

Granules Production – Easy and Cost-effective



Dipl.-Chem. Dr. Peter Nold studied chemistry at Friedrich-Alexander-Universität in Erlangen. For more than 20 years he worked at Didier-Werke AG in Wiesbaden. Since 2000 he has been working for Maschinenfabrik Gustav Eirich.



Dipl.-Ing. Ralf Löbe studied chemical process technology at Fachhochschule Mannheim. For five years he worked at Drais-Werke, Mannheim. In 1994 he changed to Maschinenfabrik Gustav Eirich, Hardheim. There he is head of the Test Center.



Dipl.-Ing. Marcus Müller studied chemical engineering at Friedrich-Alexander-Universität in Erlangen. Since 1992 he has been working in the Test Center of Maschinenfabrik Gustav Eirich in Hardheim. As Ralf Löbe, he also has great practical experience in the field of granulation.

P. Nold, R. Löbe, M. Müller

Abstract

There are various ways of producing granules from fine-grained substances or dusts. The authors compare the relatively simple and cost-effective processes of granulation in the oblique intensive mixer, disk pelletizers and drum pelletizers. Here, theory only assumes marginal importance. On the contrary, a long series of examples shows in which applications mixing granulators respectively disk and drum granulators are used worldwide today and yield a prof-

it to the users over decades. A further process described enables straightforward water-free agglomeration using organic solvents, as well as the production of high-density granules from a suspension (Evactherm process). Similarly to mixing, which cannot be calculated but needs to be observed and investigated in experiments, the process of granulation can only be approached via practical studies.

Refractories Manual 2003

Kurzfassung

Es gibt verschiedene Möglichkeiten, aus feinkörnigen Stoffen oder Stäuben Granulate herzustellen. Die Autoren stellen die vergleichsweise sehr einfachen und kostengünstigen Verfahren des Granulierens im schräg stehenden Intensivmischer sowie auf Pelletiertellern oder in Pelletiertrommeln dar. Dabei wird nur wenig auf die Theorie Wert gelegt. Vielmehr wird an einer langen Reihe von Beispielen gezeigt, in welchen Anwendungen Granuliermischer bzw. Granulierteller und -trommeln heute weltweit im Ein-

satz sind und den Anwendern über Jahrzehnte Nutzen bringen. Vorgestellt wird auch ein Verfahren, das es zum einen erlaubt, mit einfachen Mitteln mit organischen Lösemitteln wasserfrei zu agglomerieren, und das es zum anderen ermöglicht, aus einer Suspension kommend sehr dichte Granulate zu erzeugen (Evactherm-Verfahren). So wie man „Mischen“ nicht rechnen, sondern nur experimentell erfahren kann, ist auch „Granulieren“ stets ein Vorgang, der in Versuchen angegangen werden muss.

Keywords: preparation, granulation, pelletization, intensive mixers, disk pelletizers, drum pelletizers

1 Introduction

In many industries granules represent an important preliminary stage in the manufacturing of a product. In some cases the granule is a final product intended for direct sale. Reasons for transforming powders or dusts into granules can be, for instance:

- Improvement of flow properties
Example: Agglomeration of filter dusts
- Increase of bulk density
Example: Agglomeration of microsilica
- Avoidance of demixing effects
Example: ceramic press bodies

Sometimes it is also necessary to agglomerate mixing components in order to avoid demixing effects while mixing or during transport. Kaye, for instance, describes that particles differing in size by a ratio of more than 1:3 begin to exhibit demixing effects during the mixing process [1]. For this reason, fine-grained additives are transformed into granules before they are mixed in small amounts into coarser substances. Granules can be produced [2] from:

- powders:
 - dry powders by build-up agglomeration
 - moist or dry powders by press agglomeration
- liquefied material, as e.g.
 - by press granulation
 - by prilling
- suspensions
 - by spray drying
 - by fluidized-bed build-up agglomeration.

This paper deals mainly with the production of granules by means of build-up agglomeration from dry powders occurring in different technical processes or dry grind-

ing processes. Moreover, a novel, still largely unpublished method is described, in which very dense granules are produced by the drying of suspensions.

Build-up agglomeration in the mixing granulator offers particular advantages in comparison with thermal granulation processes (spray drying etc.). These are in detail:

- drying expenses are eliminated to a large extent
- no demixing due to different densities of raw materials, as may occur when processing slurry
- the first batch after commissioning already supplies correctly sized grains
- the process can be started and stopped as required
- simple change of batch or formula
- solid grains are produced
- the grain size range can easily be influenced
- machine sizes ranging from 3 l up to 5 m³ are available.

Thermal granulation was invented as a "spray drying process" at a time when energy (fuel oil) was still cheap and the mixing technology was in its fledgling stages. At that time, the aim of the process was not the production of compressible granules but drying. Advantages of the spray drier were described as follows [3]:

- lower labour costs as staff for mechanical water removal in filter presses is not required
- the usual grinding systems for filter cakes were no longer necessary.

In the 1970s, even plastic mass for the production of fine stoneware and art ceramics was produced by a method in which raw materials were transformed into slurry, then partly spray-dried, the sprayed grain then processed in the mixer with the remaining slurry to a plastic mass [4, 5]. Plastic porcelain masses as well,

Table 1	
Industrial sectors using mixing and agglomeration	
Ordinary ceramics (grain sizes >0.1–0.2 mm)	
Bricks, conventional refractory materials	
Fine ceramics (structural constituents <0.1 mm)	
Advanced Ceramics	High-tech ceramics
	Structural ceramics
	Functional ceramics
	Electroceramics
	Cutting ceramics
	Bioceramics
Chinaware	
Decorative ceramics	
Sanitary ceramics	
Wall and floor tiles	
Ceramic-based abrasives	
Note: In Anglo-Saxon language usage, the term "ceramics" additionally includes glass, enamel, glass ceramics and inorganic binders (cement, lime, gypsum)	

which were widely used at that time and proceeded from spray-dried finished granule, were prepared in the intensive mixer [6, 7].

As mixing technology advanced, the advantages of dry preparation – without the pre-stage spray grain – were soon recognised [8].

Following the advance of press technology and the conversion from plastic and semi-plastic shaping processes to dry pressing, as early as 1980 build-up granulation was described as an economical alternative to thermal granulation [9]. It was described how "through the enormously increased costs for heating energy during the last few years" [9] the importance of build-up granulation progressively increased. Plants built at this time, e.g. for tile press bodies, continue to operate up to this day.

Thermal granulation offers the advantage that the spray towers can be built in nearly any size. In the case of products manufactured in large quantities without changes in their composition, thermal granulation remains valid today. Build-up granulation or build-up agglomeration is always of interest where limited quantities of granules are to be prepared – e.g. in a tile factory or in the field of oxide or non-oxide ceramics.

2 The History of Build-Up Agglomeration

According to Pietsch [10] agglomeration reflects an interdisciplinary field of science. In earth's prehistory, many rocks arose from agglomeration processes. Among life-forms in nature, termites, for instance, use such processes in building their nests, as does the scarab (dung beetle) in building its dung ball. Using the example of baking bread, Pietsch describes the entire process to characterise agglomeration methods:

- grinding = preparation of grain size range; activation of binder (starch)
- mixing with addition of binders (water)
- forming into a green agglomerate
- aftertreatment = baking.

The authors wish to confine their explanations to mixing and agglomerating in applications in the ceramic industry. This comprises the industry sectors listed in Table 1 [11].

According to another classification (Table 2) the fields of silicate ceramics, structural materials and functional ceramics [12] are distinguished.

Today, shaping processes are carried out in many of these industries with granules produced thermally or by build-up agglomeration.

3 Foundations of Agglomeration

Information on the foundations of agglomeration is available from many sources. In addition to the recently published book "Agglomeration Processes" by W. Pietsch [10] and various informative events offered by W. Pietsch, seminars of the "Fraunhofer-Institut für Keramische Technologien und Sinterwerkstoffe (IKTS)" in Dresden are a source of information. Such seminars deal with the characterisation of granules, in addition to their production. Furthermore, the Technische Akademie Wuppertal (Technical Academy Wuppertal) regularly offers events dealing with "Agglomeration of powders and dusts".

By means of various binders and additives, today the characteristics of a granule can be adjusted within wide limits. For trouble-free handling of the moist granule a sufficient green strength is essential. This green strength can be achieved through different binders (Table 3).

In addition, substances/groups of substances such as polyethyleneglycol (PEG), carboxymethylcellulose (CMC), sodium water glass, alginates or polyvinyl pyrrolidone (PVP) are used.

With corresponding pressing aids, a homogeneous matrix can be developed, guaranteeing – if desired – that the structure of the granules does not become apparent in the final press body. Friction processes between the grains and/or between grain and wall are reduced effectively.

Table 2		
Industries according to "Re-structuring of the DKG (German Ceramic Society)"		
Silicate ceramics	Structural materials	Functional ceramics
Architectural ceramics / Bricks	Bioceramics	Piezoceramics
Refractory	Cutting ceramics	Ferrites
Tiles	Abrasives	Condensers
Sanitary ceramics	Filter ceramics, membranes	Sensors
Tableware	Oxide ceramics	Superconductors
Chemico-technical ceramics	Polymer ceramics	Optoelectronics
Insulator ceramics	Carbon	Electroceramics
	Composite materials	Ceramic resistors
	Ballistic ceramics	Non-silicious insulation ceramics
	Nuclear materials	
	Nanomaterials	
	Non-oxidic structural ceramics	

Fig. 1:
Mixing principle
of the intensive
mixer for mixing,
granulation,
coating, and
kneading purposes

Table 3 Binders [13]	
Group of substances	Product example
Polymer dispersions	product KB 2132
Polyvinyl alcohols	product KB 2061
Polysaccharides	OPTAPIX PS 13 OPTAPIX PS 21 OPTAPIX PS 94
Acrylates	OPTAPIX AC 95 product KB 2112
Wax emulsions	ZUSOPLAST WE 8 ZUSOPLAST WE 36 ZUSOPLAST WE M
Lignosulphonates	OPTAPIX L 23

In the case of raw materials with a high fines content it is often possible to granulate without adding binders.

The influence of the particle sizes and particle size distributions on the granulating process:

The particles should be as small as possible, 100 µm are usually the natural limit for a build-up agglomeration.

Coarser particles (such as unground returns, nucleation agglomerates etc.) are included only if a sufficient amount of fines is available. The bigger particles are thus coated by the smaller particles.

Close particle size distributions, the so-called monodisperse distributions, are often difficult to agglomerate.

Basically, four different mechanisms of grain enlargement [14] are distinguished:

- interfacial forces and capillary pressure at freely moveable liquid surfaces
- attractive forces between solid particles
- adhesive and cohesive forces in binders not freely moveable
- solid-state bridges.

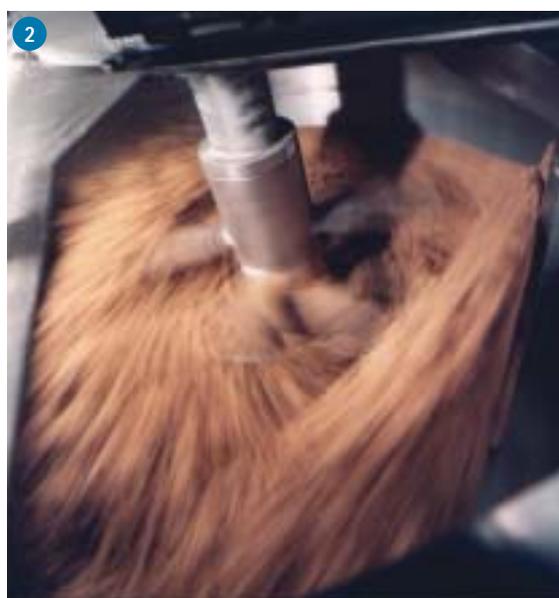
To activate the various forces, the individual solid particles must be moistened and brought close to each other. The available moisture content is of particular importance for the binding mechanism. The maximum "granulating moisture", which is always to be determined in tests, fills about 95 % of the agglomerate's pore volume. An – even slightly – higher percentage will cause the granules to be transformed into a plastic state, i.e. into sludge.

Solid-state bridges generally first appear at aftertreatment of the green pellets by chemical reaction, hardening of binders or sintering.

4 Machines that can be Used for Granulation Purposes

Two basic machine types can be used for build-up agglomeration today:

- the mixer
- the disk pelletizer or drum pelletizer



4.1 Granulation in the Mixer

For approximately 30 years, mixing granulators, which were developed from disk pelletizers, have been available on the market (Figs. 1 and 2).

The material is entrained in the inclined rotating mixing pan by way of wall friction. At the highest point it is thrown off by the wall scraper towards the fast-turning rotor. Through intensive "mixing", the particles are brought close together – under addition of the necessary quantity of water. As soon as the granulation nucleus has developed, the speed of the rotor is considerably reduced. Supported by the slowly rotating mixing tool, the mixed material rolls over the mixing pan bottom and wall. Here, the actual build-up agglomeration starts.

If the speed of the mixing tool is not reduced, a state of equilibrium will develop between particle coalescence (agglomeration) and destruction of already developed agglomerates (disagglomeration). This stage is used if a mixing granulator is installed ahead of a disk or drum pelletizer.

It is possible to influence the grain size distribution through the form of the mixing tool. Depending on material and grain size, granules between 0.1 mm and 8 mm can be obtained. Granules suitable for pressing e.g. 0.2–2 mm are often achieved with a yield of over 80 %. Apart from batch operation, continuous operation is also possible.

The advantage of granulating in the mixer is that no monitoring whatsoever is necessary during the process. In addition, it is possible to both pre-mix and granulate in the same unit.

If recycled material is used, it is often advantageous that the mixer can transform moist material such as sludge into granules when dry substances are added.

4.2 Granulation in the Disk or Drum Pelletizer

Granulation in the disk or drum pelletizer is a well-known and proven process. The pelletizing of ore mixes was described as early as 1912 [14]. While metallurgical processes continued to develop, the pelletizing processes remained almost unchanged. The pelletizing of ores is described under item 5.2.1 with an example on iron ore.

The formation of green pellets is influenced by:

- the kind of raw material used and its fineness
- the moisture content of the pellets
- the kind of binder used and its characteristics
- the wettability of the particles
- the movement pattern in the pelletizer.

With predetermined raw materials and machines, the formation of pellets is mainly dependent on the amount of water added. Too little water hampers the pellet formation just as too much water leads to disadvantages.

It is necessary to install a mixer ahead of the disk or drum pelletizer if the material is not only granulated but also mixed beforehand. In individual cases, for instance to improve special product characteristics, a combination of mixing granulator and disk pelletizer (or drum pelletizer) is advantageous, as described by Müller [15].

4.2.1 Granulation in the Disk Pelletizer

The material is introduced into a rotating disk with a 45–55 degree inclination and moistened (Fig. 3). Through wall friction, the material is carried upwards and rolls backwards reaching a certain point. The pellets are formed through a continuous rolling movement. As the pellets become bigger, they are moved from the pan bottom towards the surface until they are finally discharged as green pellets via the pan rim. The resulting grain size range is very narrow. Most of the time, screening is not necessary.

The disks have a diameter of up to 7.5 m with an output of e.g. 100 t/h of ore pellets 9–15 mm in diameter. By varying the disk pelletizer's angle of inclination, the rotational speed, the amount of water and material added, it is possible to adapt to changing materials or fluctuations of raw material characteristics. The pelletizing process should be monitored at all times in order to ensure that an optimum product is obtained.

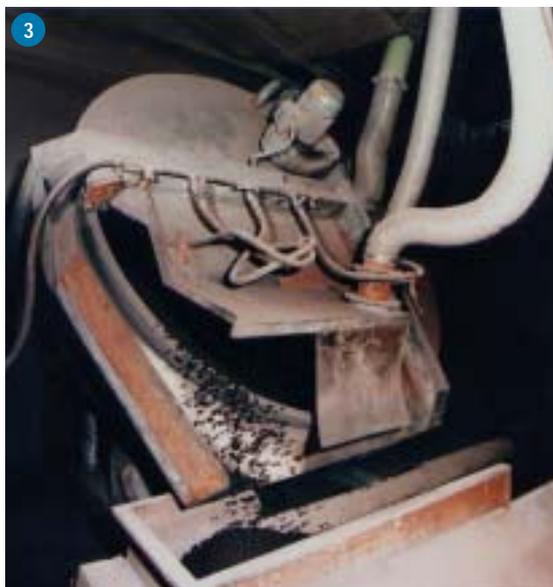


Fig. 3:
Disk pelletizer

4.2.2 Granulation in the Drum Pelletizer

If the demands on the grain size range are not very high, the use of a drum pelletizer is advantageous, as this type of pelletizer is less sensitive towards raw material and batch fluctuations.

The material is introduced into a 10 degree inclined rotary cylinder (8–14 rpm) and moistened. Through wall friction, the material is carried upwards and rolls backwards reaching a certain point. The pellets are formed through a continuous rolling movement and are discharged at the end of the drum. Mostly, a wide grain size range develops. Undersize grains are recirculated into the drum after screening.

Drums can be produced in any dimensions, e.g. with 12 m length and 3.6 m in diameter. There is a multitude of different types, with and without built-in components. The throughput rate achieves 1 t/m³ of drum volume.

4.3 Granulation in the Vacuum Mixer

The installation of an intensive mixer into a vacuum-tight housing allows mixing, granulating, and drying the material under pressure below ambient pressure. Figure 4 shows a small system ready for operation.

If suspensions are used (e.g. alumina in water) and dried, the fast-rotating rotor (peripheral speed up to 40 m/s) will break up the developing solid particles and will transform them into granules.

As the energy input into the material is highest during the plastic phase, mixing processes can be extremely efficient in this phase through intensive kneading. This is an interesting point, especially where ultra-fine material is concerned, which often cannot be mixed sufficiently in dry condition. The resulting granules have a grain size of 100 µm to several millimetres, depending on the speed of the rotor. A fundamental study has shown that this process is suitable for producing tile press bodies [16].

In some cases it was revealed that products manufactured "under vacuum" possessed new characteristics. Highly dense granules produced green bodies with

over 80 % of their theoretic density, while green bodies produced out of conventional granules showed a theoretic density of less than 54 %, even when higher pressure was applied. Correspondingly, the linear firing shrinkage will decline from 12–15 % to 3–6 %. Serkowski reported on this phenomenon during UNITECR 2001 [17].

Vacuum mixers can be operated under a protective atmosphere. This enables the operator to work with any organic solvent under potentially explosive conditions. "Waterfree" granulation is possible using corresponding binders that are dissolved in organic solvents.

5 Examples

Around 1960, processing methods were developed to agglomerate residues, fines and dusts to be used again in some other way. Thus converter dust, for example, was granulated and recirculated into the metallurgical process, basic slag was granulated with potassium salt into mixed fertilizer, calcium sulphate from the production of hydrofluoric acid was granulated and used as an additive to cement, fly ash from power plants was made transportable by way of granulation and was then used in other processes [18].

Stricter conditions on keeping the air and water clean have led to a widespread use of dry and wet dust extraction plants, as of around 1975. The accumulated dusts and sludges were treated in mixing and/or disk granulators and then recycled; in exceptional cases they were taken to dumping grounds [19]. The treated refuse included power plant boiler sludges, acid sludge, sanding dusts, and wheel swarf from the production of friction lining, filter dusts from cement plants, ashes from coal-fired power stations, and gypsum from flue gas desulfurization plants [19].

Around 1980, disk pelletizers were used [20–22] for pelletizing a whole host of products, including:

- cement filter dust
- flyash from coal-fired power stations
- lead oxide
- clay (production of swelling clay)
- fertilizers from molten-salt reactors
- mixed fertilizers
- clay-coal-mixes (porosity agent for bricks)
- zinc- and lead ores
- metal oxides
- sludges and filter cakes.

With the introduction of flue gas desulfurization plants, the accumulating gypsum was transformed through pelletization into an intermediate product for further processing [23].

Disk pelletizers have been used in the agricultural sector from a very early stage on, too. H.B. Ries describes the polishing and pilling process of sugar beet seeds [24]. Through build-up agglomeration, the seeds are transformed into high-quality seeds with

- seed coating for protection against parasites
- seed coating with nutrients and biocatalysts
- protection of seeds against climatic effects
- fixing of seed mixtures
- seed enlargement.

The intensive mixer has also already been in use for granulation purposes for a long time. Even before 1970, there were reports on the granulation of the following materials [3]:

- metal carbide powder
- activated alumina
- carbonyl iron powder
- glass powder (for feed-through sleeves in the transistor technology).

Today, the applications for mixing granulators and disk or drum granulators are spread over a wide area. However, the authors give only a few examples. The figures

Fig. 4:
Vacuum mixing
and granulating
system



5a



Alumina
Grinding balls



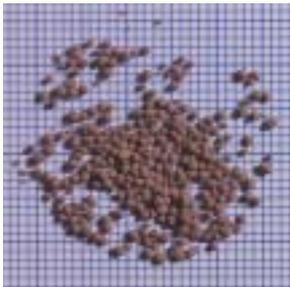
Steatite
Grinding balls



Zeolite
Molecular sieves



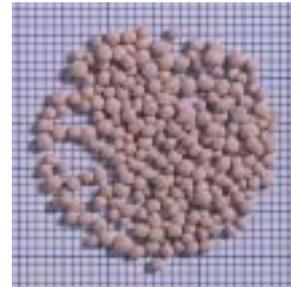
Zeolite
Molecular sieves



Bauxite
Proppants



Kaolin
Proppants



Bauxite
Moulding compound



Perlite
Lightweight aggregate



Corundum
Catalyst carrier



Clay powder
Animal food additive



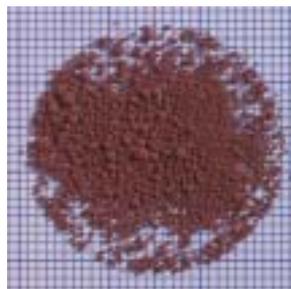
Tile press compound



Lead glass batch



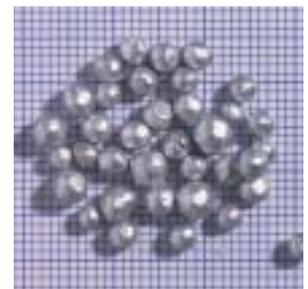
Glass powder, Carbon
Foam glass



Ferrite



Ferrite



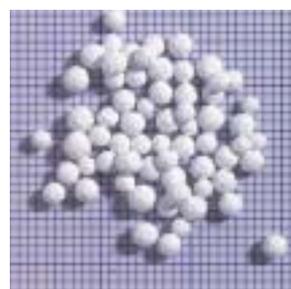
Aluminum



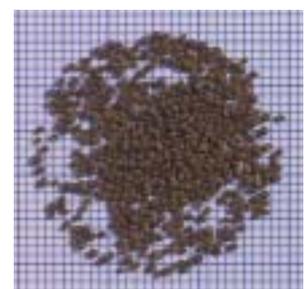
Welding powder



Zinc oxide
Varistors



Zinc oxide
Pigment

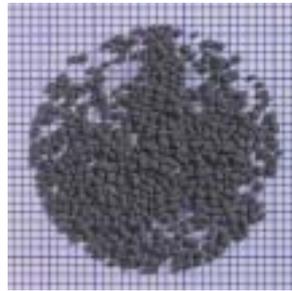


Iron oxide
Pigment

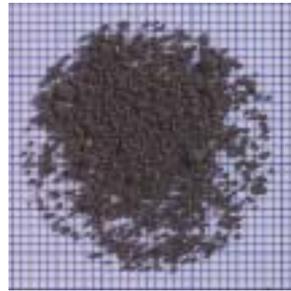
5b



Silicon carbide
Abrasives



Tungsten carbide
Hard metal



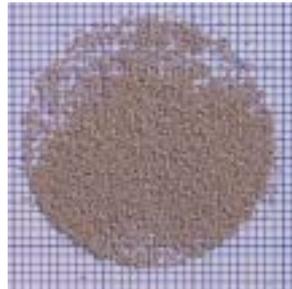
Peat fertilizer



Sugar-beet seed



Mineral powders
Animal food additive



Phosphate fertilizer



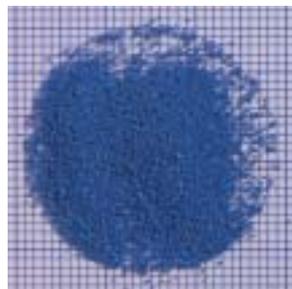
Dolomite
Fertilizer



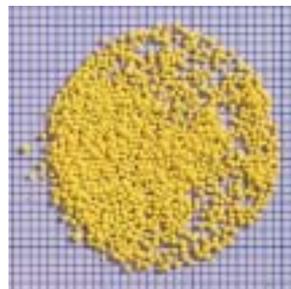
Lime
Fertilizer



Steatite, coloured
Press compound



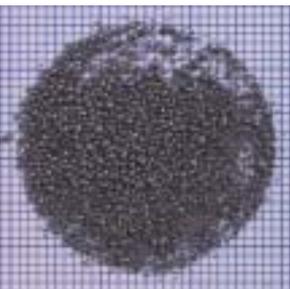
Pigment mix, blue



Pigment mix, yellow



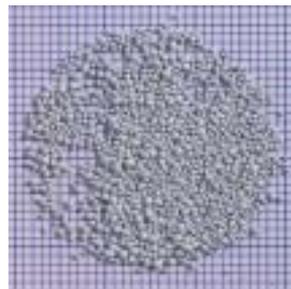
Oxide powders
Varistors



Al₂O₃-C
Iso-press compound



Oxide powders
Catalysts



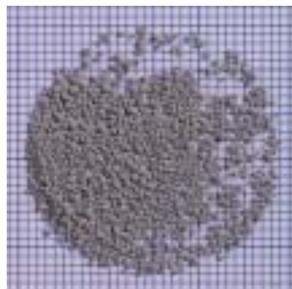
Steatite
Press compound



Porcelain
Contact compound



Cyclon dust



Cement
(granulated without water)



Kieselgur
Filter media



Gypsum
Building materials

show granules from daily practice (Figure 5 a + 5 b). Table 4 lists some further examples.

5.1 Examples from the Ceramic Industry

5.1.1 Refractory Industry

5.1.1.1 Synthetic Raw Materials

Several different raw materials were mixed and pelletized to be batched in fusion processes or passed through burning processes in shaft or tunnel kilns.

5.1.1.2 Press Bodies

Especially bodies prepared for isostatic pressing processes are granulated to make sure that the complex filling processes are performed without demixing. For example, mixes containing up to 10 components and with a graphite proportion of up to 30 % were turned into 0.2–2 mm granules.

5.1.2 Tiles

Clay and feldspar are batched into the mixer for preparation of dry press bodies and dry homogenization. Water is sprayed in and the body is moistened until the granulating moisture is reached. Then the body is mixed once more and granulation starts with particle-particle contact and adhesion due to liquid bridges. Plants of this type can be found in Germany and Mexico.

5.1.3 Molecular Sieves

Here, the use of two mixers - one on top of the other - proved to be advantageous. The zeolite raw material is batched in the first mixer together with clay-type binders and dry homogenized. Granulation starts while water is added. The fine granules are discharged into the second mixer which forms the final granules. Instead of the second mixer, it is possible to use disk pelletizers.

5.1.4 Varistor Bodies

Zinc oxide and several inorganic "additives" are batched into the mixer. After the wet mixing process, i.e. intensive kneading during the paste phase, the body is dried (Evactherm process). The density is increased from 500g/l (spray grain) to 2000g/l; the homogeneity is considerably improved.

5.1.5 Dental Ceramics

The raw materials are batched into the mixer. In order to rule out impurities through metallic abrasion, mixers with rubber lining are used. Water is then sprayed in. After nucleation, the speed is reduced and granulation starts.

5.1.6 Cutting Ceramics

The raw materials are batched into the mixer and dry homogenized. Water is then sprayed in. After nucleation, the speed is reduced and granulation starts. The resulting densities are very high.

5.1.7 Abrasives

The fine-grained raw materials are batched into the mixer and homogenized at high peripheral speed. The water-containing binder is sprayed in. After nucleation, the speed is reduced and granulation starts.

Table 4
Further examples (excerpt)

Barium titanate	Frits	Molybdenum dust
Expanded clay	Foundry dust	Incineration ash from oil
Lignite flyash	Potassium sulphate	Organic fertilizers
Cadmium selenide	Limestone powder	Carbon black
Calcium aluminate	Cat litter	Sanding dust
Chromium oxide	Caustic magnesia	Steel work dusts
Chromium steel dust	Chalk powder	Bituminous flyash
Dental ceramics	Copper powder	Detergents
Precious metal dust	Micro silica	Cement powder

5.1.8 Oxide Ceramics

The raw materials zircon oxide and stabilising additives are batched into the mixer and homogenised at high peripheral speed. Water, which can in some cases contain binders, is then sprayed in. After nucleation, the speed is reduced and the granules start forming.

5.1.9 Grinding Balls

The alumina or zirconium oxide raw materials are batched into the mixer. Binder containing water is then sprayed in and granulation is started with high speed. After nucleation, the speed is reduced and the granules start forming. As an alternative, a disk pelletizer can be used. The granules are rounded in drums in order to achieve well formed spherical grinding balls.

5.1.10 Ferrites

The dry material is fed into the mixer. Water is added at high rotor speed. After some time, granulation nuclei are produced and the granules start forming relatively slowly.

Preparing the mix in a vacuum mixer can be advantageous in the case of ferrites. The paste-like mix is produced from dry material and water. The starting materials are dispersed under the effect of high shearing forces. The energy required for drying can be applied by introducing vacuum superheated steam into the paste-like mix. The actual granulation starts after the transition phase. Granules produced this way have the following advantages over "conventional" granules:

- higher density, higher bulk density, higher calcining furnace throughput
- higher strength, less abrasion in the furnace
- homogeneous moisture distribution through the cross section
- whole grain, without shell structure
- direct calcination possible without pre-drying
- less diffusion spaces between the reactive parts, better ferrite quality.

5.2 Examples from Other Sectors

5.2.1 Pelletizing Ores

In metallurgical processes, pellets have to fulfil the following demands [14]:

- narrow grain size distribution
- no dust content

- no cakings
- predetermined bulk properties in non-fluidized beds
- predetermined flow-through conditions in fluidized and non-fluidized beds
- predetermined flow properties in fluidized beds
- predetermined floating properties in gas and liquid flows.

In order to achieve these properties, fine ground ores or ore concentrates are mixed with binders (e.g. clay) in mixers and are afterwards formed into pellets in disks or drums. Apart from the iron-ore sintering process, this process has developed into the most important agglomerating process in the metallurgical industry since 1955. The reason for this was the increasing need to smelt concentrates of poor iron ores, which could not be introduced into sintering plants due to their high proportion of ultra-fine grains [14].

It was possible to increase the output considerably by using pellets (instead of the usual classified iron sinter) in blast furnaces. According to Japanese reports, the furnace productivity more than doubled, and the demand for coke was reduced by more than 20 %.

If other raw materials, e.g. dead lime, limestone, or dolomite, are added, the pellets can take on specific metallurgical characteristics. The size of the pellets can be adjusted during the process and can reach up to 30 mm.

As early as 1980, detailed studies on iron ore pellets in blast furnace batches as well as in direct reduction processes were published [25]. The significance of pelletizing iron ore can also be seen in the fact that the pelletizing process was described at a very early stage in mathematical models [26].

In addition, iron ore pellets were described for direct reduction processes in Sweden (LKAB company) in 1980 [27]. These ores consist mainly of magnetite, which is oxidised during the burning of the green pellets. The heat released during this burning process is sufficient for calcining small dolomite or limestone additives.

The use of Eirich intensive mixers not only improved homogeneity but also resulted in a reduction in binder and bentonite consumption. For these applications, the intensive mixers operate continuously with throughputs of up to 1000 t/h. The mean processing time in the mixer ranges from 40 to 60 s. As a rule, the mixers operate continuously for 3 months; maintenance measures are carried out every 9 months.

In Japan, too, iron ore was agglomerated in Eirich mixers as early as 1991 [28]. Extensive comparative studies have shown that it is advantageous also in the sinter preparation process to mix the raw materials first in the mixer and then use the drum granulator [29]. Compared with the traditional process, the result is a considerable increase in production and a decrease in demand for coke. The savings for one steel mill can be around 100.000 /a.

5.2.2 Proppants

In order to increase the productivity of oil- and gas wells, liquids are pumped into the well hole with such a high pressure that the surrounding rock can break and crack. The liquids are mixed with proppants, which shore up

these cracks even if the pressure is relieved [30]. Proppants on the basis of natural sands are also used. However, ceramic proppants have their advantages, including higher strength, more regular sizes and better roundness. Strength, even grain size, and well formed spheres are the decisive factors that determine the extent of gas and oil permeability of the bulk [30].

Depending on the strength required, ceramic proppants are produced from various raw materials such as kaolin or bauxite. Proppants on the basis of bauxite are characterised by very high strength. Furthermore, they do not break when under pressure but are only deformed. Even if the pressure reaches 100 N/mm², less than 5 % of bauxite-proppants are destroyed [31].

Bauxite ore is ground and the powder is prepared into granules in intensive mixers. The granules are dried, classified by sieves and burned at high temperatures [31]. Typical grain sizes include 20/40 (mesh), 18/30, 16/20 and 12/20 [31]. The strength can be increased by coating the granules with phenolic resin [32].

5.2.3 Filtering Materials

For filtering and neutralising water, pre-treated products of naturally existing minerals can be used. Lime is burnt, slacked and formed in disk pelletizers into pellets of 1–3 mm diameter either solely or with the addition of further substances (such as activated carbon). In order to maintain this grain size range during continuous operation, three disk pelletizers are installed next to each other, adjusted to 1 mm, 2 mm, and 3 mm. The green pellets are dried. Through recarbonisation (by exhausts from the lime burner) specific characteristics can be achieved.

6. Grain Size and Grain Size Range

6.1 Grain Size

Through test conditions, the grain size can be influenced in broad outline and adjusted during the process. It often occurs that the individual forming processes can be successfully applied to coarser granules and that the material can be thus improved.

An example on zinc oxide ceramics illustrates this point. For the first tests, the customer requested a final grain size of <500 µm; yield (not optimised) 37 %. Three months later, during a second set of tests, the aim was a grain size of <800 µm; yield up to 80 % of correctly sized grain. Again three months later during a third set of tests (in which plans were made for a later production plant) the customer said that the maximum grain size was no longer important. The production plant was designed for a grain size of <2 mm; yield up to 90 %.

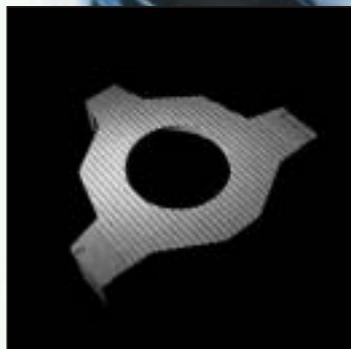
Between these test series, the customer had worked further with the material and run tests on the produced pieces during which he noticed that the size of the granules (in the given ranges) had no influence on the characteristics.

6.2 Grain Size Range

The grain size range can be influenced and also adjusted via the individual test conditions. If only a narrow grain size range or mono-grain is required, this can be achieved through a corresponding screening (and recirculation of incorrectly sized grains into the granulation process).

VISIONARY?

VACUUM MAKES IT POSSIBLE



New vacuum pressing technology for:

- Higher final density of the product
- Reduced cycling time
- Small volume of evacuation
- Reduced investment and maintenance cost

Considerably increase your market chances by being capable of fulfilling new, complex demands and reaching the highest quality requirements in the production of refractories. **LAEIS offers you the necessary support:** With our newly developed vacuum pressing technology, you can now press fine powder, granules and mixes in dry or semi-dry condition to achieve products of the highest quality.

Trier, Germany Tel. +49-651 9492-0 Fax +49-651 9492-200

LAEIS Hydraulic presses for:
Refractory products
Ceramic products
Cement bonded products

www.laeis-bucher.com

LAEIS

PART OF YOUR SUCCESS

Lately, there were reports on a process that optimises granules in terms of their grain size distribution and produces so-called "uniform granules" [33]. Quote: "Agglomerates that are too big are reduced in size while small-grained or dust particles are combined to form bigger agglomerates. Thus, oversized grains are eliminated and the amount of undersized grains is reduced, and screening is therefore no longer necessary. Throughputs of 20–4000 kg/h granules in a grain size range of 100–4000 µm can be achieved [33]."

Up to now, these units are being used in the food and pharmaceutical industry [34, 35]. If we are successful in adapting this process to applications in the ceramic industry, we can then expect considerable process simplifications (elimination of screening plants and recirculation).

7 Conclusion

As explained in the introduction, granulation in the mixer, disk or drum is a process that must be verified in experiments. This is especially true for oxide and non-oxide ceramics.

Only a few years ago, the problem was that the mixer formed "nice" granules but they were so hard that the granule structure in the pressed body was still visible. This condition has meanwhile been improved as explained in chapter 2.

Due to competition in many sectors, it goes without saying that we cannot report on the mentioned plants in detail. As for Eirich, no information is passed on to third parties. On the other hand, studies in the Eirich test centre prove that powerful mixing granulators are often surprisingly small in dimensions. An example explains this:

- For granulating 1000 t/a of oxide ceramic mixes (e.g. zircon oxide) on 200 working days with 2 shifts of 7 h each, one mixer type R 08 (75 l, approx. 80 cm pan diameter) is required.
- In order to produce twice as much, one mixer type R 09 (150 l, approx. 90 cm pan diameter) is needed.
- If the objective is to granulate 1000 t/a of oxide ceramic mixes (e.g. zircon oxide), on 200 working days with 2 shifts of 7 h each, and if the demands on the grain size range are such that only 50 % of the granules are accepted as correctly sized grain and the rest is screened and recirculated, then a mixer type R 09 (150 l, approx. 90 cm pan diameter) only is required.

Correspondingly, 500 t/a could be produced with a mixer type R 05 (40 l, approx. 50 cm pan diameter). The first batch already produces correctly sized grain. The process can be interrupted if necessary at any time.

Whether a material can be granulated or not is usually tested on laboratory mixers type R 02 (3–5 l) and RV 02 (8–10 l).

For optimum cost-effectiveness, granulation in the mixer will be chosen. In 2002, a tile production plant was started in Mexico, which processes up to 15 t/h of press granulate, produced in Eirich mixers.

The need to produce both efficiently and economically is of crucial importance today. One can only stay ahead of the competitors by utilising state-of-the-art technology.

The authors strongly suggest that those who still produce press granules with expensive conventional methods run tests with simpler granulation methods. This will give them an insight today into the challenges they will face tomorrow.

References

- [1] Kaye, B.H.: PBE International, May/June 2003, 8–12
- [2] Walzel, P.: Material for the seminar "Herstellen von Granulaten". Haus der Technik, Essen, April 2003
- [3] Ries, H.B.: Moderne Aufbereitungsverfahren für die Massen der Oxid- und Sinterkeramik. Reprint from *Angew. Mess- + Regeltechnik*, Maschinenfabrik Gustav Eirich, 1969
- [4] Lengersdorff, G., and Röhr, H.: Modern preparation method in the pottery industry. Reprint from *INTERCERAM* (1969) [3], Maschinenfabrik Gustav Eirich
- [5] Lengersdorff, G., and Röhr, H.: Moderne Aufbereitungsverfahren in der feinkeramischen Industrie. Reprint from *Keram. Z.* (1969) [7], Maschinenfabrik Gustav Eirich
- [6] Blankenburg, H.: Vergleichende Gegenüberstellung der Porzellanmasseaufbereitung nach der herkömmlichen, konventionellen Art und der neuen Aufbereitungstechnik in Bezug von sprühgetrocknetem Fertigmassgranulat. Reprint from *Keram. Z.* (1972) [1], Maschinenfabrik Gustav Eirich
- [7] Blankenburg, H.: Comparison between pottery body preparation by conventional methods and new preparation techniques employing spray-dried granulate, Reprint from *INTERCERAM* (1972) [2], Maschinenfabrik Gustav Eirich
- [8] Ries, H.B., and Balzar, U.: Trockenaufbereitung als moderne Alternative zur Nassaufbereitung keramischer Massen. Reprint from *Aufbereitungstechnik* (1978) [1], Maschinenfabrik Gustav Eirich
- [9] Ries, H.B.: Aufbereitung von Boden- und Wandfliesenmassen. Reprint from *Keram. Z.* (1980) [6], Maschinenfabrik Gustav Eirich
- [10] Pietsch, W.: *Agglomeration Processes*. Wiley-VCH Verlag GmbH: Weinheim 2002, 5–7
- [11] Website of Informationszentrum Technische Keramik, www.keramverband.de
- [12] Neustrukturierung der DKG, cfi/Ber. DKG **80** (2003) [5] D 32
- [13] Press release from P. Quirnbach, Zschimmer & Schwarz GmbH & Co, Lahnstein
- [14] Biermann, F.: Pelletieren. *Ullmanns Encyclopädie der Technischen Chemie*, Verlag Chemie, Vol. 2: Weinheim 1984, 321–334
- [15] Müller, M.: Aufbau-Agglomeration mit Pelletierteller und im Granuliermischer. *Aufbereitungstechn.* **44** (2003) [2] 22–31
- [16] Opp, A., Sax, M., Müller, M., Dürr, H. and Telle, R.: Aufbereitung von keramischen Pressmassen mit dem EVACTHERM-Verfahren. *Keram. Z.* **50** (1998) [7] 546–550
- [17] Serkowski, Müller, M., Rymon-Lipinski, T.: Proc. of Unified Internat. Techn. Conf. on Refr. Cancun, 2001, pp. 1432–1443
- [18] Ries, H.B.: Probleme bei der Aufbereitung industrieller und kommunaler Abfallprodukte. Reprint from *Industrie-Anzeiger*, Oct. 1962, Maschinenfabrik Gustav Eirich
- [19] Ries, H.B.: Granulierteller und Mischgranulatoren für die Verfestigung industrieller Stäube und Schlämme. *Eirich-Fachber.* Ausgabe 12/1277, Maschinenfabrik Gustav Eirich
- [20] Ries, H.B.: Zur Praxis der Pelletierung, *Aufbereitungstechn.* (1981) [4] 209–222
- [21] Ries, H.B.: Aufbaupelletierung – Verfahren und Anlagen. Reprint from *Aufbereitungstechn.* (1979) [12]

- [22] Ries, H.B.: Misch- und Pelletierverfahren für Erze zur Verhütung und Sekundärrohstoffe für Recycling. Reprint of a paper read at the German-Soviet metallurgy conference "Sparsame und rationelle Verwendung materieller Ressourcen im Eisenhüttenwesen und in der Buntmetallurgie", 7-13.9.1985, Maschinenfabrik Gustav Eirich, Hardheim
- [23] Ries, H.B.: Paper read at the Conference of the GVC Technical Committee from 15 to 17 March 1982, Braunschweig, Eirich-Fachber. 27/684, Maschinenfabrik Gustav Eirich
- [24] Ries, H.B.: Eirich-Fachber. Ausgabe 5/577, Maschinenfabrik Gustav Eirich
- [25] Meyer, K.: Pelletizing of Iron Ores. Springer Verlag, Berlin / Verlag Stahleisen: Düsseldorf 1980
- [26] Megwa, P.E. and Simonis, W.: Mathematische Beschreibung der Aufbauagglomeration von Eisenerzpellets. Aufbereitungstechn. (1985) {4} 197-208
- [27] Mathisson, G.: Eisenerzpellets für die Direktreduktion. Stahl u. Eisen **100** (1980) [7] 93-99
- [28] Application of powder pelletizing technology to ironmaking and steelmaking processes. Nippon Steel Techn. Rep. **48** (1991) 18-24
- [29] Improvement of granulation of raw material by using the high-agitating mixer at Kokura No. 3 sintering plant. 1995 Ironmaking Conf. Proc., 535-540
- [30] Website of CARBO Ceramics, www.carboceramics.com
- [31] Website of Clearwater Engineered Chemistry, www.cwi-chem.com
- [32] Website of Borden Chemical, www.bordenchem-oilfield.com
- [33] Uniforme Granulate, Keramiker-Handbuch 2003, Göller-Verlag Baden-Baden, p. 62
- [34] Partikelgrößenverteilungen standardisieren. CAV **5** (2003) 183
- [35] Sieblos Agglomerate optimieren. Process March 2003, 78

Received: 27.08.2003

Dipl.-Chem. Dr. rer. nat. Peter J. Nold
Maschinenfabrik Gustav Eirich,
Walldürner Straße 50, D-74736 Hardheim
peter.nold@eirich.de

Dipl.-Ing. Ralf Löbe
Maschinenfabrik Gustav Eirich,
Walldürner Straße 50, D-74736 Hardheim
ralf.loebe@eirich.de

Dipl.-Ing. Marcus Müller
Maschinenfabrik Gustav Eirich,
Walldürner Straße 50, D-74736 Hardheim
marcus.mueller@eirich.de

46th International Colloquium on Refractories 2003

12. + 13.11.2003, Eurogress, Aachen, Germany



CONFERENCE TOPIC: Refractories for Industrials

- Glass
- Cement/lime/plaster
- Ceramics
- Chemistry
- Incineration
- Refractory raw materials
- Shaped and unshaped refractories

- Processing and refractory lining service
- Wear and Corrosion
- Quality management
- Recycling
- Environmental Protection

For further information please contact:

Forschungsgemeinschaft Feuerfest e.V.
An der Elisabethkirche 27, D- 53113 Bonn, Germany
Tel.: ++49-228-91508-45 Fax: ++49-228-91508-55
e-mail: 2003@feuerfest-kolloquium.de
www.feuerfest-kolloquium.de

