical Reaction Engineering. Third Edition. s.l. : John Wiley and Sons., pp. 38-39, 1999.
- [2] Fogler, H.S. Elements of Chemical Reaction Engineering. 4th. New Jersey: Pearson Education Inc., 2006.
- [3] Lu W.H., Wu H.Z., and Ju M.Y. Effects of Baffles Design on the Liquid Mixing in an Aerated Stirred Tank with Standard Rushton Impellers. Chem. Eng. Sci., Vol. 52, pp. 3843-3851, 2013.
- [4] K J.M., Reeder M.F. and Fasano J.B. Optimize Mixing by using Proper Baffles. CEP Magazine, AIChE Publication, pp. 42-47, 2002.
- [5] Bursal N., Ertunc S., and Akay B. Process Improvement Approach to Saponification Reaction Using Statistical Experimental Design. Chem. Eng. Process., Vol. 45, pp. 980-989, 2006.
- [6] Kapoor K.L. A Textbook of Physical Chemistry. s.l. : McMillan, ND. India. Vol. 5, 2004.
- [7] Wijayarathne UPL, Wasalathilake KC. Aspen Plus Simulation of Saponification of Ethyl Acetate in the Presence of Sodium Hydroxide in a Plug Flow Reactor. Journal of Chemical Engineering and Process Technology, Vol. 5, pp. 1062-1069, 2014.
- [8] Harsh Shah, Setukumar Vavadiya, Akshay Dholariya, Bhaumik Patel, Kasim Vohra Estimation of Parameters of Kinetic Study and Arrhenius Equation for the Reaction between Ethyl Acetate with Sodium Hydroxide. International Research Journal of Engineering and Technology (IRJET), Vol. 06(03), pp. 2395-0072, 2019.
- [9] Obi Chidi and Okoye Ifeidi Peter. Kinetics and Mechanism of Ethyl Acetate Production Using Eco-Benign Solid Catalyst, DOI: 10.4172/2161-0398.1000219. J Phys Chem Biophys, 2016.
- [10] Grau M.D., Nougues J.M., and Puigjaner L. Comparative Study of Two Chemical Reactions with Different Behavior in Batch and Semibatch Reactors. Chem. Eng. J., Vol. 88, pp. 225-232, 2002.
- [11] Ahmad Mukhtar, Umar Shafiq, Ali Feroz Khan, Hafiz Abdul Qadir, and Masooma Qizilbash. Estimation of Parameters of Arrhenius Equation for Ethyl Acetate Saponification Reaction. Research Journal of Chemical Sciences, Vol. 5(11), pp. 46-50, 2015.
- [12] Kuheli D., Sahoo P., Saibaba M., Murali N., Swaminathan P. Kinetic Studies on Saponification of Ethyl Acetate Using an Innovative Conductivity -Monitoring Instrument with a Pulsating Sensor. Int. J. Chem. Kinet, Vol. 43, pp. 648-656, 2011.
- [13] Daniels F., Matthews J. and Williams J. Experimental Physical Chemistry. New York: McGraw-Hill. pp. 167-169, 1941.
- [14] Mukhtar A, Shafiq U, Qazi MO, Qadir HA. Kinetics of Alkaline Hydrolysis of Ethyl Acetate by Conductometric Measurement Approach Over Temperature Ranges (298.15-343.15K). Austin Chemical Engineering, Vol. 4 (1), 2017.
- [15] Das k, Sahoo P, Sai Baba M, Murali N, Swaminathan P. Kinetic Studies on Saponification of Ethyl Acetate using an Innovative Conductivity-Monitoring Instrument with a Pulsating Sensor. International Journal of Chemical Kinetics, pp. 1-9, 2011.
- [16] Hiroo Tsujikawa, Hakuai Inoue. The Reaction Rates of Alkaline Hydrolysis of Ethyl Acetate. Bulletin of Chemical Society of Japan, Vol. 39, pp. 1837-1842, 1966.