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## Phenomena, Technologies, Equipment

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**Cover Illustration**
Like an agglomerate, the picture on the cover is composed of many disparate components, all of which relate to the topics discussed in this book. The panels on the left and right are microphotographs of naturally agglomerated nano-particles. The top and the bottom panels depict different products from spray drying and fluid bed agglomeration. The four sectors (between the panels and the circle) represent Scanning Electron Micrographs (SEMs) of agglomerate structures as well as photographs of coated agglomerates and of granules. The top half of the circle shows products from tumble/growth agglomeration and the lower half are briquettes from roller presses as well as product from compaction/granulation. The center square includes tablets from punch and in die presses. The originals of the individual pictures from which sections are reproduced were supplied by (in alphabetical order): Albemarle Corp., Baton Rouge, LA, USA; Cabot Corp., Tuscola, IL, USA; Eirich, Hardheim, Germany; Euragglo, Qievréchain, France; Niro A/S, Soeborg, Denmark; Norchem Concrete Products, Inc., Fort Pierce, FL, USA; Köppern GmbH & Co, KG, Hattingen, Germany. Their support is appreciated and acknowledged.



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## Dedication, Acknowledgements and References

When this book was first planned, the idea was to combine in one volume concise descriptions of the agglomeration phenomena, technologies, equipment, and systems as well as a compilation of the applications of agglomeration techniques in industry. The latter was intended to demonstrate the widespread natural, mostly undesired occurrences of the phenomena, including possibilities to avoid them, and discuss the varied old, conventional, and new beneficial uses of the technologies.

However, it soon became obvious that, in its entirety, this project became too voluminous and required much more time than anticipated. Therefore, it was decided to split the subject's presentation into two volumes whereby both books will be "stand alone" publications that are also complementary.

The first volume, available here, covers the fundamental phenomena that define agglomeration as well as the industrial technologies and equipment for the size enlargement by agglomeration. Applications are mentioned in a general way throughout the text of this presentation but without going into details. These applications will be presented in a separate book entitled "Agglomeration Technologies – Industrial Applications" that is scheduled for publication in 2003. A preliminary table of contents is given in Section 13.4.

Many persons, institutions, and companies have contributed to the two volumes of this book.

First and foremost, I wish to thank my wife Hannelore for her support and understanding while, thorughout my professional career, I was compiling various papers and books (see Section 13.3). All are dedicated to her. Without my wife's active participation in preparing almost all publications, in elaborating the textbook entitled "Size Enlargement by Agglomeration" [B.42], which is a major reference for this publication (see also below), and her, if sometimes reluctant, acceptance that I was not available for long hours on many days during almost four decades, the books, in particular, could not have been completed.

It is impossible to acknowledge all the help, extensive and small, that was provided by a large number of individuals and companies. In Section 14.1, a list of vendors and other organizations is compiled which mentions those who have, in one way or another, contributed as well as some others who may be of interest as potential contacts for the readers of this book. While I have decided not to clutter the text with references, sources have been acknowledged if figures or tables were provided by or are based

on information from particular companies. The Discialmer at the beginning of this book (see page IV) should be referred to when using such cross-references.

Regarding references to literature, Chapter 13 should be consulted. The earlier textbook "Size Enlargement by Agglomeration" [B.42] contains treatments as well as many references relation to the developing science of the unit operation and covers in some detail the sizing of and scale-up methods for agglomeration equipment. Since the emphasis of the new book is on practical considerations and industrial applications, not theory, the earlier book "Size Enlargements by Agglomeration" (Wiley, 1991) should always also be referred to. Information on the availability of reprints is available at the beginning of Section 13.1 and as a footnote later in the same Section.

Since Size Enlargement by Agglomeration is one of the unit operations of Mechanical Process Technology (see Chapter 1) and, for the design and construction of agglomeration systems of any kind, many or all of the other unit operations are required, together with the associated transport and storage technologies, often even in multiplicity, and the analytical methods are applied for process evaluation and control, the reader who is interested in the topic of this book should also learn about or have access to information on the other fields of Mechanical Process Technology. This is also emphasized in Chapter 13. At this point I wish to acknowledge two books of general importance to which I have contributed chapters on agglomeration and of which major portions were included in this book. They are: "Handbook of Powder Science and Technology" M. E. Fayed, L. Otten (eds,), 1st ed., Van Nostrand Reinhold Co., New York, NY (1983) and 2nd ed., Chapman & Hall, New York, NY (1997). Source references can be found in [B.21] and [B.56], Section 13.1.

Finally, I like to mention with gratitude the following individuals who, as professionals and experts in their own fields, are or have been colleagues and/or partners in several continuing education courses over many years in the USA as well as in Europe. They have agreed that statements during their presentations and the elaborations for their course notes can be used directly, adopted, or modified for this book. They are, in alphabetical order: T. van Doorslaer, W. E. Engelleitner, M. E. Fayed, M. Gursch, D. C. Hicks, S. Jagnow, R. H. Leaver, R. Löbe, K. Masters, S. Mortensen, H. B. Ries, F. V. Shaw, J. Storm, R. Wicke, and R. Zisselmar.

For additional references and acknowledgements please refer to Sections 13.1 and 13.2.

Naples, November 2001 *Wolfgang Pietsch*

# Contents

# 1 Introduction

In 1957, under the leadership of Professor Dr.-Ing. Hans Rumpf at the Technical University (TH) of Karlsruhe, Germany, **Mechanical Process Technology** or **Particle Technology** [B.11] was first introduced as a field of science in its own right. It comprises the interdisciplinary treatment of all activities for the investigation, processing, and handling of solid particles as well as the interactions of such particulate solids. Four unit operations and associated techniques were defined (Fig. 1.1). Other common English names for this field of science, which was quickly adopted around the world, are **Mechanical Process Engineering, Powder Technology**, or **Powder & Bulk Solids Technology.**

**Size enlargement by agglomeration** is the generic term for that unit operation of mechanical process engineering which is characterized by "combination with change in particle size" (Fig. 1.1). The author of this book had the privilege to become one of the first assistants of Professor Rumpf. For several years he was responsible for the research and development of size enlargement by agglomeration at the Institute of

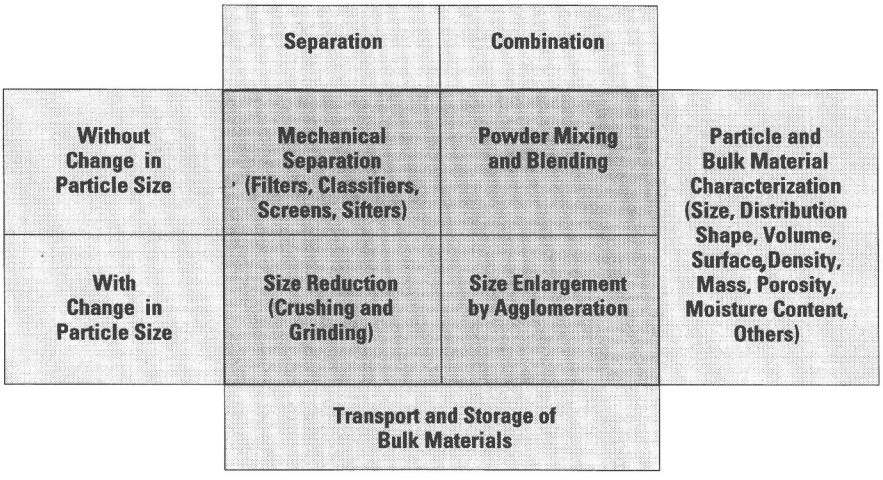


**Fig. 1.1** Unit operations and associated techniques of Mechanical Process Technology

Mechanical Process Engineering and earned his PhD with a doctoral thesis on specific aspects of a binding mechanism [1.1] of agglomeration.

Webster's Unabridged Third New International Dictionary [1.2] defines *the verb* **agglomerate** as: "to gather into a mass or cluster; to collect or come together in a mass; to collect into a ball, heap, or mass, specifically: clustered or growing together but not coherent", and *the noun* **agglomerate** as: "a cluster of disparate elements; an indiscriminately (= randomly) formed mass". A technical dictionary [1.3] defines **agglomeration** as: "sticking or balling of (often very fine) powder particles due to short range physical forces. Therefore these forces become active only if the individual particles (forming the agglomerate) are brought closely together by external effects".

These definitions distinguish the term **size enlargement by agglomeration** from the more general **size enlargement** such that particle growth occurring, for example, during crystallization or the production of particulate solids by melt solidification are not part of this unit operation of Mechanical Process Technology.

# 2
# A Short History of Agglomeration

As a *basic physical effect,* agglomeration has existed since particulate solids were first formed on Earth. Binding mechanisms between solid particles cause the stability of wet and dry soil and (often under the influence of heat and pressure) participate in the development of rock formations. Sandstone is the most easily recognized "agglomerate". Agglomeration as a *phenomenon,* e.g. the natural caking and build-up of particulate solids, must have been observed and has been used by higher developed organisms and later by humans since prehistoric times. Sea creatures covered themselves with protective coats, birds as well as other animals built nests, and humanoids formed artificial stones, all from various solids, sand, clay and different binders that were often secreted by the creature itself. As a *"tool" to improve powder characteristics,* agglomeration was used by ancient "doctors" in producing pills from medicinal powders and a binder (e.g. honey) or by food preparers during the making of bread from flour whereby the inherent starchy components act as binder.

In spite of this long "history", agglomeration as a *technology* is only about 150 years old today (excluding small scale pharmaceutical and some little-known ancient, mostly Chinese applications as well as brick and bread making). Agglomeration as a *unit operation,* defined within solids processing, started around the middle of the nineteenth century as a method to recover and use coal fines.

Agglomeration as a *science* is very young. It began in the 1950s with the formal definition of the binding mechanisms of agglomeration, interdisciplinary collection of knowledge relating to all aspects of agglomeration, and fundamental research which was no longer application oriented [B.42]. At approximately that time, the first recurring series of professional meetings were organized which were exclusively devoted to the science and technology of agglomeration (International Briquetting Association (IBA), – today Institute for Briquetting and Agglomeration (IBA) –, beginning in 1949 with biennial meetings and proceedings; International Symposia on Agglomeration, initiated in 1962 with proceedings, (see also Section 13.1)). Since that time, agglomeration science, technology, and use have experienced rapid growth but still without finding a corresponding awareness at institutions of higher learning and in the technical or process engineering communities.

This book is the second by the author on the general subject of size enlargement by agglomeration. While frequently referring to fundamentals and specifics which are

covered in more detail in the first book [B.42], this new text tries to provide an updated, comprehensive summary of the state-of-the-art of agglomeration, its basics, technologies, and applications, at the beginning of the 21st century.

# 3
# Agglomeration as a Generic, Independent, and Interdisciplinary Field of Science

As mentioned in the previous chapter, size enlargement of particulate solids by agglomeration is as old as the existence of solids themselves. Originally, agglomeration happened naturally during the development of soil, stone, and rock formations. Later, unwanted agglomeration occurred during handling and storage of particulate matter particularly when hygroscopic and/or soluble materials (such as salt) "set-up" into lumps or large, more or less solidified masses. In the animal world agglomeration was used to develop protective coatings (e.g. many marine worms, Fig. 3.1), to build nests (e.g. swallows, termites, Fig. 3.2), and to provide a nourishing and protective environment for the offsprings (e.g. dung beetles, Fig. 3.3).

Humans most probably first used agglomeration during the making of bread by taking flour (= particulate solids including an inherent binder, starch) and liquid additives (= additional binder for plasticity and "green" bonding), mixing and forming the mass, and, finally, "curing" the product, the removal of much of the moisture that was added during the mixing and agglomeration steps, to obtain structure and permanent bonding during baking. The **technology of bread making** combines all com-

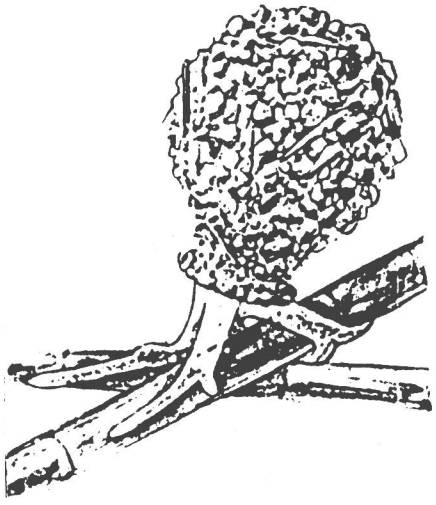

**Fig. 3.1** Protective agglomerated coating of a Rhizopod, a creping marine Protozoan (Difflugia urceolata)

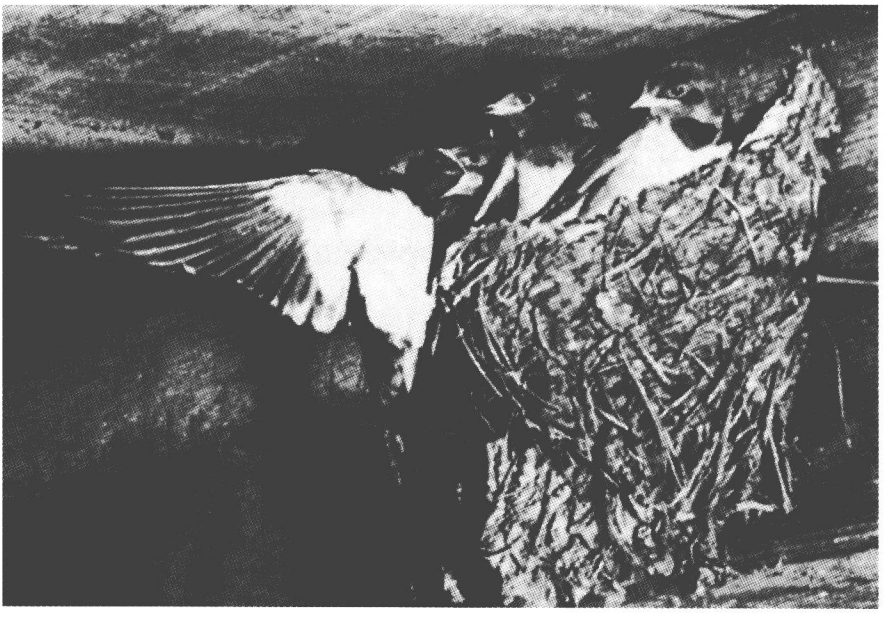

**Fig. 3.2a** Nest of swallows made by agglomeration from mud, the bird's saliva as a binder, and organic fibers for strengthening

**Fig. 3.2b** Nest of termites made from earth as well as the animal's excrements and/or secretions as binder

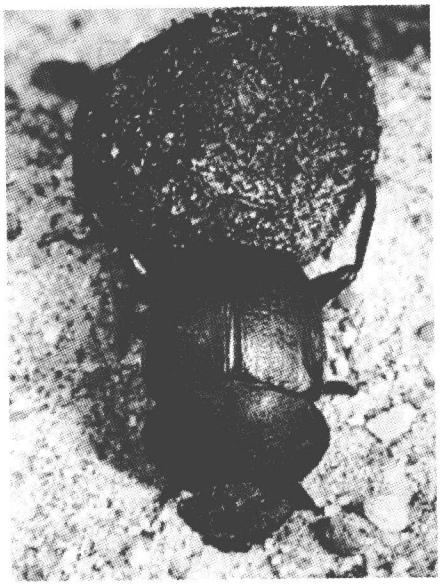

**Fig. 3.3** Dung beetle, Scarabaeus Sacer, "pelletizing" dung

ponents of a complex agglomeration process including preparation of solid feed particles by milling (= adjustment of particle size and activation of the inherent binder, starch), mixing of particulate solids with additional binder(s), forming the mass into a "green" agglomerate, and a "post-treatment" (curing = baking = heating and cooling) to provide strength and texture. Very early it was also found that the porosity of the final product could be modified (= increased) by making use of gases that are produced during fermentation (initiated by sour dough or yeast) and result in bubbles in the green mass. These voids are stabilized by "strengthening" the bread during post-treatment (baking).

For the construction of permanent shelter, humans may have observed the activities of animals that formed nests and protective "walls" from wet clay which hardened during drying (Fig. 3.2). By copying this behavior, wet clay, which was soon reinforced and made more water resistant by mixing-in straw or other fibrous material, was filled into a framework of wood branches and let harden during natural drying. To make building activities independent of the location of clay "mines", during prehistoric times bricks were already produced from clay and sand and, after hardening, transported to building sites. Since fire was known for providing heat, the accidental "firing" of a piece of clay most probably resulted in the adaptation of an improved post-treatment that yielded waterproof bricks for areas where rock was not easily available, thus allowing the development of villages and, during the 4th millenium B.C. in Mesopotamia, cities with large brick structures.

By experience, humans learned that certain natural materials helped cure specific illnesses. Minerals as well as dried animal and plant matter were ground to powder and "formulated" to yield medicines. Since powders cannot be easily consumed orally, natural binders, such as honey which, incidentally, also masked the unpleasant taste of

some of the medicinal components, were mixed with the powder and the resulting plastic mass was rolled by hand into little balls (= pills). The sticky binder(s) caused pills to adhere to each other; therefore, it was soon found that coating the pills by rolling them in flour or pollen solved this problem (see also Section 10.1).

These three, well known ancient agglomeration techniques were used with little change through the ages of human history. Many other, lesser known and somewhat more recent processes could be added. However, it is not the objective here to produce a history book. Rather, these examples relating to three major modern "industries", food, building materials, and health products, were selected to show that humans always lived with and used agglomeration. As a result, agglomeration technologies as all the other unit operations and associated techniques of Mechanical Process Technology (see Fig. 1.1) were considered to be "normal activities" which, with the beginning of industrialization in the 18th and 19th centuries A.D., were merely mechanized by simulating what was done manually before. During these early modernization efforts it was not considered necessary to question the fundamentals of the processes and "improvements" were based on empirical developments.

Until very recently, agglomeration technologies had been developed independently in the particular industries in which they were applied. Because the process requirements are fundamentally different in such unlike industries handling, for example, coal and ores on one hand or food and pharmaceuticals on the other, no interdisciplinary contact and exchange of information took place. In fact, although agglomeration techniques developed along similar lines, application related "theories" were defined which were derived from investigations of specific requirements and their solutions together with a terminology that was often incomprehensible and, therefore, not useable by the "agglomeration expert" of another industry (see Chapter 4).

Agglomeration as a science began when an effort was made to interdisciplinarily combine the extensive knowledge that had been accumulated during sometimes hundreds of years in specific fields of human activities. This approach showed that (in alphabetical order, not indicating importance):

*Baking:* A thermal post-treatment process, does not only induce the development of final bonding, structure, and consistency in bread but produces similar characteristics during the heat curing of any "green" agglomerate.

*Briquetting:* Is not predominantly a technique for the enlargement of coal fines for beneficial use but equipment which was specifically developed for that application is also suitable for such diverse uses as, for example, the briquetting of salt for the regeneration of water softeners, the briquetting of flaked DMT to decrease the bulk volume and improve handling and shipping, the briquetting of frozen vegetable pulps to be used as rations for field kitchens, the hot briquetting of sponge iron to reduce this commodity's reactivity and allow open handling and storage, or the production of fertilizer spikes and the manufacturing of artificial fireplace logs.

*Coating:* Is not only suitable for the modification of surface characteristics or the control of dispersion and dissolution of medicinal specialties but also to achieve similar properties in agrochemicals as well as human and animal foods, among others.

*Compacting:* Is not only applicable for the production of "green" bricks or other ceramic bodies prior to firing but finds many uses in powder metallurgy or for the production of battery cathodes, etc.

*Granulating:* Is not primarily a method to improve flowability of powders and formulations in the pharmaceutical industry but also in the fertilizer and bulk chemical industries as well as for carbon black, silica fume, and many other materials.

*Instantizing:* As an example of a relatively new agglomertion process, is not limited to applications in the food industry for easily dissolvable drink and soup mixtures but is equally important for pigments, insectizides, fungizides, and many more.

*Pelleting:* Originally developed for the shaping of animal feed formulations by extrusion, is also applicable for the production of catalyst carriers and other materials requiring uniform size and shape together with relatively high porosity.

*Sintering:* When going back to the fundamentals of this process, was found to be not only a high temperature process for the agglomeration of ores but, at much lower temperatures, also for plastics and other man made powders with low melting points or softening ranges, and, quite obviously, for powder metallurgy, mechanical alloying, or the like at many different temperature levels including extremely high ones for refractory metals.

The above is only a small selection of the many diverse applications of particular agglomeration methods which, in all the different environments, follow the same fundamentals, apply the same rules, and use essentially the same equipment and systems if looked at from an interdisciplinary point of view. Although these facts become more and more known, there is still the understandable preconceived notion of, for example, somebody working in an ultraclean environment, such as the pharmaceutical, food, or electronic industries, that developments, expertise, and know-how gained in the "dirty" plants of, for example, minerals or metals production and processing, can not be considered as valid information that may be applied for the solution of a "clean" problem – and vice versa. In the case of "dirty" industries, a typical concern is that the often more deeply and completely investigated technologies originating in "clean" industries can not be applied because the production capacities are too small, the process may be batch, the equipment too complex, the execution and the materials of construction too expensive, etc., etc.

However, as will be shown among other topics in this book, methods for the selection of the most suitable agglomeration process for a specific application (see Section 11.1) are the same for all projects. While some requirements, for example in regard to equipment or system capacity, or on the shape, size, and special properties of the products, may result in the definition of "cleaner" or "more heavy-duty, rugged" processes already in the preselection phase, the normal approach is to determine the preferred method and/or technique by considering the fundamentals as well as an interdisciplinary pool of expertise and know-how first. Conditions of the particular application such as, for example, "hot and dusty large volume processing", or the opposite, "clean, small capacity operation with cGMP (= current Good Manufacturing Practice) and CIP (= Cleaning In Place) capabilities" are special design criteria that can be added to most of the systems later during the engineering phase.

Nevertheless, for manufacturing reasons and sometimes also because of special requirements on the company's test facilities (see Section 11.2), some vendors specialize in equipment for one or the other industry. This is a decision of convenience by the individual supplier and does not indicate the existence of a fundamentally different technology. In fact, techniques or apparatus that were developed for a specific industry can be adopted for use in areas with different environment and requirements while still maintaining the fundamental underlying principle as well as the general machinery and process. Examples are flaking (see Section 8.4.3), instantizing (see Section 5.4), spheronizing (see Section 8.3), and spray dryer agglomerators (see Sections 7.4.3 and 7.4.4).