Particle Technology Series

Henk G. Merkus
Gabriel M.H. Meesters *Editors*

Production, Handling and Characterization of Particulate Materials

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Particle Technology Series

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José Manuel Valverde Millán, University of Sevilla, Spain

Many materials exist in the form of a disperse system, for example powders, pastes, slurries, emulsions and aerosols, with size ranging from granular all the way down to the nanoscale. The study of such systems necessarily underlies many technologies/products and it can be regarded as a separate subject concerned with the manufacture, characterization and manipulation of such systems. The series does not aspire to define and confine the subject without duplication, but rather to provide a good home for any book which has a contribution to make to the record of both the theory and applications of the subject. We hope that engineers and scientists who concern themselves with disperse systems will use these books and that those who become expert will contribute further to the series.

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Production, Handling and Characterization of Particulate Materials

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Preface

Particulate products make up around 80 % of all chemical products, from all industry sectors. The solid particles may be amorphous or have different crystalline forms, all of which have different properties. They originate from mining or other natural occurrences or are manufactured in crystallization, precipitation, agglomeration, or atomization processes. Prior to application they may undergo filtration, flotation, or classification and/or be subjected to compaction, extrusion, or comminution in order to bring them in the desired particle size range. Most often, they undergo transport and storage in some kind of equipment. “Particles” may not only be solid but also consist of entities like small droplets (by emulsification or atomization) or gas bubbles in another phase. The physical and mechanical behavior of single particles generally are reasonably well understood.

Base materials and products not only occur in the form of dry powders but also as dispersions, such as suspensions, emulsions, air bubbles in liquids, foams, or aerosols. In addition to the properties of the particles themselves, the properties of these products strongly depend upon the type of system, particulate concentration, and type and viscosity of the continuous phase, as well as upon particle size distribution and particle shape. Base materials and products have a complex nature as a result of the many possible mutual interactions between particles and of particles with chemical components in the system. Although much progress in understanding has been made over the past decades, the physical and mechanical properties of these complex systems are still much less understood than those of gases and liquids. Therefore, adequate product characterization is a challenging task. Sometimes, the products are characterized by techniques that have some more or less direct relationship with the property of interest. Often, however, various parameters derived from measured particle size distributions are linked to the properties of interest through empirical correlations. Selecting the optimum parameter(s) and measurement method(s) for characterization is one of the challenges. Finding the best correlation with behavior is the other challenge.

Although several books and many articles have been written that are specialized to one of the above unit operations or to a specific material or product, hardly any comprehensive book describes the ins and outs of all unit operations in comparison. That is the aim of this book. Specialized authors have written the various chapters. We are very grateful to all of them for their contribution and hope that our book will pay a useful contribution to further optimization of the process equipment applied for particulate materials and products.

Pijnacker, The Netherlands — Henk G. Merkus
Delft, The Netherlands — Gabriel M.H. Meesters

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Dr. Grant Ballantyne (Senior Research Fellow, the University of Queensland, Julius Kruttschnitt Mineral Research Centre (JKMRC)) is an early career researcher at the Julius Kruttschnitt Mineral Research Centre (JKMRC). Grant's three research themes are energy reduction, integration of comminution and flotation, and site optimization. His work is focused on the reduction of energy consumed through hard-rock mining, specifically rock-breakage processes. The measurement of energy use and efficiency is intrinsically involved in this objective. Grant is passionate about equipping the next generation of metallurgists through undergraduate lecturing, postgraduate supervision, and professional development courses. Grant has been involved in successful site-based industry research over the past 10 years covering four continents and eight commodities. Grant's research has gained industry recognition, especially through an international non-for-profit group called the Coalition for Eco-Efficient Comminution (CEEC).

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Em. Prof. Norman Chigier was awarded M.A. (1960), Ph.D. (1961), and Sc.D. (1977) degrees from the University of Cambridge. He has held teaching appointments at Sheffield University and Technion-Israel Institute of Technology. He was appointed William J. Brown Professor of Mechanical Engineering at Carnegie Mellon University, Pittsburgh, Pennsylvania, in 1982. Dr. Chigier was a recipient of the ASME's Lewis F. Moody Award (1965), the Institute of Fuel's Lubbock-Sambrook Award (1968 and 1975), and the Tanasawa Award (1988). Prof. Chigier's first book, *Combustion Aerodynamics* (1972), was coauthored with Prof. John Beer of Massachusetts Institute of Technology and was subsequently translated into Japanese (1976). His textbook, *Energy*, *Combustion and Environment*, was published by McGraw-Hill (1981). He is the author or coauthor of over 300 papers. In 1974, he founded the international review journal, *Progress in Energy and Combustion Science*. In 1992, Professor Chigier was named a fellow of the ASME. He is editor of the *Combustion: An International Series*, published by Hemisphere Publ. Corp. He was one of the founding members of the International

Institute of Liquid Atomization and Spray Systems (ILASS) and founding editor of the archival research journal, *Atomization and Sprays*. In 2006 he received the Arthur Lefebvre Award for distinguished contributions in the field of atomization and sprays.

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Dr. Tevfik Gemci was awarded B.Sc. (1983) and M.Sc. (1985) degrees in mechanical engineering from Istanbul Technical University. He was a recipient of a research fellowship from the German Academic Exchange Council (DAAD – Deutscher Akademischer Austauschdienst) from 1987 to 1989. He received his Ph. D. (1993) degree in mechanical and environmental engineering from the Technical University of Kaiserslautern in Germany. He was appointed as assistant professor of environmental engineering at Sakarya University in Turkey from 1996 to 1998. In 1999 he joined Carnegie Mellon University, Pittsburgh, Pennsylvania, as a research professor and worked with Dr. Norman Chigier in the Spray Systems Technology Center till 2004. He continued his academic research as research professor in the Mechanical Engineering Department at the University of Nevada Las Vegas from 2004 to 2007. He is the author or coauthor of over 40 papers. From 2008 to 2015 he has worked as an expert consultant in the field of thermal fluids science and spray and aerosol dynamics on a wide variety of industrial projects of mechanical, environmental, pharmaceutical, biomedical, and biotechnological nature. Since 2015 he started to work as a senior quality assurance and validation engineer at B. Braun Medical Inc.

Prof. Karen Hapgood spent 5 years in the US pharmaceutical industry before joining Monash University in 2006, where she cofounded the Monash Advanced Particle Engineering Laboratory. Prof. Hapgood is most well known for her work on nucleation during wet granulation, and her broader research interests include drug delivery via powders, granules, and tablets. She received the AAPS New Investigator in Pharmaceutics and Pharmaceutical Technology Award (2006), is a fellow of IChemE and Engineers Australia, and is an executive editor of *Advanced Powder Technology*.

Dr. Marko Hilden (Senior Research Fellow, the University of Queensland, Julius Kruttschnitt Mineral Research Centre (JKMRC)). Marko's research interests cover a range of topics in mineral processing and comminution including Semi-Autogenous Grinding and High-Pressure Grinding Roll modeling and optimization, mathematical modeling of mineral liberation, conceptual circuit design and simulation, and comminution energy efficiency. In 2007 he was awarded a Ph.D. studying industrial screen separation at the JKMRC. His earlier experience includes a process-improvement engineering role at iron ore operations in Western Australia's Pilbara region and as a coal technology researcher based in Melbourne.

Prof. George E. Klinzing holds degrees in chemical engineering from the University of Pittsburgh and Carnegie Mellon University and a Ph.D. degree from the later. He recently was given an honorary doctor of engineering degree from the University of Newcastle in Australia. He joined the University of Pittsburgh as a professor on its USAID Ecuador project at Universidad Central in Quito. After 3 years he returned to the main campus of the University of Pittsburgh and began his teaching and research career that has span 50 years with summer employment in the energy industries. He helped to produce 54 M.S. theses and 25 Ph.D. dissertations in the areas of mass transfer and particle technology. His research spans a wide spectrum of topics in particle technology mostly in the experimental field addressing challenging problems from pressure behaviors to electrostatic influences. He has written two books on pneumatic conveying. In 1987 he joined the administrative team of the Swanson School of Engineering as the associate dean for research followed by a 17-year stint as the vice provost for research for the University. He helped to establish university-wide interdisciplinary centers: the Center for Energy, the Petersen Institute for NanoScience and Engineering, the Center for National Preparedness, and the Center for Simulation and Modeling. He began several multidisciplinary initiatives on such topics as suffering, geriatrics and ambulatory and cognitive capacity, wisdom and aging, cybersecurity and immunology, business of humanity, nanoparticle safety, and synthetic biology. Prof. Klinzing is a fellow of the American Institute of Chemical Engineering and the American Association for the Advancement of Science. He holds the Life Time Achievement Award from the Particle Technology Forum of the A.I.Ch.E. as well as the Gary Leach Award and the McAfee Award. He serves on the editorial board of *Particulate Science and Technology* and *Powder and Bulk Engineering*. At the Swanson School of Engineering he holds a Whitford Professorship. He has produced over 250 research publications and holds 3 US patents and 6 US copyrights.

Dr. Ing. Ger J.M. Koper is associate professor at the Department of Chemical Engineering of the Delft University of Technology. He became electronics engineer in 1975 and worked as such in the Department of Histochemistry and Cytochemistry of the Leiden University from 1976 to 1985. He then moved to theoretical physics and received his Ph.D. degree from Leiden University in 1990 on *aging in spin glasses*. From 1990 until 2000 he was senior lecturer at the Physical and

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His main teaching emphasis lies on applied and advanced thermodynamics, specializing on nonequilibrium phenomena, as well as *interfacial engineering*, focusing on colloidal stability and particle synthesis. He regularly (co-)organizes national and international conferences as well as training schools for graduate students, postdocs, and professionals.

His research field is colloid and interface science with emphasis on aggregation and adsorption phenomena. His experimental expertise includes optical techniques such as transient electro-optical birefringence (Kerr effect), reflectometry, microscopy, and static and dynamic light scattering as well as dielectric spectroscopy and electro-kinetics. His current research topics are in the field of materials research for polyelectrolyte membrane fuel cells, in particular nanostructure formation, and applied thermodynamics.

His topics of particular interest include investigation of aggregation phenomena of colloidal particles and binding of protons and metal ions by polyelectrolytes.

He served in many committees and boards and currently is both member of the management team and working group leader of the COST Action CM1101 *Colloidal Aspects of Nanoscience for Innovative Processes and Materials* and newsletter editor for the *International Association of Colloid and Interface Scientists*.

Dr. Roman Latsuzbaia currently holds a position as scientist at Netherlands Organization for Applied Scientific Research, TNO, in Zeist, the Netherlands. In 2010 he received his M.Sc. degree in applied chemical engineering from the University of Manchester with a thesis on the synthesis of catalyst nanoparticles for direct methanol fuel cells. In 2011 he moved to Delft University of Technology, group of Advanced Soft Matter, and received his Ph.D. under the supervision of Dr. Ger J.M. Koper on a topic of fuel cell catalyst production and regeneration. His main areas of expertise are in fuel cells, electrochemistry, colloid and interface science, and production and characterization of nanomaterials. His current focus is in the field of electrochemical synthesis of specialty chemicals.

Dr. Lian X. Liu (Senior Research Fellow, the University of Queensland, Julius Kruttschnitt Mineral Research Centre (JKMRC)) obtained her bachelor and master's degree in mineral processing from Northeastern University, China, which is the premier institution in mining and minerals. Her master's thesis was focused on comminution processes, specifically on grinding kinetics and modeling. She did her Ph.D. at the University of Queensland in the area of population balance modeling in granulation including particle breakage processes. Dr. Liu has developed a strong research profile and expertise since her Ph.D. in granulation, breakage, and compaction of particles, through many applied research projects funded by the industry, ARC and CSIRO. She is now working at JKMRC in the areas of ore characterization and comminution process modeling.

Prof. Don McGlinchey is the head of the Centre for Industrial Bulk Solids Handling and leader of the Design, Process and Manufacturing Research Group at Glasgow Caledonian University. He has undertaken consultancy projects for both multinational companies and small to medium enterprises (SMEs) and delivered short courses in Europe and the USA. He is a chartered physicist, with a B.Sc. in physics and a Ph.D. in on the subject of the effect of vibration on particulate materials. Don is the editor of two books and has authored over 50 research articles. He is a participant in the European Federation of Chemical Engineers Working Party on the Mechanics of Particulate Solids (EFCE – WPMPS) and a member of the Institution of Mechanical Engineers (IMechE) Bulk Materials Handling Committee. His current research interests include multiphase flow instrumentation and the application of computational fluid dynamics (CFD) techniques to gas-solids conveying.

Dr. Gabriel M.H. Meesters has a B.Sc. and M.Sc. in chemical engineering with a major in bioprocess technology from the Delft University of Technology. He has a Ph.D. in particle technology also from the Delft University of Technology. He worked at biotechnology companies like Gist-Brocades in the Netherlands, as well as for Genencor International and currently at DSM in research and development in the Netherlands. In all these functions he was working on formulation and product development. Since 1996 he holds a part-time position at the Delft University of Technology, as assistant professor at the faculty of Applied Sciences, first in the Particle Technology group, later the Nanostructured Materials Group, and currently in the Product and Process Engineering group. He supervised over 15 Ph.D. students and more than 50 M.Sc. students. He published around 60 refereed papers, holds around 15 patents and patent applications, and is coauthor and coeditor of the book *Particulate Products: Tailoring Properties for Optimal Performance* (2014; Springer). He (co-)organized several international conferences in the field of particle technology and was president of the World Congress on Particle Technology in 2010.

Em. Prof. Dr. Henk G. Merkus graduated in physical organic chemistry at the University of Amsterdam. He worked several years at the Royal Dutch Shell Laboratories in Amsterdam on research in the field of detergents and industrial chemicals, followed by development work on thermal wax cracking for production of C_2–C_{14} olefins and on acid-catalyzed synthesis of carboxylic acids from C_3–C_6 olefins. Then, he made the change to analytical chemistry, involving both measurements and method development with a large variety of techniques and methods, first at Shell's process development department in Amsterdam and later in the chemical engineering department of Delft University of Technology. Gradually, the analytical horizon widened: first surface area and porosity measurements were added to chemical analysis, later followed by particle size analysis. He is author of the book *Particle Size Measurements: Fundamentals, Practice, Quality* (2009 Springer) and many journal articles, as well as coauthor and coeditor of the book

Particulate Products: Tailoring Properties for Optimal Performance (2014 Springer). Moreover, he participates in standardization activities regarding particle size measurement, both in the Dutch NEN and the International Organization for Standardization (ISO) (TC24).

Dr. Sam Palaniandy (Senior Research Fellow, the University of Queensland, Julius Kruttschnitt Mineral Research Centre (JKMRC)). His main research interest is in stirred milling and fine grinding technology. He has experience in conducting performance evaluation of stirred mills and has conducted stirred mill circuit surveys in Australia, South America, Africa, and Europe. His current activities include site surveys, development of ore characterization method for fine particles, small-scale test for stirred mills, and process modeling.

Prof. Dr. Karin Schroën obtained her Ph.D. degree in food process engineering from Wageningen University (still agricultural at the time) in 1995, working on an emulsion membrane bioreactor within the group of Klaas van 't Riet. Since then she has worked as a postdoc at University College London (UK) focusing on downstream processing of two-phase bioconversions and in the Biotechnology group of Wageningen University, studying fundamentals and process design for enzyme-catalyzed antibiotic synthesis. She became an assistant professor within the laboratory of food process engineering at Wageningen University in 2001, an associate professor in 2010, and was appointed full professor in September 2012. Her fields of expertise comprise emulsification, microtechnology, encapsulation, membrane separation, modeling, surface modification, and food process engineering in general. She teaches courses at various levels, has more than 120 scientific publications to her name, and holds seven patents.

Prof. Dr. Dietmar Schulze is professor of mechanical process engineering at the Ostfalia University of Applied Sciences Braunschweig/Wolfenbüttel since March 1996. He is an expert in the areas of powder characterization and handling (e.g., silo design). Dietmar Schulze has published more than 100 papers on powder technology including a recent book named *Pulver und Schüttgüter* and its English translation *Powders and Bulk Solids*. He received the Technology Transfer Award from the local Chamber of Industry and Commerce in 1994 and the Arnold Eucken Award from VDI in 1995.

Dietmar Schulze studied mechanical engineering with focus on mechanical process engineering at the Technical University of Braunschweig and graduated in 1985. From 1985 to 1991 he worked as scientific assistant of Prof. Jörg Schwedes at the Institute of Mechanical Process Engineering in Braunschweig. He finalized his thesis on silo stresses and discharge in 1991. After this, he and Prof. Jörg Schwedes founded the consultancy "Schwedes + Schulze Schüttguttechnik" focusing on powder characterization and silo design. Since 1993 Dr. Schulze has been developing and manufacturing ring shear testers for the measurement of flow properties of powders.

Dr. Fengnian Shi (Principal Research Fellow, the University of Queensland, Julius Kruttschnitt Mineral Research Centre (JKMRC)), a Chinese by birth, joined the JKMRC in 1988 and has been associated with the JKMRC for the past 27 years. He was awarded a Ph.D. degree at the University of Queensland in 1995. His research covers wide areas in comminution for the mineral and coal industries, including breakage characterization for ore, coal, and coke, mathematic modeling of impact crusher, SAG mill, ball mill, HPGR and vertical spindle mills, circuit simulation, and plant optimization. He jointly holds two international patents on the rotary breakage tester (JKRBT) and the breakage characterization method, part of which is presented in Section 6.4. He is a team leader of electrical comminution, developing the technology of ore pre-weakening, coarse particle liberation, and ore pre-concentration using high-voltage pulses. He has published more than 100 papers, the majority being in the peer-reviewed journals and refereed conference proceedings.

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Before joining DSM he had been working as assistant professor in the Industrial Pharmacy at the University of Groningen, where the focus of the research was on solid processing aspects of pharmaceutical production (fluid bed and high shear agglomeration and coating for controlled-release applications). He holds a Ph.D. in Chemical Engineering from the University of Groningen with the subject of multicomponent diffusion in bio-separations (ultrafiltration and chromatography).

Prof. Dr. Richard Wakeman (B.Sc., M.Sc., Ph.D., D.Tech., C.Eng., FR.Eng., F.I. Chem.E.) is a consultant chemical engineer (www.richardwakeman.co.uk) who has worked internationally in Europe, Asia, and the USA since 1971. He is an emeritus professor at Loughborough University (UK), was previously professor of process engineering at Exeter University, and has been a visiting professor at Pardubice University (Czech), a university professor at Chung Yuan University (Taiwan), and the Golden Jubilee Fellow at the University of Mumbai (India). He was awarded an honorary doctorate of technology by Lappeenranta University of Technology (Finland) in 2012.

He obtained a Ph.D. in chemical engineering from the University of Manchester Institute of Science and Technology (UMIST) after working in the chemical industry as a troubleshooting engineer; he then joined Exeter University where he later became professor of process engineering. His interests are in solid/fluid separation science and technology, membrane processes, and particle processing. He has published some 400 papers, patents, and books, and his work has received recognition through several awards, including the Junior Moulton Medal (1978), Moulton Medals (1991 and 1995) and Arnold Greene Medal (2008) of the Institution of Chemical Engineers, the Suttle Award (1971) and the Gold Medal (1993, 2003, and 2005) of the Filtration Society, and the Chemical Weekly Award of the Indian Institute of Chemical Engineers (2005).

He has been the executive editor of *The Transactions of The Institution of Chemical Engineers* and is an editor of *Filtration*. He is a fellow of the Institution of Chemical Engineers, a chartered engineer, and was elected a fellow of the Royal Academy of Engineering in 1996. He was the honorary secretary of the Filtration Society between 2000 and 2013 and is a past chairman.

Dr. Ralf Weinekötter studied chemical engineering in Karlsruhe (Germany) and Nancy (France). His Ph.D. thesis at the *ETH* Zurich (Swiss Institute of Technology) covered the “continuous mixing of fine particles.” Part of the research was executed at the Particle Technology group of DUPONT in Delaware (USA). During his time as senior lecturer at the Institute of Process Engineering *ETH* Zurich), he published with H.R. Gericke the book *Mischen von Feststoffen* (Springer Verlag 1995). This book was followed by *Mixing of Solids,* Kluwer Academic Publishers (2000). In 1994 Weinekötter joined the R&D group of Gericke AG, Switzerland. He became responsible for the development of powder mixers. Over the years he has held various positions within this international company which specialized on feeding, mixing, and conveying installations for powders. Today he is managing director of Gericke AG. He contributed to the latest edition of Perry’s *Chemical Engineers’ Handbook* with the chapter on solids mixing.

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13 years' industrial and academic experience through conducting successful industry-based research work. Planning and implementing multidisciplinary projects in comminution, flotation, and thickening for a number of industrial projects provided him a strong desire to conduct applied research underpinned by sound fundamental understanding. Since 2011, when he joined JKMRC, his research is focused mainly on two areas, namely, surface breakage of rocks and liner wear modeling. Although Mohsen is an early career researcher at the University of Queensland, he has a strong track record of publications by publishing more than 10 journal papers and presenting over 35 papers in high-profile international conferences and winning many awards for his research and papers.

Chapter 1
Introduction

Henk G. Merkus

Abstract A great variety of industrial products is based on particulate materials. Behavior and performance of such products depend upon particle size and shape in the base materials, in addition to chemical and structural characteristics. An overview of the major production and handling processes of particulate materials as well as the basic characteristics for their design, monitoring and control is presented in this book. It is meant to facilitate easy comparison and good choices of production and handling processes and of equipment. Topics for particulate production comprise size enlargement – crystallization, precipitation, granulation and extrusion – as well as size reduction, viz. comminution, atomization and emulsification. In the chapters on handling, mixing and segregation, filtration and sedimentation, flotation, classification, storage and transport are discussed. The quality of batches of base materials and products is assessed through measurements in samples. Adequate care of the sampling process to obtain representative samples is required, especially in cases where the batches show segregation, i.e. when the composition is different at different locations in the batch. Then, deviations in non-representative samples may be substantial. The quality of products is often described in terms of PSD parameters in addition to descriptors that are more closely related to behavior. Unfortunately, the choice of PSD parameters is often made without good reasoning, causing the chosen parameters to be sub-optimal. Therefore, suggestions are given for optimum choices. The behavior of bulk powders and concentrated dispersions depends in addition to particle characteristics upon the degree of particle packing and the inter-particle forces, and for dispersions also upon the particulate concentration, the surface type and the zeta-potential. Optimum rheological behavior of such concentrated systems, powders as well as liquid dispersions, is of prime importance, during processing as well as for product quality. Some examples are presented for the parameters that have an empirical relationship to practical situations. The choice of PSD parameters that are typically used for control of processes is easier than that for product quality, since usually only adequate repeatability and instrument robustness are important for good

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Table 1.1 Examples of particulate materials

Dry powders	Sugar, flour, starch, sand, cement, coal, pigments, polymer beads, peas, diamonds, toner powder, etc.
Liquid mixtures	Emulsions (L/L) + suspensions (S/L): milk, butter, margarine, creams, bacteria, blood, paint, etc.
Solid mixtures (S/S)	Ores, sediments, pharmaceuticals, etc.
Aerosols (L/G + S/G)	Fog, mist, sprays, etc., which may consist of dust, salt, sand, coal, ore, inhalers, etc.
Gas bubbles in medium	Whipped cream (G/L), insulating foam (G/S), etc.

control. This chapter gives an introduction to the general background and the challenges of selecting adequate processes and to the relevant particulate parameters.

1.1 Objective of This Book

Particulate products make up around 80 % of all chemical products. They have in common that they contain one or more particulate base materials, which may come from a wide variety of sources. Sometimes the source is natural, sometimes the materials are man-made through a chemical reaction (see Table 1.1).

Often the particles are solid, sometimes tiny droplets or air bubbles. Always, some kind of production process is involved to give the materials the desired size range and/or shape of the particles, composition and performance quality of the ultimate product.

In addition to the production processes, mechanical processes take care of mixing (to counteract segregation and/or reach a homogeneous composition), transport and storage. Optimum particle size range and shape are essential for best product quality, since these properties relate to product behavior and performance [13].

Many textbooks and articles have been written on the techniques applied for the production and handling of particulate materials. They provide a wealth of detailed information but are nearly always restricted to a single technique, base material or field of application. The objective of this book is to give an overview of all techniques with sufficient background to provide insight and understanding. Thus, it is meant to facilitate comparison of all techniques and equipment applied for improved implementation in design and control of particulate processes.

1.2 Production and Handling Processes

A wide variety of particulate materials and products exists, as illustrated in Table 1.1. Some originate from natural sources, others are man-made through some chemical reaction. Most often some mechanical processing is involved.

Given the vast differences between these materials, it is clear that they require different production techniques. The main production processes can be identified as [16, 17, 19]:

- **Size enlargement**. Particles can be formed from solutions through crystallization and precipitation. Most often, purification in addition to particle formation is the objective.

  Crystallization is executed in both batch and continuous processes. Its main goal is production of crystalline particles of high purity, usually from solutions in which other components are present as well. In batch processes crystals are formed by cooling, by solvent evaporation of saturated solutions, through addition of anti-solvents or by changing the pH; often, seeding with small particles is applied in order to start the crystallization. Continuous processes typically proceed by solvent evaporation. Here the 'seeds' are often the result of attrition of the formed larger particles. The particle size of the product typically approaches the millimeter range (see further Chap. 2).

  Precipitation results from the reaction between two (soluble) ionic compounds, the product of which is insoluble in the medium, usually water. The resulting particle size of the precipitate is very small (nanometer range), since its solubility product is very abruptly overstepped by the addition of the one dissolved compound to the other. Precipitation processes usually also involve agglomeration or aggregation of the primary particles, through which particle size is significantly enlarged (see further Chap. 3). Typically, precipitation occurs in batch processes.

  Polymerization of aqueous monomer emulsions may be considered as a variety of precipitation, since the monomers are very slightly soluble whereas the solubility of oligomers and polymers is zero. The polymerization typically starts in the continuous aqueous phase. At the point that the formed oligomers become insoluble, they form emulsion droplets (primary particles as a dispersed phase).

  During these precipitation and polymerization processes spontaneous agglomeration, aggregation or coalescence of primary particles often occurs already at early stages of particle formation and growth. This is especially true if the primary particles have little or no electric charge or are insufficiently stabilized by surfactants.

  Solid particles in the micrometer range can be enlarged to millimeter sizes by agglomeration/aggregation, granulation and extrusion. These processes are very helpful to reach the desired properties – e.g. flowability and particle shape – for the end product.

Agglomeration is the formation of clusters having point contacts between the primary particles. It often occurs not only in dry powders when the particles are smaller than about 10 μm or are humid (by formation of liquid bridges), but also in liquids when the particles have little or no surface charge. Here, it is induced by Brownian motion and stirring. The bonding strength can be substantially increased by the formation of solid bridges coming from dissolved material.

Aggregation is the formation of clusters in which the particles have a stronger bonding than in agglomerates due to the presence of side contacts. Aggregation typically takes place in liquid dispersions. Here too, solid bridges can be formed coming from dissolved material.

Note that agglomeration and aggregation may be counterproductive when they occur unintentionally as they can give rise to undefined lumps.

Granulation and tabletting is applied to improve the flow characteristics of products as well as to homogenize mixtures of ingredients. Granulation is applied for a wide variety of products. It is typically executed in equipment where the ingredient particles are continuously mixed and sprayed with binder liquid, the combination of which leads to clustering and compaction of the particles. The compacts may be used as such, but they can act also as precursors for a tabletting stage, in which they are further compressed for better strength and shape (see further Chap. 4).

Extrusion was first introduced in the processing of plastic granules. Soon after, its application expanded to metals and rubber. Gradually, it has also become very popular for food applications in e.g. cereals, pastas and snacks, since subsequent steps in the preparation process can be executed in the same extruder. In this technique use is made of the plastic deformability of products. Sufficient plasticity is usually reached at somewhat elevated temperatures, for some products in combination with the available water content in the feed (see further Chap. 5).

- **Size reduction**. Often, the dry solid particles produced are too large for their envisaged application in a product. One example is when they result from mining of ores. Another reason may be that the drying process involved in particle production has caused the presence of large lumps that consist of agglomerates and/or aggregates. Then, particle size must be reduced by *comminution (crushing or milling)*, which involves dry or wet processes of breakage, attrition and de-agglomeration. Comminution operations usually consume a lot of energy. Besides differences in equipment, this causes that the total comminution operation often is split into several stages for better economy (see further Chaps. 6 and 11).

  Crushing involves usually dry breakage of large pieces from up till about 1 m into cm-sized particles. Crushers of various types are typically applied for (fairly) hard materials, cutters for tough ones.

  Milling is executed in both dry and wet state to result in millimeter-sized or smaller particles. Dry milling usually is performed in roller mills, wet milling in ball mills or bead mills. The advantage of wet processes is that the resulting small (micrometer-sized) particles can be stabilized to prevent re-agglomeration and aggregation.

Dispersion of dry powders in a liquid is also often called milling. It is applied to break up agglomerates and when stable particulate suspensions are required. It involves application of energy and dispersants/surfactants to reach sufficient de-agglomeration and suspension stability.

Atomization, spraying and nebulization are processes of producing droplets from a liquid in an air stream. If solid particles are aimed at, then the liquid is a melt or a solution, and the droplet formation is followed by cooling or drying. The resulting size of droplets and particles may vary from millimeters to smaller than micrometers (see further Chap. 7).

- **Emulsification**. Here, the particles are in liquid form. They are dispersed in another, immiscible liquid, typically through application of high energy/shear in various types of equipment and stabilized by means of surfactants/emulsifiers. This is a special way of size reduction for liquids. Particle size is usually in the nano- to micrometer range. Typical applications of emulsions are in food, pharmaceuticals and lubricants. Adequate stabilization is required to guarantee a long shelf life (see further Chap. 8).

In addition to production processes, some kind of mechanical processing is often involved, in which particle size is not intentionally altered:

- **Mixing**. Mixing is an important process step for particulate materials. Its goal is to reach a homogeneous product. For single particulate materials, the reason for mixing is the existence of (undesired) differences in particle size distribution at different positions within a product batch. This may be caused by process fluctuations, but more often it is mainly the result of segregation (see below). Always, the degree of segregation shall be checked in batches of particulate materials.

  Segregation is the opposite process from mixing. It usually occurs in dry, free-flowing materials in relation to differences in particle size and/or density. It is induced by vibration of the materials during transport or by aeration upon storage in silos. The detrimental effect of such heterogeneity on product quality can be severe, since product properties relate to particle size and its distribution. Note that some degree of segregation especially in free-flowing materials has to be accepted in practice since ideal mixtures do not exist in practice.

  Mixing of a segregated material batch is required to reach a sufficiently homogeneous size distribution within the batch. Moreover, it is essential for particulate products when they are composed of different ingredients. For such products, mixing is necessary to reach a homogeneous product composition, in addition to particle size distribution. Especially for products, which are composed of small quantities of a given component and a majority of other particulate material(s), such as active material and excipients for pharmaceutical pills, adequate mixing before further processing is essential. Decreasing the particle size to below about 20 μm or making the powder humid can be used to counteract segregation during mixing. Both measures increase the particle-particle interactions and make powders cohesive, thus resulting in lower or no sensitivity to segregation. Note that cohesive powders may greatly challenge steady transport and adequate silo charge and discharge. Thus, these powders require adequate choices in the design and operating conditions of the equipment (see further Chap. 9).

- **Filtration/sedimentation**. Filtration and sedimentation are typically applied to separate solid particles from a liquid or air, in which they are produced or dispersed. Sometimes, the goal is collection of the particles, sometimes it is obtaining a clear liquid or clean air. This may be the case for protection of the environment or for obtaining a clean room. Typically, the efficiency of collection is expressed in a mass percentage of collected particles in comparison to originally present particles. The quality of clearing of a liquid is usually expressed in a degree of remaining turbidity or percentage transmission, for clean gas or clear liquids in the mass or number of particles smaller than a given size per unit volume, e.g. *PM10*.

  Filtration uses the porosity of mechanical filters or particulate filter beds for particle collection or removal from suspensions. Typically, removal of particles from suspensions concerns particle sizes larger than about 1 μm; in air, it is usually below 1 μm. Mechanical filters in the form of sieves can only remove particles if they have smaller aperture sizes than the size of the particles. Therefore, industrial sieves are limited to collection and removal of larger particle sizes. An advantage of sieves is that the particles can be easily separated from the sieves. Filter cloth and particulate filter beds remove particles based on the existing void sizes and the possibilities for adsorption. Because of the presence of very small voids and the adsorption possibilities, also submicrometre particles can be removed. Well known examples of application of filter beds are beer filtration and application of sand beds for filtration of drinking water. Filter cloth is e.g. applied for obtaining clean air. Recovery of the particles from the cloth or the beds is usually incomplete (see further Chap. 10).

  Sedimentation is the result of positive density differences between particles and suspension liquid, which cause settling of particles when they are larger than about 1 μm (depending on density). Sedimentation of smaller particles or when the density difference is small can be enhanced in centrifuges. The opposite case – particle density smaller than that of the liquid – is named *creaming*. A well known example is the creaming of milk. Sedimentation can be applied for clearing of liquids as well as for particle collection. The latter typically requires some kind of filtration following sedimentation, and drying of the resulting cake. Various types of sedimentation are also applied in classification (see below and also Chap. 10).

- **Flotation** is another way to separate solid particles from a liquid. It is usually applied in cases where specific minerals are to be separated, or Brownian motion prevents particles from adequate sedimentation, or the density difference between particles and liquid is small. In this process, various types of chemicals (pH adjusters, surfactants and ionic substances) are applied to make the surface

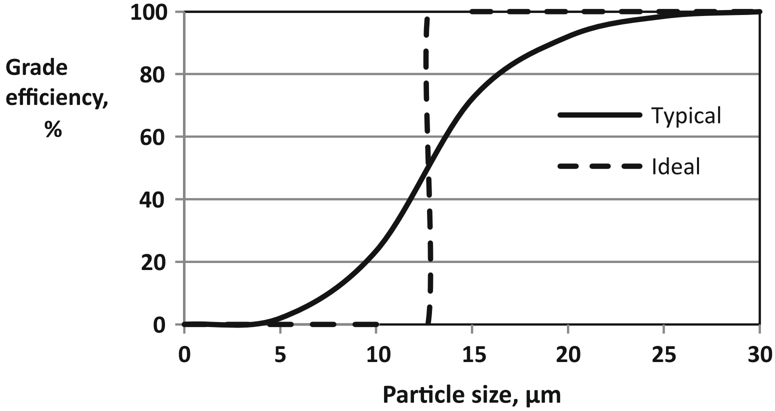

Fig. 1.1 Grade efficiency curve for a gas cyclone, showing typical and ideal separation (Adapted from [16])

of target (mineral) particles hydrophobic and to generate air bubbles to which the hydrophobic particles are to be attached. This air causes the particles to float as a froth at the liquid surface, which facilitates their removal from the liquid. This process is e.g. widely applied in the production of valuable minerals from ores and in waste water treatment (see further Chap. 11).

- **Classification**. Classification of produced particulate materials is applied if their size range does not match the desired size range, by removing fines or coarse particles, by separating particles having a different density, or to remove particles completely from a gas or liquid stream. Typically, (hydro)cyclones, sieves or gravitational or centrifugal sedimentation units are applied, in particulate mixtures with air or liquid. For dry mixtures, also magnetic and electrostatic separators are used. (Hydro)cyclones typically operate at particle sizes in the range of about 1–500 µm (depending on particle and fluid density). The grade efficiency for separating particles having a size distribution into fractions of fine and coarse product is often used to sketch the separation efficiency against particle size. As an example, a typical grade efficiency or Tromp curve for a gas cyclone is presented in Fig. 1.1 (see further Chaps. 10 and 12).
- **Storage and powder discharge**. Intermediate storage in bins, silos or heaps is required when production and usage are not in a direct sequence but at different times or locations. Silos have the advantage that they provide for a closed environment so that effects by humidity or oxygen can be avoided. Note that bulk storage always leads to some kind of consolidation of the lower parts of the stored material, which increases its bulk density and may decrease its flowability. Note also that segregation often occurs during the total of feeding, storage and discharge. Moreover, poor silo and hopper design typically leads to problems upon discharging the particulates. Then, this discharge from the silo may proceed only for a small part while the product near the silo walls remains stagnant. In order to avoid such problems, flow properties and bulk density of the

product to be stored should be determined and hoppers and silos, including inlet, outlet and additional equipment, be designed in relation to potential stresses and segregation. This is essential for optimum operation (see further Chap. 13).

- **Transport/conveying**. Often, production, storage and further usage do not occur at the same location but at some distance. Then, transportation of the material is necessary. Different types of equipment are applied, which all have their economical and technical pros and cons. Pneumatic and hydraulic conveying in pipelines is often favored for short and medium long distances (up till a few kilometers). If the transport distance is short, belts are used also. For long distances, product batches are transported in bags, trucks, wagons and ships. Note that segregation and dusting often occur during transport and, thus, should be taken care of. Note further that generation of electrostatics in powder flows may be strong, which can cause dust explosions resulting in dramatic casualties and losses of property. Prevention of dust explosions makes adequate care for good grounding and nonconductive piping, etc. in pneumatic conveying essential (see further Chap. 14).

Note: One of the founding fathers of particle technology, Professor Hans Rumpf of the University of Karlsruhe, named the above unit operations mechanical process technology (mechanische Verfahrenstechnik), because they deal with the transformation of material systems by predominantly mechanical operations [18]. Still, they are the subject of R&D and education in the corresponding departments of many German technical universities. In many other countries, at least some of the unit operations are studied and taught in other departments, such as chemical, civil and equipment engineering. Major companies typically form project groups of experts from different departments for the task to make optimum choices for all processing equipment and the appropriate operating conditions for a given material or product.

Typically, the processing of particulate materials is considered as very complex in view of the many parameters that can play a role, such as different phases (solid, liquid and gas), particle size distributions, different particle shapes, often high concentrations causing particle-particle interactions, etc. It is impressive to see that, despite this complexity, significant progress has been made in the development of modeling and simulation packages that approach reality, although they are still mainly based on empirical equations (see Chaps. 2, 3, 6, 12, and 14).

1.3 Characterization of Particulate Bulk Products and Processes

As shown in Table 1.1, particulate materials in a wide variety form the basic materials for many industrial products, in almost all industrial areas. Typically, each product requires a specific particle size range, particle shape as well as bulk properties for its base material(s) in addition to specific other components in the end products. The reason is that the performance quality of particulate materials and products depends upon particle size, size distribution, shape and bulk properties, since these relate to the way that the particles interact with each other and the surrounding medium. Quality aspects of bulk products involve for example [13, 19]:

– rheological properties of powders (important for steady flow and dosage)
– fluidization behavior (important if a fluid bed is applied in its application)
– dusting behavior (related to health hazards and explosion risks)
– particle packing density (related to e.g. bulk density, concrete strength, resistance of filter beds to fluid flow and rheological behavior)
– rheological behavior of dispersions, especially at high particulate concentrations (important during processing as well as with respect to e.g. taste or paint application)
– optical properties (related to color, gloss, transparency and hiding power of paint and sunscreen)
– surface properties (chemical and physical) and zeta-potential
– dissolution rate
– sensorial characteristics (e.g. taste of food, sweets and beverages).

Characterization of industrial materials is required in order to test whether they comply to their required quality and specifications, or for monitoring and control of the process, or with respect to environmental requirements in view of health and explosion behavior. This characterization usually involves both the particles and the bulk product. Measurement of particle size distribution (PSD), shape and/or porosity of the particles is often the first approach as it is fairly easy. Characterization of the bulk properties is usually second; it is more complex and product specific, but at least as important. It regards bulk density and rheological properties, which are essential in relation to handling and processing (see further Chaps. 5, 13, and 15).

Typically, test samples are used to characterize the quality of material batches. Always, such samples shall be representative for the product batch and contain a sufficient number of particles over the full size distribution. Most often such representativeness can only be obtained, due to segregation and/or process fluctuations, by collecting samples at different times during particulate flow or from different locations in a bulk, mixing them to reach a composite sample and, if necessary, dividing this composite sample to reach a smaller test sample. Note that sometimes very small test samples are used for analysis. In such small samples, the number of particles present may severely limit the quality of the results. ***Especially highly segregated materials require adequate quality of the sampling procedure, which always should be checked***. For compliance testing, the materials should preferably be analyzed after production as well as before application, in view of potential segregation during transport and storage (see Chaps. 9 and 15).

The quality criteria for the total analytical method, including sampling, sample preparation and measurement, are dictated by the performance requirements of the ultimate product. In general, the analysis aims at characteristics of the particles (size distribution, particle shape and porosity), at bulk solids properties as bulk density, flowability and wall friction, and for dispersions at particulate concentration and rheological behavior.

Measurement of the PSD is often the first approach for product quality control in view of the ease of execution and its relationship with both bulk properties and product quality. Its measurement method requires, besides obtaining a representative test sample, also adequate dispersion, concentration range, size range, analysis time, precision (both repeatability and reproducibility), accuracy, resolution and sensitivity. Note that inadequate sampling and dispersion as well as incompetent analysts, who deviate from given instructions or overlook unexpected behavior during analysis, may strongly deteriorate the quality of the analysis results [12]. Moreover, the optimum control of many processes requires very fast availability of measurement results, for which PSD parameters are often adequate. To reduce human involvement, various kinds of commercial samplers and on- and in-line PSD measurement instruments have been developed for application in the control of these industrial processes. On the other hand, analysis of the bulk properties is essential when particle-particle interactions play an important role in the product performance.

The properties of single particles strongly depend upon particle composition, type and size. For near-spheres, the relationships are generally well understood. For example, color and transparency strongly depend, in addition to chemical nature, upon both crystal type and particle size, especially when the size is in the same range as the wavelength of the light. Good examples are pigments in paints and sunscreens [13]. For non-spherical particles, however, the understanding is hampered by the influence of particle shape.

The behavior of bulk particulate materials and products may be very complex since the inter-particle distance is often small and mutual interactions of the particles and with the surrounding medium play a role. Mechanical interactions between particles dominate in dry powders. If the particles are regular and have sizes larger than about 20–50 μm in a (medium-) narrow size distribution, the powders show a behavior that depends mostly upon bulk density and size distribution (degree of particle packing and strength of attractive forces). They are usually free flowing, since the inter-particle attractive forces are small in comparison to particle mass. The consequences are that they can move easily but also segregate easily.

An example of the influence of particle size is sketched in Fig. 1.2 for the fluidization behavior of powders [6, 13].

If the particles in a powder show a broad size distribution, which is the case for most industrial products, then the bulk density strongly depends upon the history of aeration, consolidation and vibration. Aeration during storage often leads to relatively small bulk densities especially when the particles take their first position while leaving a lot of empty space. Tapping, vibration and consolidation generally result in significantly greater bulk density than in the loose state after pouring. This is because the smaller particles may fill the voids in between the larger ones, but only after breaking the attractive inter-particle forces by the induced external forces. Higher bulk density also means that bed porosity is significantly smaller and mechanical interactions larger. Near maximum packing the mobility of the particles is strongly reduced due to mutual hindrance.

Note that particles having a substantial static charge at their surface behave in the same way as with a high degree of aeration since repulsive forces between the

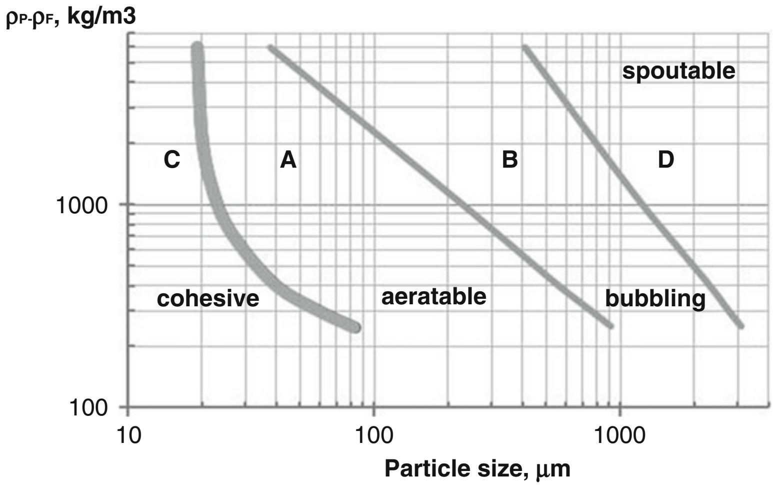

Fig. 1.2 Geldart fluidization diagram [13] (Adapted from [6]); (Copyright Springer; reproduced with permission)

particles dominate in the behavior. An example is polymer powder, which can build up charge by friction. This charge causes that particles repel each other and cannot reach close proximity.

Dry powders, where the particles have sizes smaller than about 20 μm and/or have shapes like plates or fibers, behave quite differently from powders containing larger, regular particles. They are cohesive already in fairly loose packing, since the inter-particle forces are large in comparison to particle mass and, thus, dominate in the behavior. This makes such powders very resistive to flow, especially after some kind of consolidation (as e.g. occurs during storage), when inter-particle distances are decreased. Presence of small amounts of water in a powder enhances this phenomenon of cohesivity.

Several performance quality aspects of products, for example cohesivity, flowability, rheological behavior, shear strength and taste, can be measured more or less directly through standardized measurement methods (see further Chaps. 5, 13, and 15).

The rheological behavior of bulk powders is usually characterized through shear tests and measurements of bulk density (free and tapped), angle of repose and wall friction. Taylor et al. [22] have concluded through principal component analysis that a combined flowability index based on measurements of critical orifice, compressibility and angle of repose provides a better product characterization than the individual tests. The different facets of the flow properties of powders also can be illustrated in so-called spider diagrams, based, for example, on measured characteristics of bulk density, wall friction, shear strength, Hausner ratio and some equipment characteristics such as hopper wall angle for mass flow and critical outlet size. Such diagrams present a nice overview of powder flow behavior and equipment characteristics through ranging the various parameters in terms of easy, modest and poor flow. Thus, they also highlight specific aspects that deserve more attention. This is advantageous for application in hopper design (see further Chap. 15).