

LEQ III

2020/2021

Reactors Module

EXPERIMENT 1 – R1A and R1B (2 laboratory classes):

R1A (1 class) – Experimental determination of the Residence Times Distribution (RTD) for a tubular reactor, testing different volumetric flow rates, and using the technique of an impulse tracer injection in the reactor feed. Use of the obtained RTD for reactor pattern flow diagnosis. Modelling of the real reactor.

R1B (1 class) – Study of the real reactor performance carrying out the reaction of peroxodisulphate with iodide, at steady state. Comparison of the obtained real conversion with the predicted one for the ideal plug flow case, trying to correlate differences in the reactor performance to the deviations detected in the patten flow (experiment R1A).

EXPERIMENT 2 – R2A and R2B (2 laboratory classes):

R2A (1 class) – Experimental determination of the Residence Times Distribution (RTD) for a continuous stirred tank reactor, testing different agitation conditions, and using the technique of a negative step tracer injection in the reactor feed. Use of the obtained RTD for reactor pattern flow diagnosis.

R2B (1 class) – Study of the real reactor performance carrying out the reaction of ethyl acetate with sodium hydroxide, at steady state. Analysis of this performance dependence on the agitation conditions. Comparison of the obtained real conversion with the predicted one for the ideal perfectly mixed case, trying to correlate differences in the reactor performance to the deviations detected in the patten flow (experiment R2A).

R1A

Objectives

Experimental determination of the Residence Times Distribution (RTD) for a tubular reactor, testing different volumetric flow rates, and using the technique of an impulse tracer injection in the reactor feed. Use of the obtained RTD for reactor pattern flow diagnosis. Modelling of the real reactor.



Experimental procedure R1A :

- 1) Stabilize the pattern flow of distilled water across the tubular reactor and measure the corresponding volumetric flow rate, at the exit of the reactor.
- 2) Calibrate the spectrophotometer at 650 nm: i) adjust 0 % of transmittance with no radiation beam reaching the detector; ii) adjust the 100 % transmittance (zero absorbance) with water passing continuously across the flux cell.
- 3) The tracer concentration at the exit of the reactor will be followed by spectrophotometry, measuring continuously the absorbance of the solution effluent of the reactor as a function of time after the injection of the tracer.
- 4) Inject 0.4 mL of tracer (concentrated solution of methylene blue) in the reactor feed, and initiate time counting at that moment.
- 5) The evolution of the absorbance of the reactor effluent, as a function of time, will be registered in the data acquisition system, until the absorbance reaches approximately zero.
- 6) Repeat the preceding procedure from 1) to 5) for different volumetric flow rates.

RESULTS

» Measures of the volumetric flow rate

1st experience: Q (mL/min) =

2nd experience: Q (mL/min) =

3rd experience: Q (mL/min) =

» The recorded file in the data acquisition system, for each experiment, can be read with Excel.

TOPICS FOR TREATMENT AND DISCUSSION OF THE RESULTS (R1A):

» Calculate the experimental values of the RTD function, E(t), for each reactor working condition:

$$E(t) = \frac{C(t)}{\int_0^{\infty} C(t) dt} \quad \text{or} \quad E(t) = \frac{A(t)}{\int_0^{\infty} A(t) dt}$$

If necessary, carry out a base line correction, and then calculate $\int_0^{\infty} A(t) dt$.

» Compare the experimental RTD with that of the ideal Plug Flow Reactor.

» Calculate the real mean residence time, for each experience: $\bar{t} = \int_0^{\infty} tE(t) dt = \frac{V_{\text{effective}}}{Q_{\text{effective}}}$

and estimate the effective volume of the reactor, considering the practical impossibility of the existence of any by-pass in the studied reactor (so, $Q_{\text{effective}} = Q_{\text{measured}}$).

» Discuss the effect of volumetric flow changing in the pattern flow analysis, taking the Plug Flow Reactor as the ideal reference.

» Suggest and test a real reactor model, composed by an association of ideal reactors whose RTD is close to the experimentally obtained RTD for the studied tubular reactor.

Data: Volume of tubular reactor = 0,470 L

References

F. Lemos, José Madeira Lopes, Fernando Ramôa Ribeiro, "Reatores Químicos", IST Press, Lisboa, 2014.

Froment, Bischoff, "Chemical Reator Analysis and Design", Wiley, Nova York.

O. Levenspiel, "Chemical Reaction Engineering", Wiley, Nova York.

H. Scott Fogler, "Elements of Chemical Reaction Engineering", Prentice-Hall.

TREATMENT AND DISCUSSION OF THE RESULTS (R1B):

- » After each sampling, calculate the concentration of iodine in the reactor effluent.
- » Calculate the experimental conversion at steady state from the previous results.
- » Calculate the expected conversion with the model of ideal Plug flow reactor, for the same volumetric flow rate and volume of the real reactor.
- » Compare the previously calculated conversions (experimental and ideal one), relating eventual differences with the detected deviations in the pattern flow (R1A).

Data: kinetic rate equation $r = k [S_2O_8^{2-}] [I^-]$ where $k(M^{-1}min^{-1}) = 1,38 \times 10^7 \exp(-E_A/RT)$,

$E_A = 10300 \text{ cal/mol}$; Vol. Reactor = 0,505 L.

References

- F. Lemos, José Madeira Lopes, Fernando Ramôa Ribeiro, "Reatores Químicos", IST Press, Lisboa, 2014.
- Froment, Bischoff, "Chemical Reator Analysis and Design", Wiley, Nova York.
- O. Levenspiel, "Chemical Reaction Engineering", Wiley, Nova York.
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R2A

Objectives

Experimental determination of the Residence Times Distribution (RTD) for a continuous stirred tank reactor, testing different agitation conditions, and using the technique of a negative step tracer injection in the reactor's feed. Use of the obtained RTD for reactor pattern flow diagnosis.



Experimental procedure R2A:

Prepare 5L of an aqueous solution of NaOH 0.1 M, to be used as a tracer.

- 1) Fill the feed vessels with distilled water.
- 2) Fill the reactor with the prepared NaOH 0.1 M solution.
- 3) Define the file to be used in the acquisition data system for each experiment.
- 4) Keep both volumetric water flow rates controllers in position 10 and initiate the reactor feeding, with the agitator in its maximum rotation, beginning the time counting (and the registration of data in the file) after 5 s.
- 5) Follow the evolution of the tracer concentration, NaOH (measured by electric conductivity), as a function of time, until electric conductivity reaches values close to zero. During this evolution, measure both volumetric flow rates.
- 6) Repeat the procedure 1) to 5) with different agitation conditions.

RESULTS:

- » Feeding volumetric flow rate: experience 1 –
experience 2 –
experience 3 –

» The recorded data file for each experience contain the evolution of the effluent conductivity $K(t)$, proportional to the tracer (NaOH) concentration, as a function of time from the beginning of the negative step ($t=0$) until the absence of tracer at the exit of the reactor, and can be read with Excel.

TREATMENT AND DISCUSSION OF THE RESULTS (R2A):

» Calculate the experimental values of the $H(t)$ function for each experiment of tracer negative step input: $H(t) = K(t)/K_0$

» In order to obtain the experimental values of the RTD, calculate $E(t) = -dH(t)/dt$ for each experiment (use numerical procedure first). Probably, it will be necessary to fit an equation to the $H(t)$ experimental values, and then make the derivative of this equation, so that the experimental points errors are not enormously amplified.

» For each case (each RTD), make a diagnosis of the working condition concerning the pattern flow inside the reactor:

- if there is a by-pass, it will be expressed in the experimental $E(t)$.
being $H(0^+) = Q_b/Q$ and Q_b (caudal de "by-pass") = $Q - Q_e$

- calculate the effective mean residence time $\bar{t} = \int_0^{\infty} tE(t)dt$

- calculate the effective volume of the reactor, in each case, being $\bar{t} = \frac{V_{efetivo}}{Q}$, where Q is the

total volumetric flow rate fed to the reactor, and compare with the expected reactor volume (measured): V (fluid inside reactor) = 1360 mL.

- Compare the real RTD with the ideal $E(t)$ typical of a continuous perfectly mixed reactor:

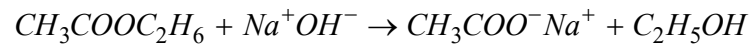
$$E(t) = \frac{1}{\theta} e^{-\frac{t}{\theta}} \quad \text{where} \quad \theta = \frac{V_{medido}}{Q_{total\ medido}}$$

References

- F. Lemos, José Madeira Lopes, Fernando Ramôa Ribeiro, "Reatores Químicos", IST Press, Lisboa, 2014.
- Froment, Bischoff, "Chemical Reator Analysis and Design", Wiley, Nova York.
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R2B

Study of the real reactor performance carrying out the reaction of ethyl acetate with sodium hydroxide. Analysis of this performance dependence, at steady state, on the agitation conditions. Comparison of the obtained real conversion with the predicted one for the ideal perfectly mixed case.



Experimental Procedure (R2B):

- 1) Prepare 2x 5 L of an aqueous NaOH solution 0.1 M and measure the corresponding conductivity.
- 2) Prepare 2x 5 L of an aqueous EAc solution 0.15 M.
- 3) Define the file for data acquisition, where the values of conductivity and temperature will be collected.
- 4) Fill each feeding vessel with the corresponding reactant solution: NaOH – pump 1 and EAc – pump 2.
- 5) Regulate both volumetric flow rates at the position 10. Regulate the agitator for its maximum rotation.
- 6) Follow the evolution of the reaction mixture conductivity at the reactor exit until constant values are obtained. Meanwhile measure both flow rates during the transient evolution.
- 7) Repeat the procedure from 4) with a different agitation condition. Follow the evolution of the conductivity at the exit of the reactor until a new steady state is reached, measuring again both volumetric flow rates.
- 8) After reaching steady state at 7), turn off the feeding pumps, keeping the reactor (that became a batch reactor) under strong agitation and measure the conductivity of the mixture (K^∞) reached when all the reference reactant was consumed.

RESULTS :

» Volumetric flow rates, conductivity and temperature at steady-state:

experience 1 - $Q_{NaOH} =$ $Q_{EAc} =$ $K =$ $T =$
experience 2 - $Q_{NaOH} =$ $Q_{EAc} =$ $K =$ $T =$

» Conductivity of the feeding NaOH solution: $K_{NaOH} =$

» Conductivity of the reaction mixture after complete conversion of the limiting reactant: $K^\infty =$

TREATMENT AND DISCUSSION OF THE RESULTS (R2B):

» The conductivity values are related to the reactant NaOH concentration, at steady-state, by:

$$C_{NaOH} = C_{NaOH\ entry} \frac{K - K^\infty}{K^0 - K^\infty}, \text{ so that the steady-state conversion will be } \mathcal{X} = \frac{K^0 - K}{K^0 - K^\infty},$$

where K is the measured conductivity of the reaction mixture at the steady-state,

K^0 is the conductivity of the mixture for zero conversion, which is given by:

$$K^0 = K_{\text{solutionNaOH}} \frac{Q_{NaOH}}{Q_{NaOH} + Q_{EAc}}$$

K^∞ is the conductivity of the reaction mixture for total conversion of NaOH (final measure of step 8) of the procedure).

» Calculate the experimental steady state conversion obtained for each working condition.

» Calculate the predicted conversion in an ideal perfectly mixed reactor.

» Compare both conversions (real and ideal) for each working condition, relating eventual differences to changes in the pattern flow detected in the analysis of the corresponding RTD in R2A.

Data: kinetic law $r = k [EAc] [NaOH]$ where $k(T) \text{ (m}^3 \text{ mol}^{-1} \text{ s}^{-1}) = 7.2 \times 10^4 \exp(-49724 / 8.314 T)$

References

F. Lemos, José Madeira Lopes, F. Ramôa Ribeiro, "Reatores Químicos", IST Press, Lisboa, 2014.

Froment, Bischoff, "Chemical Reactor Analysis and Design", Wiley, Nova York.

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