Conversion Expressions for Electrochemical Reactors which Operate under Mass Transport Controlled Reaction Conditions—Part II: Batch Recycle, Cascade and Recycle Loop Reactors*

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In a previous paper, graphical plots were used to calculate the fractional conversion of reactant to product in batch and single pass electrochemical reactors under mass transport controlled conditions. This paper extends the approach to treat batch recycle, cascade and recycle loop reactors. These cases are common both in industry and in pilot laboratory scale in universities. The working curves developed provide a rapid route to the calculation of reactor performance. These simulated curves, produced via standard spreadsheet/plotting software, help to illustrate a chemical engineering approach to science and technology students and short course delegates. The use of a family of working curves avoids the tedious calculation of fractional conversion over a range of process conditions and helps students to appreciate the use of graphical displays to describe the fractional conversion over electrochemical reactors.

NOMENCLATURE

$\begin{array}{c} A\\ A_1\\ A_2 \end{array}$	Effective electrode area Electrode area used in single pass Electrode area to achieve a known conversion over a recycle loop reactor with a recycle ratio R	${f m}^2 {f m}^2 {f m}^2 {f m}^2$
а	Surface area of electrode per unit volume of reactor	m^2/m^3
с	Bulk concentration of reactant	$molm^{-3}$
$c_{(IN)}$	Concentration of reactant at the reactor inlet	$molm^{-3}$
C _(OUT)	Concentration of reactant at the reactor outlet	$molm^{-3}$
c(IN,0)	Concentration at reactor inlet (batch recycle) at time zero	$molm^{-3}$
$c_{(IN,t)}$	Concentration at reactor inlet (batch recycle) at time t	$molm^{-3}$
c ₍₀₎	Initial concentration of reactant (batch reactor)	$molm^{-3}$
c _(t)	Concentration of reactant at time t (batch reactor)	$mol m^{-3}$
F	Faraday constant (96485)	$C \text{ mol}^{-1}$
L	Length of reactor	m
Ι	Current	А
1	Distance into the reactor	m
Κ	A constant in equation (17)	
k _m	Average mass transport coefficient	ms^{-1}
Ν	Number of identical reactors in a cascade arrangement	(dimensionless)
N_T	Number of recycles of electrolyte through the tank, $N_T = t/T$)	(dimensionless)

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Q	Volumetric flow rate of electrolyte	$m^{3} s^{-1}$
$\tilde{\mathbf{Q}}_0$	Volumetric flow rate entering (and leaving) the system	$m^{3} s^{-1}$
\tilde{Q}_2	Volumetric flow rate recirculated through the reactor loop	$m^{3} s^{-1}$
R	Recirculation ratio, $R = Q_2/Q_0$	(dimensionless)
t	Time	S
V _R	Volume of electrolyte within the reactor	m ³
V_{T}	Volume of electrolyte within the mixer tank	m ³
X_A^{Batch}	Fractional conversion of reactant in a batch system	(dimensionless)
X_{SP}^{PFR}	Fractional conversion of reactant over a single pass PFR	(dimensionless)
X _{SP} ^{CSTR}	Fractional conversion of reactant over a single pass CSTR	(dimensionless)
X_{BR}^{PFR}	Fractional conversion of reactant for a PFR in batch recycle	(dimensionless)
X_{BR}^{CSTR}	Fractional conversion of reactant for a CSTR in batch recycle	(dimensionless)
X_N^{PFR}	Fractional conversion of reactant over a cascade of N identical PFRs	(dimensionless)
X_{N}^{CSTR}	Fractional conversion of reactant over a cascade of N identical CSTRs	(dimensionless)
X_R^{PFR}	Fractional conversion of reactant for a PFR with recycle loop	(dimensionless)
	Fractional power in equation (18)	(dimensionless)
	Fraction in equation (17)	(dimensionless)
	Current efficiency	(dimensionless)
R	Mean residence time of electrolyte in the tank $(R = V_{R/Q})$	S
Т	Mean residence time of electrolyte in the reactor $(T = V_T/Q)$	S

SUMMARY OF EDUCATIONAL ASPECTS OF THIS PAPER

- 1. In Part 1 [1], a new training tool was provided for teaching in electrochemical engineering. Working curves showed the effect of mass transport coefficient and electrode area (hence process conditions) on the fractional conversion in an electrochemical reactor operating in the batch or single pass mode while supporting a single electrode reaction under completely mass transport controlled conditions.
- 2. Here, the approach is extended to more sophisticated reactors. This allows students to appreciate that batch recirculation, use of a number of identical reactors in hydraulic series ('cascade') and a recycle loop over the reactor can be exploited to provide greater operational flexibility, improved control stability or an enhanced overall conversion.
- 3. The level of students using the material has ranged from undergraduate chemistry, materials engineering and chemical engineering undergraduates through to Masters and Doctoral candidates. The approach has been adopted in workshops and short courses to industry and in tutorial sessions at international conferences.
- 4. The use of simple working curves, obtained by the use of an EXCEL spreadsheet and standard graph plotting routine, allows students to appreciate the engineering convenience of representing the design equations for fractional conversion in a graphical format.
- 5. The material can be used to illustrate specific fractional conversion ranges in important applications, such as environmental treatment and clean synthesis.
- 6. Comparison of the fractional conversion in recycle and cascade modes of operation with

the simpler batch and single pass reactors (treated in Part 1) shows the advantages of the more sophisticated operational modes.

INTRODUCTION

IN PART 1 [1], we described a training tool for teaching electrochemical engineering. Working curves were used to illustrate the design equations which describe the fractional conversion of reactant in electrochemical reactors (batch, plug flow reactor-PFR, and continuous stirred tank reactor-CSTR). For simplicity, the reactors were assumed to operate in the batch or single pass mode while supporting a single electrode reaction (involving z electrons per reactant species) under completely mass transport controlled conditions. The reaction rate reaches the maximum when the electrode operates at a limiting current, I_L, controlled by the rate at which reactant species reach the electrode [1]. At any time and for a fixed concentration, c, next to the electrode, the product of the average mass transport coefficient (a function of the limiting current) and electrode area (k_mA) was highlighted as the important performance factor:

$$k_m A = \frac{I_L}{zFc}$$
(1)

where F is the Faraday constant.

Here, the approach is extended to more sophisticated reactors. This allows students to appreciate that batch recirculation, use of a number of identical reactors in hydraulic series ('cascade') and a recycle loop over the reactor can be exploited to provide greater operational flexibility, improved control stability or an enhanced overall conversion. The assumption of a single electrode reaction under complete mass transport control is retained.

As in the case of the first paper, graphical plots have been developed to express fractional conversion for various process conditions, in the case of reaction which is under full mass transport control. In each case, a mass balance (per unit time) must be set up for the system and the corresponding differential equations can then be solved (see Part 1). In all cases, a steady volumetric flow rate of electrolyte together with a constant electrode area and known mass transport coefficient are assumed. An analytical solution to the design equations is possible in all cases, leading to the expressions summarised in Table 1. A larger number of data points has been calculated using customised functions in Microsoft Excel 4.0 and on a 486 or (more recently) version 5.0 EXCEL on a Pentium PC. The curves can then be interpolated between data points to provide information on a particular conversion under a fixed process condition.

BATCH RECYCLE

The use of the batch recirculation (or batch recycle) mode (Fig. 1) is a versatile strategy that allows the operator to adjust the volume of the system and aid feedstock preparation, monitoring, temperature control and product extraction [2]. In this case the reactant concentration is a complex function, and it is important to develop an adequate model for the prediction of experiment results. Despite its importance, relatively few contributions have appeared for electrochemical systems [3–6]. For a PFR in batch recycle, the reactant concentration is a function of both position and time due to coupling of the PFR, where

concentration is a function of position with the well mixed tank where concentration is a function of time. The mass balance $(mol m^{-1} s^{-1})$ for this case, formulated using the same methodology as in the previous paper [1], leads to [3]:

$$A\frac{\partial c(t,l)}{\partial t} = -Q\frac{\partial c(t,l)}{\partial l} - k_{m}Aac(t,l) \qquad (2)$$

The term on the left-hand side concerns the variation of concentration with time, the first term on the right concerns concentration variation with position inside of the reactor and the last term on the right-hand side is the reaction rate (assuming complete mass transport control of a single electrode process).

This differential equation can be solved using Laplace transform techniques leading to complicated expressions that are difficult to work with. Alternatively, approximate models are available [7, 8] that result from deliberately ignoring the J_t term in equation (2). In this case, the resulting expression is:

$$X_{BR}^{PFR} = 1 - \frac{c_{(IN,t)}}{c_{(IN,0)}}$$

= 1 - exp\{ -\frac{t}{\tau_T} [1 - exp(-k_m A/Q)]\} (3)

or

$$X_{BR}^{PFR} = 1 - exp\left\{-N_T X_{SP}^{PFR}\right\} \tag{4}$$

Where X_{SP}^{PFR} is the fractional conversion per pass through the reactor, and τ_T is the residence time in the mixer tank. The term $N_T = t/\tau_T$ is the number of recycles of electrolyte through the tank. Therefore, equation (3) shows that conversion for a PFR in a batch recycle is equal to the conversion per pass, X_{SP}^{PFR} , multiplied by the number of recycles t/τ_T .

Table 1. Expressions for fractional conversion in electrochemical reactors under complete mass transport controlled reaction conditions for batch recycle, cascade and recycle loop modes of operation. The expressions all contain the performance factor, $x = k_m A/Q$. In the batch recycle mode, the number of recycles through the tank, $N_T = t/_T$ is important. Cascade operation allows the overall conversion to be increased by the use of N identical reactors. The conversion in PFRs containing a recycle loop is influenced by the recycle ratio, R

Reactor Type	Expression for Overall Fractional Conversion of Reactant	Equation Number
PFR in batch recirculation	$X_{BR}^{PFR} = 1 - \frac{c_{(IN,t)}}{c_{(IN,0)}} = 1 - exp \bigg\{ -\frac{t}{\tau_{T}} [1 - exp(-x)] \bigg\}$	(3)
CSTR in batch recirculation	$X_{\text{BR}}^{\text{CSTR}} = 1 - \frac{c_{(\text{IN},t)}}{c_{(\text{IN},0)}} = 1 - \exp\left\{-\frac{t}{\tau_{\text{T}}}\left[1 - \left(\frac{1}{1+x}\right)\right]\right\}$	(6)
Cascade of N identical PFRs	$X_N^{PFR} = 1 - \frac{c_{(OUT,N)}}{c_{(IN)}} = 1 - exp(-Nx)$	(13)
Cascade of N identical CSTRs	$X_{N}^{CSTR} = 1 - \frac{c_{(OUT,N)}}{c_{(IN)}} = 1 - \frac{1}{\left[1 + x\right]^{N}}$	(15)
PFR with recycle loop	$X_{Recycle}^{PFR} = 1 - \frac{c_{(OUT)}}{c_{(IN)}} = 1 - \frac{1}{[(1+R)\exp{x(1+R)}] - R}$	(16)



Fig. 1. Definition sketch for an electrochemical reactor in the batch recirculation mode of operation.

For a CSTR, the non-steady state mass balance $(mol s^{-1})$ for the reactor can be written as [3]:

$$LA\frac{dc_{(OUT)}}{dt} = Q(c_{(IN)} - c_{(OUT)}) - k_m A c_{(OUT)}$$
(5)

where L is the effective length of the reactor. An approximate solution leads to [2]:

$$X_{BR}^{CSTR} = 1 - \frac{c_{(IN,t)}}{c_{(IN,0)}}$$
$$= 1 - \exp\left\{-\frac{t}{\tau_{T}}\left[1 - \left(\frac{1}{1 + k_{m}A/Q}\right)\right]\right\} (6)$$

or

$$X_{BR}^{CSTR} = 1 - \exp\{-N_T X_{SP}^{CSTR}\}$$
(7)

The term in parentheses represents the product of the number of recycles through the tank and the single pass conversion across the reactor. Figures 2 and 3 show conversion achieved for a PFR and CSTR, respectively, in batch recirculation as a function of the number of recycles. The conditions chosen include a high flow rate through the reactor and a low conversion per pass. For these cases, it is interesting to consider all systems as a batch



Fig. 2. Fractional conversion vs. number of single passes through a PFR in batch recirculation, showing the importance of the factor k_mA/Q in equation (3).



Fig. 3. Fractional conversion against number of single passes through a CSTR in batch recirculation, showing the importance of the factor k_mA/Q in equation (6).

reactor and a first order rate decay expression to predict reactor concentration:

$$\frac{\mathbf{c}_{(t)}}{\mathbf{c}_{(0)}} = \exp{-\frac{\mathbf{k}_{m}\mathbf{A}}{\mathbf{V}_{R}}t}$$
(8)

The fractional conversion at any time, t, is then:

$$X_A^{\text{Batch}} = 1 - \frac{c_{(t)}}{c_{(0)}} = 1 - \exp\left(\frac{k_m A}{V_R}t\right) \quad (9)$$

The differences between equations (3) and (9) can be seen in Fig. 4, where the percentage error in the conversion has been defined as:

% Error in
$$X_A^{PFR} = 100 \left[\frac{X_A^{Batch} - X_A^{PFR}}{X_A^{PFR}} \right]$$
 (10)

As can be seen in Fig. 4 the error decrease in an exponential fashion as flow rate is increased. The error is smaller at low values of k_mA/Q and, hence, at small degrees of conversion. A similar trend is observed in Fig. 5 for a CSTR, where the percentage error has been defined as:

% Error in
$$X_A^{CSTR} = 100 \left[\frac{X_A^{Batch} - X_A^{CSTR}}{X_A^{CSTR}} \right]$$
 (11)

but a comparison between Figs 4 and 5 shows a greater error in the case of assumption of a simple batch model when treating a CSTR in batch recirculation. This is again due to the fact that the CSTR model considers the concentration inside the reactor to be equal to the outlet concentration [13], as discussed in the previous paper [1]. Figures 4 and 5 lead to the conclusion that the simple batch model is appropriate for reactors in batch recycle, when the flow through the reactor is high and the conversion per pass is low. In such cases, application of the simple batch model greatly simplifies the data analysis [2, 3, 14, 15].

THE CASCADE MODE OF OPERATION

The arrangement of identical reactors in hydraulic series (i.e., a cascade of reactors) (Fig. 6) is a convenient mode when it is necessary to increase overall conversion. In the case of the PFR, the successive application of equation (12) describing the fractional conversion over a single pass PFR:

$$X_{SP}^{PFR} = 1 - \frac{c_{(OUT)}}{c_{(IN)}} = 1 - exp\left(-\frac{k_m A}{Q}\right) \quad (12)$$



Fig. 4. Percentage error in determining the fractional conversion for a PFR in a batch recirculation, when considered as a simple batch reactor, as a function of volumetric flow rate, Q (Equation (10); $V_T = 3000 \text{ cm}^3$, t = 1000 s).

over a series of identical reactors in hydraulic series (cascade) leads to:

$$X_{N}^{PFR} = 1 - \frac{c_{(OUT,N)}}{c_{(IN)}} = 1 - exp\left(-N\frac{k_{m}A}{Q}\right) \quad (13)$$

Therefore, a cascade of N PFRs should show the same behaviour as a single PFR with an of area N times greater than that of the single pass reactor.

In the case of a cascade of N identical CSTRs, successive application of equation (14):

$$X_{SP}^{CSTR} = 1 - \frac{c_{(OUT)}}{c_{(IN)}} = 1 - \frac{1}{1 + (k_m A/Q)}$$
 (14)

leads to an expression for a cascade of N identical CSTRs:

$$X_{N}^{CSTR} = 1 - \frac{c_{(OUT,N)}}{c_{(IN)}} = 1 - \frac{1}{\left[1 + (k_{m}A/Q)\right]^{N}}$$
(15)

Figure 7 displays the conversion achieved versus the number of reactors in cascade; the value km/Q also plays an important role, as can be seen in the figure. An increase of the number of identical CSTR in series allows higher values of conversion to be achieved for a fixed value of k_mA/Q . This behaviour has been practically demonstrated, in the case of rotating cylinder electrode reactors for metal ion removal [16–19]. This is due to the shift towards PFR behaviour for a sufficient number of CSTRs; in the limiting case of an infinite number of reactors, the same conversion is achieved for a PFR having the same area as the sum of the individual CSTRs.

REACTORS WITH A RECYCLE LOOP

A reactor with a recycle loop, shown in Fig. 8, is characterised by part of the product steam being returned to the reactor inlet. In this case, the conversion achieved is given by [6]:

$$X_{\text{Recycle}}^{\text{PFR}} = 1 - \frac{1}{\left[(1+R) \exp \frac{k_m A(R+1)}{Q} \right] - R}$$
(16)



Fig. 5. Percentage error in determining the fractional conversion for a CSTR in batch recirculation, when considered as a simple batch reactor, as a function of volumetric flow rate, Q (equation 11); $V_T = 3000$.

where R is the recirculation ratio, defined as R = Q2/Q0, where Q0 is the volumetric flow rate entering the system and Q2 the volumetric flow rate recirculated (Fig. 8). For R0, equation (16) leads to a single pass PFR. On the other hand, for R the system approximates to a stirred reactor with a correspondingly lower conversion. The adjustable recirculation ratio of the loop reactor is fully exploited in important engineering applications.

In the case of a CSTR, a recycle loop cannot improve the (ideal) mixing conditions—and the rate of mass transport remains constant. However, the loop can act as a means of stabilising the control of a reactor about a set point operation. For example, a recycle loop around a rotating cylinder electrode reactor, used to remove silver from photographic fixing solution, allowed a constant metal ion extraction rate to be achieved, despite some variation in the silver level of the feed stream [17].

Figure 9 shows a decrease in the conversion as a function of the recirculation rate. This happens for reactions with a positive reaction order relative to the reactants such as in the case of mass transport,



Fig. 6. Definition sketch for a cascade of N identical CSTRs.



Fig. 7. Fractional conversion achieved over a cascade of identical CSTRs as a function of the number of reactors, showing the importance of the factor k_mA/Q in equation (15).

where the reaction rate depends upon the limiting current, which is a function of the bulk concentration. This lower conversion value, achieved at high reaction rates, is due to dilution of the feed. In some cases, however, dilution of the feed helps to increase reaction rate, as in the case of autocatalysis [9]. The mass transport coefficient km is a function of flow rate (and electrode/reactor geometry). Therefore, an increase in the overall reaction rate will require an increase in the mass transport coefficient, km, and/or the electrode area, A, to achieve a certain conversion. The mass transport coefficient is expected to be a strong



Fig. 8. Definition sketch for a reactor with a recycle loop.



Fig. 9. Fractional conversion achieved over a PFR with a recycle loop as a function of the recycle ratio R. A constant value of k_m is assumed in equation.

function of flow rate according to a power law expression:

$$\mathbf{k}_{\mathrm{m}} = \mathbf{K} \mathbf{Q}^{\gamma} \tag{17}$$

where depends on the flow regime and the electrode/reactor geometry; typical values are 0.4-0.5 for laminar flow and 0.8-1.0 for turbulent flow [12]. The relationship between the area necessary to achieve a particular conversion in single pass (A1) and the area necessary for the same purpose with recycle (A2) is [10-12]:

$$\frac{A_2}{A_1} = \frac{K_1}{K_2} (R+1)^{1-\beta} \frac{\ln\left\{\frac{c_{(IN)} + Rc_{(OUT)}}{c_{(OUT)} + Rc_{(OUT)}}\right\}}{\ln\frac{c_{(IN)}}{c_{(OUT)}}}$$
(18)

Figure 10 shows the application of equation (18) for laminar and turbulent flow conditions assuming that $c_{(OUT)} = 0.1c_{(IN)}$ (90% conversion) and that K is proportional to the flow rate raised to the same power. The electrode area necessary to

achieve a particular conversion decreases at higher values of recycle ratio.

A large difference in behaviour is observed between laminar flow (=0.5) and turbulence flow, (=0.8). It must be understood that an increase in flow rate is needed to improve km, as shown in (17), but this leads to a decrease in the residence time in the reactor, $_{\rm R} = Q/V_{\rm R}$). Furthermore, Q is a function of the recirculation rate. The effect of volumetric flow rate, Q, is, therefore, complex [6, 7].

SUMMARY

- Batch recycle, cascade and recycle loop electrochemical reactors have been considered in order to develop working curves showing the conversion achieved under mass transport controlled reaction conditions.
- 2. As in Part 1, Table 1 shows how the parameter k_mA is repeated throughout the design expressions and a comparison of equations from this



Fig. 10. Comparison of electrode areas required for single pass and recycle operations of a plug flow reactor to achieve a fractional conversion of 0.90, showing the effect of the factor ? in equation (18)?.

table with those in the previous paper [1] shows how both the PFR and the CSTR in batch recycle can achieve a fractional conversion equal to the number of recycles multiplied by conversion per pass through the reactor.

- 3. For the batch recycle mode, it has been demonstrated that assumption of a simple batch model is a reasonable approximation, involving a low error, when the flow rate through the reactor is high and the conversion per pass is low.
- 4. In a cascade arrangement, it has been shown that the overall conversion increases with the number of reactors.
- 5. A cascade of identical CSTRs behaves as a PFR in the case of a sufficiently large number of reactors.
- 6. In the case of a loop reactor, the recycle ration influences the electrode area necessary to achieve a particular fractional conversion.

- 7. Conversion strongly depends upon the recycle ratio for a reactor with a loop. When the ratio tends to infinity, the system effectively behaves as a perfectly mixed tank; for a ratio of zero, the system can be viewed as a PFR in the single pass mode.
- 8. The use of graphical plots neatly illustrates the design expressions for electrochemical reactors under mass transport control and allows easy estimation of fractional conversion under a range of operational conditions.

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