Static mixers: Mechanisms, applications, and characterization methods – A review

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A B S T R A C T

Static mixers and multifunctional heat exchangers/reactors (MHE/R) are qualified as efficient receptacles for processes including physical or chemical transformations accompanied by heat transfer due to their high productivity and reduced energy expenditures. The present work reviews recent conceptual and technological innovations in passive static mixers and continuous in-line reactors. Current industrial applications are discussed from a process intensification perspective, focusing on mixing and mass transfer performance. Typical experimental techniques employed to characterize and quantify the mixing process are explored. The work is complemented by a review of mixing fundamentals, knowledge of which allows the development of theoretical models crucial for the analysis of experimental data, like the chemical probe mixing assessment method. Considering the development of continuous flow equipment in numerous processes, advances in this field will certainly be of increasing interest to the scientific and industrial communities.

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1. Introduction

Static mixers and multifunctional heat exchangers/reactors (MHE/R) are being increasingly incorporated in process industries for their mixing and heat transfer capabilities. Process intensification is a chemical engineering purpose that consists in seeking processes with higher productivity, safer operating conditions, reduced waste production, and lower energy consumption. New applications are being explored and new on-line exchanger/reactor designs are being developed offering several advantages compared to batch processing and mechanically stirred vessels. The small space requirement, low equipment operation and maintenance costs, sharp residence time distribution, improved selectivity through intensified mixing and isothermal operation, byproduct reduction, and enhanced safety are the main features that have promoted the use of these devices in chemical, pharmaceutical, food processing, polymer synthesis, pulp and paper, paint and resin, water treatment, and petrochemical industries (Anxionnaz et al., 2008; Bayat et al., 2012; Ferrouillat et al., 2006a,b; Shi et al., 2011; Thakur et al., 2003).

Characterizing mixing in industrial processes is an important issue for various economic and environmental considerations (Anxionnaz et al., 2008; Lobry et al., 2011; Stankiewicz and Moulijn, 2000) since it governs byproduct effluents and consequently process efficiency. In addition, due to the wide range of applications of mixers and micro-structured mixers, such as homogenization, chemical reaction, dispersion and emulsification, and heating or cooling processes, the mixing efficiency in these devices is a decisive criterion for overall process performance. Indeed, mixing affects various process parameters including heat and mass transfer rates, process operating time, cost and safety, as well as product quality.

To describe the mixing mechanism, Fournier et al. (1996a) and Baldyga and Bourne (1999) introduced macromixing, mesomixing and micromixing as three parallel mixing stages of different scales. Macromixing concerns homogeneity at the reactor scale and is generally described by the residence-time-distribution (RTD) method (Castelain et al., 1997; Habchi et al., 2009a; Villermaux, 1986) as a signature of velocity field uniformity. At the intermediate scale, mesomixing reflects the coarse-scale turbulent exchange between the fresh feed and its surroundings governed by turbulent fluctuations, so it is characterized by the RMS of velocity fluctuations or the turbulent kinetic energy (TKE) (Habchi et al., 2010), and the length scale of these fluctuations. When the fluid aggregates are reduced in size by the turbulent cascade to the Kolmogorov scale, micromixing starts by engulfment in the smallest vortices; it is then achieved in the viscous-convective subrange by laminar stretching and folding, associated with thickness reduction by striation, up to molecular diffusion at sub-Batchelor scales that rapidly dissipates the concentration variances (Batchelor, 1953). The turbulence micro-scales are directly related to the turbulence energy dissipation rate ε (Hinze, 1955; Lemenand et al., 2005; Streiff et al., 1997). In this sense, the Kolmogorov scale is a key parameter for the selectivity of chemical reactions in the turbulent regime, since the limiting mechanism of the whole mixing process occurs at the smaller turbulence scale, hence governing species contact at the molecular scale, (Baldyga and Bourne, 1988, 1989, 1999; Baldyga and Pohorecki, 1995; Falk and Commenge, 2010; Guichardon and Falk, 2000; Komori et al., 1991; Villermaux, 1986).

Qualitative investigation of the mixing process using optical techniques can give valuable information on the flow hydrodynamics. However, understanding and quantifying the mixing mechanism is essential in designing industrial processes involving fast reactions that can present characteristic reaction times smaller than the characteristic mixing time. This fundamental property of the turbulent field (Wallace, 2009) can be determined by classical velocimetry methods such as laser Doppler anemometry, particle image velocimetry, or hot-wire anemometry, all of which give access, in three-dimensional space, to the contributions of the turbulent energy dissipation rate. Alternative methods to characterize mixing based on observations of a chemical system have been recently developed, especially by Baldyga and Bourne (1990), Bourne et al. (1992a,b), and Fournier et al. (1996a): Villermaux–Dushman reactions or the iodide/iodate method (Baldyga and Bourne, 1989; Durandal et al., 2006; Dushman, 1904; Guichardon and Falk, 2000; Guichardon et al., 2000; Mohand Kaci, 2007; Oates and Harvey, 2006; Wheat and Posner, 2009). These techniques, called "chemical probe methods", are based on the competition between mixing and well known chemical kinetics by the straightforward observation of reaction selectivity through monitoring the secondary product concentrations. Under optimal conditions, the slowest reaction time is equal to the mixing time. From the knowledge of the mechanism, kinetics, and stoichiometry of the chemical reaction, the local turbulent energy dissipation rate can readily be derived from the measured selectivity by using phenomenological mixing models (Bourne et al., 1992a; Fournier et al., 1996a; Guichardon and Falk, 2000).

The following sections present an overview of static mixers and multifunctional heat exchangers/reactors, their applications and mixing capabilities. Then mixing fundamentals and experimental techniques developed for its assessment are reviewed. The iodide/iodate method based on the concept presented above is then detailed and the adaptive procedure and mixing models are discussed. The final section includes concluding remarks on static mixers, their present state and future opportunities, with comments on the mixing characterization techniques presented.

2. Static mixers for industrial processes

2.1. Distributive mixing in static mixers

A static mixer can be a hollow tube or channel with a specific geometrical construction that influences the flow structure in a manner to promote secondary transverse flows that enhance mass and heat transfer in the cross-section. Another type of static mixer concept is the insert-type configuration in which the typical design is a series of identical, stationary inserts, called *elements*, and that can be installed in pipes, channels, or ducts. The purpose of the elements is to redistribute the fluid in the directions transverse to the main flow, these are the radial and tangential directions. Static mixers divide and redistribute streamlines in a sequential fashion using only the pumping energy of the flowing fluid.

The inserts can be tailored and optimized for particular applications and flow regimes. Commercial designs typically use standard values for the various parameters that provide high performance throughout the range of possible applications.

Static mixers were not widely used in the process industry before the 1970s, although some patents are much older. A patent dating to 1874 describes a single-element, multi-layer motionless mixer used to mix gaseous fuel with air (Sutherland, 1874); An early French patent used staged/helical elements to promote mixing in a tube (Les Consommateurs de Petrole, 1931), and another shows a multi-element design for blending solids (Bakker, 1949). In the early 1950s, staged elements designed to promote heat transfer were patented (Lynn, 1958). Since then, major petrochemical companies made development efforts and presumably used their own designs, before any commercialization (Stearns, 1953; Tollar, 1966; Veasey, 1968).

There are more than 2000 U.S. patents and 8000 literature articles describing motionless mixers and their applications (Thakur et al., 2003). Nowadays, static mixers have become standard equipment in the process industry. They are used in continuous processes as an alternative to conventional agitation since similar and sometimes better performance can be achieved with lower cost. Motionless mixers typically exhibit lower energy consumption and reduced maintenance requirements because they do not include moving parts. They require smaller space, lower equipment cost, and no power except pumping. They can provide homogenization of feed streams with a minimum residence time and can be manufactured from most materials of construction so as to meet various standards and to adapt with harsh working conditions.

However, stirred vessels remain powerful tools in process industry and find vast applications especially for processing highly viscous products (Aubin and Xuereb, 2006; Cabaret et al., 2007). Numerous recent studies investigate their hydrodynamics with Newtonian as well as rheologically complex fluids (Alliet-Gaubert et al., 2006; Aubin et al., 2000, 2001; Fangary et al., 2000; Torré et al., 2007). New impeller and mixing vessel configurations and innovative operating methods are
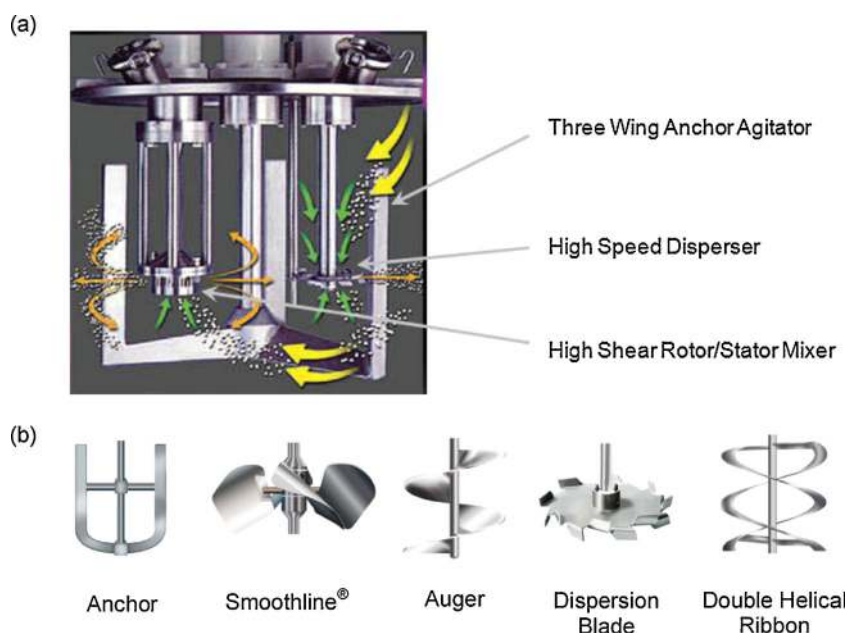
Fig. 1 – Stirred-tank reactor batch mixing, (a) Model VMC-100 multishaft vacuum mixer (Ross Engineering, Inc.) and (b) advanced impeller designs (Robbins & Myers, Inc.).
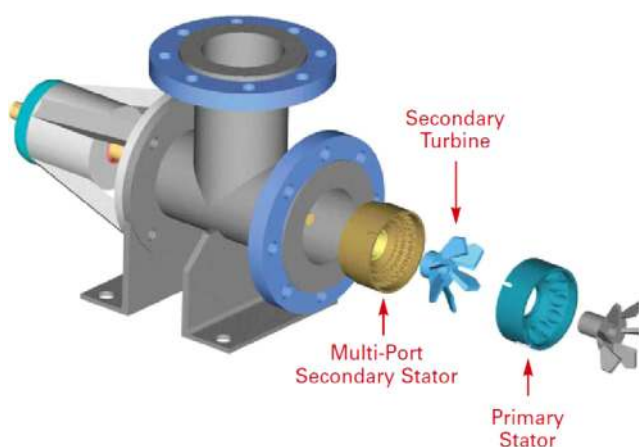

Fig. 2 – Greerco Pipeline Mixer (Chemineer, Inc.): a hybrid high-shear continuous in-line mixer with mechanically-driven parts.

being introduced to enhance their mixing efficiency, safety, and overall productivity (Aubin et al., 2006; Fentiman et al., 1998; Torré et al., 2008). Multishaft batch mixers (Fig. 1a) can simultaneously carry out agitation, dispersion, and high-shear emulsification operations. Fig. 1(b) shows modern impeller designs optimized for specific considerations including high shear requirement, energy efficiency, close clearance, particle size constraints, high viscosity and rheological sensitivity, fouling potential, cleanability, interchangeability, and ease of installation.

In addition, some processes require high productivities that cannot be provided by batch mixers, together with high shearing levels that exceed the capabilities of in-line static mixers. For this purpose, hybrid mixer designs that can be mounted in-line and incorporate mechanically driven parts have been developed (Chemineer Inc., 2004). Fig. 2 shows the Greerco Pipeline Mixer (Chemineer, Inc.) providing the advantages of both continuous and batch processing for demanding applications, though at greater manufacturing and operating costs.

Commercial static mixers are of various types: open designs with helices (Helical Kenics (Chemineer, Inc.),...), open designs with blades or vortex generators (Low Pressure Drop (Ross Engineering, Inc.), Custody Transfer mixer (Komax Systems, Inc.), High-Efficiency Vortex (Chemineer, Inc.),...), corrugated-plates (SMV (Sulzer, Inc.),...), multi-layer designs (SMX and SMXL (Sulzer, Inc.),...), closed designs with channels or holes (Interfacial Surface Generator (Ross Engineering, Inc.),...), or designs based on metallic foam inserts (Ferrouillat et al., 2006c), offset strip fins, or microstructured parallel plates. Fig. 3 illustrates some of the available commercial designs and Table 1 lists the top static mixer manufacturers.

The applications of these devices can range from mixing of miscible fluids to interface generation between immiscible phases (high shear rate dispersive mixing), in addition to heat transfer operation and thermal homogenization.

Motionless inserts such as blades or corrugated plates induce changes in the fluid streamlines. Inserts with holes, channels, helical elements, and oblique blades cause local acceleration and stretching of the fluid. They split the incoming fluid into layers and then recombine the layers in a new sequence. Multilayer designs with blades and baffles split the fluid in multiple layers. These various mixing actions cause distributive mixing, by convection rather than diffusion; although to the extent that distributive mixing is high, diffusion is better able to achieve homogeneity on a molecular scale. The striation thickness (Mohr et al., 1957) is used to quantify distributive mixing in laminar flows and the RTD (residence time distribution) for both laminar and turbulent regimes.

At Reynolds numbers (based on the plain tube diameter) greater than a few hundreds, flow instabilities lead to downstream oscillations and pseudorandom behavior. Even in creeping flow, mixing elements in series asymptotically approach a condition known as chaos where the downstream location of a fluid element becomes essentially unpredictable based on its upstream location.

KMX-V (Chemineer, Inc.)

SMX (Sulzer, Inc.)

Custody Transfer (Komax Systems, Inc.)

Standard LPD (Ross Engineering, Inc.)

Kenics KM (Chemineer, Inc.)

ZT-MX (ZelenTech, Ltd)

SMI (Sulzer, Inc.)

Corrugated channel reactors
(Laboratory pilots)

Fig. 3 – Commercially available in-line static mixers.

The industrial application thus governs the choice of the device. In this sense, laminar and turbulent flows must be distinguished because the basic mixing mechanisms vary widely between the two flow regimes.

2.1.1. Mixing in laminar flows

Laminar flows are encountered in the case of high-viscosity fluids like in food, paint, dairy, cosmetic, polymer, adhesive, and detergent industries. The low levels of mixing

| Table 1 – Commercially available static mixers and their corresponding manufacturers.	
Manufacturer	Static mixer models
Chemineer	KM, HEV, KMX, KME, Thermogenizer, Ultratab, WVM
Sulzer	SMF, SMN, SMR, SMRX, SMV, SMX, SMXL, SMI, KVM, CompaX
Ross Engineering	ISG, LPD, LLPD
Komax Systems	Komax, Custody Transfer, Ozone Mixer, GGM, FRP, Channel Mixer, Triple Action Mixer
Alfa Laval	ART Plate Reactor
Westfall Manufacturing	Model 2800 Plate Type Mixer
Fluitec	CSE-XR
ZelenTech	ZT-MX
Wymbs Engineering	HV, LV
Lightnin	Inliner Series 45, Inliner Series 50
EMI	Cleveland
Bran and Luebbe	N-form
Toray	Hi-Toray Mixer
Prematechnik	PMR
UET	Heliflo (Series, I, II and III)
Noritake	N10, N16, N26, N60
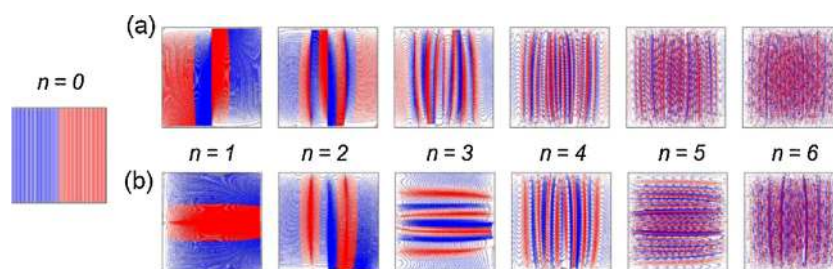
Fig. 4 – Simulations of laminar mixing in two SAR configurations, (a) SAR-1 and (b) SAR-2 with *n* the number of splitting and recombination steps (Ghanem et al., 2013b).

in undisturbed laminar flow have the obvious consequence of giving spatial non-homogeneities in composition. In low-Reynolds-number flows, for viscosity or residence-time purposes, it is necessary to provide solutions based on kinematic mixing through the primary flow topology, such as split-and-recombine reactors (SAR). The concept is based on passive liquid stream division, then rotation in bends of opposite chiralities, and finally recombination, achieving stretching/folding following the baker's transform. Mixing is efficiently ensured by diffusion without generating prohibitive pressure drops. Fig. 4 illustrates the SAR principle in two laminating mixer configurations showing the creation of additional intermaterial area with each step, promoting fluid mixing by molecular diffusion (Ghanem et al., 2013b).

Conventional static mixers are designed to homogenize the fluid by redistributing it in the radial and tangential directions. Undisturbed laminar flow also gives temporal non-homogeneities in the sense that molecules leaving the tube at a given instant have entered at different times. The same fluid redistribution that gives spatial mixing also gives temporal mixing. In the ideal case of plug flow, entering segregated phases will be uniformly redistributed when they leave the mixer, and all the molecules leaving together will have entered together. The extent to which actual static mixers are close to ideal can be characterized using the residence time distribution for temporal variations (Castelain et al., 1997; Aubin et al., 2005) and using the striation thickness distribution for spatial variations (Sokolov and Blumen, 1991; Aubin et al., 2005).

Chaotic advection is a phenomenon that can be created in laminar flows by geometric modifications of classical configurations, for instance the alternation of bend chiralities in simple ducts (Le Guer and Peerhossaini, 1991; Mokrani et al., 1997) in order to obtain complex flow trajectories in which particles follow radial and tangential paths that have been shown to produce global enhancement of transport phenomena (Aref, 1984; Jones et al., 1989; Peerhossaini et al., 1993; Carrière, 2006).

2.1.2. Mixing in turbulent flows

In turbulent flows, motionless mixers promote turbulence and generate intense radial mixing, even near the wall. When the turbulent regime can be attained, eddy diffusion gives sufficient mixing for most industrial processes. Mixer vendors often claim that static mixers can significantly reduce contact time or increase heat transfer compared to a plain pipe. This enhancement is more important for laminar flow, and the relative intensification decreases in turbulent regimes (Ghanem et al., 2013a). It is true, however, that static mixers can increase the level of turbulence without changing pipe diameter and flow rate, albeit with a higher pressure drop. In turbulent flows, motionless mixers are generally used for

process intensification. That is, they allow the same operations to be performed with a somewhat smaller, in-process inventory. Three important applications are gas mixing in the turbulent regimes, blending of aqueous solutions in turbulent flow, especially for water treatment, and blending of polymer melts or solutions. They are also used as reactors, particularly for polymerizations. The presence of inserts and perturbators usually produces a complex vortex system in which concomitant phenomena simultaneously enhance mass and heat transfer. An example of such system is the High-Efficiency Vortex HEV (Chemineer Inc., 1998) static mixer and heat exchanger (Ghanem et al., 2013a; Lemenand et al., 2010). Similarly, Fig. 5 illustrates the different mixing mechanisms in the Komax Triple Action Mixer.

2.1.3. Pure mixing in industrial applications

The most common use of static mixers in industry is in mixing of miscible fluids. Two or more fluids or a reacting mixture are blended to reduce and even eliminate concentration gradients (Goldshmid et al., 1986), for example to incorporate the enzyme in milk in making yogurt. Static mixers can be used for solid blending, in which they are fed by gravity, and for the blending of particulate solids such as cereal grains, bread and cake mixes, and concrete components (Baker, 1991; Bakker, 1949). Spray evaporation, gas mixing and gas/liquid dispersion like ozonation of potable water are common applications in modern industry. Fig. 6 shows static mixer configurations commonly used in gas-handling processes that provide effective contact, small bubble size, and global homogeneity with minimum length requirement (see Fig. 7).

Homogenization in laminar flows is another common process. Additives such as plasticizers and internal lubricants, stabilizers, colorants, fillers and flame retardants are
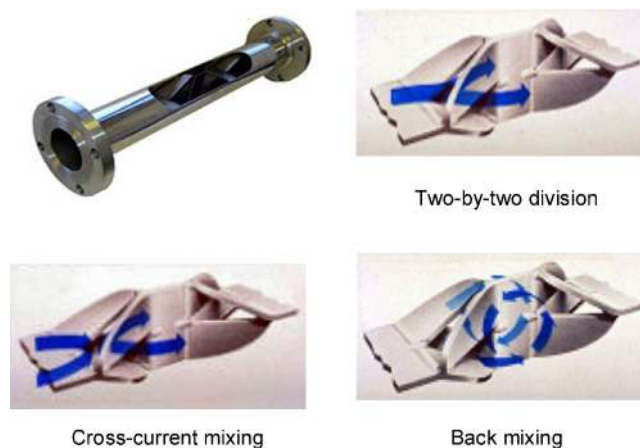

Fig. 5 – Triple action mixer (Komax Systems, Inc.) and the associated mixing mechanisms.

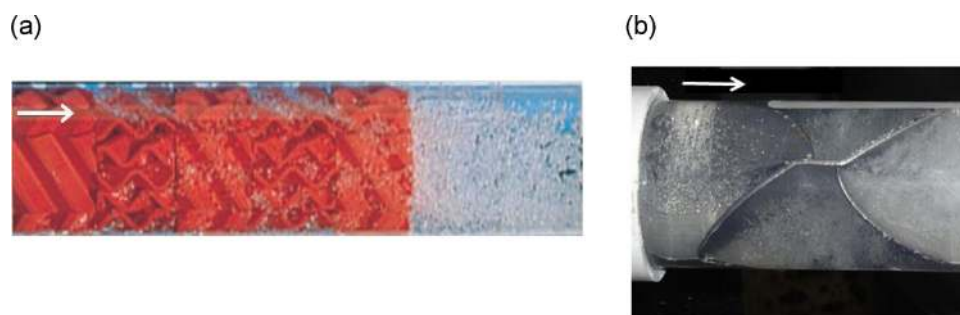Fig. 6 – Static mixer designs for handling gaseous-phase fluids.

Fig. 7 – Gas/liquid dispersion and contacting experiments, (a) Sulzer SMV-type elements and (b) Ozone Mixer (Komax Systems, Inc.).

commonly blended into polymer melts. The compact additive-dosing mixers shown in Fig. 8 provide efficient solutions for many polymer-related processes and many other applications. Secondary fluids are injected into the main flow and rapidly mixed by a combination of vortex shedding and shear zone turbulence. Some of the models can be installed between pipe flanges and feature very small length and space requirements. The typical combination of a gear pump and motionless mixer replaces an extruder at the end of

a polymerization line. Such operations are closely linked to applications of thermal homogenization discussed later since the same device simultaneously homogenizes both concentration and temperature. Motionless mixers are also used to process glues (Schneider et al., 1988), and a familiar household application is the use of disposable static mixers to blend two-part epoxy resins.

Applications of static mixers in the food industry are numerous. Food products are typically highly viscous and

Fig. 8 – Compact static mixers with additive-dosing feature for secondary fluid injection.

SMR for chocolate cooling
(Sulzer, Inc.)

In-line sanitary static mixer
(Komax, Inc.)

SMX plus (Sulzer, Inc.)

Fig. 9 – Insert-type static mixers adapted for the food industry.

non-Newtonian (Holdsworth, 1993) and are usually processed in the laminar regime. Cybulski and Werner (1986) reported that static mixers are used to mix acids, juices, oils, beverages, chocolate, milk drinks, or sauces in food formulations. Fig. 9 shows some static mixers adapted for the food industry. Their surfaces are usually electropolished and they are mostly made from 316 stainless steel and housed in sanitary tubings of similar materials.

Water clarification and sludge treatment can also make use of these devices. Turbidity in potable water is caused by suspended solid particles at low concentrations. Static mixers are used to disperse a flocculating agent, such as alginate, as a first step in clarification. In this case flows are in the turbulent regime, but excessive shear, as might be caused by mechanical agitation, can damage the flocculates, leading to higher consumption of the flocculating agent.

2.1.4. *Mixing in presence of chemical reaction*
Static mixers are also good tools for mixing gases and prevaporized liquid fuels prior to a reaction. Indeed, this is the first recorded use of a static mixer (Sutherland, 1874). Despite the high diffusivities of gases, mixtures do not immediately achieve homogeneity and additional mixing may be needed for good combustion. Increasing the residence time after the gases have been metered together will accomplish the necessary mixing, but this also increases the in-process inventory and can lead to safety problems in the event of a back fire. Additional active mixing is therefore required. Static mixers are often used for pre-reactor feed blending to improve reaction yields. Baker (1991) discussed their use in nitric acid production. Static mixers placed upstream of a reactor to mix air with ammonia increase nitric acid yield and eliminate hot spots that can damage the costly platinum catalyst. Many chemical reactions involving gases can be improved using static mixers, such as those for making vinyl chloride, ethylene dichloride, styrene, xylene and maleic anhydride (Baker, 1991). Static mixers have been reported to have great potential in reducing NO emission in combustors (Braun et al., 1998). A conventional application of a static mixer is found in the nuclear industry to improve sampling and analysis of contaminants in an air flow (McFarland et al., 1999).

Applications of static mixers in polymerization reactions have been suggested (Grace, 1971; Phillips et al., 1997). Sulzer designed a polystyrene process that makes extensive use of motionless mixers, particularly of the SMR type; Tein et al. (1985) report some details of this process. Static mixers are also used in post-reactors and in devolatilization pre-heaters in other polystyrene processes. An academic study on styrene polymerization in a static mixer reactor was reported by Yoon and Choi (1996). Fleury et al. (1992) studied the polymerization of methyl methacrylate while Baker (1991), Khac Tien et al.

(1990), Myers et al. (1997), and Schott et al. (1975) describe the use of motionless mixers in making polystyrene, nylon, urethane and sulfonated compounds.

Most of the applications concern highly exothermic polymerizations. However, the majority of industrial installations concern the reaction injection molding (RIM) of polyurethanes, for which the reaction is moderately exothermic. Commercial RIM machines use an impingement mixer followed by a static mixer to blend the reactive components quickly (Kolodziej et al., 1982). An academic study by Hoefsloot et al. (2001) treated polypropylene degradation in a static mixer-reactor. Other types of chemical reactions can benefit from the use of static mixers. A reactive extrusion process for glycol glucoside synthesis can be improved using static mixers (Subramanian and Hann, 1996). An application to the lactase treatment of whole whey has been reported (Fauquex et al., 1984; Metzdorf et al., 1985), and Lammers and Beenackers (1994) investigated using a static mixer-reactor for producing starch ethers such as hydroxypropyl starch for the food and pulp and paper industry. Grafelman and Meagher (1995) reported liquefaction of starch using a single-screw extruder and a post-extrusion static mixer reactor. Cultivation of attenuated hepatitis A virus antigen in a titanium static mixer is described by Junker et al. (1994). Reaction applications suggested for static mixers include cracking of heavy and crude oils (Jurkias, 1998) or the controlled hydrolysis of whey proteins by trypsin (Margot et al., 1998).

2.2. *Heat transfer in static mixers*

Static mixers constitute a part of a broader family of devices introduced above as multifunctional heat exchangers/reactors (MHE/R) in which mixing, chemical reaction, and heat transfer can occur simultaneously in the same apparatus. Food processing production lines often incorporate such devices for cooking, cooling, sterilization, or pasteurization of food products. They are also used for thermal regulation and heat injection or evacuation in catalysis or esterification processes or any other applications in which heat transfer is coupled with reaction.

Many traditional designs such as stirred tanks already involve heat transfer generally at the vessel wall; however, in these devices there is often a significant distance between the heat-transfer device and the site of the chemical reaction where heat is generated causing considerable thermal non-homogeneities within the vessel making isothermal operation difficult. The aim of the heat exchanger/reactor (HEX reactor) is to eliminate this distance by supplying or removing heat almost as rapidly as it is absorbed or generated by the reaction.

One application is the traditional thermal homogenization and heat transfer in heat exchangers involving viscous

Fig. 10 – **Commercial static mixers qualifying as multifunctional heat exchangers/reactors.**

fluids in the laminar regime, such as polymer solutions. Static mixing elements can also be used in turbulent flow to reduce the exchanger size and increase the intensity of turbulent systems. They can be used for highly exothermal chemical reactions where rapid heat evacuation is critical for safety reasons and product quality (Phillips et al., 1997). These devices function in two ways. They increase the metal to fluid surface area in a manner analogous to the use of externally finned tubes. They also change the hydrodynamics by redistributing the flow within the tube cross-section, and the combination of effects can significantly increase heat transfer coefficients. Fig. 10 shows some static mixer configurations with heat transfer capabilities that thus qualify as HEX reactors.

2.2.1. Thermal homogenization

For an undisturbed laminar flow in a plain pipe, thermal diffusion is the only heat transfer mechanism in the radial direction. A great variety of motionless inserts have been used to promote radial flow and thus reduce radial temperature gradients in process fluids. One common application

is the installation of a static mixer immediately downstream from a screw extruder in order to obtain a thermally homogeneous polymer melt. A typical application of thermal homogenization is for film blowing or sheet extrusion because thermoplastics require a uniform temperature distribution to eliminate position-dependent variations in the product (Chen, 1975). Fig. 11 is a schematic diagram illustrating the use of Sulzer SMV mixing elements in a through-air-drying (TAD) system to provide uniform air temperature, a decisive factor in final product quality.

Schott et al. (1975) suggested using motionless mixers for polyethylene, polypropylene, and polystyrene processing, and such uses of motionless mixers in extrusion have become standard practice. Their main purpose is thermal homogenization but they can also alleviate composition differences resulting from polymer blending and coloring. The need is primarily for radial mixing, but flow patterns in classic extruders produce polymers that lack tangential symmetry. Thus, some tangential mixing is needed as well, and this is automatically provided by most motionless mixers. Myers et al. (1997) reported applications in the turbulent regime to enhance
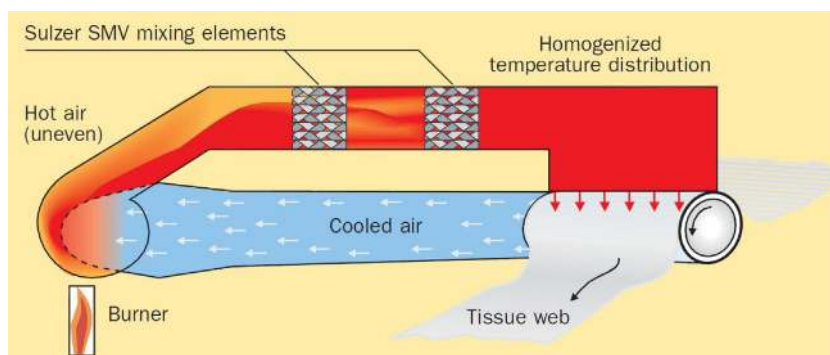
Fig. 11 – Schematic diagram illustrating a through-air-drying (TAD) system incorporating Sulzer Chemtech SMV mixing elements.

thermal homogenization. Some typical uses of static mixers/heat exchangers in the polymer industry include:

- heating and cooling of polymer and polymer solutions;
- heating polymer solutions prior to devolatilization;
- cooling polymer melts before filling plants to avoid thermal damage to packaging;
- melt viscosity adjustment by temperature variation for optimal process conditions;
- cooling polyester melts between the reactor and the fiber spinning unit; and
- cooling polymer melts before granulation to raise the viscosity.

Mixing elements are most beneficial in deep laminar flow (Ishikawa and Kamiya, 1994; Joshi et al., 1995), and most applications have been in this area. Literature descriptions of their use largely concern the reactive flows covered in the next section. Static mixers have a distinct advantage over plain tubes in such applications because they provide a more uniform residence time distribution. If interest is limited purely to heat transfer, inserts specifically designed for this purpose seem better than general-purpose devices (Habchi et al., 2012a). The greatest interest has been in helical twisted tape and offset strip fins (Bergles, 1995). These devices can be used for turbulent flow and boiling heat transfer as well as for laminar flow to improve heat transfer coefficients.

2.2.2. Heat transfer in presence of chemical reactions

In addition to the polymerization, Lammers and Beenackers (1994) suggested using a continuous tubular reactor containing static mixers to produce starch ethers for food and pulp. Static mixers can be used in turbulent flow reactors, for example in the catalyst tubes of a reformer furnace. Static inserts are said to increase heat transfer coefficients, eliminate channeling within the catalyst bed, avoid cocking, prevent catalyst deterioration due to hot spots and improve yield. Applications to condensation (Fan et al., 1978) and boiling heat transfer (Azer and Lin, 1980) have been reported. Another example is provided by Gough and Rogers (1987), who discussed the treatment of coal tar oil residues using static mixer heat exchangers. The residues contain heat-sensitive phenolic compounds that can readily polymerize. Similar residues result from distillate bottoms from naphtha cracking and can eventually include carbon solids. These fluids are preheated before being burned in a reducing atmosphere to produce carbon black. Motionless mixers are potential solutions to many problems encountered in heat exchanger operations. In cooling processes, skinning due to boundary-layer

solidification may be alleviated due to the better radial mixing (Baker, 1991). Fouling in reactive systems is caused by long residence times in the wall vicinity and high temperature differences between the wall and the bulk fluid. Crystallization, polymerization or biological growth may occur, and the resulting films have low thermal conductivity and cause significant resistance to heat transfer. Gough and Rogers (1987) have shown that static mixers can significantly reduce the above effects.

Furthermore, by integrating heat exchanger technology into the static mixer continuous flow, high -ield reactors that go beyond batch reactor limitations to make possible safe, environmentally friendly and cost-effective process intensification can be developed. In modern processing it is important that each individual molecule be exposed to an identical, carefully controlled processing environment in order to ensure consistent product properties and high productivity. The ART Plate Reactor (Alfa Laval) shown in Fig. 12 is an example of such devices; here a serpentine channel design comprising multiple direction changes provides uniform plug flow and residence time distribution. High heat transfer capacity allows optimal thermal control of exothermic reactions, improving safety and product quality.

However, for process intensification, proper optimization should consider the overall expected gain. Mixing inserts have several significant disadvantages compared to plain tubes: higher pressure drop, greater potential for fouling, relative difficulty of cleaning and greater cost. Note that thermal diffusivities are several orders of magnitude higher than molecular diffusivities, so that most heat transfer operations are feasible using tubes of reasonable diameter and length and with reasonable residence times. The use of mixing inserts is justified when there is a strong need to minimize in-process inventory. Examples include the need to suppress detrimental reactions when the process material is particularly dangerous or expensive. Fig. 13 depicts some of the large-scale static mixer and heat exchanger applications where the equipment size intensifies the losses and complicates implementation and maintenance.

The present study focuses on the usage of these devices as static mixers/reactors rather than heat exchangers. The following section introduces the basic mixing mechanisms encountered in the corresponding flows.

3. Mixing fundamentals

Mixing is a central issue in process engineering and many industrial fields. In fact, the way in which reagents are mixed,
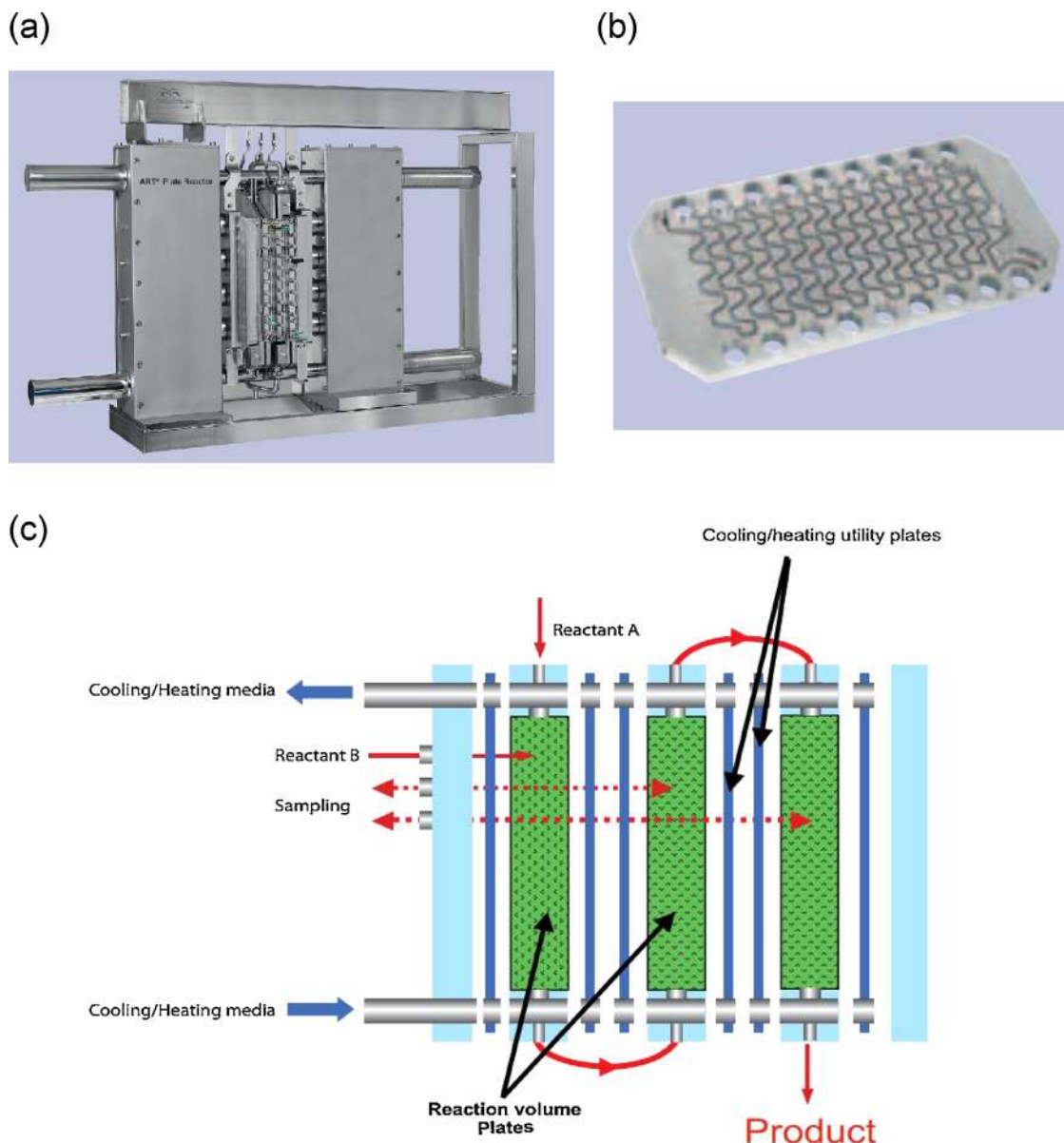
Fig. 12 – ART Plate Reactor 37 (Alfa Laval, AB), (a) global view, (b) serpentine process plate, and (c) working principle.

whether at the reactor scale, affected by the flow structures, or at molecular scales, influences the selectivity and hence the productivity of reactions.

3.1.   Energy analysis

To understand the mixing mechanisms through an energetic approach, we distinguish two mechanisms by which energy is dissipated in turbulent flow: the energy dissipation due to boundary layers at the walls and surfaces of inserts, and the energy dissipation in the bulk fluid, that is the region of approximately homogeneous core turbulence. It is this second form of energy dissipation that is most important in distributive mixing (Goldshmid et al., 1986). Consider an unmixed feed stream containing black and white liquid. Turbulence will quickly intermingle the initially unmixed feed and disperse it down to the size of the smallest eddies. In concept, the fluid in these smallest eddies remains black or white before becoming gray due to molecular diffusion. The size of the

smallest eddies is proportional to the Kolmogorov length scale, λ_K:

$$\lambda_K = \left(\frac{\mu^3}{\rho^3 \bar{\varepsilon}} \right)^{1/4} \tag{1}$$

where μ is the viscosity, ρ is the density, and $\bar{\varepsilon}$ is the power dissipation per unit mass of fluid. The weak dependence on power dissipation, which is to the 0.25 power, means that λ_K varies over a relatively narrow range, from 5 to 50 μm for the great majority of industrial processes. It happens that the constant of proportionality between the Kolomogorov scale and the smallest eddy size is of the order of 1 by definition. Given typical diffusion coefficients for low viscosity fluids (those for which turbulence is possible), complete mixing will occur in milliseconds. Except for very fast reactions, the reaction rate will be governed by the intrinsic kinetics regardless of mixing effects. The total power dissipation, $\bar{\varepsilon}$, is divided into two

SMV mixer for optimized NO$_x$ removal
(Sulzer, Inc.)

Large-scale channel mixer
(Komax Systems, Inc.)

SMR-type heat exchanger for fiber cooling
processes (Sulzer, Inc.)

SMR reactor for polystyrene synthesis
(Sulzer, Inc.)

Fig. 13 – Large-scale applications of static mixers with gaseous and liquid media.

parts: ε_1 in the boundary region and ε_2 in the core region (Eq. (2)).

$$\bar{\varepsilon} = \varepsilon_1 + \varepsilon_2 \qquad (2)$$

Only the second contribution, ε_2, enhances the rate of fast chemical reactions. The aim is therefore to achieve high ε_2 values at a low pressure drop level, which means that the ratio $\varepsilon_2/\bar{\varepsilon}$ must be high (Bourne et al., 1992b). Bourne and his co-workers have devised a set of fast, competitive consecutive reactions, the selectivity of which is very sensitive to mixing on the molecular scale. They used these reactions to show that $\bar{\varepsilon}$ in a plain pipe is about 5 W/kg, while SMV and SMXL static mixers can generate respectively about 800 and 500 W/kg when the fluid velocity is 2 m/s. For these mixers, ε_2 values calculated by Bourne et al. (1992b) were 33% and 66% of the $\bar{\varepsilon}$ values respectively. This shows that the SMV mixer, which uses a corrugated-plate design and causes the highest pressure drop, is less efficient for reaction enhancement than the SMXL mixer, which uses a multilayer design.

3.2. Mixing scales

Mixing, as a physical phenomenon, is a complex process. For a deeper understanding, it is inevitable to distinguish and describe some simpler stages of mixing, namely macromixing, mesomixing, and micromixing illustrated in Fig. 14 (Baldyga and Bourne, 1986, 1992, 1999; Fournier et al., 1996a).

3.2.1. Macromixing

Mixing on the scale of the whole vessel is called "macromixing". This determines the environment concentrations by convecting the fluid particles in the flow domain. The mixing process depends directly on the transfer efficiency of the "mean" flow at different scales (Baldyga and Bourne, 1999). Macromixing consists in the dispersive capacity of the flow at the heat exchanger/reactor scale, and is generally characterized by the residence time distribution (RTD) method as a signature of velocity field uniformity (Castelain et al., 1997;

Habchi et al., 2009a; Villermaux, 1986). In fact, the RTD is directly related to the global motion of the flow since it represents the time the fluid particles take to migrate from the device inlet to the outlet. This large-scale motion, caused by the mean flow velocity, drives the fluid particles between high- and low-momentum regions in the heat exchanger/reactor volume, determining the large-scale convective transfer called macromixing.

The RTD sharpness, hence the macromixing, can be enhanced by generating a radial convective transfer, for instance longitudinal vortices (vortex generators) or baffles that perturb the fluid path (Mutabazi et al., 1989; Fiebig, 1995; Peerhossaini and Bahri, 1998; Ajakh et al., 1999; Toe et al., 2002; Lemenand et al., 2003, 2005; Momayez et al., 2004, 2009, 2010; Ferrouillat et al., 2006a; Mohand Kaci et al., 2010; Habchi et al., 2010, 2012b).

3.2.2. Mesomixing

At the intermediate scale, mesomixing reflects the coarse-scale turbulent exchange between the fresh feed and its surroundings governed by the turbulent fluctuations (Baldyga et al., 1995); a fast chemical reaction is usually localized near the feed point, forming a plume of fresh feed. This plume is at intermediate scale between the micromixing scale and that of the system or the reactor. Spatial evolution of the plume can be identified with the process of turbulent diffusion. Another aspect of mesomixing is related to the inertial-convective process of disintegration of large eddies. Mixing by inertial-convective disintegration of large eddies proceeds without any direct effect of molecular mixing. However, there is an effect of inertial-convective mixing on the micromixing process.

Fully understanding and describing mesomixing is not a simple task. Several approaches can be adopted since the inertial-convective mixing process depends on various parameters including turbulent kinetic energy k, length scale of turbulent fluctuations L, and their combination in the turbulent diffusivity D_t. Locally, it is also sensitive to specific operating conditions like pipe diameter, ratio of feed stream velocity to mean velocity of flow surrounding the feed point
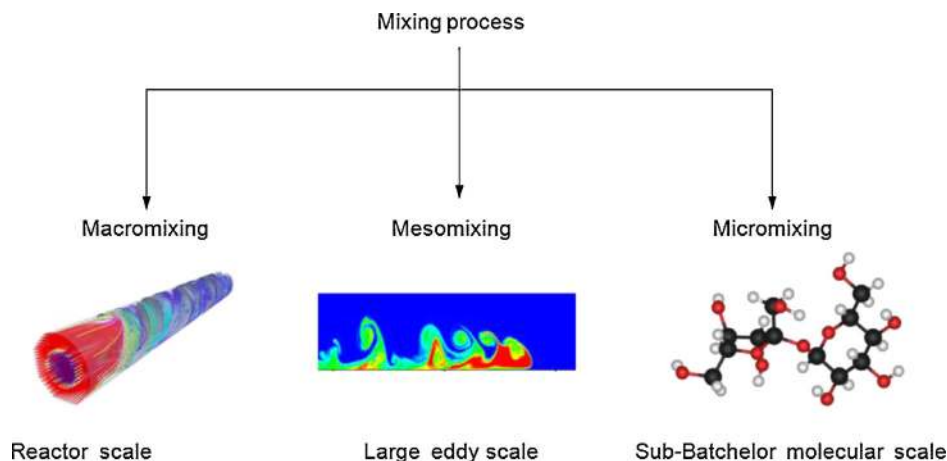
Fig. 14 – Three different mechanisms of the mixing process and their corresponding length scales.

and eventual back-mixing into the feed pipe (Baldyga et al., 1993). Two mechanisms have been proposed: a turbulent dispersion mechanism and an inertial-convective disintegration mechanism. Further details can be found in Baldyga and Bourne (1999).

The simplest models (Pohorecki and Baldyga, 1983; Villermaux and David, 1983) of inertial-convective mixing assume erosive diminishing of the blobs of fresh feed, followed by micromixing. In the more complex models (Baldyga, 1989; Baldyga and Bourne, 1989; Baldyga et al., 1994), only the large eddies of the inertial-convective subrange determine the environment for micromixing.

Intermediate structures, which can be turbulent cascade vortices or fractal structures in a chaotic laminar flow (Aref, 1984; Habchi et al., 2009a,b; Jones et al., 1989; Lemenand and Peerhossaini, 2002; Muzzio et al., 1992), contribute to mesomixing. In a chaotic laminar flow, the fractal structures are characterized by the Lyapunov exponent (Carrière, 2006), while in turbulent flows they can be characterized by the TKE and the Reynolds strain-rate tensor (Habchi et al., 2010; Mohand Kaci et al., 2009; Schäfer et al., 1997). Mesomixing is thus basically governed by the turbulent fluctuations and the random path of the fluid particles except the mean flow. It is related to the magnitude of velocity fluctuations of the eddies and the length scale of these fluctuations. Consequently, one of the simplest ways to capture the essential features of mesomixing is to link it to the RMS of velocity fluctuations or the TKE, k (Habchi et al., 2010):

$$k = \frac{1}{2}\sum_{i=1}^{3}\overline{u_i u_i} \tag{3}$$

3.2.3. Micromixing

Micromixing, the last of the mixing stages, consists of viscous-convective deformation of fluid elements that accelerates the aggregate size reduction up to the diffusion scale (Baldyga and Bourne, 1989).

The selectivity of chemical reactions depends on micromixing: how the reagents mix on the molecular scale (Baldyga and Bourne, 1999). This mechanism entails the engulfment and deformation of Kolmogorov microscale eddies and is the limiting process in the reduction of local concentration gradients. It can be characterized by a micromixing time that is directly related to the turbulence energy dissipation rate (Baldyga and Bourne, 1999). Following

the Hinze and Kolmogorov theory, which is based on the idea of energy cascade, the drop breakup in multiphase flows can also be characterized by the turbulence kinetic energy dissipation rate (Hinze, 1955; Lemenand et al., 2013; Streiff et al., 1997). Thus, an increase in turbulence kinetic energy dissipation favors the micromixing process, enhancing the selectivity of fast chemical reactions and also reducing the maximum drop size in multiphase flows. The local turbulence kinetic energy dissipation rate, ε, which can be used to quantify micromixing, can be related to the strain rate tensor components by:

$$\varepsilon = \frac{1}{2}\upsilon\overline{\left(\frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i}\right)^2} \tag{4}$$

4. Methods to characterize mixing in continuous flows

Understanding and describing the degree of mixing is a crucial issue. In addition, process control and optimization sometimes necessitate parameterization, measurement, and quantification of mixing. This urge has led to the development of several qualitative and quantitative measurement techniques that are sometimes case-specific; each of which is adapted to a certain type of flow; some of them have been the subject of extensive research, the list of references cited hereafter is not exhaustive.

4.1. Qualitative techniques

Qualitative characterization techniques are mostly optical and necessitate transparent devices or at least devices with transparent viewing windows. The information obtained on mixing quality can give an indirect approximation of mixing time.

4.1.1. Acid–base or pH indicator reactions

The generation of color change in acid-base reactions relies on the presence of a pH indicator in the basic or acidic solution. A number of different pH indicators have been used for characterizing mixing in micromixers like methyl orange, bromothymol blue, and phenolphthalein.

Branebjerg et al. (1995) estimated a mixing time by recording the time of color change. Kockmann et al. (2006) considered a mixing length defined as the downstream length in the mixer where color change was no longer visible. In this latter study, mixing of two fluids was visualized with the
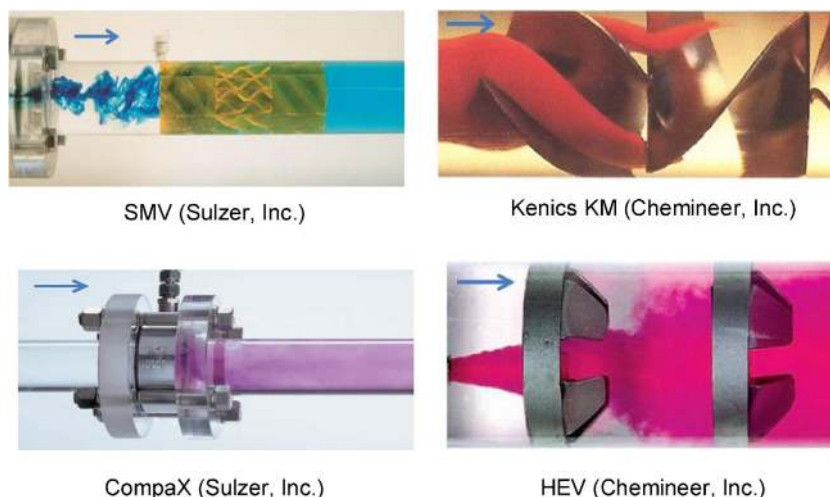
Fig. 15 – Flow visualization using colored dyes in different static mixer configurations.
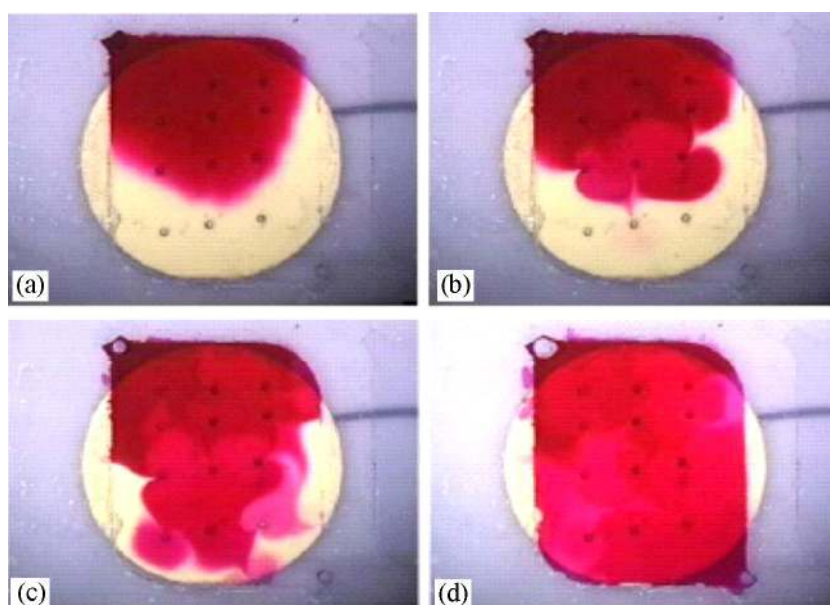

Fig. 16 – Photographs showing acoustic microstreaming visualization in a microchamber at different times, (a) 0 s, (b) 28 s, (c) 67 s, and (d) 106 s.
Source: Liu et al. (2002).

pH-indicator bromothymol blue for good optical contrast. The redox reaction of an alkaline solution of di-sodium hydrogen phosphate (pH 8) with deionized water and bromothymol blue (pH 7) resulted in a mixture with a pH value of 7.5, indicated by a blue color. The concentration of the reactants was very low to avoid significant influence on fluid properties. The mixing time, as defined in this work, indicated when mixing was complete. The color change of the bromothymol blue solution was optically observed to determine the mixing length, where color change was no longer visible. This indicated complete
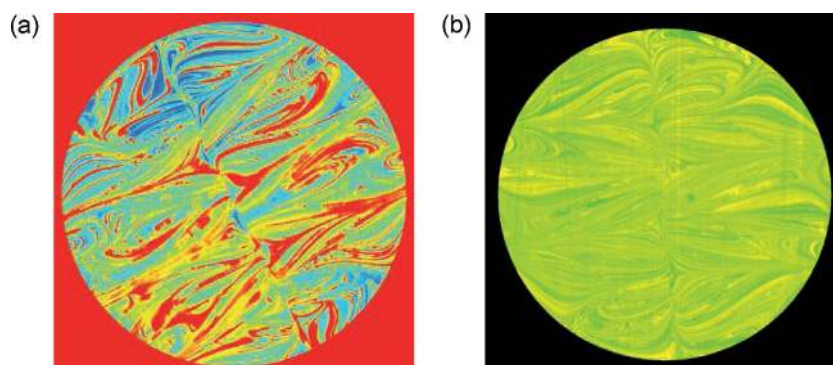

Fig. 17 – Laminar mixing and homogenization efficiency shown using Laser Induced Fluorescence (LIF), (a) Sulzer SMX and (b) Sulzer SMX plus.

mixing at a certain length in the mixing channel. The mixing length was divided by the average velocity in the mixing channel to calculate the mixing time.

Using this technique, the mixing process visualization is perpendicular to the flow direction. Consequently, no information on the spatial mixing quality through the depth of the device is obtained. Results depend on the acid/base concentration ratios used. This technique is predominantly used for mixing in single-phase systems. The quality and resolution of the images are also important for evaluation of mixing performance and determination of the endpoint, especially if a statistical analysis (e.g. calculation of the coefficient of variance) is done on the concentration field.

4.1.2. Dilution of colored dyes

Using this method, concentrated solutions of colored dye are contacted with colorless solutions or simply pure water in the studied reactors. Fig. 15 depicts the visual evidence for the mixing process produced in different commercial devices. Mixing quality and flow structures can be clearly observed at different positions along the mixer. Flow instabilities and vortices can also be visually spotted.

Suitable imaging techniques are needed to accompany the development of transparent micromixers. The main issues are usually to achieve a simple, fast analysis rich in contrast and complex in information based on visual inspection or microscopic imaging. Hessel et al. (2003) proposed an approach relying on aqueous solutions with high concentrations of water blue and pure water, which were fed through the interdigital micromixers of interest. These blue-colored solutions provide excellent contrast due to high dye solubility and large extinction coefficient. They are, in particular, suitable for imaging multilaminated systems arranged parallel to the direction of observation and multiphase systems such as gas/liquid and liquid/liquid flows.

Schönfeld et al. (2004) described this method in which visualization of the mixing process is conducted perpendicular to the flow direction. Consequently the information obtained is spatially averaged over the micromixer depth. Assuming a layer structure that is horizontal or tilted with respect to the observer, it is difficult to distinguish perfectly mixed systems from the complex multi-layered mixing patterns, and this difficulty may yield overly short mixing times. Another disadvantage of using the water blue imaging is that, due to the large molecular weight of this dye, its diffusion certainly differs from that of low-molecular-weight species.

Colored dyes are appropriate for characterizing mixing in single and two-phase systems. This technique is also useful for monitoring the evolution of dynamic systems over time or at different process steps. Fig. 16 illustrates the transient evolution of the mixing process induced by acoustic microstreaming in a microchamber by using a colored dye.

4.1.3. Dilution of fluorescent materials

Here, high energy light (e.g. laser) is required to induce fluorescence, which is monitored using fluorescence microscopy. Typical fluorescent materials are fluorescein and rhodamine B.

Using conventional microscopy, the resulting information is averaged on the optical axis over the device depth. The method is associated with confocal scanning microscopy which enables point-wise measurement of the fluorescent dye through the depth of the micro device. Peerhossaini and Wesfreid (1988) used laser-induced fluorescence (LIF) for
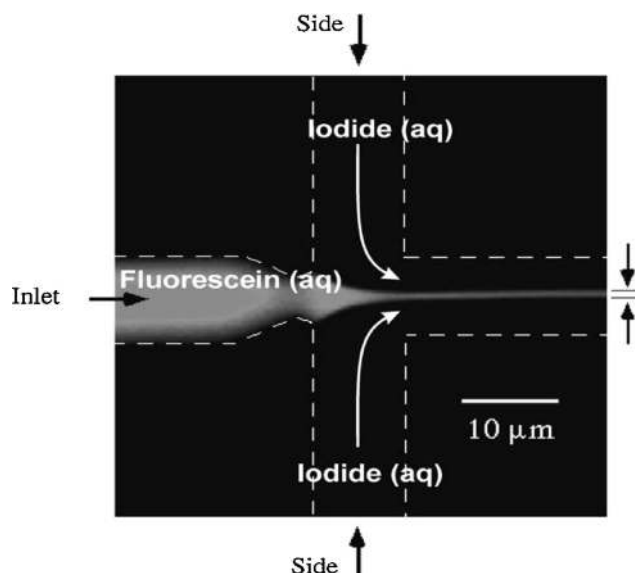

Fig. 18 – Characterization of diffusive mixing by fluorescent Hydrodynamic Focusing (Knight et al., 1998).

visualizing, for the first time, the Görtler vortices embedded in a concave boundary layer. Lemenand et al. (2003) used the same technique for visualizing the streamwise vortices generated behind vortex generators in a HEV static mixer. Fig. 17 illustrates the laminar mixing process visualized by LIF in the Sulzer SMX and SMX-plus configurations.

A faster alternative to turbulent mixing is to reduce the length scale over which the fluids must diffusively mix. Knight et al. (1998) used a hydrodynamic focusing technique incorporating fluorescent species to characterize diffusive mixing in a microfluidic, continuous-flow mixer capable of mixing times less than 10 ms by mere diffusive length scale reduction. As shown in Fig. 18, a fluorescent dye labels the flow from the inlet channel and appears bright, while nonfluorescent buffer flows from the side channels. The side flow squeezes, or "hydrodynamically focuses", the inlet flow into a thin stream that exits the intersection sheathed in buffer fluid. At such small length scales, molecules from the side flow rapidly diffuse across the inlet stream, resulting in fast mixing. Fluid flow was imaged using standard epifluorescence and confocal scanning microscopy. In both cases, the inlet flow was labeled with the fluorescent dye 5-carboxyfluorescein.

Johnson et al. (2002) reported using fluorescence imaging of the rhodamine dye through a research fluorescence microscope equipped with a 10× objective, a mercury arc lamp, a rhodamine filter set, and a video camera. They also proposed elementary mixing quantification through calculation of the percentage of mixed fluid using Eq. (5):

$$\text{percentage mixed} = \left(1 - \frac{\sqrt{(1/N)\sum_{i=1}^{N}(I_i - I_i^{\text{Perf.mix}})^2}}{\sqrt{(1/N)\sum_{i=1}^{N}(I_i^0 - I_i^{\text{Perf.mix}})^2}} \right) \quad (5)$$

where N, I_i, I_i^0, and $I_i^{\text{Perf.mix}}$ are respectively the total number of pixels, the intensity at pixel i, the intensity at pixel i if no mixing or diffusion were to occur, and the intensity of the perfectly mixed solution at pixel i. The experimental data for a perfectly mixed solution were obtained by placing the same fluorescent buffer, with half of the normal rhodamine concentration, in both inlet channels and then applying the various electric fields under investigation.

Both Johnson et al. (2002) and Knight et al. (1998) were able to retrieve three-dimensional concentration data throughout the mixer length. Fluorescent species can also be used to study mixing in single- and two-phase systems.

4.1.4. Reactions yielding colored species

This method is used to characterize single phase systems. A practical requirement of the test reaction is the formation of a soluble colored species. One test reaction reported in the literature to characterize a split-and-recombine micromixer is the reduction of potassium permanganate in alkaline ethanol (Mensinger et al., 1995).

Another test reaction is the iron rhodanide reaction, which has been used to characterize mixing in interdigital micromixers (Hessel et al., 2003). Mixing was recorded by digital recording with an optical microscope equipped with a video camera. A defined illumination was established by using a light guide system with goosenecks. Equipment was calibrated for each experimental run with each mixer by using standard dye solutions of known concentration. The images of both calibration and experimental solutions were converted to greyscale format. Therefore, the concentration distribution was gathered by analyzing the images taken with fixed illumination with images of the calibration solutions, and thus concentration profiles along the channel cross-section were obtained.

Differing from the classical colored dye dilution and water blue solutions, the formation of the brownish complex in the iron rhodanide reaction potentially provides information on fluid layer formation both at the very beginning and during the course of mixing. The advantage of this reaction over dilution-based methods is that the colored product appears only after the reactants are completely mixed at the molecular scale. This avoids measurement errors with complex multi-layered mixing patterns or tilted lamellae that are inherent in most other visualization techniques.

The drawback inherent in the conventional imaging and microscopic techniques employed for visualization is that no information on the spatial mixing quality throughout the micromixer depth can be obtained, which can lead to under- or over-estimation of mixing times and lengths. Some reactions may form a solid product, limiting their use for characterizing micromixers.

The limitations of any one method can be compensated for by using several techniques to examine the same case. In this way, complementary information can be obtained from each technique, offering a broader view on the course of mixing. For example, dilution-type experiments can be complemented by reactive methods. Fig. 19 shows the flow patterns in a split-and-recombine mixer obtained by using the dye water blue and those revealed by the iron rhodanide reaction. Comparing the results helps point out technical artifacts and pinpoint the real physical phenomena taking place.

4.2. Quantitative techniques

Using these methods, the characteristic mixing time is linked to segregation levels in a reactive flow or a flow containing chemical species whose concentrations are influenced by the mixing qualities. The concentrations are measured by optical techniques, and following the associated case-specific physical phenomena, the mixing time is calculated using physical models.

4.2.1. Monitoring species concentration

The spatial or temporal in situ monitoring of species concentration is a quantitative method for measuring mixing and is largely used when evaluating the performance of stirred tanks and macro-scale static mixers using the coefficient of variance (Paul et al., 2004).

The temporal or spatial mean and variance can be calculated to give a measure of the segregation intensity and therefore the "evenness" of concentration (Men et al., 2007).

One-dimensional profiles or two-dimensional maps of species concentration, depending on the measurement technique used are obtained to determine a striation thickness distribution, thus giving information about the scales of segregation. For one-dimensional concentration profiles, the measurement is representative of the concentration averaged through the channel or device depth. It is pertinent only if the flow structure is two-dimensional and the concentrations are constant throughout the channel depth. Both types of information allow the characteristic fluid lamellae scale in the device to be identified, which in turn can be related to mixing time.

The species concentration is typically detected using photometric and fluorescence methods. Salmon et al. (2005) used Raman spectroscopy for the analysis of reaction–diffusion dynamics. Confocal Raman imaging is a nonintrusive technique that can give the local concentrations of chemical species by scanning a sampling volume of a few micrometers in size throughout the entire system. Raman spectroscopy is routinely used in chemical analysis and has recently been coupled with microfluidics. Salmon et al. (2005) analyzed interdiffusion of pure liquids by coupling Raman imaging and microfluidics on quantitative grounds. Raman spectra at a given location in the microchannel were recorded using a confocal Raman microscope. The samples were illuminated by radiation from an argon-ion laser. Backscattered light was collected by the objective, and a grating dispersed the Raman spectrum onto a charge-coupled device. The local concentrations of the fluids could be probed from the intensities of nonoverlapping Raman bands specific to each liquid. Concentration maps were then obtained using x–y microactuators to displace the microchannel under the microscope objective.

For photometric, fluorescence, and Raman (using visible light) detection techniques, the device must be optically transparent. For infrared detection, the device must be transparent to infrared radiation wavelengths.

4.2.2. Competing parallel reactions

This technique, namely the chemical probe, entails competitive-consecutive or competitive-parallel reactions, which are based on the chemical result of the local injection of a reagent in stoichiometric deficit in the main flow. The principle is to carry out two reactions in parallel that both use a common reactant, which they compete for. One of the reactions should be very fast (quasi-instantaneous) with characteristic time t_{r1}, so that it proceeds only if mixing is ideal (extremely rapid). The other reaction should be fast (but slower than the first) and takes place when there is an excess of the common reactant, when mixing is slow and non-ideal. The second reaction has characteristic time t_{r2} close to the mixing time t_m. The local chemical reaction thus results from a competition between mixing at microscales and the reaction kinetics. Quantitative information can be obtained on the yield of the secondary (slower) reaction. Consequently, mixing performance is characterized by the
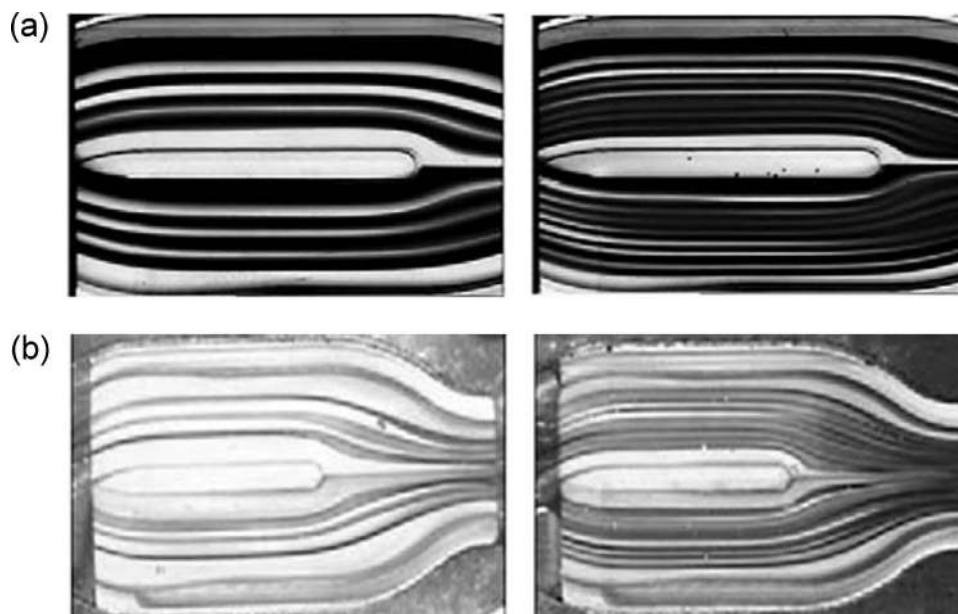
Fig. 19 – Multi-lamination flow patterns of a split-and-recombine mixer, (a) dilution-type experiment using the dye water blue and (b) reaction-type experiment using the iron rhodanide system (Schönfeld et al., 2004).

amount of secondary product formed: the greater the yield of the secondary reaction, the poorer the mixing quality.

No direct information on mixing time is accessible and the performances of mixers have most often been compared on the basis of the segregation index X_S, an indicator of mixing quality. In perfect mixing, the common reagent is totally consumed by the quasi-instantaneous reaction so that $X_S = 0$. In total segregation, an overconcentrated zone of the injected common reagent occurs and the slower reaction can react with this common reagent, so that $X_S = 1$, as shown in Fig. 21. The segregation index can be related further to the mixing time by using physical models (Baldyga and Bourne, 1984, 1989, 1999; Durandal et al., 2006; Falk and Commenge, 2010; Fournier et al., 1996b).

Several types of competing parallel reaction systems have been reported for this method. Baldyga and Bourne (1990) first studied the factors determining the product distribution of parallel reactions such as imperfect mixing, choice of reactor, or sequence of reagent introduction. They carried out experiments on the competition for hydroxide ions between the reactions of ferric hydroxide precipitation and the alkaline hydrolysis of ethyl chloroacetate. In the same work, the authors also reported the competitive nitration of benzene and toluene. Other competing parallel reaction systems, also based on the alkaline hydrolysis reaction, were later proposed as means of characterizing mixing performance. Bourne and Yu (1994) replaced the Fe^{3+} used by Baldyga and Bourne (1990) with H^+. Brucato et al. (2000) simulated stirred-tank reactors through computational fluid dynamics, using Cu^{2+} instead of Fe^{3+}. Computational fluid dynamics and numerical simulations (Fig. 20) can give access to qualitative and quantitative information concerning mixing performance through simply following particle trajectories, by examining concentrations or residence time distributions, or even by simulating complete reactive systems. The present work, however, focuses on the experimental techniques developed in the field of mixing characterization.

Bourne et al. (1992a) also proposed using the simultaneous diazo coupling of 1- and 2-napthol with diazotized sulfanilic acid to characterize mixing. This is known as the second Bourne reaction scheme and was also originally developed for studying micromixing in stirred tanks. Although this reaction system has been successfully applied to characterize the mixers performance (Ehlers et al., 2000), it is relatively difficult to use since it incorporates four consecutive reactions.

Another method is based on the precipitation of barium sulfate developed by Barthole et al. (1982), which offers the advantage of using non-toxic and cheap reactants. However the reaction times are relatively long (of the order of seconds) making it difficult to characterize systems with good mixing qualities where mixing times are much shorter (Mohand Kaci, 2007), which explains the preference for the Villermaux/Dushman method.

The reactive system, known as the Villermaux/Dushman or the iodide/iodate method is based on a technique originally developed for the micromixing characterization in stirred tanks (Fournier et al., 1996a,b; Guichardon and Falk, 2000) and has been the most commonly used method for evaluating reactor performance (Aoki and Mae, 2006; Baccar et al., 2009; Ehrfeld et al., 1999; Falk and Commenge, 2010; Ferrouillat et al., 2006b; Keoschkerjan et al., 2004; Kockmann et al., 2006; Kölbl et al., 2008; Mohand Kaci, 2007; Nagasawa et al., 2005; Panic et al., 2004; Schaer et al., 1999).

This competing-parallel-reactions chemical probe method offers several advantages:

- relatively simple reactions;
- precise knowledge of the reaction mechanism and kinetics (Guichardon et al., 2000);
- product concentration accessible through simple spectrophotometry;
- reaction rates sufficiently high enabling short mixing time measurement; and
- better accuracy among other chemical probe methods (Durandal et al., 2006).

However, quantitative treatment of the experimental data requires a robust kinetic model. Comparison of published
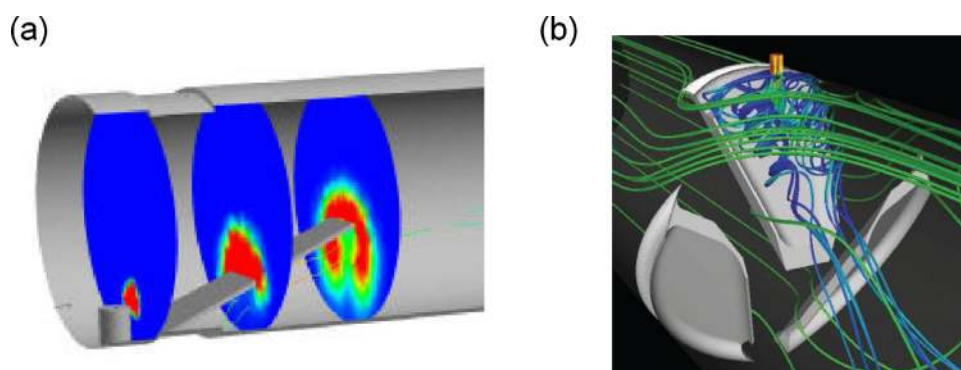
Fig. 20 – Computational fluid dynamics (CFD) as a powerful tool for mixing analysis, (a) concentration contours in the Chemineer WVM pre-distribution tab configuration and (b) turbulent streamlines in the Sulzer CompaX additive-dosing mixer.
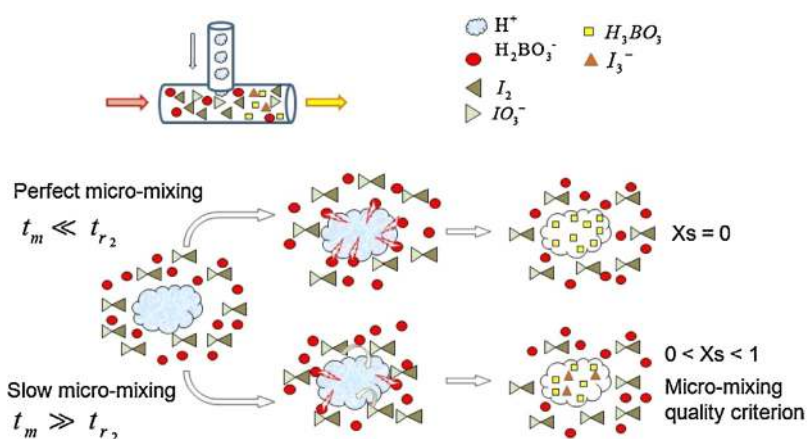

Fig. 21 – Schematic diagram of the micromixing process in the chemical probe.

models shows differences due to the sensitivity of the iodine-forming reaction to mixing conditions (ionic strength, concentration, etc.). The ideal approach is to re-determine the reaction kinetics using the specific parameters of the system adopted employing modern techniques for fast reactions (Bourne, 2008). Despite this fact, the Villermaux/Dushman method for characterizing the extent of micromixing through examining the iodine yield gives qualitatively consistent and intelligible results. With special attention given to the uniformity of reactive system characteristics, this method is suitable for ranking different mixers or different operating conditions (Bourne, 2008). Quantitative comparison of mixing times measured in different reactor configurations using different methods remains a delicate procedure that must take the above remarks into consideration.
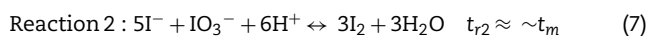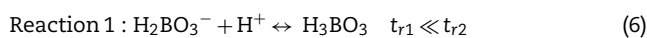
5. Iodide/iodate chemical probe method

This chemical probe method, developed by Fournier et al. (1996a,b) to study partial segregation in stirred tanks, implements a system of parallel competing reactions producing iodine. The coupling of the borate neutralization and the Dushman reaction in this system allows the measurement of mixing efficiency in reactors by monitoring the amount of iodine produced. As mentioned before, this technique has been extensively used in different types of reactors. A novel adaptive procedure recently developed by the authors to improve the reliability of the iodide/iodate method will be presented and discussed.

5.1. Chemical system and mixing models

5.1.1. Iodide/iodate reaction system

In this method, borate neutralization (Eq. (6)) is quasi-instantaneous with characteristic time t_{r1}; the second, the Dushman reaction (Eq. (7)) (Dushman, 1904) is slower and has characteristic time t_{r2} close to the mixing time t_m. The balanced reactions can be modeled as follows:

$$\text{Reaction 1}: H_2BO_3^- + H^+ \leftrightarrow H_3BO_3 \quad t_{r1} \ll t_{r2} \tag{6}$$

$$\text{Reaction 2}: 5I^- + IO_3^- + 6H^+ \leftrightarrow 3I_2 + 3H_2O \quad t_{r2} \approx \sim t_m \tag{7}$$

The iodine I_2 further reacts with iodide ions I^-, yielding I_3^- ions following the quasi-instantaneous equilibrium reaction:

$$\text{Reaction 3}: \quad I^- + I_2 \overset{K_B}{\longleftrightarrow} I_3^- \tag{8}$$

The kinetics of the three reactions was established by Guichardon et al. (2000). The characteristic times of reactions (1) and (2) are:

$$t_{r1} = \frac{\text{Min}([H_2BO_3^-]_0; \quad [H^+]_0)}{(r_1)_{t=0}} \tag{9}$$

$$t_{r2} = \frac{\text{Min}((3/5)[I^-]_0; \quad 3[IO_3^-]_0; \quad 1/2[H^+]_0)}{(r_2)_{t=0}} \tag{10}$$

where the brackets denote the reagent concentration and r_1 and r_2 are the respective rates of reactions (1) and (2):

$$r_1 = k_1[H_2BO_3^-][H^+] \tag{11}$$

$$r_2 = k_2[H^+]^2[I^-]^2[IO_3^-] \tag{12}$$

where k_1 and k_2 are the reaction rate constants of reactions (1) and (2), respectively. Here $k_1 = 10^{11}\,L\,mol^{-1}\,s$ and k_2 is a function of ionic strength, λ as defined by Guichardon et al. (2000):

$$\text{For} \quad \lambda < 0.166\,M: \quad \log_{10}(k_2) = 9.28105 - 3.664\sqrt{\lambda}$$

$$\text{For} \quad \lambda > 0.166\,M: \quad \log_{10}(k_2) = 8.383 - 1.5112\sqrt{\lambda} + 0.23689\lambda \tag{13}$$

The principle of this method is to add, in stoichiometric deficit, a small quantity of sulfuric acid H^+ to an initial mixture of I^-, IO_3^-, and $H_2BO_3^-$. In perfect mixing, the injected H^+ is totally consumed by the quasi-instantaneous reaction (1) in Eq. (6) and hence there is no formation of iodine I_2. When the mixing process is not sufficiently fast to sustain reaction (1), the local overconcentration of H^+ produces iodine I_2 by reaction (2) (Eq. (7)), which reacts with iodide ions I^- to yield I_3^- ions (Eq. (8)): it modifies the absorbance of the final solution that is detected by the spectrometer, used to deduce the concentration of produced I_3^-. Therefore, the selectivity in I_2 is a measure of molecular-scale segregation and indicates mixing quality.

The characteristic segregation index X_S is defined by the expression:

$$X_S = \frac{Y}{Y_{ST}} \tag{14}$$

where Y is the ratio of the quantity of H^+ transformed into I_2 following the second reaction to the total quantity of injected H^+ and Y_{ST} is the value of Y in the case of total segregation. X_S is given for open loop flows by Villermaux (1986) as:

$$X_S = \frac{2([I_2] + [I_3^-])}{[H^+]_0}\left(\frac{Q_p}{Q_{H^+}} + 1\right)\left(\frac{[H_2BO_3^-]}{6[IO_3^-]_0} + 1\right) \tag{15}$$

where Q_p and Q_{H^+} are respectively the flow rate of the main stream and the injected sulfuric acid.

The mass balance on iodine I_2 atoms leads to the following expression (Fournier et al., 1996a,b):

$$[I_2]^2 - \left(\left(\frac{3}{5}\right)[I^-]_0 - \frac{8}{5}[I_3^-]\right)[I_2] + \frac{3}{5}\frac{[I_3^-]}{5K_B} = 0 \tag{16}$$

where K_B is the equilibrium constant of reaction (3) (Eq. (8)) which depends on the solution temperature:

$$\log_{10}K_B = \frac{555}{T} + 7355 - 2575\log_{10}(T) \tag{17}$$

At $T = 298\,K$ the equilibrium constant $K_B = 702\,mol^{-1}$.

5.1.2. Adaptive procedure

Recently, the conventional chemical probe procedure was used to compare two curved $2 \times 4\,mm$ rectangular ducts with different radii of curvature (Habchi et al., 2011). A first experiment, with distributed wall injections and a given set of species concentrations fixing a "high rate" for reaction (2),

demonstrated that the selectivity did not vary with Reynolds number. In fact, the channel walls are corrugated, and near the solid walls the flow is governed by the roughness size. The chemical probe could not discriminate between the geometries when a wall injection is applied. A second experiment, with a slow ("low-rate") reaction (2) and unique injection point at the duct inlet, also did not show variation with Reynolds number, because the measurement volume depends on the flow velocity. The Reynolds numbers were relatively low due to the small size of the duct sides, so that the measurements are inevitably hindered by mesomixing effects. The challenge was thus to adapt the chemical method for characterizing global mixing by enlarging the measurement volume so as to capture and take into account all the mixing scales. To meet this challenge, a new protocol was proposed that involves pre-adjusting the measurement volume to the different Reynolds numbers by adapting the kinetics of reaction (2) to each Reynolds number, leading to more relevant results for the segregation index X_S. The second reaction time t_{r2} can be adjusted by varying the initial reagent concentrations; however, as it is quite difficult to vary the initial concentrations of I^-, IO_3^-, and $H_2BO_3^-$ constituting the initial solution because they are linked by the pH, the choice was made to vary the H^+ concentrations injected into the main flow. Accordingly, for each Reynolds number, the second reaction time is calculated in such a way as to obtain a constant measurement volume computed from the length over which reaction (2) takes place, $L_{r2} = Wt_{r2}$. Therefore, when the Reynolds number and the flow speed increase, the second reaction time is decreased by the same factor by increasing the H^+ concentration. This method reveals the relative mixing improvement when the Reynolds number is increased, and allows comparing different reactor geometries.

Finally, the measured segregation index is related to the mixing time by the engulfment model (Baldyga and Bourne, 1989) for turbulent regime and by the stretching efficiency model (Falk and Commenge, 2010) for laminar one.

5.2. Micromixing models

The segregation index X_S provides qualitative information on global mixing dependent on the chemical system. The related quantitative parameter is the intrinsic mixing time, t_m independent of the reactive conditions, which can be identified through mixing models devised on phenomenological grounds.

5.2.1. Engulfment model

Several models exist for determining mixing time and turbulent energy dissipation rate. Baldyga and Bourne (1984, 1989) have developed a complex model, the engulfment model (E-model), based on the assumption of first-order-law growth of the incorporated reagent volume into spiral marbled structures, shown in Fig. 22, that are later homogenized under the stretching effects until near-Batchelor scales, and then molecular diffusion.

The E-model is used for the turbulent flow regime. The first of its steps is the growth of the volume V_i containing the species i:

$$\frac{dV_i}{dt} = EV_i \tag{18}$$

where the engulfment rate E, the inverse of the mixing time, is related to the TKE dissipation rate, ε and the kinematic
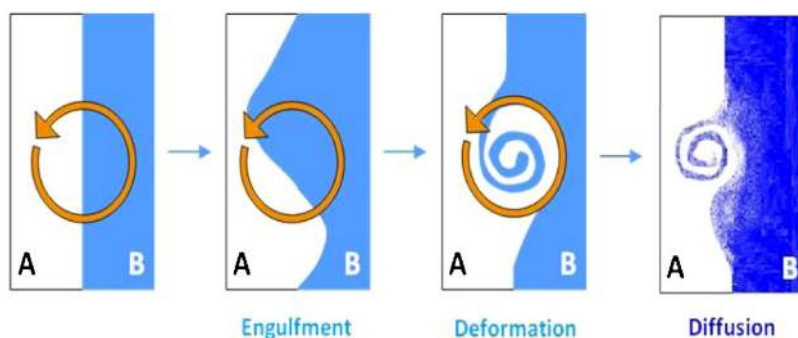
Fig. 22 – Principle of the engulfment model.
Adapted from Baldyga and Bourne (1999).

viscosity, υ following the scale law analysis by Baldyga and Bourne (1989):

$$E = \frac{1}{t_m} = 0.05 \left(\frac{\varepsilon}{\upsilon} \right)^{1/2} \tag{19}$$

Expressing Eq. (18) in terms of mass balance, the growth of the uniform mixing zone of concentration c_i is given by the temporal differential equation:

$$\frac{dc_i}{dt} = E(\langle c_i \rangle - c_i) + R_i \tag{20}$$

where $\langle c_i \rangle$ refers to the mean concentration value of i in the environment when mixing is completed and R_i is the rate of formation of substance i by the chemical reaction.

To find the rate of variation of each species, Eq. (20) must be solved as a function of time for each species involved in reactions until the sulfuric acid concentration tends to zero resulting in a system of nine nonlinear equations for the different reagents in the iodide/iodate reaction system. The Newton–Raphson iterative method is employed and computations are made with MATLAB software. The calibration curve is obtained by the set of X_S solutions found by sweeping the mixing-time parameter, t_m. From the experimental X_S value (Eq. (15)), the mixing time can be determined by the inverse function $X_S = f(t_m)$. The corresponding curve can be used to find the mixing time, t_m when the segregation index value is available from experiments.

5.2.2. Stretching efficiency model

In laminar shear flow, it is known that the characteristic dimension of the structure decreases in the direction orthogonal to the elongation. Diffusion and convection are then competitive processes, but according to the shear rate value, for large segregation scales, diffusion process is slow compared to convection and mixing is almost controlled by stretching. At fine segregation scales, diffusion becomes the controlling step. This problem has been addressed by several authors (Baldyga and Bourne, 1983; Li et al., 2005; Ou and Ranz, 1983a,b) and reinvestigated by Baldyga and Bourne (1984), who considered the total mixing time to be the simultaneous contribution of the diffusion time and the shear stretching characteristic time and proposed to calculate the mixing time of intertwined lamellae by considering the striation thickness and the shear rate in the reactor.

Based on this approach, the stretching efficiency model proposed for laminar regimes, by Falk and Commenge (2010) is used to calculate the mixing time. Baldyga and Bourne (1984)

suggest calculating the mixing time in laminar flow by the following equation:

$$t_m = \frac{\text{arcsinh}((0.76\dot{\gamma}\delta_0^2)/D_m)}{2\dot{\gamma}} \tag{21}$$

where δ_0 is the initial striation thickness, D_m the molecular diffusion of water and $\dot{\gamma}$ the "efficient" generalized shear rate for mixing in the reactor. In this sense, $\dot{\gamma}$ is assumed to represent the rate of creation of intermaterial area:

$$\dot{\gamma} = \frac{1}{a_v} \frac{da_v}{dt} \tag{22}$$

where a_v is the intermaterial area per unit volume.

However, determination of $\dot{\gamma}$ is not trivial and numerical or analytical solutions are needed. Falk and Commenge (2010) estimate the mean power dissipation rate per unit mass of fluid $\bar{\varepsilon}$ and the "efficient" generalized shear rate for energy dissipation $\dot{\gamma}_m$ in laminar flow from the Hagen–Poiseuille law. Hence, $\bar{\varepsilon}$ and $\dot{\gamma}_m$ can be derived in terms of the channel hydraulic diameter D_h, the mean flow velocity W, and the kinematic viscosity of the fluid υ:

$$\bar{\varepsilon} = \frac{32\upsilon W^2}{D_h^2} \tag{23}$$

$$\dot{\gamma}_m = \left(\frac{\bar{\varepsilon}}{2\upsilon} \right)^{1/2} \tag{24}$$

The mean power dissipation rate per unit mass of fluid $\bar{\varepsilon}$ can also be calculated, for all flow regimes, from the net pressure drop over the entire reactor length ΔP by:

$$\bar{\varepsilon} = \frac{Q\Delta P}{\rho V} \tag{25}$$

where Q is the volume flow rate of the main flow and V is the total fluid volume between the two pressure-drop measurement points.

The initial striation thickness δ_0 depends in turn on the experimental procedure and the injection mode. Under optimal conditions, the acid is injected in the direction of the main flow with an equal velocity to avoid a jet impact with the flow. In this case, the initial striation thickness can be estimated using:

$$\frac{\pi\delta_0^2}{4} W = Q_{inj} \tag{26}$$

where Q_{inj} is the volume flow rate of the injected acid solution. Given the above conditions, the initial striation thickness thus equals the injection needle diameter d.

For the ideal case where the entire mean shear rate of the flow contributes to the mixing process, application of the relation in Eq. (21) gives the following expression for the mixing time:

$$t_m = \frac{D_h}{8W} \text{arcsin} h \left[3.04 \left(\frac{d}{D_h} \right)^2 Pe \right] \tag{27}$$

where $Pe = WD_h/D_m$ is the Péclet number.

In reality, however, several objections can be raised based on flow phenomena. To calculate the mixing time, the initial concentration field is considered to be constituted of interwined lamellae of the two streams. This state is not achieved directly after the injection point and needs additional time for full development.

Also, the model assumes that the slabs constantly undergo constant shearing rates. This is a crude assumption, since the lamellae do not always undergo constant deformation rate; they are stretched and rotated in usually complex three-dimensional flow fields. In addition, the orientation of the deformation field plays an important role in the mixing process. A fluid lamella may go by in a state in which it is perpendicular to the stretching field, increasing its striation thickness and hence reducing the concentration gradient and consequently the mixing rate.

These considerations lead to the conclusion that only part of the mechanical energy consumed is used for mixing. Falk and Commenge (2010) use the concept of mixing efficiency η proposed by Ottino et al. (1979):

$$\eta = \frac{\dot{\gamma}}{\dot{\gamma}_m} = \frac{\dot{\gamma}}{(\bar{\varepsilon}/2\upsilon)^{1/2}} \tag{28}$$

Taking the above relation into consideration, the expression of the mixing time introduced in Eq. (21) finally becomes:

$$t_m = \frac{D_h}{8W\eta} \text{arcsin} h \left[3.04 \left(\frac{d}{D_h} \right)^2 \eta Pe \right] \tag{29}$$

Comparison of the experimental values of t_m to those calculated using the model in Eq. (29) gives access to the mixing efficiency η.

6. Conclusions

This review reports on the static mixing process in industrial applications—the mechanisms, the devices, the characterization methodologies—in an attempt to summarize the basic information needed to handle a mixing application on an industrial scale.

The first part of the study surveys the available commercial models of static mixers and heat exchangers/reactors and their fields of application. All have the common characteristics of safe operating conditions, compactness, good reaction control, high yields and selectivities, energy savings, fewer unit operations, and cheaper processing.

The emergence of computer-assisted design and computational fluid dynamics facilitates the evolution of conceptual designs and the refinement of the principal configurations. This process would have been much slower had it relied merely on physical experiments; in addition, experiments

cannot provide the precise local measurements that are needed to understand the effects of complex geometries. There is still, of course, substantial room for further improvement and new designs and principles. For example, some innovative designs promote axial rather than radial mixing by approximating the residence time distribution of a continuous stirred-tank reactor (Nauman et al., 2002). Even the most widely studied geometries can still be further optimized. For Kenics-type helices, for example, the optimal angle of twist, the ratio of the element's length to its diameter, the offset angle between two consecutive elements, and the homogeneity of the geometric parameters for all elements as opposed to sequential position-dependent variations are all decisive factors for the overall performance that have yet to be determined.

Fouling resistance, corrosion, maintenance, and cleaning operations are at present problems in such devices. Academia seems overly obsessed with theoretical gains in mixing and heat transfer, and the tools to address technical complications in the implementation and production phases are still immature and need extensive development. Moreover, laboratory-scale optimization processes are leading to new designs that prove more potent than the available models, yet industrialization seems slow: sometimes it can take years for research prototypes to become available in the markets, if ever. Another issue is the scalability and extrapolation potential for each of the proposed configurations, especially those featuring a wide variety of geometric parameters and operating conditions like the microstructured heat exchanger/reactors, where only few technologies have been applied to industrial syntheses.

Even so, more robust and professional designs are needed and field trials must be conducted to evaluate the potential in industrial environments. For instance, helical inserts are promising in terms of heat transfer capacity, yet in most cases, studies have examined the insertion of elements in only a single channel; such studies in an entire heat exchanger/reactor could be interesting. Conditions may vary widely between a factory and a research facility. Currently, the trend is to develop and commercialize generic models for a wide variety of applications; such models exhibit satisfactory performance. The future need is for designs that are optimal, in some sense, for a particular application where they can operate with maximum efficiency.

The second part of the review is devoted to the fundamentals and measurement techniques for mixing. Several approaches have been proposed to understand the mixing mechanism, the most pertinent of which is the definition of three different mixing scales, macro-, meso-, and micromixing. The intrinsic parameters of the largest and smallest scales have been more or less unveiled, but the link between the two, namely the mesomixing scale, remains hard to conquer. At this scale, inertial-convective mixing depends on several parameters and is sensitive to various conditions; its mastery requires further investigation using modern analytical approaches and measurement techniques.

Mixing characterization is of increasing importance in modern applications for various reasons, the most basic of which is to differentiate and classify the available devices so as to be able to choose the proper configuration for a specific application. This report surveys existing experimental methods available for characterizing mixing and its outcomes, some of which have been adopted from chemical engineering, biology, or analytical chemistry. Information concerning

the main principles, the type of information obtained, and the limitations of each technique is provided, thus allowing identification of mixing performance characteristics that cannot be measured with existing techniques. Most of the experimental techniques employ optical methods, which require transparent devices or at least transparent viewing windows. In practical applications, however, transparent construction materials may not be well adapted to the harsh industrial environment of the chemical and physical processes encountered, so that these characterization methods may not be applicable. This shows the importance of coupling academic studies with industrial issues, when possible, for the improvement and control of process performance.

Quantitative characterization needs robust theoretical models to support and process the experimental findings. Developing adaptive procedures and modernizing scientific databases with state-of-the-art techniques is crucial to ensure maximum precision of the results delivered by classical methodologies, as in the case of the chemical probe method whose outcome strongly depends on the specific experimental conditions and parameters adopted in each individual study.

Static mixers and multifunctional heat exchangers/reactors are currently at the center of active research endeavors due to their promising capabilities in a world where energy efficiency is no longer a mere detail. Alarming environmental degradation and resource depletion, accompanied with continuing high-standard lifestyle requirements, necessitate proper exploitation of available power to produce high-quality commodities while minimizing compromises and damage to delicate ecosystems. It is hoped that the present work will help guide the potential industrial or academic user of static mixers in choosing a device and the relevant mixing characterization method based on an understanding of the associated mixing mechanisms in the context of energy-efficient processing.

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