

The Iodide Iodate Method to Characterize Microstructured Mixing Devices

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The iodide iodate method (also known as Villiermaux method) was used to characterize multilamination-type mixers, manufactured at the Institute for Micro Process Engineering of the Forschungszentrum Karlsruhe. Mixing sensitive results were obtained using different reactant concentrations. It is shown that the experimentally obtained data cannot be treated as quantitative data in sense of a "mixing quality." © 2008 American Institute of Chemical Engineers *AICHE J*, 54: 639–645, 2008

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Introduction

Effective mixing is of major importance in many fields in chemical engineering. The conversion and selectivity of chemical reactions in the liquid phase might heavily depend upon mixing. With the same mechanism, mixing devices and processes can be characterized by the use of competitive (either consecutive or parallel) reaction schemes. There are two prominent examples: the azo dye stuff coupling of 1-naphtol and 2-naphtol with diazotized sulfanilic acid¹ and the so-called iodide iodate reaction scheme.^{2–7} Both systems were originally developed for the characterization of macroscopic dimensioned stirred tanks.

For static microstructured mixers, the azo dye stuff coupling method had been applied to devices similar to those presented in this publication.^{8,9} Further work using this characterization method on microstructured mixing devices has not been published. Furthermore, bromination of an aromatic compound was used to characterize a microstructured cyclone-type mixer.¹⁰ All other publications on the characterization of microstructured mixing devices with competing chemical reactions use the iodide iodate reaction system.^{11–18} The present publication reports on the characterization of micro mixing devices with the iodide iodate method with a

special emphasis on the impact of the reactant concentrations on the sensitivity of the method. This issue has not been investigated in detail in previous publications.

Description of the Iodide Iodate Method

The iodide iodate reaction scheme, also named the "Villiermaux/Dushman method"¹⁶ or "Villiermaux-reaction"¹⁸ was first proposed in 1991.⁷ It is based on the parallel competition of two reactions:

(a) The neutralization of dihydroborate ions



(b) and the comproportionation reaction of iodate and iodide to iodine (also named the "Dushman reaction"¹⁹)



Reaction 1 is virtually instantaneous. Reaction 2 is also fast but by orders of magnitude slower than reaction 1. In a perfectly mixed system, the product distribution is solely governed by the chemical kinetics resulting in very small iodine yields. Sulfuric acid is mixed with a solution of boric acid, sodium hydroxide, potassium iodate, and potassium iodide. The occurrence of a significant amount of iodine is attributed to a local excess of H^+ , that is, when all H_2BO_3^-

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in this local volume is consumed by reaction 1, there is still H^+ left for reaction 2. By the amount of iodine produced, mixers can be compared qualitatively. When suitable model ideas of mixing processes like the incorporation model for stirred vessels are available, quantitative treatment of experimental results of competing reactions is possible.²⁰

The iodide iodate method was originally developed for the investigation of mixers of macroscopic size, where a small volume of concentrated sulfuric acid is injected in a large stirred volume containing the rest of the chemicals over a long time (typical several hundreds of seconds; dV_1/dt is very small) in order to ensure micro mixing controlled results (Figure 1a).

In 1999, Ehrfeld et al. reported a first attempt to adapt the method for microstructured mixing devices, where two fluids streams in a ratio of 1:1 are mixed, one containing diluted sulfuric acid and the other fluid stream, the other essential chemicals (Figure 1b, $dV_1/dt = dV_2/dt$).¹² As opposed to the originally proposed boric acid buffer, an acetic acid buffer system was used, which adjusts the pH value in the resulting mixture, where iodine is formed after the mixing process is completed.²¹

Panic et al. have proposed initial concentrations for the chemicals (containing the original borate buffer system), suitable to characterize microstructured mixer devices in order to obtain maximum sensitivity of the method and a final pH of the resulting mixture that produces stable iodine concentrations.¹⁶

The iodine produced according to Eq. 2 reacts with excessive iodide according to Eq. 3. The temperature dependence of the chemical equilibrium constant (Eq. 4) is given by Eq. 5.²²



$$K_B = \frac{[I_3^-]}{[I_2][I^-]} \quad (4)$$

$$\log_{10} K_B = \frac{555}{T} + 7.355 - 2.575 \log_{10} T. \quad (5)$$

The value of K_B for a temperature of 20°C is 787 L/mol.

The triiodide concentration in the resulting mixture can be detected by means of UV/VIS spectrometry. The spectrum of

the triiodide ion exhibits two distinct maxima at 290 and 353 nm.²³ In most of the published literature on the iodide iodate method, the absorption value at a wave length of 353 nm is used for analytical purposes. The relation between the absorption values, A , the molar absorption coefficient at a wave length of 353 nm, ϵ_{353} , and the concentrations of triiodide, $c_{I_3^-}$, is given by Beer's law (Eq. 6).

$$c_{I_3^-} = A/\epsilon_{353}l. \quad (6)$$

Considering the chemical equilibrium (Eq. 4), the total amount of iodine (= iodine in form of iodine and triiodide molecules, $I_2 + I_3^-$) can be calculated. With the help of the derived concentrations, a "segregation index," X_S , can be calculated (Eq. 7)

$$X_S = \frac{Y}{Y_{ST}} = \frac{c_{I_3^-} + c_{I_2}}{c_{H_0^+}} \cdot \left(2 + \frac{c_{H_2BO_3,0}}{3 \cdot c_{IO_3^-,0}} \right). \quad (7)$$

The segregation index^{3,24} divides the iodine produced (Y) by the maximum yield (Y_{ST}) in the case of worst mixing, named state of total segregation, based on the model idea that a certain number of partial volumina are perfectly mixed (V_{PM} , no iodine is produced), while in the other partial volumina solution 1 and 2 are totally segregated (V_{ST} maximum of iodine is produced).

Using the segregation index X_S , another model parameter can be derived: the "micro mixedness ratio" α (Eq. 8) given by the ratio between perfectly mixed volumes (V_{PM}) and volumes with total segregation (V_{ST}).^{2,5}

$$\alpha = \frac{V_{PM}}{V_{ST}} = \frac{1 - X_S}{X_S}. \quad (8)$$

The link between the micromixedness ratio (experimentally determined by utilizing the iodide iodate reaction scheme) and the micro mixing time (experimentally determined by optical methods like Laser Doppler Velocimetry and Particle Image Velocimetry) for stirred vessels is purely empirical and shown by Rousseau et al.²⁴ (using the concentrations proposed by Fournier et al.³) to be represented by Eqs. 9–11.

$$2 < \alpha < 5 \text{ for } t_m = 0.73 \alpha^{-2.26} \quad (9)$$

$$5 < \alpha < 7 \text{ for } t_m = 0.82 \alpha^{-2.3} \quad (10)$$

$$2 < \alpha < 20 \text{ for } t_m = 0.158 \alpha^{-1.45}. \quad (11)$$

Since there is no link between model ideas of mixing in microstructured mixers and the experimentally determined triiodide concentrations, most of the published literature on the application of the iodide iodate method on microstructured mixers solely gives the pure spectrometer signal (absorption A) except Rakoczy et al.¹⁸ and Men et al.¹⁴ This contribution only uses the absorbance A . It has been shown for stirred vessels that the calculated segregation index is a function of the concentration of the chemicals used.³ So, the segregation index is no quantitative measurement of a "mixing quality," as claimed in literature.¹⁴ Different devices can only be compared qualitatively, when measuring at the same concentrations. It is not allowed to compare absorbance

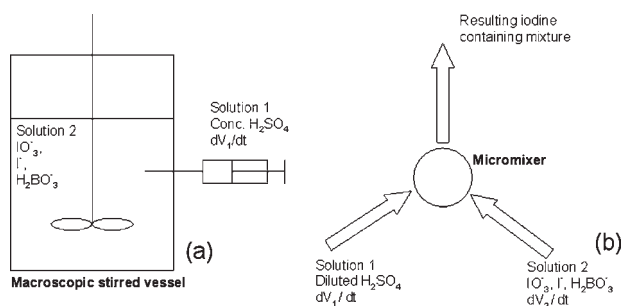


Figure 1. Scheme of applying the iodide iodate method on macroscopic stirred tanks (a) and scheme of applying the iodide iodate method on microstructured mixing devices (b).

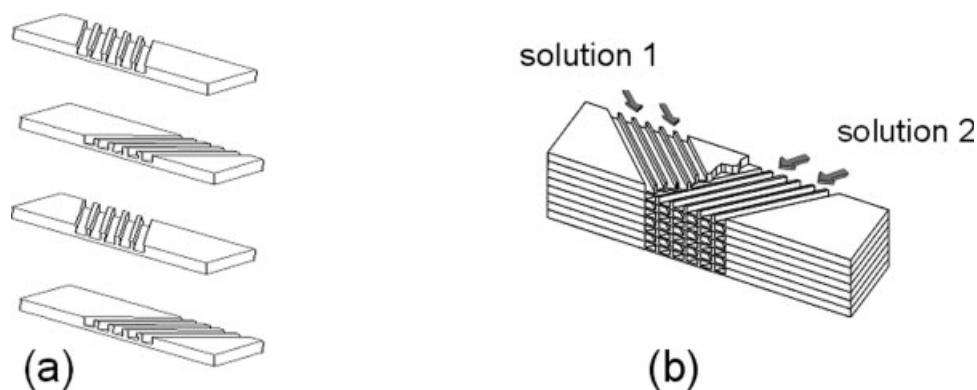


Figure 2. Construction principle of microstructured V-type mixers: (a) stainless steel foils with micro channels and (b) stack of stainless steel foils.

values measured at different reactant concentrations or different buffer systems.¹³ Comparison can be achieved utilizing the pure spectrometer signal (absorbance A). Another major drawback using the segregation index X_S as opposed to the spectrometer signal is that only measurements with absorbance values below 2 ($A < 2$) can be used, since the quantification of triiodide concentration is prerequisite to the determination of the segregation index. Absorption values above 2 are out of the range of Beer's law,²⁵ although there is a defined functional dependence even at higher values of the absorption. This contribution uses the absorbance value A for comparison purposes.

Description of the Micro Mixing Devices

In this work, mixing devices made by the Karlsruhe Research Center have been used.

Figures 2a, b show the construction principle of the examined V-type mixer inlays. The microchannels were micro machined in stainless steel foils (Figure 2a). Those micro-machined foils were stacked, and the stack was diffusion bonded (Figure 2b). Mixing of liquids^{8,9,18} and gases²⁶ is performed in the exit of the microchannel system. Liquids are filtered with a mesh of 40- μm width.

Mixing occurs by diffusion of the species into adjacent fluid layers. In the case of V-type mixers, mixing is enhanced by disturbing the exiting fluid layers mutually. The theoretical thickness of those layers is as big as the thickness of the foils used (200 μm for the case of the mixing inlays used for this study yielding an effective diffusion path of maximally 100 μm for a given molecule). The top and the bottom layer are different regarding the theoretical diffusion path of the species.

Figure 3a shows a photo of the diffusion-bonded foil stack, which serves as an inlay for a flange adapter (Figure 3b). This flange adapter can be conveniently attached to an experimental setup. Additionally, the outlet of the flange adapter serves as a mixing chamber at the exit of the microchannel system (8-mm diameter).

The examined V-type mixer inlays denoted s200-12foils, s50-12foils, s100-6foils, s100-24foils, and s100-12foils differ in the geometric parameters of the microchannel system. The geometric parameters of the mixer inlays are given in Table 1.

The parameters given in Table 1 are the channel width b , the channel height h , the number of channels per foil z_k , the fin width s separating the channels, the bottom thickness d and the number of foils per inlay stack z_f . Figure 4 shows a

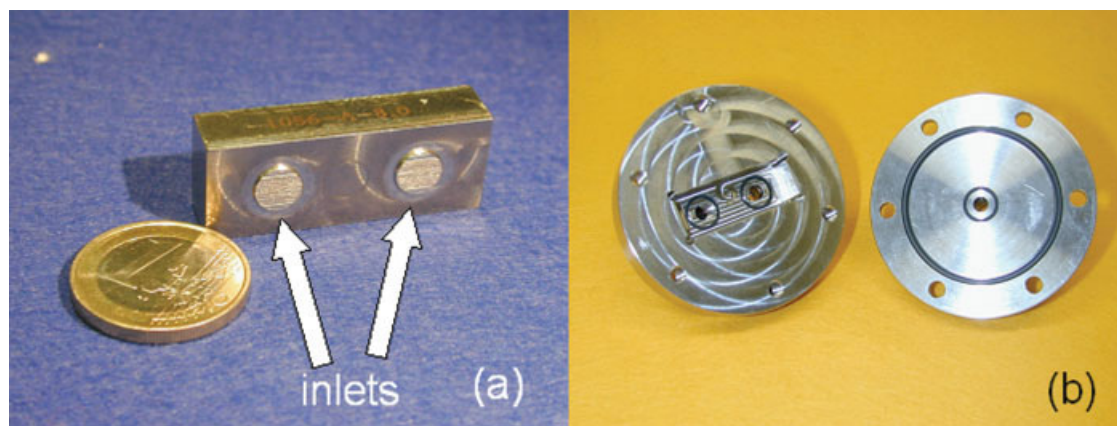


Figure 3. V-type mixer inlay (a) and flange adapter (b).

[Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Table 1. Geometric Parameters of the Examined V-type Mixer Inlays

Device Number	b (μm)	h (μm)	z_k	s (μm)	d (μm)	z_f
S200-12foils	100	70	10	200	130	12
S100-12foils	100	70	10	100	130	12
S50-12foils	100	70	10	50	130	12
S100-6foils	100	70	10	100	130	6
S100-12foils	100	70	10	100	130	12
S100-24foils	100	70	10	100	130	24

SEM of the exit of a mixer inlay with some of the geometric parameters.

The channels of all micro mixer inlays possess the same geometry with respect to height, width, and length. Furthermore, all micromixer inlays have 10 channels per foil and the bottom thickness is $130\ \mu\text{m}$ for each channel. The inlays differ in the numbers of foils per stack, which are 6 (s100-6foils), 12 (s100-12foils), and 24 (s100-24foils) at a constant fin width of $100\ \mu\text{m}$. Furthermore, the fin width was varied at a constant number of 12 foils per stack. Mixer inlays with a fin width of $50\ \mu\text{m}$ (s50-12foils), $100\ \mu\text{m}$ (s100-12foils), and $200\ \mu\text{m}$ (s200-12foils) have been examined. Variation of the parameters “channel number” or “number of foils” and “fin width” at constant values of the other set of parameters allow for the examination of their influence on the mixing performance of the V-type mixers.

In addition to the V-type mixers, a parallel type mixer has been examined.

The construction principle is shown in Figure 5a. The P-type mixer consists of 10 foils (five for each passage) containing 10 channels each. The cross section of each channel is $100\ \mu\text{m} \times 100\ \mu\text{m}$. The channels are separated by fins of $s = 100\ \mu\text{m}$. The bottom thickness is $100\ \mu\text{m}$.

Description of the Test Apparatus and Operating Conditions

Figure 6 shows a schematic of the experimental setup. The test apparatus was constructed of stainless steel. The reaction media are pumped with micro annular gear pumps (HNP Mikrosysteme GmbH, type 7206). The mass flow rates are

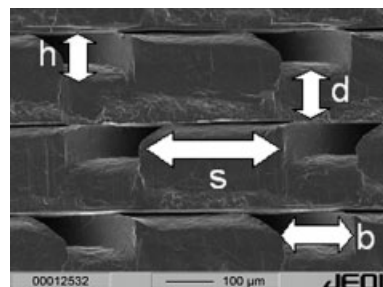


Figure 4. SEM of the exit side of a mixer inlay.

controlled with Coriolis mass flow meters (Endress and Hauser, Promass 63). Temperature control prior to the mixing element was achieved using a refrigerated circulating bath (Thermo Haake, DC 50/K20) with a fluid circulating through micro heat exchangers. The feeds were combined at a temperature of 20°C , using the same mass flow for each feed, in a microstructured mixing device. Mixing was always achieved against atmospheric pressure. The pressures were measured by pressure transmitters (Bourdon and Haenni E 913). The resulting mixtures were collected at the mixer exit and examined in a quartz cell (Hellma type 110 QS) using a UV/VIS spectrometer (Hewlett-Packard HP 8453).

The concentration of the reactant used to prepare the solutions are listed in Table 2. The abbreviation 1SC represents the “standard concentration” used for the characterization experiments. At all other solutions mentioned, use multiple concentrations of those compounds (e.g., twice the concentration for each compound in 2SC and thrice the concentration of each compound in 3SC).

Experimental Results and Discussion

Sensitivity of the method

Varying the reactant concentrations using the iodide iodate method on characterizing mixing devices, we observed that the concentration of the chemicals used has a significant impact on the sensitivity of the method.

Figure 7 shows the absorbance of resulting mixtures at a wave length of $353\ \text{nm}$ using the mixer inlay s100-24foils,

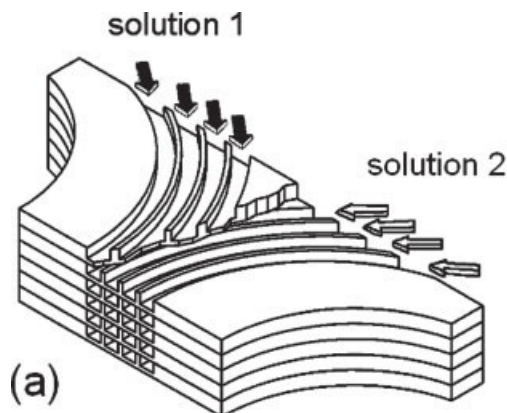


Figure 5. Construction principle (a) and photo (b) of parallel mixer (P-type mixer).

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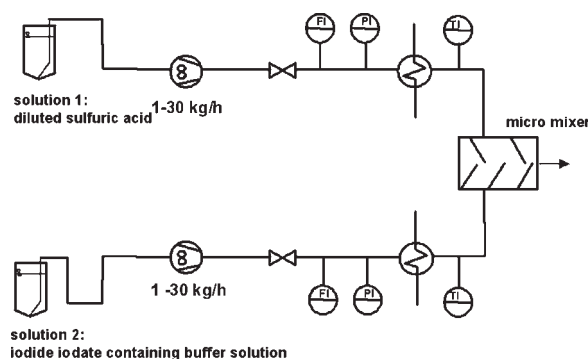


Figure 6. Schematic of the experimental setup.

measured at various reactant concentrations at total mass flow of 9 and 18 kg/h, respectively. It can be clearly seen in Figure 7, that increasing the reactant concentrations increases the sensitivity of the method.

Variation of reactant concentrations

The experimental results obtained using the “standard concentrations” (1SC) given in Table 2 are presented in Figure 8, where the experimentally obtained absorbance (A) values are given as a function of the total mass flow.

The experimental results obtained using twice the “standard concentrations” (2SC) (see Table 2) are presented in Figure 9.

As can be seen from Figures 8 and 9, the variation in reactant concentrations has a major impact on the results of the mixing experiment. Using the standard concentration (1SC), the results for mixers s200-12foils, s100-12foils, and s50-12foils are nearly indistinguishable suggesting independence of “mixing efficiency” from geometrical parameters. However, if double the standard concentration is used (2SC), the experimentally obtained values for different mixing inlays differ much more (see S50-12foils vs. S100-12foils) compared to using the lower concentration. Choosing inappropriate reactant concentrations might lead to a wrong interpretation when comparing two mixing devices.

Variation of the parameter channel number

Figure 10 compares the experimentally obtained absorption values of mixer inlays, which possess the same geometrical channel parameters (b , h , s , d , z_k ; see Table 1) but differ in number of microstructured foils (i.e., the total number of channels). The compared mixers show the same experimental

results over linear fluid velocity. A similar match with a slightly higher scattering of the experimental values was obtained using twice the standard concentration. Measuring at higher reactant concentrations (e.g., 3SC or 4SC) leads to triiodide concentrations in the resulting mixtures, which are too high to be within the measurement range of the UV/VIS spectrometer for all devices except s100-6foils using thrice the standard concentration (3SC). Since the fluid elements have the same linear momentum and the same energy dissipation rate is achieved, it can be claimed that the mixers can easily be scaled by adding foils. This is an important point regarding the portability of laboratory experiments into industrial application.

Variation of the parameter fin width

The comparison of mixers with different fin widths at a constant number of channels is shown in Figure 11 (1SC) and Figure 12 (2SC). The experimentally obtained differences are rather small using the standard concentration (Figure 11). It can be shown that choosing higher reactant concentrations (Figure 12), the difference in mixing performance of the devices used is clearer shown (especially in case of comparing the mixer inlays s50-12foils and s100-12foils). Furthermore, it can be shown that the bigger the fin widths, the better the achieved mixing results. A possible explanation for this phenomenon could be that larger fin widths are beneficial for the formation of eddies that enhance mixing between the channel exits. Further fluid dynamic calculations might elucidate that assumption.

Comparison of a parallel mixer vs. a V-type mixer

Furthermore, the mixer inlay s100-6foils was compared with the P-type mixer described above using thrice the standard concentration (3SC), because both devices cannot be compared using twice the standard concentration given in Table 2 (Figure 13). Taking only the measurements at twice the standard concentration (2SC) into consideration, it could be concluded that both devices mix equally well at the same rates of the mass flow (open symbols). Using thrice the standard concentrations (filled symbols), different mixing properties of both devices are revealed. This example shows clearly that the data obtained experimentally cannot be treated as quantitative data.

The exiting jets from the P-type mixer form more or less closed layers of fluids with a thickness of the foil thickness. Presuming perfect channels (which can be done for machined channels), the distribution perpendicular to the flow axis of the fluids should be near homogenous.

Table 2. Concentrations of the Reactants in the Feed Streams Used for Applying the Iodide Iodate Method on Mixing Devices

	Standard Concentration “1SC” (mol/L)	Twice the Standard Concentration “2SC” (mol/L)	Thrice the Standard Concentration “3SC” (mol/L)
Solution 1			
Sulfuric acid H_2SO_4	0.015	0.03	0.045
Solution 2			
Potassium iodide KI	0.016	0.032	0.048
Potassium iodate KIO_3	0.003	0.006	0.009
Sodium hydroxide NaOH	0.045	0.900	0.945
Hydroboric acid H_3BO_3	0.045	0.900	0.945

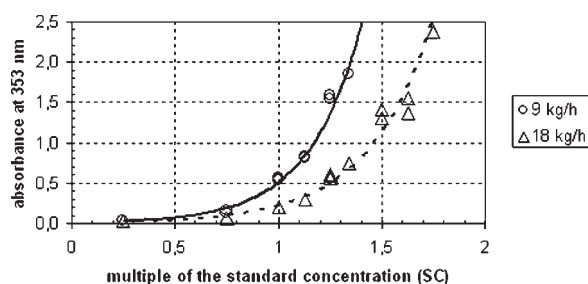


Figure 7. Absorbance over reactant concentrations at constant total mass flow rates.

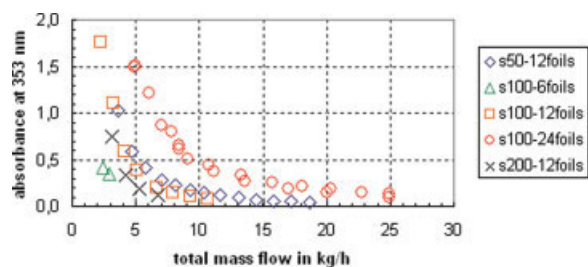


Figure 8. Experimental results obtained by utilizing the “standard concentrations” (1SC).

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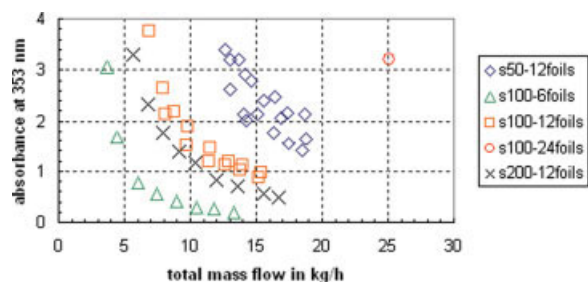


Figure 9. Experimental results obtained by utilizing twice the “standard concentrations” (2SC).

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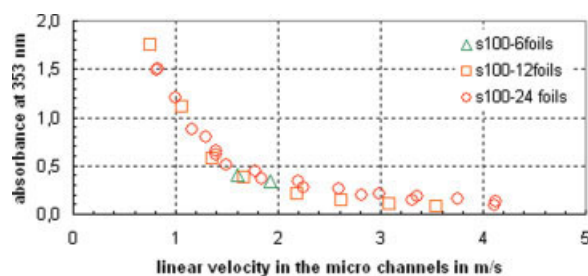


Figure 10. Comparison of mixer inlays with different numbers of channels with respect to fluid velocity in the micro channels at the “standard concentration” (1SC).

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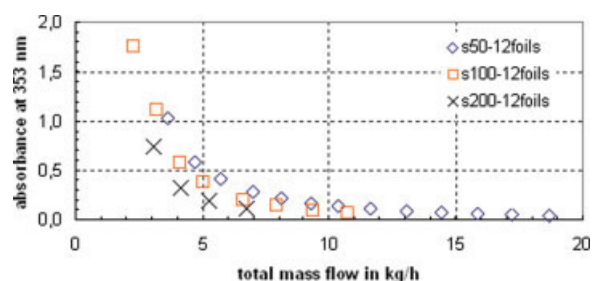


Figure 11. Comparison of mixer inlays with different fin widths s at the “standard concentrations” (1SC).

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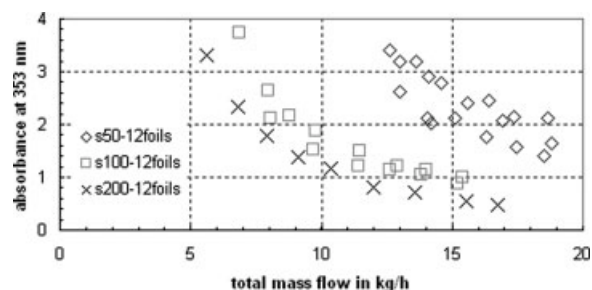


Figure 12. Comparison of mixer inlays with different fin widths s at twice the “standard concentrations” (2SC).

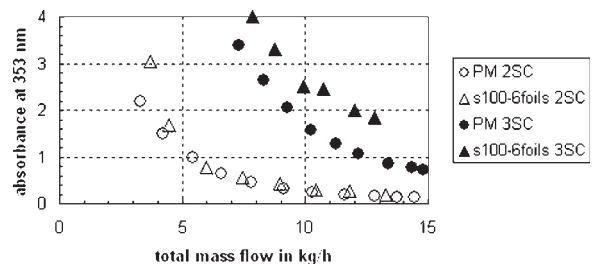


Figure 13. Comparison of a P-type and a V-type mixer at two different concentrations (2SC and 3 SC).

The fluid jets exiting the V-type mixer form an angle of 90° . An additional force is expected coming from the fluid dynamics of this angle.

However, experimental results show a better performance of the P-type mixer. This could be due to the fact that only 10 channels run in parallel. A boundary effect could occur, that the outer channels of the V-type mixer are not experiencing the additional mixing force and pointing away from the general flow axis that leads to an excess of each fluid at the respective wall of the mixing chamber. Further experiments in estimating this effect are in course.

Conclusion

Multilamination type mixers have been examined utilizing the iodide iodate reaction scheme at three different reactant concentrations.

It is shown that different experimental results for structurally different V-type mixers can be attributed to structural details (viz. the total channel number and the fin width of the mixer inlays).

Furthermore, it is shown that the concentration of the chemicals used needs to be carefully chosen in order to obtain optimal characterization of the examined devices. Good mixing devices require higher concentrations of the chemicals used to achieve mixing sensitive results. At high flow rates and low reactant concentrations, little iodine is formed and no distinct maxima are found in the UV spectrum. The worse a device is mixing at given flow rates, the lower the reactant concentrations are that are required for a full characterization due to the upper detection limit of UV/VIS spectrometers. High concentrations lead to more sensitive results. Devices that cannot be distinguished at lower concentrations (standard concentration) can be distinguished at higher concentrations (twice or thrice the standard concentration).

Comparing the V-type mixer s100-6foils at constant mass flow rates with a P-type mixer, it is clearly shown that the obtained spectrometer signals cannot be treated quantitatively in the sense of a "mixing quality." The use of higher reactant concentrations reveals the better mixing performance of the P-type mixer at constant mass flow rates.

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