

Sucrose Inversion: An Experiment on Heterogeneous Catalysis*

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Illustration of heterogeneous catalysis concepts in laboratory courses is not usually simple or economical. For our undergraduate senior lab course we have developed an environmentally friendly experiment dealing with several aspects of heterogeneous catalysis, having in mind the use of readily available and relatively inexpensive equipment and chemicals on a compact setup, which students can safely operate. The experiment deals with the acid-catalyzed sucrose inversion, performed in packed bed chemical reactors, where the catalyst is a cation-exchange resin in the H⁺ form. An additional reactor is included for illustrating an enzyme-catalyzed system. The conversion achieved is determined using the Flow Injection Analysis technique.

NOMENCLATURE

C_{sucr}	sucrose concentration, within the catalyst pellet (mol/m ³)
C_{sucr}^s	sucrose concentration at the catalyst surface conditions (mol/m ³)
D_e	effective diffusivity (m ² /s)
d_p	particle diameter (m)
$E(t)$	residence time distribution function
f	dimensionless sucrose concentration profile
F	feed flow rate (m ³ /s)
k	intrinsic rate constant (s ⁻¹)
k^{obs}	observed rate constant (s ⁻¹)
L	bed length (m)
Pe	Peclet number
r	radial distance (m)
r_0	particle radius (m)
t	time (s)
u	superficial velocity (m/s)
V	reactor volume (m ³)
X_{sucr}	sucrose conversion
z	dimensionless radius direction

Greek Symbols

ε	bed porosity
ϕ	Thiele modulus
η	internal effectiveness factor
μ	fluid viscosity (N s/m ²)
ρ	fluid density (kg/m ³)
τ	space-time (s)

INTRODUCTION

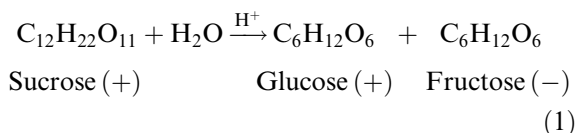
HETEROGENEOUS CATALYSIS is a topic of great industrial importance in Chemical Engineering and consequently plays a significant role in theoretical undergraduate courses. In fact,

heterogeneous catalysis is present in most of chemical reaction engineering (CRE) handbooks, e.g. [1–4], and is an essential part of the majority of the undergraduate CRE courses [5–9]. This paper describes an experimental set-up on heterogeneous catalysis, running at a chemical engineering laboratory discipline, taught during the 1st semester, 4th curricular year of the 5 years long Chemical Engineering course at the University of Porto, Portugal.

All the experiments now available at this laboratory were designed having in mind: high safety, low investment and operation costs, reduced environmental impact and high didactic content.

The sucrose inversion (hydrolysis) is acid-catalyzed and can be conducted in a fixed bed, packed with a cation-exchange resin in the protonic form. This is the kind of experiment that fits perfectly the above mentioned principles and succeeded to capture the attention of an international company dedicated to the manufacture and commercialization of this kind of equipment [10].

The resin's three-dimensional network forms a macroporous structure in which the ionically linked H⁺ cations are the acid sites. Sucrose undergoes acid hydrolysis into glucose and fructose (stereo-isomers) according to a pseudo-first order reaction [11, 12]:



The optical rotation of sucrose is positive ($[\alpha]_D^{20} = +66.5^\circ$) and its hydrolysis with an acid yields a 1:1 ratio of D-(+) glucose and D-(–) fructose, with positive and negative optical rotations, respectively. Since fructose has a greater optical rotation than glucose (-92.4° versus 52.7°),

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the optical rotation of the reaction mixture will change sign from positive to negative as the reaction proceeds to completion. This is why it is usually called sucrose inversion reaction.

Sucrose inversion on cationic exchange resins was studied by Reed and Dranoff [11], using Amberlite IR 120, and by Gilliland *et al.* [12], using Dowex 50W-X8. These researchers found that the reaction performance could, under certain circumstances, be controlled by the diffusion transport of sucrose along the resin's pores, rather than by the kinetics of the hydrolysis reaction itself. Moreover, no change in the overall reaction rate was noted as the particle Reynolds numbers ($\rho u d_p / \mu$) varied from 0.14 to 4.8, indicating external (film) mass transfer was unimportant [11].

An experimental set-up was built to conduct the sucrose inversion under diffusion-controlled transport conditions and then it can be used to illustrate important concepts such as the 'effectiveness factor' and the Thiele modulus, apart from other issues such as flow pattern in a packed bed reactor. The concepts addressed also include determination of kinetic parameters, reactor design, comparison of ideal vs. real reactors and residence time distribution, which are fundamental in the modern CRE curricula [5, 6, 8, 13]. Emphasis is also given to computer-assisted experimentation (data acquisition) and to the use of computer applications for problem solving, therefore meeting the current trends in laboratory instruction [8].

To make this experiment even more interesting, another packed bed reactor was added, containing a bio-catalyst, and its performance is compared with the ion exchange based catalyst. Invertase immobilized into small beads of calcium alginate can be used as the biocatalyst. In this case, the reaction kinetics follows the Michaelis-Menten equation.

THEORETICAL BACKGROUND

A heterogeneous catalyzed reaction is controlled by the internal diffusion transport when the reactants react faster (or the products are formed faster) than they diffuse inward (or outward), through the pores in a catalyst pellet. This diffusional resistance causes the average distribution of reactant/product within the resin particle to become non-uniform (e.g., lower concentration of reactant at the center), thus originating a decreased average reaction rate. A quantitative description of this effect can be given by an 'internal effectiveness factor', η defined as the ratio between the actual reaction rate (i.e., with pore diffusion resistance) and the reaction rate under particle surface conditions (i.e., without diffusion resistance). This quantity is a function of the Thiele modulus, ϕ , which for a first-order reaction is given as:

$$\phi = r_0 \left(\frac{k}{D_e} \right)^{1/2} \quad (2)$$

where r_0 is the particle radius, k is the reaction rate constant and D_e is the effective pore diffusion coefficient. The relationship between η and ϕ is, for spherical particle geometry and isothermal conditions [2]:

$$\eta = \frac{3}{\phi} \left(\coth(\phi) - \frac{1}{\phi} \right) \quad (3)$$

The Thiele modulus evidently compares the rates for chemical reaction and diffusion. For low values of ϕ the system is in the kinetic regime and $\eta \rightarrow 1$. On the other hand, diffusion regime is attained for sufficiently large ϕ , and $\eta \rightarrow 3/\phi$.

A straightforward way to evaluate the importance of diffusion resistances to mass transfer in a heterogeneous reaction system consists in performing kinetic measurements using two different particle sizes, say r_{01} and r_{02} . If the attained catalytic performances differ, diffusion limitations may be present. The following expression, for the ratio of the two observed kinetic constants, can be obtained from Equation (3) and from the definition of the effectiveness factor (since $k^{obs} = \eta \times k$):

$$\frac{k_1^{obs}}{k_2^{obs}} = \frac{\eta_1}{\eta_2} = \frac{r_{02}}{r_{01}} \frac{\coth \phi_1 - \phi_1^{-1}}{\coth \left(\phi_1 \frac{r_{02}}{r_{01}} \right) - \left(\phi_1 \frac{r_{02}}{r_{01}} \right)^{-1}} \quad (4)$$

where ϕ_2 was replaced by a function of ϕ_1 :

$$\phi_2 = \phi_1 \frac{r_{02}}{r_{01}} \quad (5)$$

by assuming a constant effective diffusion coefficient. Equation (4) can be solved numerically to compute ϕ_1 from the measured apparent kinetic constants. The other variables (ϕ_2, η_1, η_2) are then obtained from Equations (5) and (3).

In order to compute k_1^{obs} and k_2^{obs} , one must be able to relate the reactor's outlet conversion to the reaction's kinetic parameter. This obviously depends on the particular flow pattern in the reactor and on the reaction kinetics. For ion exchange based packed bed reactors, the simplest approach is to describe it as an ideal plug flow reactor, and therefore the conversion is given by [1]:

$$X_{sucr} = 1 - e^{-k^{obs} \tau} \quad (6)$$

where τ is the space-time, based on the reactor volume ($\tau = V/F$). It is assumed that the active sites are homogeneously distributed in the packed bed.

When axial dispersion cannot be neglected, a more complete model should be used for prediction of the reactor conversion. One approach is to use the total segregation model, which assumes that elements of different ages do not mix, thus remaining segregated, until they exit the reactor. In such circumstances, the mean conversion is given by [1]:

$$\bar{X}_{sucr} = \int_0^{\infty} X_{batch} E(t) dt \quad (7)$$

where the first factor in the integral is obtained from the batch reaction kinetics and $E(t)$ is the residence time distribution function. For first-order kinetics and for a semi-infinite axially-dispersed plug flow reactor, Equation (7) yields [14]:

$$X_{sucr} = \int_0^{\infty} (1 - e^{-k^{obs}t}) \frac{\sqrt{\tau Pe}}{2\sqrt{\pi t^3}} e^{-(Pe(\tau-t)^2/4\tau t)} dt \quad (8)$$

where Pe is the Peclet number, which can be estimated from any available correlation. After evaluation of the experimental sucrose conversion, a numerical iterative procedure must be used to compute k^{obs} .

As explained, after computation of k^{obs} , students can determine the Thiele modulus and the effectiveness factors for both catalyst pellets. This allows them to evaluate the importance of diffusion limitations and to identify which regime, between internal diffusion controlled and reaction rate determined, is predominant for each particle size.

It is also very interesting to ask students to determine the sucrose concentration profiles within the pellets. For first-order reaction and spherical geometry, resolution of the steady-state

diffusion equation (i.e., steady-state mole balance) provides the following dimensionless concentration profile [1]:

$$f = \frac{C_{sucr}}{C_{sucr}^s} = \frac{1}{z} \frac{\sinh(\phi z)}{\sinh \phi} \quad (9)$$

where, C_{sucr}^s is the sucrose concentration at the surface conditions and $z = r/r_0$ is the dimensionless direction, oriented from the center to the surface.

EXPERIMENTAL SET-UP

Packed bed catalytic reactors

The experimental set-up developed can be seen in Fig. 1. It consists of three reactors, named for easy as A, B and C. The reactors consist in jacketed glass columns (from Omnifit), 0.025 m in internal diameter and 0.25 m in length, equipped with one adjustable stopper, what allows for the use of different amounts of catalyst. Reactors A and B, the chemical catalytic reactors, are packed with Amberlite IR 120 resin of two different diameters, while reactor C, the biological reactor, is packed with invertase immobilized on calcium alginate beads. The two Amberlite IR 120 particle diameters (8.8×10^{-4} m and 3.1×10^{-4} m) were

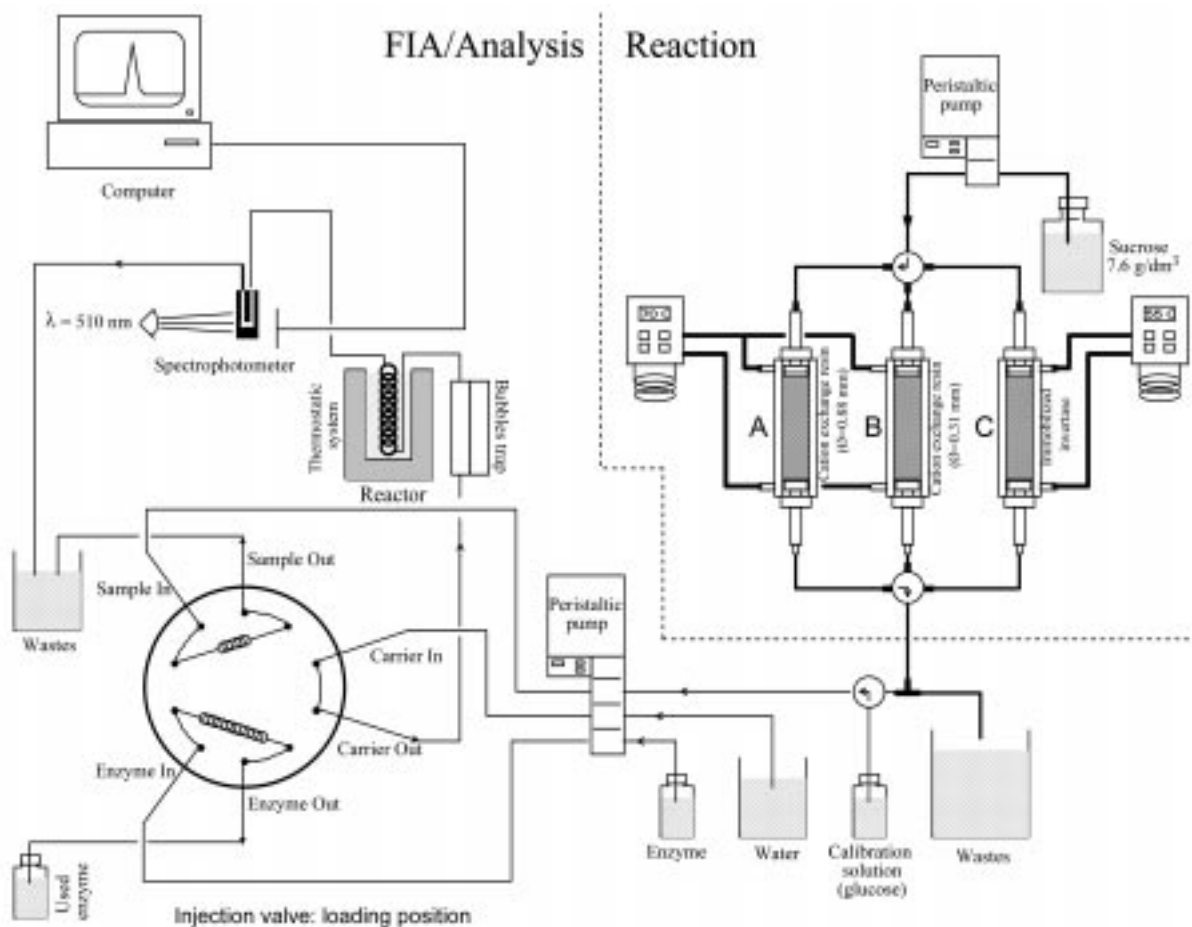


Fig. 1. Experimental set-up sketch.

obtained by sieving the original sample. The resin's capacity (that measures the active sites concentration) is about 1.9 meq/cm^3 of resin bed [15] and its apparent density is $1.27 \times 10^3 \text{ kg/m}^3$. The calcium alginate beads were made dropping, drop by drop and with the help of a peristaltic pump, a sodium alginate aqueous solution (1% w/w) containing invertase (β -fructosidase, 270,000 amu from Boehringer Mannheim, 0.1% w/w) into a calcium chloride solution (0.2 M). The exchange of sodium by calcium led to a macroporous networked alginate bead, where the invertase is trapped.

The three reactors are kept at a constant temperature with the help of a recirculating thermostatic bath. The reactant solution (aqueous sucrose) is fed to the reactors via a four-cylinder peristaltic pump (from Watson-Marlow). In Fig. 1 one can see how the use of two four-way valves allows for directing the flow through each reactor. A small air space is allowed at the column's top to account for catalyst and solution expansion upon heating while the small amount of solution above the catalyst is allowed to stop the entrance of bubbles. The reactors' conversion is analyzed using a FIA (Flow Injection Analysis) system, described below.

After each experiment, water must be flown through each reactor for some minutes, in order to remove the remaining sucrose and avoid accumulation of bacterium. The same applies to all the tubing that contacts the sucrose solution.

We have observed that resin regeneration (with a concentrated HCl solution) is not necessary during one semester.

Flow injection analysis

FIA is a recent analytical method that can be efficiently used, in this case, to evaluate the conversion of sucrose into glucose and fructose. In general terms, the method involves the reaction of a specific reactant with the sample component that one wishes to quantify, originating a product that can be detected by spectrophotometry, conductometry or other methods. A remarkable advantage of this system consists in the fact that it can be used for on-line automated measurements, requiring small amounts of sample and specific reactant. We will give here some attention to our particular FIA implementation, since we believe that its relative low cost and simplicity make it quite suitable for student laboratories.

Glucose is the component to be analyzed by the FIA system. It will thus be reacted with a buffered solution of two enzymes, glucose-oxidase and peroxidase (Peridochrom Glucose GOD-PAP from Boehringer Mannheim), giving a colored compound that has a maximum absorbency at 510 nm.

Typically, a FIA setup is composed of four sections [16–18]:

1. *Pumping system.* One peristaltic pump is used for continuously feeding three components to

the FIA system: the sample (solution to be analyzed), the specific reactant (enzyme solution) and the carrier (water in our case).

2. *Injection valve.* A two position, 10-way valve allows for predetermined amounts of sample and specific reactant to be mixed into the carrier stream.
3. *Reaction system.* That's where the reaction between the sample and the specific reactant occurs, after injection. It is actually just a coiled tube, going from the valve into the detector. It should be maintained at constant temperature during an experiment (note the thermostatic system in Fig. 1).
4. *Detection system.* After reaction, the colored product is detected by a spectrophotometer. Other detection systems can be used in FIA (conductivity, refraction index, etc.), depending on the nature of the reaction product.

In the 10-way injection valve used, two pairs of ports have been looped together. In the load position (see Fig. 1), the sample and specific reactant streams go through the respective loops, while the carrier stream enters and leaves the valve without mixing. In the injection position, the carrier goes through the two loops, carrying the reactant and the sample to be analyzed. The internal diameter of the tubing in the FIA system is 0.8 mm and made of Teflon. Mixing between the sample and the reactant occurs due to axial and radial dispersion in the coiled tube between the valve and the detector. We work with a 1-m long tube and a residence time of about three minutes. Also, the reactant loop is about four times larger than the sample loop (respectively 16 and 4 cm long).

Since FIA works with quite low flow rates, an appropriate peristaltic pump is necessary. We've used one with 12 rolls (Ismatec), operating typically at 10 rpm. The injection valve (Valco) was operated manually, even though an automatic, computer driven, system could be easily implemented. The spectrophotometer (Jenway 6300) was equipped with an $18 \mu\text{L}$ flow-through cell and set to measure absorbance at 510 nm. A computer was connected to the detector via the RS-232 port, allowing for on-line graphical visualization of the output signal (using the LabView software, by National Instruments).

The enzyme solution is a bit expensive (a box with ten 100 ml flasks costs about €145, without VAT). However, it can be reused (on average a volume of 1 to 2 flasks is used 6 times per week), as long as it is kept refrigerated overnight.

The analytical method has to be calibrated prior to the conversion measurements. To do this, one uses a set of glucose solutions of known concentration. We've used a two-way valve in order to direct the sample flow from the reactors' outlet stream or from the reference solution vessel (see Fig. 1). A calibration curve (glucose concentration versus maximum intensity of the detector signal) is then obtained.

Table 1. Operating conditions and physical data for each reactor

	Reactor		
	A	B	C
Particle diameter (m)	8.8×10^{-4}	3.1×10^{-4}	3×10^{-3}
Temperature (°C)	70	70	55
Bed length (m)	0.212	0.208	0.210
Internal diameter (m)	0.025	0.025	0.025
Bed porosity	0.52	0.59	–
Feed flow rate (m ³ /s)	2.08×10^{-7}	1.94×10^{-7}	1.80×10^{-7}

The outgassing of streams in a FIA system interferes with the spectrophotometer measurements and can cause important delays for removing the bubbles. To overcome this, a simple homemade Teflon membrane module can be used to remove most of the small bubbles which form.

DATA TREATMENT AND DISCUSSION

Table 1 shows the operating conditions used to obtain the results presented below, with a concentration of sucrose in the feed of 7.6 kg/m^3 . Enough acetic acid was added to the sucrose solution fed to the bioreactor so that the pH was 4.6, for which the invertase activity is maximum. A buffered solution of acetic acid and sodium acetate cannot be used for obtaining this pH value because the sodium exchanges with the calcium ion present in the alginate beads, destroying the network. Besides, the calcium ion interferes with the method of analysis.

For calibration of the FIA method, four reference glucose solutions were used, with known concentrations of about 1, 2, 3 and 4 kg/m^3 . Notice that if sucrose was completely converted, the glucose concentration in the outlet stream would be 4.0 kg/m^3 . The carrier used was distilled water.

In order to make a regression of the calibration curve, it should be drawn in such a way that the more precise figures are set in the *xx* co-ordinate [19]. In the present case it should be the reference glucose solutions concentrations, where a second-order polynomial fits quite well (Fig. 2). The maximum of the absorbance peaks, for each reference glucose solution, was measured several times and the data shown in Fig. 2 represent average values (which errors are about 3%, obtained from the *t* distribution at a 95% confidence level).

Students are asked to start the calibration procedure with the more concentrated reference sample and to adjust the flow rate in the pump in order to maximize the peak absorbance value without saturating the signal. This gives them some perception to the effect of the flow rate on the analysis and provides the necessary correction due to eventual decay on the enzyme’s activity over time. The criterion for accepting a measurement is to obtain two consecutive readings within 1% agreement. While performing the calibration, students start up the thermostatic bath and, as soon as the intended temperature is reached, turn on the feed pump so that the sucrose solution starts flowing through reactor A. This avoids having to wait for reactor A to reach steady-state conditions, after the calibration is finished. Steady state should be attained after a time of about 1.5τ .

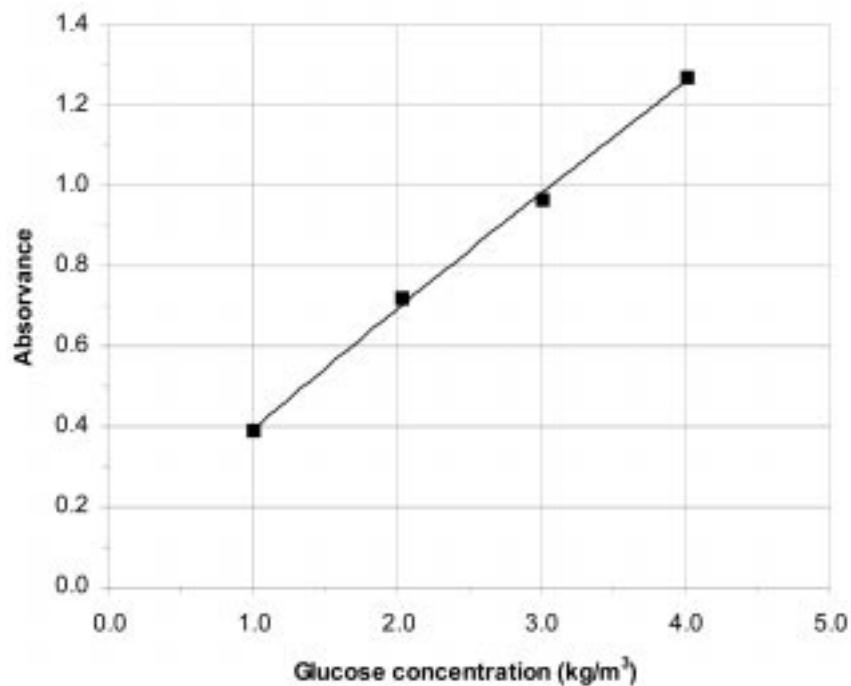


Fig. 2. Calibration data and fitting curve.

At least two FIA measurements (with a five-minute interval) of the reactor's outlet stream are then performed to verify that the concentration is constant. Afterwards, the feed stream is directed to reactors B and C and the same procedure repeated.

While waiting for the steady state to be attained, students measure the flow rate leaving the reactor. The full experiment can be performed in less than three hours. One aspect that should be taken into consideration is the accumulation of air bubbles within the bed due to the high temperature in the reactor, which affects the mass transfer by creating an additional external resistance. In order to minimize this effect, the sucrose solution can be placed in an open flask and heated up in the thermostatic bath, so that it releases the dissolved air.

Using the calibration data, students can estimate right away the sucrose conversion on each reactor (by taking into account the stoichiometry of reaction 1), therefore concluding that reactor B yields a higher conversion, despite having essentially the same amount of catalyst as reactor A. They are asked to discuss this result, emphasizing a qualitative description of the phenomena involved and then they compare these results with the conversion attained with the bioreactor. This opportunity is also used to ponder the effect of different operating variables on conversion, like temperature or flow rate, and on whether these may affect the conditions for the occurrence of the diffusion regime.

Once the conversions are computed, one must estimate the observed (apparent) kinetic constants, k^{obs} , based on the total bed volume, for both reactors A and B. Obtaining the Michaelis-Menten parameters is more involving and we did not ask our students to estimate them.

The apparent kinetic constants of both ion-exchange based reactors are calculated from Equations (6) and (8). The results obtained from these two models (ideal plug flow and plug flow with axial dispersion, respectively) are then compared, and the effect of axial dispersion discussed. The computation of k^{obs} from Equation (8) can be done without much effort using, for instance, any currently available spreadsheet with iterative equation solving capabilities (e.g., the Solver routine in Excel). The Peclet number associated with the model can be estimated from any correlation reported in the literature, for instance the one proposed by Chung and Wen [20]:

$$Pe = \frac{0.2 + 0.011 \left(\frac{\rho u d_p}{\mu} \right)^{0.48}}{\varepsilon} \frac{L}{d_p} \quad (10)$$

In this calculation, the properties of water, at the reactor temperature, are used.

Then, from Equations (4), (5) and (3), the remaining variables (η_1 and η_2) are computed, using the kinetic constant computed from the more realistic model, i.e., Equation (8).

Table 2. Computed results

	Reactor		
	A	B	C
X_{sucr} (%)	59.5	85.8	51.1
τ (s)	500.9	525.8	573.9
Pe	97.5	234.4	–
k^{obs} (s^{-1}) from Eq. (6)	1.80×10^{-3}	3.71×10^{-3}	–
k^{obs} (s^{-1}) from Eq. (8)	1.82×10^{-3}	3.74×10^{-3}	–
ϕ	7.4	2.6	–
η (%)	35.2	72.4	–

Table 2 shows the results obtained for the previously mentioned operating conditions. The conversion obtained by the bioreactor is higher than 50%. It is interesting to note that about 100 mg of invertase catalyze the sucrose inversion almost as good as about 100 cm³ of the 0.81 mm diameter cation-exchange resin.

From the data reported in Table 2 one may also see that the apparent kinetic constants computed for both ion exchange reactors, using the two flow pattern models, are essentially identical. Indeed, as indicated by the high values of the estimated Peclet number, axial dispersion can be neglected, and the simpler ideal plug flow model is sufficient to describe the flow pattern in the reactor. Students' attention is drawn to this issue, which may save computation time and effort in real-world engineering situations.

As predicted, the two ion exchange reactors show quite different efficiency factors, η . Reactor B, with the smaller resin particles, has an efficiency factor that is about twice as that for reactor A. Even so, reactor B is still operating in an intermediate regime, since only for $\phi \leq 1/3$ the efficiency would approach 100%, i.e., the chemical regime would be attained (see Fig. 3). This would imply having particles with a diameter not larger than about 4×10^{-5} m (from Equation (5)). Students should realize, however, that, in practice, reducing the particle size in a bed may cause secondary problems, like too high a pressure drop. A trade-off situation must be explored. As shown in Fig. 3, for particles larger than that used in reactor A the diffusion regime is achieved (linear zone), as for sufficiently high Thiele modulus $\eta \approx 3/\phi$.

With the Thiele modulus computed for both catalysts, evaluation of the sucrose profiles within the pellets allows students to better understand the competition between the two considered phenomena: diffusion and reaction inside the catalyst particles. The sucrose concentration profiles, computed from Equation (9), are shown in Fig. 4. It is interesting to note that, for the larger particles, the diffusion resistance leads to a more marked concentration profile because sucrose cannot diffuse in from the bulk sufficiently rapid. In this case, the large value of the Thiele modulus indicates that the surface reaction is rapid and that the reactant is consumed very close to the external

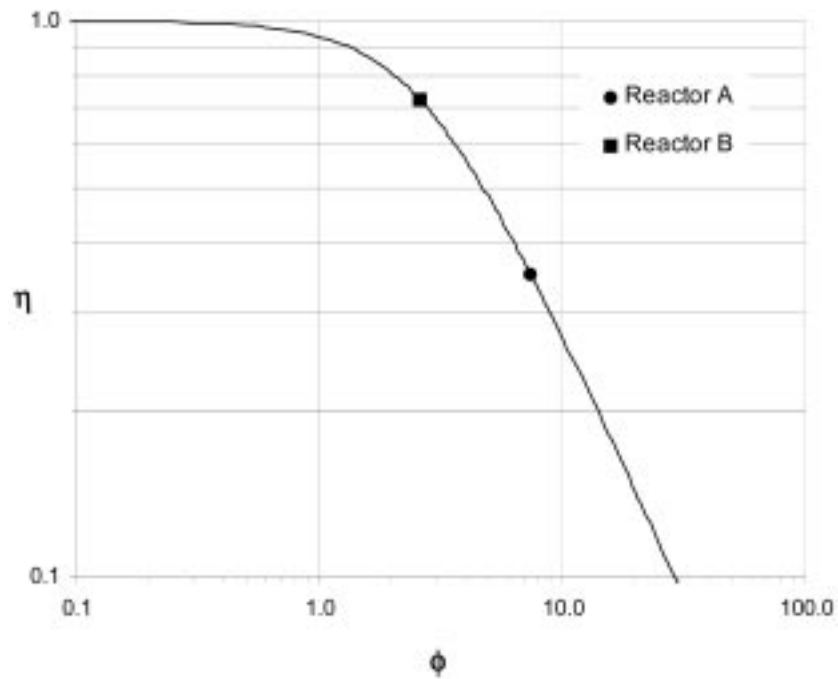


Fig. 3. Effectiveness factor as function of Thiele modulus (through Equation (3)) and experimental data recorded for both chemical reactors.

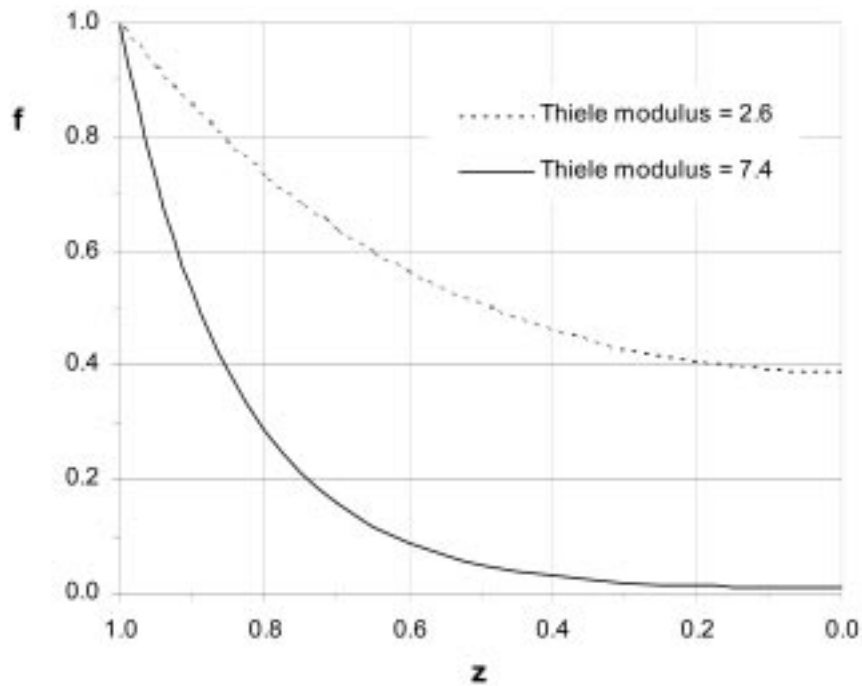


Fig. 4. Sucrose concentration profiles within the two catalyst pellets (through Equation (9)).

pellet surface and very little penetrates into the interior of the pellet. Indeed, for $\phi = 7.4$, the sucrose concentration at the center of the pellets is almost null (cf. Fig. 4), thus decreasing the observed catalytic performance and consequently the effectiveness factor. Smaller particles (with smaller ϕ) have a small diffusion resistance, thus leading to a rather flat curve. Since the rate of reaction at any point in the pellet depends on C_{sucr} ,

this profile causes an increased average rate and a higher η (cf. Table 2).

Finally, students are asked to estimate the sucrose diffusion coefficient and to compare it with data reported in the literature. From the effectiveness factors shown in Table 2 one may compute the intrinsic reaction rate constant ($k = k^{obs}/\eta$). A value of $5.17 \times 10^{-3} \text{ s}^{-1}$ was obtained (for both reactors). Then, from the definition

of the Thiele modulus (Equation (2)) one gets $D_e = 1.85 \times 10^{-7} \text{ cm}^2/\text{s}$. Students may then critically compare the computed values for k and D_e with others found in the literature (e.g., those from Gilliland *et al.* [12] or from Reed and Dranoff [11]).

Students find this experiment very attractive because it allows them to interiorize complex concepts such as the effectiveness factor or the Thiele modulus and it provides a wide practical perspective on the theoretical concepts taught on heterogeneous reactors. Also, the presence of a bioreactor introduces for most of the students basic concepts related with bioengineering. Moreover, this experiment is having the recognition of several other schools, which are now trying to buy or to assemble themselves similar experiments. We must remark that the overall cost of the experimental setup shown in Fig. 1 is about €10,000 (excluding VAT).

CONCLUSIONS

This lab experiment is a simple, compact, economic and environmentally friendly illustration of some important principles of heterogeneous catalysis, including chemical- and bio-catalysis. It has several instructional capabilities which include the

understanding of the principles of packed bed catalytic reactors, the effect of catalyst particle size on the competitive effects that occur between reaction and mass transfer inside the catalyst pellet (through evaluation of the Thiele modulus and the effectiveness factor) and the examination of steady-state catalysis. It is also possible, with the described set-up, to further illustrate important concepts of CRE, namely the flow pattern in a packed bed reactor (for instance with tracer studies) or the effect of some important operating conditions (e.g., flow rate, temperature or feed concentration) on the steady-state conversion. In addition, it uses a basic, but effective, implementation of an important analytical method: flow injection analysis, which is a very advantageous technique when sampling continuous processes. Experimental execution is unelaborated and can be completed within a three-hour class time.

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REFERENCES

1. H. S. Fogler, *Elements of Chemical Reaction Engineering*, 3rd edition, Prentice-Hall, New Jersey (1999).
2. G. F. Froment and K. B. Bischoff, *Chemical Reactor Analysis and Design*, 2nd edition, John Wiley & Sons, New York (1990).
3. O. Levenspiel, *Chemical Reaction Engineering*, 3rd edition, John Wiley & Sons, New York (1999).
4. J. Villiermaux, *Génie de la Réaction Chimique—Conception et Fonctionnement des Réacteurs*, Tec & Doc—Lavoisier, Paris (1993).
5. J. L. Falconer and G. S. Huvar, Important concepts in undergraduate kinetics and reactor design courses. *Chem. Eng. Ed.* **38**, 1999, pp. 138–140.
6. H. S. Fogler, An appetizing structure of chemical reaction engineering for undergraduates. *Chem. Eng. Ed.* **27**, 1993, pp. 110–116.
7. R. P. Hesketh, D. Bosak and L. Kline, Automotive catalytic reaction engineering experiment. *Chem. Eng. Ed.* **34**, 2000, pp. 240–245.
8. M. Shalabi, M. Al-Saleh, J. Beltramini and D. Al-Harbi, Current trends in chemical reaction engineering education. *Chem. Eng. Ed.* **30**, 1996, pp. 146–149.
9. M. P. Dudukovic, Undergraduate reaction engineering laboratory. *AIChE Symp. Ser.* **75**, 1979, pp. 71–80.
10. <http://www.armfield.co.uk/index.shtml>.
11. E. W. Reed and J. S. Dranoff, Ion exchange resin catalysis of sucrose inversion in fixed beds, *Ind. Eng. Chem. Fundam.* **3**, 1964, pp. 304–307.
12. E. R. Gilliland, H. J. Bixler and J. E. O'Connell, Catalysis of sucrose inversion in ion-exchange resins. *Ind. Eng. Chem. Fundam.* **10**, 1971, pp. 185–191.
13. J. M. Lopes, F. Lemos, C. Pinheiro, F. R. Ribeiro, F. D. Magalhães, A. Mendes and C. Costa, Teaching residence time distributions in the laboratory, *Int. J. Eng. Ed.*, **18**, 2002, pp. 674–681.
14. A. E. Rodrigues, Theory of residence time distribution, in A. E. Rodrigues, J. M. Calo and N. H. Sweed (eds.) *Multiphase Chemical Reactors, Vol. I Fundamentals*, NATO Advanced Study Institute Series, N° 51, Sijthoff Noordhoff, 1981, pp. 225–284.
15. F. Helfferich, *Ion Exchange*, McGraw-Hill, New York (1962).
16. M. Valcarcel and M. D. Luque de Castro, *Automatic Methods of Analysis—Techniques and Instrumentation in Analytical Chemistry, Vol. 9*, Elsevier, New York (1988).
17. A. Araújo, J. C. Lima, J. Alonso-Chamarro and M. J. Bartroli, Flow injection system based on the sandwich technique for saving expensive reagents, *Clin. Chim. Acta*, **203**, 1991, pp. 67–76.
18. B. Karlberg and G. E. Pacey, *Automatic Methods of Analysis—Flow Injection Analysis, a Practical Guide, Vol. 10*, Elsevier, New York (1989).
19. S. C. Chapra and R. P. Canale, *Numerical Methods for Engineering*, 3rd edition, chapter 17.1.6, McGraw-Hill, Boston, (1998).

20. S. F. Chung and C. Y. Wen, Longitudinal dispersion of liquid flowing through fixed and fluidized beds. *AI. Ch. E. J.*, **14**, 1968, pp. 857–865.

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