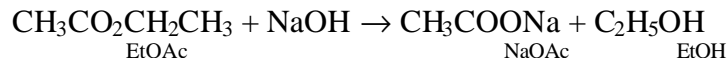


# CBE CHEMICAL CORPORATION

**To:** CE428 Team  
**From:** Dr. Ima Manager  
**Date:** January 20, 2006  
**Subject:** **Kinetics of Saponification Reaction**

Our process engineering division has begun work to develop a new process to produce SuperChem®. Unfortunately, this new process has an undesirable side reaction, the saponification of ethyl acetate. Your assignment is to do a preliminary study of the kinetics of this reaction by estimating the reaction rate constant and the temperature dependence of this rate constant. I also request that you compare your results with literature results (see Keusch Reference).

The side reaction in question is the reaction of ethyl acetate (EtOAc) and sodium hydroxide (NaOH) to produce sodium acetate (NaOAc) and ethanol (EtOH).



It is known to be an irreversible second-order reaction with a reaction rate expression of

$$-r_{\text{NaOH}} = kC_{\text{EtOAc}}C_{\text{NaOH}}$$

I would like your team to use two of the lab-scale reactor set-ups we already have in the fume hood of 116 Jarvis. One is a constant volume batch reactor (CVBR) and the other is a continuous stirred reactor (CSTR). Obviously, you should get equivalent information from both reactors but I would like you to make some suggestions about the suitability of each set-up for collecting additional data.

Our technical staff has suggested the use of 0.1 N NaOH and 0.1 M EtOAc in your preliminary studies. They also suggest quenching your reactor samples in 5 ml of 0.1N HCl solution and then titrating the quenched samples using 0.1 N NaOH and a phenolphthalein indicator to monitor the concentration of NaOH in the reaction. I would like your team to suggest an alternate analytical method for future studies.

Additional suggestions:

## For CVBR experiment

1. The reactor should be charged with 100ml of 0.1N NaOH and 100ml of 0.1M EtOAc. If running the reaction at a temperature below or above room temperature, pre-cool or pre-heat the reactants in separate containers by placing the containers, **carefully**, in the reservoir of the circulating bath.

2. Run the reaction at three temperatures between 10 and 30 °C.
3. Use mild agitation of 100-125 rpm in the reactor.
4. Prepare a series of reaction quench containers by adding 5 ml of 0.1N HCl to either the 50 or 125 cc Erlenmeyer flasks.
5. Take 10 ml samples of the reaction mixture at the following times after mixing: 2, 5, 10, 15, and 20 minutes.
6. Titrate the samples using 0.1N NaOH to estimate the unreacted NaOH and EtOAc in the reactor.

For the CSTR experiment (Perform at ONE temperature only)

1. The reactor should be filled to 140 ml with 0.1 N NaOH and 0.1 M EtOAc. Assume 50% by volume for each.
2. Set the temperature of the circulating water bath to reproduce the temperature of one of the batch reactor runs.
3. Use mild agitation of 100-125 rpm (Estimate rotation by comparing to stirrer for batch reactor)
4. Use a feed rate of 10 cc/min for each reactant.
5. Adjust outlet flow rate to maintain constant volume in reactor.
6. Prepare reaction quench containers as described above.
7. Collect periodic samples of reactor effluent beginning after 7 minutes of continuous operation. Analyze using titration procedure described above. Run until steady state is attained.

For Both Experiments

**Transfer the reaction products to the 5-gallon carboy provided for the experiment. Do not dispose of this material in the sink or drain!**

**Cleanup is essential, including all of the titration glassware!**

I will expect your memo two weeks following your team's scheduled laboratory session.

Attachments: Titration Procedure  
List of References

#### Appendix 1. Titration Procedure

Measure 5 ml of 0.1 N HCl into 125 ml flasks or beakers. This is the solution that will quench the reaction for titration.

Take a 10 ml effluent sample.

Titrate the sample with 0.1 N NaOH and a phenolphthalein indicator until the solution turns pink. Name the volume of titrant X.

$$\text{mol/l NaOH} = \frac{(5\text{ml HCl} \times 0.1\text{M HCl}) - (X \text{ ml titrant} \times 0.1\text{M NaOH})}{10\text{ml sample}}$$

#### Reference:

Levenspiel, O., *Chemical Reaction Engineering*, John Wiley & Sons, New York 1962.

Fogler, H. S., *Elements of Chemical Reaction Engineering*, 3<sup>rd</sup> Edition, Prentice Hall, Upper Saddle River, New Jersey 1999.

Keusch, P., 2003, "Kinetics- Alkaline hydrolysis of Ethyl Acetate-Second Order Reaction", Internet posting at [http://www.uni-regensburg.de/Fakultaeten/nat\\_Fak\\_IV/Organische\\_Chemie/Dida ktik/Keusch/chembox\\_etac-e.htm](http://www.uni-regensburg.de/Fakultaeten/nat_Fak_IV/Organische_Chemie/Dida ktik/Keusch/chembox_etac-e.htm) , University of Regensburg.