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1.1 Introduction

Process technology and microsystem technology are both interdisciplinary engineering and natural science branches connecting physics, chemistry, biology, engineering arts, and management techniques to an enabling toolbox for various applications. Process engineering embraces orientating calculations for process and equipment design under general orientation, and system-orientated, cross-linked thinking. Process engineers are working in various areas ranging from the food industry through biotechnology to pharmaceutical products, from analytical and laboratory equipment through energy conversion to industrial chemistry for the production of millions of tons of chemicals [1, Chapter 1]. Chemical process engineering covers not only the design and implementation of chemical production and analytical processes but also deals with the equipment design, the appropriate materials, the fabrication, and operation of various chemical production processes. The aims of process technology are the economical and safe production of the desired products with the intended form and composition.

Microsystem technology, coming from information technology and miniaturization of data-processing devices, has now entered many fields in our daily life. Silicon chips and sensors can be found in cars, washing machines or smart cards with various functions. Besides the data-processing function, microsystems have taken over other tasks like sensing and analyzing, actuating or controlling larger systems. Microsystem engineering comprises besides engineering skills like design, simulation, or material knowledge also a deep physical and chemical knowledge for the fabrication and functional design issues. Also medical and biological skills are useful for the growing application fields for analysis, diagnostics, and therapeutics. A good overview about the state-of-the-art in microsystem technology

is given in [2]. For the control and manipulation of still smaller systems, microsystem technology is a major link to nanotechnology [3, 4].

Figure 1.1 gives an impression of the wide field and complexity of both disciplines, but also illustrates the multiple interfaces and common fields. The fruitful ideas from both sides may inspire the further development in both disciplines and result in an enlargement of possibilities and applications for the innovation across the borderlines.

Chemistry in *miniaturized equipment* is an emerging discipline coming together from microsystem technology and from chemical engineering, but also an established discipline of chemical analytics. Starting at the end of the nineteenth century a group of researchers at the University of Delft around Behrens [4a] and at the Technical University of Graz around Prof. Emich and Prof. Pregl developed the chemical analysis of very small amounts of reagents. In 1900 Prof. Behrens wrote his book “Mikrochemische Technik” [4a] about micro chemical techniques. In 1911 Prof. Friedrich Emich published the textbook “Lehrbuch der Mikrochemie” [5] and Prof. Fritz Pregl was rewarded in 1923 by the Nobel price for his fundamental work in microchemical analysis. In the middle of the last century in nuclear science small structures were developed for the separation of isotopes, see [6]. From this work, among others, the LIGA technology emerges at German research institutes.

Dealing with very small geometrical structures is also a well-known area in process engineering. The adsorption technology and chemical reactions at catalytic surfaces are based on the flow and adhesion processes in nanoscale pores [7, Chapter 4]. Transformations and transfer processes on the molecular scale are called “micro processes” in contrast to a “macro process” where convection

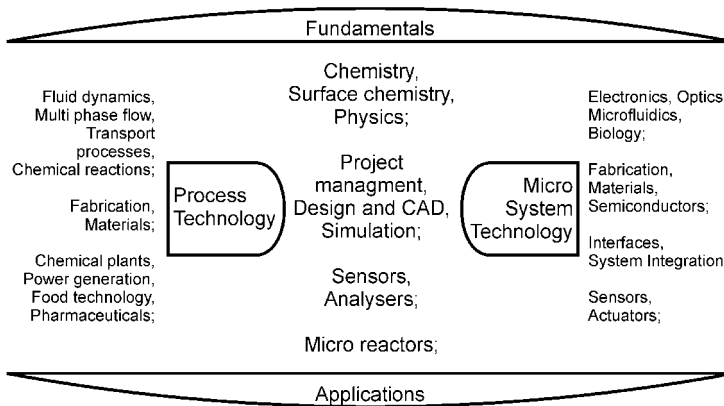


Fig. 1.1 Disciplines of process engineering and microsystem technology, differing and common overlapping areas (middle column). The lists are not complete and the future will certainly bring new applications and new common fields and applications.

plays the major role. Some typical length scales for process technology, chemistry and microtechnology are given in Fig. 1.2.

Figure 1.2 illustrates the different wording in process engineering, microsystem technology, and nanotechnology, especially the different meaning of “micro”. The micropores in adsorption media are one characteristic example on the nanometer scale. Microstructured equipment has internal characteristic dimensions like channel diameter or gap height within the micrometer range. A clear definition of “micro” does not exist, but it is not necessarily required for all applications and areas.

In the process industry, there are several applications of structures with typical dimensions below 1 mm, like compact plate and fin heat exchangers or structured packings in separation columns for enhanced heat and mass transfer. This is often summarized under the key word of process intensification. However, the miniaturization of conventional technology is limited by two major restrictions: the fabrication possibilities for the small structures at reasonable costs and the increased fouling probability, the high danger of blocking, and total failure of these structures. The first restriction has been widened with the enhanced fabrication possibilities, but the risk of fouling and blocking is still there and should not be underestimated.

The elementary setup of microstructured and conventional equipment is similar and displayed in Fig. 1.3. Process plants consist of process units, which themselves are made of equipment like heat exchangers or vessels with internal structures. The basic geometrical elements of the internal structures in conven-

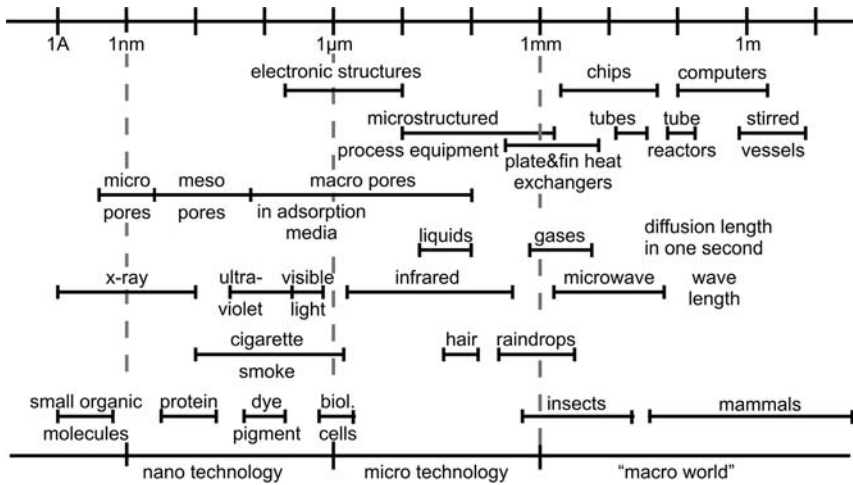


Fig. 1.2 Characteristic length of important processes and equipment in chemical engineering and microsystem technology. The top and bottom line indicate also the different wording of micro processes in the two disciplines, adopted from [8].

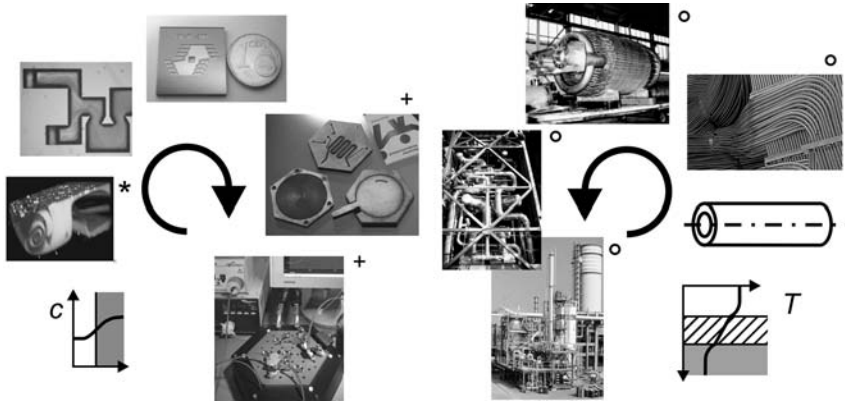


Fig. 1.3 Setup of microstructured and conventional equipment from single microstructures through combined elements to an entire plant. The principle of the active area

can be applied in both cases (Sources: * from [9]; + courtesy of FhG-ICT, Pfingsttal, Germany; o courtesy of Linde AG, Wiesbaden, Germany).

tional technology are the tube, the plate, and the film, on or in which the transport processes and transformations happen. The layout of process equipment and process steps follow this scheme from small elementary active areas (“micro process”) over the process space of the device (“macro process”) to the balancing of the complete process.

The parallel arrangement of microstructured channels or elements is called internal numbering-up, which is the most frequent way to increase the throughput of an apparatus. The parallelization of microstructured devices is called external numbering-up, applied to bypass the flow distribution problems within the equipment. A relatively new concept is the equal-up concept, the parallelization of similar effects [8]. The numbering-up and equal-up concepts facilitate the scale-up process from laboratory equipment to production equipment, but still have their own problems of flow distribution in manifolds, see Chapter 8.

1.2 Unit Operations and Beyond

The consecutive groups or steps in a process plant can frankly be named for many cases as

- pretreatment or conditioning of the incoming substances,
- transformation of the reagents in chemical, physical, or biological processes,
- separation of the received components, and
- purification and conditioning of the products, see Fig. 1.4.

The physical and chemical processes in the various steps may be the same or similar, like heat transfer or extraction. They are called *unit operations* that are playing a

major role in the research and development of process engineering. The unit operations can be combined and connected in different forms. The concept of unit operation combines a macro process with the apparatus to a process unit. It allows us to treat all micro processes within the process space in the same manner and to derive scientifically based design rules and calculation instructions. For an entire process plant the unit operations are combined and switched in a proper way and integrated for efficient material and energy use. Besides the energy and mass flow integration the appropriate process control and automation determines the economical performance and safety of the plant. This gives a very complex picture of a chemical or process technology plant, which is illustrated in Fig. 1.4. For a proper design and operation of a plant, many disciplines have to work closely together.

The unit operations can be categorized into three major groups according to the employed physical effects and major driving forces for combination or separation of substances: the mechanical, the electromagnetic, and the thermal unit operations (molecular driving forces) see Table 1.1. This list does not claim to be complete, especially the separation processes from analytics are only shown schematically. Probably in the next years further operations will be developed enabled by enhanced fabrication and integration possibilities. In adsorption of species or membrane separation, chemical processes may also be involved for mass-transfer processes in microstructures, see [11, Chapter 3]. The consequent treatment of unit operations allows the methodological design with help of the following principles. The *principle of continuity* of substances, phases, energy and momentum includes the preference of continuous processes opposite to batch processes. The *principle of balancing* of the relevant transport processes gives the energy, momentum, and mass fluxes in differential or black-box form. The *principle of scaling* and *similarity* of processes gives a calculation tool for transferring experimental, analytical, and numerical results to processes on different scales with the help of dimensionless numbers and groups.

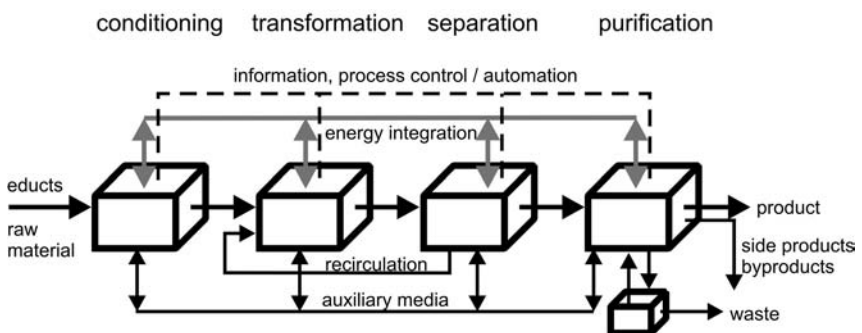


Fig. 1.4 Main process steps in a chemical production plant with pretreatment, conversion, separation, and purification of the products, adapted from [10]. The system integration includes the energy management, auxiliary media as well as information for the process control and automation.

Table 1.1 Main mixing and separation unit operations.

Unit operations	Molecular/thermal	Mechanical/ext. force	Electromagnetic
Mixing and aggregation	diffusion ¹⁾ dissolving ²⁾	spraying ²⁾ aeration ²⁾	electrophoretic mixing ¹⁾
Combination	extracting ^{2,c)}	stirring ²⁾	mixing with magnetic beads ²⁾
Control of segregation	desorption ^{2,c)}	mixing ^{1,2)} dosing ^{1,2)}	
Separation	thermodiffusion ¹⁾ countercurrent diffusion ¹⁾	sedimentation ²⁾ cycloning ²⁾ centrifucation ²⁾ pressure diffusion ^{1,2)}	eledcodeposition ²⁾ magnetodeposition ²⁾ electrofiltration
<i>Employed phases:</i>	condensation ^{2,a)} evaporation ^{2,a)}	(ultracentrifuge) filtration	electroosmosis electrophoresis
1) single phase	crystallization ^{2,a)}	osmosis	magnetostriction
2) multiple phase	distillation/ rectification ^{2,a)}	gas permeation classification	
a) with own cophase	drying ^{2,b)}	sorting	
b) own + additional cophase	absorption ^{2,c)} adsorption ^{2,c)}		
c) additional cophase	extraction ^{2,c)} ion exchange ^{2,c)} membrane processes		

More detailed operation description and further reading in [10, 13–15]; additional thermal unit operations, which are closely related to the listed:

Condensation: partial condensation^{2,a)}

Evaporation: flash evaporation^{2,a)}, vacuum evaporation^{2,a)}

Drying: freeze-drying^{2,a)}, radiation drying^{2,a)}, superheated steam drying^{2,a)}

Distillation: outside/secondary steam distillation^{2,b)}, molecular distillation^{2,a)}, reactive distillation

Rectification: extractive rectification^{2,b)}, azeotropic rectification^{2,b)}

Absorption: chromatography, desorption

Membrane processes: permeation, pervaporation, dialysis, osmosis and reverse osmosis, micro- and ultrafiltration.

The *principle of an active area* indicates the platform of the driving forces in molecular and thermal processes. It provides a description for the transfer processes with linear correlations between the flux and the driving force, also called the kinetic approach [12, Chapter 1]. The processes act in basic geometrical elements like the vessel, the tube, a channel, pipe, pores, or plates, which are combined to form the process space in the chemical equipment. Within these elements the fluid itself forms geometrical elements like beads, drops, bubbles, films or thin layers, which determine the transfer processes and which are confined by the geometry, see Fig. 1.5. The three phases of a pure substance allow the following combinations for phase mixtures of a carrier fluid and a dissolved

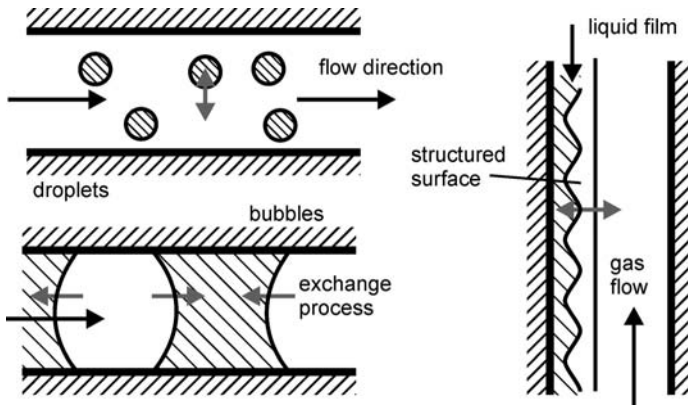


Fig. 1.5 Geometrical elements for transport processes between two phases: droplets, bubbles and a falling film on a structured surface.

phase: gas – gas, gas – liquid (droplets and aerosols), gas – solid (aerosols) and liquid – gas (bubbles and foam), liquid – liquid (miscible or immiscible, emulsion), and liquid – solid (suspension).

The *principle of technical enhancement* and *process intensification* compared to the natural driving forces gives the opportunity to control the transfer rates and state conditions in a way that is optimal for the desired results. The *principle of a selective phase* enormously enlarges the process space by adding a new component, which enforces a new equilibrium within a process (drying, extraction, stripping etc.). The *principle of flow guidance* in the equipment and process space (cocurrent, countercurrent, crosscurrent, mixed arrangement or recirculated flow) in addition with various switching possibilities (series, parallel, cascading) gives the basis for the effective exploitation of the existing driving force. The heuristic application of these principles can facilitate the system design and encourage the process engineering and microsystem engineering research and development [10].

Mixing can be treated as a major unit operation, which is fundamental for many other processes. Mixing can adopt many forms like homogenization, dispersing, or suspending; mixing can occur with or without chemical reactions, or as a precursor for chemical reactions, in combustion, or polymerization. A modern, general definition of mixing is the control of segregation, which describes the general role of mixing in process technology. The potential of microstructures in mixing processes, like the short diffusion length, a fast mixing or controlled flows, will be shown in various parts of this book.

A further major process step, the transport of fluids, is not listed in Table 1.1, but is partially included in the unit operation description. Active devices for fluid transport are pumps and compressors, which possess a wide variety of possibilities depending of the fluid, the viscosity, the required pressure increase, and the volume flow. Inside conventional equipment, field forces are employed

for the fluid transport: a density or pressure difference, centrifugal or inertial forces. Additionally, capillary forces can be used for fluid transport due to the channel geometry and the surface characteristics of the material, see [16].

Chemical reactions are more heterogeneous than the above presented unit operations. There exist some segmentation proposals similar to the unit operations that follow physical or physicochemical aspects like heat release (exo-, endothermic), rate constants (fast, slow), kind of initiation (photo, electro, ...), or the component phases. Vauck and Müller [1] count up to 27 chemical reaction types, which can hardly be classified. The reactors, the equipment with chemical reactions inside, can be categorized with the help of the operation: batchwise within a vessel, stopped flow for many analytical applications, or continuous flow in a pipe, in a fixed or fluidized bed. The continuous-flow operation is the predominant mode for microstructured equipment due to the small hold up of media inside the reactor. A comprehensive overview of chemical reactions in microstructures is given in [17, Chapters 3 to 5], which describe 21 different reactor types with 23 gas-phase reactions, 36 different reactor types with 95 liquid-phase reactions, and 12 different reactor types with 28 gas-liquid reactions.

A large field of chemical reactions deals with catalytic transformations. In homogeneous catalysis, the catalyst acts in the same phase as the reagents. Examples are enzymatic reactions where the liquid catalyst enforces biological transformations. Hessel et al. [17, Chapter 3] count 24 reactor types for catalyst screening, which is a popular application for microstructured devices. The majority of catalytic reactions (>80%) are dealing with more than one phase, especially with solid or immobilized catalysts. In these reactions the mass transfer is the major limiting process. Fuel-cell reactors are summarized by Hessel et al. in [18, Chapter 2] with about 12 reactor types and 63 reactions.

At the end of this chapter, the scaling behavior of processes as well as the benefits and the potential for miniaturization is sketched for selected unit operations together with a more detailed description. The combination of the various units leads to a complex, hierarchic unit of processes and equipment that can show emerging abilities not possible within a single unit. The whole system is more than the sum of all elements. Hence, the concept of unit operations has its limits and should be complemented by a holistic integrated process design.

1.3 Balances and Transport Equations

The starting point of process engineering calculations and the design of process equipment are the conservation and balance equations of mass, species, momentum, and energy as well as the definition of the entropy. The conservation laws of mass (continuity equations) and energy (First Law of thermodynamics) hold in the scope of chemical processes dealt with in this book. They can be described by words in the following scheme:

$$\left[\begin{array}{c} \text{System change} \\ \text{with time} \end{array} \right] = \left[\begin{array}{c} \text{Incoming} \\ \text{Flow} \end{array} \right] - \left[\begin{array}{c} \text{Outgoing} \\ \text{Flow} \end{array} \right] + \left[\begin{array}{c} \text{Source or} \\ \text{Sink} \end{array} \right] \quad (1.1)$$

The source or sink of the flow property depends on the system and the parameter itself and is described later together with other possible simplifications.

1.3.1

Statistical Mechanics and Boltzmann Equation

Before introducing the balance equations of the various parameters, a short excursion to the molecular origin of these equations starts with the derivation of the Boltzmann transport equation for the thermodynamic equilibrium. In an ideal gas the molecules are regarded as hard spheres interacting only by very short hits with other molecules or with the boundary (wall, surface, or other limiting elements). It can be assumed that the probability of a molecule moving with the velocity w in a certain direction is equal for all three space coordinates, see also [19, p. 148]. This can be expressed by the constant ratio of the derivation of the probability distribution function (PDF) to the function $f(w)$ itself and the velocity component w ,

$$\frac{f'(w)}{wf(w)} = \frac{d \ln f(w)}{w dw} = -2\gamma, \quad \Rightarrow \quad \ln f(w) = c_i - \gamma \cdot w^2 \quad (1.2)$$

The integration constant is set to -2γ and determined with the kinetic energy of the molecules,

$$\gamma = \frac{M^*}{2kT} = \frac{M}{2R_m T} \quad (1.3)$$

The integration constant c_i is determined by normalizing the sum of the probability to unity. The integration gives the probability distribution for one velocity component w , which stands for the other components as well.

$$f(w) = \left(\frac{M}{2\pi R_m T} \right)^{\frac{1}{2}} e^{-\frac{M}{2R_m T} w^2} \quad (1.4)$$

The integration over a sphere of the three space coordinates gives the probability of the absolute velocity c , independent of the direction,

$$\varphi(c)dc = 4\pi c^2 F(c)dc \quad \Rightarrow \quad \varphi(c) = 4\pi c^2 \left(\frac{1}{2\pi RT} \right)^{\frac{3}{2}} e^{-\frac{1}{2RT} c^2} \quad (1.5)$$

The most probable velocity of a molecule is determined by $c_{mp} = \sqrt{2RT}$. The mean velocity from the kinetic energy is given by $\bar{w} = \sqrt{3RT}$. With the number of molecules in a unit volume N_A and the collision cross section of the spheri-

cal molecule $\pi\sigma^2$, the number of hits between the molecules and the mean time between these hits can be determined. Multiplied with the mean velocity, an estimation of the mean free path of a molecule, the average length between two collisions, can be derived:

$$\lambda = \frac{kT}{\sqrt{2}n\pi\sigma^2} . \quad (1.6)$$

The mean free path divided by a characteristic length gives the dimensionless Knudsen number Kn , which is used later to estimate the influence of the molecular mobility on the fluid behavior inside microstructures, see Section 3.3. A closer look at the probability distribution f for the location $\bar{x} = \bar{x}(x, y, z)$ and the velocity space of a particle $\bar{w} = \bar{w}(u, v, w)$ varying with time gives a better image of the forces and energy distributions in an ideal gas. The integration of the PDF over the velocity space results in the number of particles in the control volume [20, p. 4],

$$\int f(t, \bar{x}, \bar{w}) d\bar{w} = N(t, \bar{x}) \quad (1.7)$$

The integration of the PDF over the velocity space, divided by the mass, results in the fluid density,

$$\rho = \frac{1}{m} \int f(t, \bar{x}, \bar{w}) d\bar{w} \quad (1.8)$$

The integration of a state variable multiplied by the PDF over the velocity space gives the mean value of this variable. The total derivative of the PDF in an external field (for example a gravitation field) is determined by the collisions of the molecules in a control volume, in detail, the current of gain and loss due to the molecule collisions,

$$\frac{df}{dt} = \frac{\partial f}{\partial t} + \bar{w} \cdot \frac{\partial f}{\partial \bar{x}} + \frac{F}{m} \frac{\partial f}{\partial \bar{w}} = J_{\text{gain}} - J_{\text{loss}} = \Delta J_{\text{coll}} \quad (1.9)$$

This equation is called the Maxwell–Boltzmann transport equation, an integro-differential equation. The determination of the loss and gain current, the collision integral, and the construction of the PDF are the main problems in solving Eq. (1.9), see also [21, 22]. Regarding the collision of two molecules with the relative velocity w_{rel} , with the probability f and f_1 before and the probability f' and f'_1 after the collision, the integration over the volume element and the velocity space of both molecules after the collision determines the left side of Eq. (1.9), the collision integral, see [19, p. 263].

$$\Delta J_{\text{coll}} = \int \frac{\sigma^2}{2} \bar{w}_{\text{rel}} (f' f'_1 - f f_1) d\bar{w}_1 d\bar{w}' d\bar{w}'_1 \quad (1.10)$$

This collision term is also used and implemented in the numerical lattice methods, see [23]. The first moments of the PDF f_j , for which the collision term will vanish in the case of local equilibrium, are also called the collision invariants and will be used to yield the first solutions of Eq. (1.9). With these variables f_j the Boltzmann equation can be simplified to:

$$\frac{\partial}{\partial t}(\rho f_j) + \frac{\partial}{\partial \vec{x}}(\rho f_j \bar{w}) + \frac{\rho \bar{F}}{m} \frac{\partial f_j}{\partial \bar{w}} = 0 \quad (1.11)$$

For $f_j=1$ the mass conservation equation is derived,

$$\frac{\partial \rho}{\partial t} + \text{div}(\rho \bar{w}) = 0 \quad (1.12)$$

For the velocity components $f_j=u, v, w$, the momentum conservation equation for all three space coordinates is derived,

$$\rho \left(\frac{\partial \bar{w}}{\partial t} + \bar{w} \frac{\partial \bar{w}}{\partial \vec{x}} \right) + \frac{\partial p_j}{\partial \vec{x}} + \text{div} \left(\frac{\partial \bar{w}}{\partial \vec{x}} \right) - \frac{\rho \bar{F}}{m} = 0 \quad (1.13)$$

This equation is the basis for the Navier–Stokes equations of fluid dynamics. The collision invariant $f_j=1/2 w^2$, which describes the continuity of the kinetic energy during the collision process, leads to the following equation,

$$\frac{\partial}{\partial t} \left(\frac{\rho}{2} \overline{w^2} \right) + \frac{\partial}{\partial \vec{x}} \left(\frac{\rho}{2} \overline{w^2} \bar{w} \right) + \frac{\rho \bar{F}}{m} \bar{w} = 0 \quad (1.14)$$

With the following simplifications for the velocity mean values [19, p. 266]

$$\overline{w^2} = \frac{3p}{\rho} + w^2 \quad \text{and} \quad \overline{w^2} \bar{w} = \frac{2}{\rho} (-\lambda \text{grad } T) \quad (1.15)$$

the energy conservation equation can be written as

$$\rho \frac{\partial \dot{q}}{\partial t} + \bar{w} \frac{\partial \dot{q}}{\partial \vec{x}} + \text{div}(-\lambda \text{grad } T) + p \text{div} \bar{w} + \dot{e}_q = 0 \quad (1.16)$$

where the term \dot{e}_q describes the shear and deformation tensors in the energy equation, which can be summarized with the viscous dissipation. A more detailed discussion on the energy equation is given in [19, p. 266]. Here, the illustration of the linkage between the microstate Boltzmann equation (1.9) and the macrostate balance Eqs. (1.12) to (1.16) is sufficient and gives a good insight for molecular processes. The macroscopic equations are valid, if enough molecules act together to receive a smoothed signal from the collisions ($N > 10^6$). A further and detailed discussion of special microeffects can be found in [24]. In Chapter 2 of this book the limits of continuity are treated in more detail.

1.3.2

Macroscopic Balance Equations

The macroscopic balance equations for process equipment can be formulated on various levels as shown in Fig. 1.6. The equipment level is the basic calculation niveau for plant design and layout. Elements of the equipment like tubes or trays can be balanced on the level of a differential length, which refers to the “micro process” description. The third and base level is represented by the differential element (here for all three dimensions with flow in the z -direction), which allows the continuum approach and the integration over the balance space. These three levels may take different forms regarding the relevant problem.

In Fig. 1.6 the balanced variables in a differential element are given by the parameter X , which stands for the mass, species, momentum, or energy. The general balance equation for a system and a differential element is written as:

$$\frac{\partial}{\partial t} X = X dy dx - \left(X dy dx + \frac{\partial}{\partial z} X dz dy dx \right) \quad (1.17)$$

with X as the general balanced value.

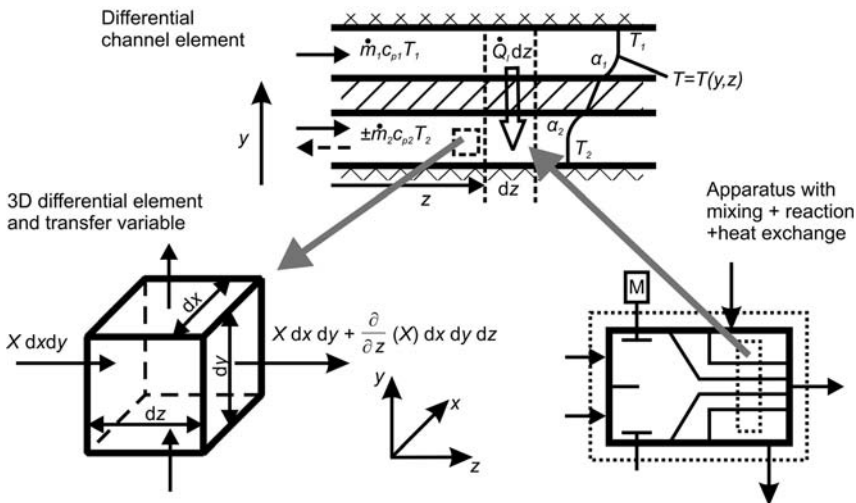


Fig. 1.6 Overview of the various balancing volumes in process engineering: 3D differential element for general calculations, 1D differential element depending on the geometry of the active area (channel, tube, plate, film, interface, see Fig. 1.5), and entire equipment for process balances (right).

1.3.3

The Mass Balance

Within the systems treated in this section no mass is generated or destroyed, hence no sink or source appears in the balance equation (1.1). The mass balance for a process element is written as

$$\sum m_{\text{in}} = \sum m_{\text{out}} - \sum m_{\text{conv}} = \text{const.} \quad (1.18)$$

For time-dependent mass flow rates, Eq. (1.8) can be written as

$$\frac{\partial m}{\partial t} = \dot{m}_{\text{in}} - \dot{m}_{\text{out}} \quad (1.19)$$

In a one-dimensional differential element, the balanced value X is set to the density multiplied by the velocity. The balance equation (1.17) can accordingly be written as

$$\frac{\partial(\rho A)}{\partial t} = - \frac{\partial(\rho w A)}{\partial z} \quad (1.20)$$

For a constant cross section or for a cubic differential element the mass balance can be written as

$$\frac{\partial \rho}{\partial t} + \text{div}(\rho \vec{w}) = 0 \quad (1.21)$$

This equation is also known as the continuity equation [25, p. 2] and is one of the fundamental equations of fluid dynamics. For an incompressible fluid the continuity equation can be simplified to

$$\frac{\partial(\rho A)}{\partial t} = 0 = - \frac{\partial(\rho w A)}{\partial z} \quad (1.22)$$

This equation is also called the equation of continuity for the mass in a system. It will help to simplify other balance equations.

1.3.4

The Species Equation

If the fluid density is replaced by the concentration of the relevant species, the species balance of a system can be derived from the mass balance. For an arbitrary process volume this equation can be written as

$$\sum \dot{n}_{\text{in}} = \sum \dot{n}_{\text{out}} - \sum \dot{n}_{\text{conv}} = \text{const.} \quad (1.23)$$