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Comparison between physical and chemical methods for the measurement of mixing times

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Abstract

The methods used to measure mixing times can be divided into two groups: physical and chemical methods. Theoretical and experimental comparisons between these two types of methodology are presented in this paper. The theoretical hypotheses used to compare physical and chemical mixing times were determined by the local mass transfer equations with boundary and initial conditions. In the chemical case, stoichiometric equivalence is not achieved simultaneously at every point in the region of scrutiny of the probe. Therefore, the measured mixing time depends on when the probe is considered to be under stoichiometric equivalence conditions. For a conductivity cell with an electrolytic tracer, the same physical and chemical mixing times are obtained when, in the chemical method, the minimum conductivity measured is assumed to be the equivalence point and a particular definition of deviation from homogeneity is used. Experimental measurements performed in an agitated reactor confirm the theoretical results. $© 1997$ Elsevier Science S.A.

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1. Introduction

The mixing time is a useful quantity for measurement of the blending of a phase. However, it is difficult to compare the mixing times obtained by different researchers because they are strongly dependent on the definition and method of measurement of the system non-homogeneities, the probe type, the device used to introduce the tracer, its location, etc.

Firstly, it is important to distinguish between methods which require the presence of a chemical reaction, i.e. chemical methods *(*e.g. decolorization method, pHmetric method*)*, and those in which a reaction is absent, i.e. physical methods *(*e.g. thermic method, conductimetric method*)*.

In physical methods, the tracer is injected into the system. One or more probes in the reactor measure a quantity which is proportional to the concentration of the tracer; in this case the mixing time is the time interval from the introduction of the tracer to a fixed deviation from homogeneity.

Chemical methods use an instantaneous chemical reaction. Reactant A, in stoichiometric excess, is introduced into a system, in which reactant B is already present in a uniform concentration; in this case, the mixing time is the time

necessary to achieve equivalence conditions in the whole reactor or a definite zone. The equivalence point is noted, for example, by the colour change of the indicator *(*decolorization method*)* or by measurement of the conductivity or pH *(*the latter method has disadvantages such as a high electrode time constant*)*.

The measured mixing time decreases as the reactant excess increases. If the reactant excess increases, its concentration in the system will be greater; therefore, under the same fluid dynamic conditions, equivalence conditions will be achieved more rapidly.

A comparison of the two types of method has previously been reported by other workers. Ruszkowski and Muskett *[*1*]* have indicated that the chemical mixing time is shorter than the physical mixing time, but no theoretical explanation was given for this experimental result. Takao et al. *[*2*]* carried out an experimental study on the relationship between the physical mixing and instantaneous reaction in a stirred tank reactor. Their experimental results confirmed Käppel's theory *[*3*]*.

The aim of this work is to compare theoretically and experimentally these two types of method, in order to determine the conditions under which physical and chemical methods can be considered to be equivalent and to provide experimental evidence for the theoretical results.

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2. Theoretical part

2.1. Mixing time and deviation from homogeneity

In order to define a mixing time, we need to specify exactly the deviation from system homogeneity. Several definitions of non-homogeneity (see Hiby [4] and Käppel [3]) are possible corresponding to different mixing times. The following definition is considered in this work for physical methods:

$$
d_{\rm s}(\Re_{\rm s};\,t) \equiv \max\left(\frac{|C_{\rm Ts}(\Re_{\rm s};\,t) - C_{\rm Tx}|}{C_{\rm Tx}}\right) \tag{1}
$$

By fixing the deviation from homogeneity as δ , the mixing time t_{δ} is defined as the longest period of time from injection of the tracer to when the deviation d_s (\mathcal{R}_s ; *t*) reaches δ. This means

$$
t_{\delta s} \equiv \max(t)
$$
 so that $d_s(\Re_s; t) = \delta \Rightarrow t_{\delta s} = f(\Re_s, \delta)$ (2)

Definition Eq. *(*1*)* does not consider the non-homogeneity inside the region of scrutiny \mathcal{R}_s . Therefore, when the measurement volume of the region of scrutiny \mathcal{R}_s is increased, the non-homogeneity is neglected for a greater volume, implying a decrease in the mixing time. This effect has been studied by Thyn et al. *[*5*]*.

2.2. Mixing time with physical and chemical methods

In chemical methods, by assuming a homogeneous irreversible chemical reaction and the following reaction scheme

 $\nu_{\rm A}A+\nu_{\rm B}B\rightarrow\nu_{\rm P}P$

a single partial differential equation,suitable for a comparison between physical and chemical methods, can be obtained from the local material balances of the two reactants *(*A and B*)*. If the molecular diffusion coefficients of reactants A and B are equal, using the same method as employed by Toor *[*6*]*, we obtain

$$
\frac{\partial C_{\text{Ch}}}{\partial t} + \vec{\nu} \cdot \vec{\nabla} C_{\text{Ch}} = D \nabla^2 C_{\text{Ch}}
$$
\n
$$
C_{\text{Ch}} = -\frac{\nu_{\text{B}} C_{\text{A}}^{\text{i}}}{\nu_{\text{A}} C_{\text{B}}^{\text{i}}} \quad \vec{x} \in \mathfrak{R}_{\text{A}}^{\text{i}} \quad t = 0
$$
\n(3)

$$
C_{\text{Ch}} = 1 \quad \vec{x} \in \mathfrak{R}_{\text{B}}^{i} \quad t = 0
$$

$$
\vec{\nabla} C_{\text{Ch}} \cdot \vec{n} = 0 \quad \vec{x} \in S_{\text{T}}(t) \quad t \ge 0
$$

where C_{Ch} is

$$
C_{\rm Ch} \equiv \frac{\nu_{\rm A} C_{\rm B} - \nu_{\rm B} C_{\rm A}}{\nu_{\rm A} C_{\rm B}^{\dagger}} \tag{4}
$$

In general the total boundary surface $S_T(t)$ is a function of time (for example, when $S_T(t)$ includes the surface of the stirrer*)*. Eq. *(*3*)* is also valid in turbulent systems when the molecular diffusion coefficients of the reactants are different. In this case, the diffusion terms are negligible.

In the case of the physical method, from the material balance of the tracer at $t=0$ and $t \rightarrow \infty$, we obtain

$$
C_{\rm T}^{\rm i} V_{\rm T} = C_{\rm T\infty} (V_{\rm T} + V_0) \tag{5}
$$

By setting

$$
C_{\rm Ph} \equiv \frac{1 - C_{\rm T}/C_{\rm T\infty} - \delta}{1 - \delta} \tag{6}
$$

from Eqs. *(*5*)* and *(*6*)*, we obtain

$$
\frac{1 - C_{\rm T}^{\rm i}/C_{\rm T\infty} - \delta}{1 - \delta} = -\frac{V_0 + \delta \cdot V_{\rm T}}{V_{\rm T}(1 - \delta)}\tag{7}
$$

By replacing Eqs. *(*6*)* and *(*7*)* in the localmaterial balance of the tracer, we obtain

$$
\frac{\partial C_{\text{Ph}}}{\partial t} + \vec{\nu} \cdot \vec{\nabla} C_{\text{Ph}} = D \nabla^2 C_{\text{Ph}}
$$
\n
$$
C_{\text{Ph}} = -\frac{V_0 + \delta \cdot V_{\text{T}}}{V_{\text{T}}(1 - \delta)} \quad \vec{x} \in \mathfrak{R}_{\text{T}}^{\text{i}} \quad t = 0
$$
\n
$$
C_{\text{Ph}} = 1 \quad \vec{x} \in \mathfrak{R}_{0}^{\text{i}} \quad t = 0
$$
\n(8)

 $\vec{\nabla} C_{\text{Ph}} \cdot \vec{n} = 0 \quad \vec{x} \in S_{\text{T}}(t) \quad t \ge 0$

In order to obtain the same solution of both Eq. *(*8*) (*physical methods*)* and Eq. *(*3*) (*chemical methods*)*, the partial differential equations must have the same boundary conditions. By imposing

$$
\frac{\nu_{\rm B} C_{\rm A}^{i}}{\nu_{\rm A} C_{\rm B}^{i}} = \frac{V_{\rm B} + \delta \cdot V_{\rm A}}{V_{\rm A} (1 - \delta)}
$$
\n(9)

and by taking δ from Eq. (9), we obtain

$$
\delta = \frac{C_{A}^{i}V_{A} - \frac{\nu_{A}C_{B}^{i}V_{B}}{\nu_{B}}}{C_{A}^{i}V_{A} + \frac{\nu_{A}C_{B}^{i}V_{A}}{\nu_{B}}} = \frac{n_{A} - n_{A}^{\text{ste}}}{n_{A} + n_{A}^{\text{ste}}V_{A}} \tag{10}
$$

When $\mathcal{R}_T^i = \mathcal{R}_A^i$, $\mathcal{R}_0^i = \mathcal{R}_B^i$ ($V_T = V_A$, $V_0 = V_B$) and Eq. *(*10*)* is imposed, the solution is the same in both the physical and chemical cases

$$
C_t(\vec{x},t) \equiv C_{\text{Ph}}(\vec{x},t) = C_{\text{Ch}}(\vec{x},t)
$$
\n(11)

By defining $t^*_{\delta s}$ as the maximum time at which $C_{ts}(\mathcal{R}_s,t) = 0$, we obtain

 $t_{\delta s}^*$ = max(*t*) so that $C_{ts}(\Re_s, t) = 0$

$$
\Rightarrow \left\{ \frac{C_{\text{Tx}} - C_{\text{Ts}}(\Re_s, t)}{C_{\text{As}} \left(\frac{C_{\text{Tx}}}{\nu_{\text{A}}}\right)} = \delta \text{ (physical case)} \atop \nu_{\text{A}} \left(\frac{C_{\text{Bs}}(\Re_s, t)}{\nu_{\text{B}}} \text{ (chemical case)} \right) \right\} \quad (12)
$$

In the physical case, $t^*_{\delta s}$ corresponds to the mixing time obtained by considering Eq. *(*1*)* without the absolute value in the numerator as the definition of deviation from homogeneity.

In the chemical case, the presence of a finite region of scrutiny \mathcal{R}_s creates the problem of defining the achievement of the stoichiometric equivalence point. When the reaction rate is instantaneous, the simultaneous presence of reactants A and B is not possible; therefore two limiting situations are obtained.

- 1. The stoichiometric equivalence is achieved at a single point in region \mathcal{R}_s , and reactant B is present at the remaining points. In this case, $C_{ts}(\mathcal{R}_s,t)$ is positive. t^{*-} _{os} is defined as the maximum time for which this situation exists.
- 2. The equivalence condition is obtained at each point of \mathcal{R}_s , except at a single point of the region of scrutiny, and reactant A is present at the remaining points. In this case, $C_{ts}(\mathcal{R}_s,t)$ is negative. $t^{*+}{}_{\delta s}$ is defined as the maximum time for which this situation exists.

Because reactant A is introduced in stoichiometric excess, situation *(*2*)* necessarily follows situation *(*1*)*.

The experimentally measured mixing time is between t^{*} ⁻_{δ s} and t^{*} ⁺_{δ s} and its value depends on the point of equivalence used by the detection method; it may be larger or smaller than the mixing time measured with physical methods.

2.3. Mixing time using electrolytes

A conductivity cell allows measurement of the specific conductivity of an electrolytic solution with very high accuracy. As the measurement is very simple to make, an electrolytic solution represents a good tracer for the measurement of mixing times using the physical method. In this case, the mass transfer of ions, not neutral compounds, occurs. A term describing the migration due to the electric field appears in the local balances of the ionic species. By imposing the condition of electroneutrality, it is possible to obtain a local balance of the electrolyte without the migration term from the local balances of single ions *[*7*]*. We therefore obtain a partial differential equation, formally equal to Eq. *(*8*)*, where C_{Ph} is the electrolyte concentration.

In the region of scrutiny, the cell measures the average specific conductivity of the fluid which, for dilute solutions, is proportional to the mean concentration of the ions. Therefore, if the electrode is set in the most critical region, the mixing time measured uses Eq. *(*1*)* as the definition of deviation from homogeneity.

There is not a single electrolyte in the presence of a chemical reaction, and therefore diffusion and migration terms appear in the material balances. In this case, Eq. *(*3*)* is valid only if the system is turbulent and it is possible to neglect these terms.

In the case of a chemical reaction it is also possible to employ a conductivity cell; the equivalence point is identified as the absolute minimum of the average specific conductivity in the region of scrutiny.

2.4. Mixing time using an acid–base reaction (NaOH, HCl)

Conductivity measurements using a chemical reaction have been carried out by injecting a concentrated sodium hydroxide solution into a system containing hydrochloric acid. The injected reactant is the hydroxide ion OH^- and the (already present) reactant is the hydrogen ion H^+ .

In this case, Eq. *(*3*)* is valid if the system is turbulent and if the initial concentrations are sufficiently high to neglect water dissociation. As shown in Appendix A, the time necessary to achieve the minimum absolute average specific conductivity of the solution in the region of scrutiny is practically equal to the mixing time defined in Eq. *(*12*)*.

3. Experimental part

In Section 2, it was shown that, under certain conditions, the mixing times measured by chemical and physical methods are identical. Conductivity measurements of mixing times have been performed using both methods in order to test this correspondence. The region of scrutiny must be the same in order to compare the mixing times obtained fromthe physical and chemical methods; therefore, the same conductivity cell was used in both cases $(V_s = 0.16 \text{ cm}^3)$.

3.1. Experimental apparatus

The measurements were performed using the apparatus shown in Fig. 1. The vessel dimensions and probe positions are given in Tables 1 and 2. The reactor is cylindrical with a flat bottom. It has four baffles located symmetrically and a stirrer equipped with two impellers *(*Rushton type*)*. The reactant or tracer was injected under the lower impeller. The injection system consists of a tank T_1 and two electrical valves controlled by a timer.

Fig. 1. Scheme of the apparatus: T_1 , tank; V_1 , valve; E_1 , E_2 , electrical valves; P, conductivity cell; C, conductivity meter; R, recorder.

Table 1 Reactor dimensions and probe position

Table 2 Dimensions and position of the impellers

D	$a = D_s/5$	$r = D/4$	$s = 3/4D$ $H_a = D$ $H_a = 3D$		
130 mm	26 mm	32 mm	98 mm	130 mm 390 mm	

The tank T_1 , containing the solution to be injected *(approx*imately 10 ml in both methods*)*, is set to a pressure of about 7 bar by opening the valve V_1 . The electrical valve E_1 is opened to inject the tracer into the reactor. The electrical valve E_1 closes after 0.8 s and the electrical valve E_2 is opened for a few seconds. In this way, the injection tube is washed to avoid slow introduction of the injected solution.

3.2. Physical method

Several millilitres of a concentrated solution of KCl were injected into the reactor containing demineralizedwater.Specific conductivity vs. time curves were obtained on a recorder and mixing times at different δ were measured. The measured mixing times were averaged assuming a normal distribution of errors *(*eight measurements for each set of experimental conditions*)*. An example of the normalized conductivity curve obtained with the physical method is shown in Fig. 2.

3.3. Chemical method

About 10 ml of an 8 N solution of sodium hydroxide were injected into a dilute hydrochloric acid solution contained in the reactor which was prepared by introducing a known volume of concentrated 2 N HCl solution.

According to the theoretical findings, the mixing time was calculated by considering the minimum of the recorded specific conductivity curves.

The injection device did not allow the reproducibility of the exact quantity of solution injected during the experiments. As a consequence, the stoichiometric excess of injected sodium hydroxide solution was measured by making a backward pHmetric titration of the solution contained in the reactor at the end of the test. A hydrochloric acid solution, with an exactly known dilution ratio to the initial 2 N solution,

Fig. 2. Normalized conductivity vs. time (physical method, $N = 200$ $rev \text{ min}^{-1}$).

Fig. 3. Normalized conductivity vs. time (chemical method, $N = 200$ rev min⁻¹, δ = 0.250).

was used for this titration. The injected volume V_A was negligible in comparison with that of the system $(V_A \ll V_B)$; under these conditions, Eq. *(*10*)* becomes

$$
\delta \approx \frac{n_{\rm A}^{\rm ecc}}{n_{\rm A}} = \frac{V_{\rm A}^{\rm ecc}}{V_{\rm A}}
$$
\n(13)

Therefore exact knowledge of the hydrochloric acid solution concentration was not necessary. The evaluation of δ was reduced to the ratio between the volumes, and the only cause of error in the determination of δ was the volumetric measurements.

An example of the conductivity curve obtained using the chemical method is shown in Fig. 3.

3.4. Experimental measurements and results

All the tests were carried out at 200 rev min^{-1} . The system is turbulent under these conditions, so the diffusion term is negligible compared with the convection term.

In this situation, the migration term is also negligible, because its magnitude depends on the diffusion term. By making the current intensity vector equal to zero, we obtain the potential gradient of the migration term as a function of the ion diffusion flux *[*7*]*

$$
\vec{\nabla}\Phi = \frac{F}{\chi} \sum_{i} z_i (-D_i \vec{\nabla} C_i)
$$
\n(14)

If the diffusion and migration terms are not negligible, the mixing time measurements will depend not only on the system fluid dynamics, but also on the diffusion coefficients of the compounds employed in the test.

The conductivity cell was placed above the higher impeller in order to obtain high mixing times. The mixing times measured using the decolorization method in this zone were at a maximum.

The two impellers between the injection and detection devices were chosen to dampen the concentration oscillations in the region of scrutiny; this simplifies the comparison between the mixing times measured using the chemical and physical methods. As shown in Section 2, in the case of the absence of oscillations

Fig. 4. Mixing times obtained using chemical and physical methods vs. δ $(N=200 \text{ rev min}^{-1})$.

$$
t^*_{\delta s} = t_{\delta s}
$$

From the measurements obtained using the physical method, it was verified that this condition was satisfied. The results obtained for both methods are given in Fig. 4.

Fig. 4 shows that the correspondence between the mixing times obtained using the two methods is satisfied within the limits of experimental error. This result implies that, in the chemical method, the mixing time corresponding to the minimum specific conductivity coincides with $t^*_{\delta s}$; the effect of non-homogeneities in the region of scrutiny is therefore negligible with respect to the measurement error. The satisfaction of this condition provides useful information. The mixing time measured using the chemical method corresponds to $t^*_{\delta s}$ only when the region of scrutiny can be considered as being perfectly mixed *(*in this case, Eq. *(*8A*)* is the same as that obtained using a point measurement*)*. As a consequence, in the region of scrutiny the effect due to non-homogeneities is not detectable using conductivity measurements but is hidden by other experimental errors which have a major effect on the measurements. The increase in the mixing time obtained by reducing the volume of the region of scrutiny is no longer experimentally measurable. Therefore, the similarity between t^* ² values, measured using the chemical and physical methods, shows that a reduction in the volume of the region of scrutiny does not lead to a measurable variation in the mixing time.

4. Conclusions

From this experimental and theoretical study on the different methods of measurement of mixing times, it is possible to draw the following conclusions.

- The mixing times measured using physical and chemical methods refer to different definitions of deviation from homogeneity, and both measurements are influenced by the volume of the region of scrutiny.
- In the chemical conductivity method, the time necessary to achieve the minimum absolute average specific conduc-

tivity of the solution in the cell corresponds to the mixing time defined in Eq. *(*12*)* if the volume of scrutiny of the cell can be considered to be perfectly mixed so that Eq. *(*6A*)* is valid.

• The correspondence between the chemical and physical mixing times $t^*_{\delta s}$, measured using the same conductivity cell, implies that a reduction in the volume of the region of the scrutiny does not lead to a measurable variation in the mixing time.

Appendix A

The $Na⁺$ ions injected into the system do not take part in any chemical reaction. This implies that Eq. (8) is valid; therefore

$$
C_{t} = \frac{1 - C_{\text{Na}} + / C_{\text{Na}} + \infty - \delta}{1 - \delta} = \frac{C_{\text{H}} + - C_{\text{OH}}}{C_{\text{HCl}}^{i}} \tag{1A}
$$

By substituting $C_{\text{Na}^+\infty}$ with $C_{\text{NaOH}}^i V_A/V$ in Eq. (1A) and solving for $C_{\text{Na}^{+}}$, we obtain

$$
C_{\text{Na}^{+}} = \frac{C_{\text{NaOH}}^{i} C_{\text{HCl}}^{i}}{C_{\text{NaOH}}^{i} + C_{\text{HCl}}^{i}} \left[1 - \frac{C_{\text{H}^{+}} - C_{\text{OH}^{-}}}{C_{\text{HCl}}^{i}} \right]
$$

= $\alpha - \beta (C_{\text{H}^{+}} - C_{\text{OH}^{-}})$ (2A)

with

$$
\alpha \equiv \frac{C_{\text{NaOH}} C_{\text{HCl}}^{\text{i}}}{C_{\text{NaOH}}^{\text{i}} + C_{\text{HCl}}^{\text{i}}} \quad \beta \equiv \frac{\alpha}{C_{\text{HCl}}^{\text{i}}}
$$

From the electroneutrality condition of the solution, we obtain

$$
C_{Cl^{-}} = C_{Na^{+}} + C_{H^{+}} - C_{OH^{-}}
$$

= $\alpha + (1 - \beta) (C_{H^{+}} - C_{OH^{-}})$ (3A)

In dilute solutions, the specific conductivity is proportional to the concentrations of the ions present, so that, at every point of the system, we obtain

$$
\chi(\vec{x},t) = \frac{F^2}{RT}(D_{\text{H}^+}C_{\text{H}^+}(\vec{x},t) + D_{\text{OH}^-}C_{\text{OH}^-}(\vec{x},t) + D_{\text{Na}^+}C_{\text{Na}^+}(\vec{x},t) + D_{\text{Cl}^-}C_{\text{Cl}^-}(\vec{x},t))
$$
(4A)

By substituting Eqs. *(*2A*)* and *(*3A*)* into Eq. *(*4A*)*, and calculating the averaged integral in the region of scrutiny, we obtain

$$
\chi_{\rm s} = \frac{F^2}{RT}(K_1 C_{\rm H+s} + K_2 C_{\rm OH-s} + K_3) \tag{5A}
$$

with
$$
K_1 \equiv D_{H^+} + \gamma
$$
 $K_2 \equiv D_{OH^-} - \gamma$

$$
K_3 \equiv \alpha (D_{\text{Na}^+} + D_{\text{Cl}^-})
$$
 $\gamma \equiv D_{\text{Cl}^-} - \beta (D_{\text{Na}^+} + D_{\text{Cl}^-})$

From the ionic product of water, using the second order Taylor expansion of the reciprocal of C_{H^+} in C_{H^+s} , and calculating the averaged integral in the region of scrutiny, the second term becomes equal to zero and an equation formally equal to the local expression is obtained

$$
C_{\text{OH-s}} \cong \frac{k_{\text{w}}}{C_{\text{H+s}}} \tag{6A}
$$

By substituting Eq. *(*6A*)* into Eq. *(*5A*)*, and by imposing the time derivative of χ _s equal to zero, we obtain

$$
\frac{d\chi_{s}}{dt} = \frac{F^{2}}{RT} \left(K_{1} - K_{2} \frac{k_{w}}{C_{H+s}^{2}} \right) \frac{dC_{H+s}}{dt} = 0
$$
\n(7A)

Eq. *(*7A*)* implies that the time derivative of the average specific conductivity is equal to zero in two cases:

- 1. when the hydrogen ion accumulation rate (dC_{H_g}/dt) is equal to zero, meaning that in the region of scrutiny, the inlet net flux of hydrogen ions will be equal to their conversion rate;
- 2. when the value of C_{H^+s} is equal to $C_{\text{H}^+s}^*$

$$
C_{\mathrm{H}^{+}\mathrm{s}}^* \equiv \sqrt{\frac{K_1}{K_2}} k_{\mathrm{w}} \tag{8A}
$$

The value of $C_{\text{H}^+\text{s}}^*$ is very close to $\sqrt{k_{\text{w}}}$, and therefore the average specific conductivity has achieved its minimum value and $C_{ts} \cong 0$. By considering the concentration values used in Section 3, with the diffusion coefficients at 18 $^{\circ}$ C, we obtain

$$
C_{\text{H}^+ \text{s}}^* = 0.7 \times 10^{-7} \text{ eq dm}^{-3} \Rightarrow C_{\text{rs}} = -0.4 \times 10^{-7} \approx 0
$$
\n(9A)

Appendix B. *Notation*

 $\,$ 1 $\,$

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