SOLIDIFICATION

The solidification, also known under the term stabilization, is based on the physical encapsulation of the contaminants and their chemical fixation in the solid matrix of a newly generated product. The input material and waste are intensively mixed in continuous or batch wise operated mixers with additives like cement, limestone, gypsum, fly ash or blast furnace slag and water [Lakhani et al. 2014]. In the case of the physical encapsulation, the input material is merged by the binders and additives. Thereby, differentiation between micro encapsulation (encapsulation of single particles) and macro encapsulation (encapsulation of agglomerates of particles or the whole waste matter) should be made. In the case of the physical encapsulation, porosity is reduced substantially and the connecting paths to the surface are blocked, preventing water infiltration. The leaching process is limited to the surface or fracture surface of the final solidified product [Mayer and Sussa 2020]. In the case of the chemical addiction of the contaminants, the impurities are joined into the solid matrix of the agglomerated product. While the treatment of a heavy metal, precipitation processes are frequently used to allocation the impurities to a non-leachable form and to close the harmful compounds in the solidified body.

Generally, solidification is the simple technology and requires the least energy input compared to other agglomeration methods. It does not need large investment outlays and can be carried out even in small production plants. The solidification process can be divided into: (i) preparation of...
mortar, (ii) mixing the mortar with the material/waste, (iii) pouring mortar into matrix and shaking, (iv) curing of solid molds [Borowski et al. 2014].

Depending on the specific of the waste to be treated and the characteristic of the product obtained, the solidified body is utilized at site, disposed of, and stored at admitted landfills or used for construction [Johnson et al. 2014]. Furthermore, the cement mortar can be cast to mold then can be reuse [Rajgor and Pitroda 2013]. In order to improve a mortar for a specific property of concrete, various types of additives can be used: (i) inorganic additives from chemical industry, fly ash and slag, specific minerals including hazardous; (ii) organic additives from sewage sludge of food and packaging industry [Mueller et al. 2008].

Typically, the ingredients are transported via screw conveyors to the solidification unit. The screw conveyors with shaft or spiral conveyors without shaft are used depending on the application and the specification of the additives. The input material and additives are dosed via volumetric dosing units or gravimetric weighing drums into the mixer. The mixers are the core equipment of the solidification unit and ensure a proper mixing of the input material or waste with the additives and the water [Mayer and Sussa 2020]. The required mixing time, as well as the rotation speed of the mixer shaft and the adjustment of the mixing tools are controlled. The final mixture can be cast into forms (Fig. 1).

The filled forms are transported via a roller conveyor system from the solidification unit to the hardening area. The molds can be removed from the form just after 7 days, but the final strength will be achieved after approximately 28 days. The hardened molds have a very low porosity. Water infiltration is not possible further on. Leaching is limited to the surface of the concrete block [Borowski et al. 2014].

**GRANULATION**

Granulation is a widely used technique of particle agglomeration, but the growth agglomeration is used most commonly. During the granulation process, small fine or coarse particles are converted into larger agglomerates called granules. Granules are formed from the particulate materials by wetting and nucleation, coalescence or growth, and consolidation [Iveson et al. 2001]. Granulation is broadly classified into dry granulation and wet granulation, the latter being used the most widely (Fig. 2). The currently available granulation technologies include roller compaction, spray drying, supercritical fluid, low/high shear mixing, fluid bed granulation. The recent progress involves pneumatic dry granulation technology, and reverse wet granulation, steam granulation, moisture-activated dry granulation or moist granulation, thermal adhesion granulation, melt granulation, freeze granulation, foamed binder or foam granulation [Shanmugam 2015].

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**Fig. 1. Solidified concrete block [Mayer and Sussa 2020]**

**Fig. 1. Types of equipment used in wet granulation [Kumar et al. 2013]**
During granulation, the adhesion forces, usually enhanced by binders, cause particles to stick together when they collide in a stochastically moving mass of particulate solids. No externally induced directional forces or pressures act on the growing agglomerates. As a result, more or less spherical agglomerates are grown. Because of the relatively small forces caused by interactions in the tumbling charge, the porosity of the granules is high and increases as bed density decreases. Additionally, since the adhesion forces are small, so are the particles forming the granules [Pietsch 2008, Mangwandi et al. 2011].

The granulation technique usually produces regular granules of the same or similar shapes and dimensions in the range from 2 to 20 mm. Granules may have a diameter above 20 mm, but it should be noted that the granulation yield decreases rapidly with their increasing size. They have a significantly higher bulk density than the starting material and do not cause dusting or caking. There is also no segregation of ingredients, which guarantees a homogeneous composition of specific products [Kelbaliyev et al. 2013].

Individual grains form a stable agglomerate only when there are adequately high forces connecting these grains. This requires a high degree of fragmentation of the particles and maintaining its humidity. The homogeneity of the mass subjected to granulation has a very large impact on the course of granule formation and their strength. In order to increase the strength of the granulate, specific amounts of binders are added to the mixture [Medici 2000]. In addition to the preparation method of the material with the addition of a binder, the quality of the granules also depends on the type of device used and the parameters of its operation, e.g. rotational speed, inclination angle, water spraying capacity, as well as the material dispenser capacity [Zainuddin et al. 2000].

The basic disc agglomerator (pelletizer) is an inclined, flat-bottomed, shallow pan [Reynolds et al. 2005]. The motoreducer drove the shaft attached to the granulator disc by means of a belt transmission. The damping liquid (water) was delivered drop-wise onto the moving granular bed by a sprinkler. The feeder comprised a tank in which the raw material was mixed by means of the built-in stirrer and a feeding screw with a continuous regulation of rotational speed. The finished product (wet granulate), tumbling over the rim of the disc, was collected in a container. So, in the disk granulation the main factors are taken into consideration, as: disc rotations, the inclination angle, side wall height and plate diameter [Holger and Lampke 2016] (Figure 3).

A drum agglomerator represents a simple type of equipment for growth agglomeration by tumbling. They are used in industries for the processing of large amounts of bulk solids. A drum agglomerator normally consists of a cylindrical steel tube with a slight (typically up to 10” from the horizontal) slope toward the discharge end. Retaining rings are often fitted to the feed and discharge ends of the drum to avoid spill-back and to increase the bed depth of material and/or its residence time, respectively [Pietsch 2008]. A drum agglomerator typically work in a closed circuit. Green bodies leaving the drum are sized on vibrating or roller screens. Oversized load is shredded.

![Diagram](image)

**Figure 3.** Disc granulator with segregation effect: $n$ – number of rotations of the disc, $\alpha$ – the inclination angle, $H$ – height of the side wall, $D$ – diameter of the plate [Holger and Lampke 2016]
and returned to the drum collected with the screen fines. Commonly, there are granulating of waste materials, as example: silicon carbide dust, stone dust, fine coal, and fly ash [Gesoglu et al. 2012, Borowski and Ozga 2020].

EXTRUSION

Extrusion is low-pressure agglomeration for the production of caked material. This method is widely used in the pharmaceutical industry, in the agribusiness, and in the polymer industry. Recent developments evaluated extrusion for utilization of selected particulate waste materials, as: wood, plastic, fiber, textile, and biochar. A few works present the extrusion method of producing mixed polymer and plant material biocomposites for use as an alternative disposable product [Moad 2011, Mitrus et al. 2017].

Extrusion is the process of converting a raw material into a product of uniform shape and density by forcing it through a die under controlled conditions. Extrusion can be operated as a continuous process, which is capable of material flow at relatively high output. An extruder consists of the conveying system which transports the mixing material, and the die system which forms the material into the required shape. Extrusion may be generally classified into a molten system under the temperature control or a semisolid viscous system. In molten extrusion, heat is applied to the material in order to control its viscosity and enable it to flow through the die. In turn, semisolid systems are multiphase concentrated dispersions containing a high proportion of solid mixed with liquid phase [Chokshi and Zia 2004].

Extrusion can be performed using four main classes of extruders: screw, sieve and basket, roll, and ram extruders. The screw-melt extrusion equipment consists of an extruder, auxiliary equipment for the extruder, down-stream processing equipment, as well as other monitoring tools used for performance and product quality evaluation. The extruder is typically composed of a feeding hopper, barrels, single or twin screws, the die and screw-driving unit (Fig. 4). The auxiliary equipment for the extruder mainly consists of a heating/cooling device for the barrels, a conveyor belt to cool down the product and a solvent delivery pump [Chokshi and Zia 2004].

The monitoring devices on the equipment include temperature gauges, a screw-speed controller, an extrusion torque monitor and pressure gauges. The theoretical approach to understanding the melt extrusion process is, therefore, generally presented by dividing the process of flow into four sections: (1) feeding of the extruder, (2) conveying of mass (mixing and reduction of particle size), (3) flow through the die, and (4) exit from the die and down-stream processing [Chokshi and Zia 2004]. Generally, the extruder consists of one or two rotating screw inside a stationary cylindrical barrel (Fig. 5). The barrel is often manufactured in sections, which are bolted or clamped together. An end-plate die, connected to the end of the barrel, determines the shape of the extruded product. The heat required to melt or fuse the material is supplied by the heat generated by friction as the material is sheared between the rotating screws and the wall of the barrel in combination with electric or liquid heaters mounted on the barrels.

Most commercial extruders have a modular design. This enables to modify the process to

![Figure 4. Design of the extruder [Chokshi and Zia 2004]](image-url)
meet particular requirements, for example, from standard to high shear extrusion or addition of solvent and evaporating the solvent form the material. Modifying the screw designs allows the extruder to perform a mixing and reduction of particle size in addition to extrusion, so that the material can be blended into the extrudate or even dissolved [Chokshi and Zia 2004].

The extrusion channel is conventionally divided into three sections: (i) feeding zone, (ii) transition or kneading zone, and (iii) cooking or metering zone. The starting material is fed from a hopper directly in to the feed section, which has deeper flights or flights of greater pitch. This geometry enables the feed material to fall easily into the screw for conveying along the barrel. The pitch and helix angle determine the throughput at a constant rotation speed of the screws. The material is transported as a solid plug to the transition zone where it is mixed, compressed, melted and plasticized. Compression is developed by a decreasing the thread pitch but maintaining a constant flight depth or by decreasing flight depth while maintaining a constant thread pitch. Both methods result in an increased pressure as the material moves along the barrel. The melt moves by circulation in a helical path by means of transverse flow, drag flow, pressure flow and leakage; the latter two mechanisms reverse the flow of material along the barrel. The space between the screw diameter and the width of the barrel is normally in the range of 0.1–0.2 mm. The material reaches the metering zone in the form of a homogeneous plastic melt suitable for extrusion. For an extrudate of uniform thickness, the flow must be consistent and without stagnant zones right up to the die entrance. The function of the metering zone is to reduce the pulsating flow and ensure a uniform delivery rate through the die cavity [Chokshi and Zia 2004].

The twin-screw extruder has two agitator assemblies mounted on parallel shafts. These shafts are driven through a splitter/reducer gear box and rotate together with the same direction of rotation (co-rotating) or in the opposite direction (counter rotating) and are often fully intermeshing. In such case, the agitator element wipes both the surface of the corresponding element on the adjacent shaft, and the internal surfaces of the mixing chamber and ensures a narrow and well-defined residence time distribution. In general, co-rotating shafts have better mixing capabilities as the surfaces of the screws move towards each other. This leads to a sharp change in the mass flow between the screw surfaces. As the screws rotate, the flight of one screw element wipes the flank of the adjacent screw, causing the material to transfer from one screw to the other. In this manner, the material is transported along the extruder barrel. The twin-screw extruder is characterized by the following descriptive features [Chokshi and Zia 2004]: (1) short residence time, (2) self-wiping screw profile, (3) minimum inventory, (4) versatility, (5) superior mixing.

Typical twin-screw laboratory scale machines have a diameter of 1–18 mm and length of four to ten times the diameter. A typical throughput for this type of equipment is 0.5–5 mm·min\(^{-1}\). As the residence time in the extruder is rather short and the temperature of all the barrels are independent and can be accurately controlled from low temperatures (30 °C) to high temperatures (300 °C), the degradation by heat can be minimized [Chokshi and Zia 2004].

Extrusion processing involves consideration of various parameters: viscosity with shear rate and temperature, elasticity and extensional flow within nozzle. The extruders allow the in-process monitoring and control of parameters,
such as the temperature, as well as the pressure in extruder and die. The main controlling parameters are process temperature, feed rate, screw speed, motor load and melt pressure. The extrusion is a very effective means of transforming raw materials and waste in particulate form into agglomerated products of various sizes, shapes, textures, composition and content. The process parameters, in combination with screw and die selection, allow a high degree flexibility in processing [Mitrus et al. 2017].

**BRIQUETTING**

Briquetting is a pressure agglomeration that allows obtaining a high degree of concentration of matter. This requires an increase in the energy expenditure [Huang et al. 2008]. Briquetting enables to obtain homogeneous nuggets of many shapes (e.g. barrel, cushion, saddle etc.) and dimensions usually larger than 20 mm (Fig. 6). The essence of briquetting is that as a result of exerting pressure on the particulate material, the grains move closer together. Close direct contact of the grains helps adhesion, which has a significant impact on the surface joining of these grains [Purohit et al. 2006].

Briquetting results in agglomerated products of a specific shape and dimensions, and high mechanical strength. In addition to the agglomeration of mineral resources, fibrous filling and various mixtures of other particulate materials are also briquetted. The fibrous materials should be shredded to a state of loss of elasticity.

**Briquetting of fibrous material leads to the disappearance of its existing structure and modification of some properties, e.g. thermal insulation [Muazu 2017].**

Stamp, screw and cylindrical presses are the most commonly used for briquetting [Temmerman et al. 2006]. The stamp presses allow achieving high pressures. Due to their low efficiency, they are used in low volume production and in laboratory tests. The roller presses are characterized by high performance. Most often, the fine-grained mineral materials and post-production waste are briquetted [Lazaro et al. 2007, Yilmaz 2011].

Briquetting is often used to merge bulk materials with binding additives (binders) [Kumar 2012]. The addition of a binder significantly increases the mechanical strength of briquettes [Taulbee et al. 2009]. Further improvement of the mechanical properties is obtained by curing (seasoning) briquettes. Owing to this property, briquettes can be loaded, transported and stored until development, without fear of losing their consistency.

The briquetting of fine-grained minerals and industrial waste plays an important role in the economy due to enabling the rational use of raw material base and energy resources with meeting the environmental protection requirements [Weyenberg et al. 2005, Herting and Kleinebudde 2007]. The briquetting for waste utilization is widespread used, including: metal scraps, stone dust, fine coal with biomass, and fly ash.

**SINTERING**

Sintering is a process of agglomeration with high temperature post-treatment to create the ecological-safely final agglomeration products. Sintering is a binding mechanism and two technologies may be used [Pietsch 2008]: (i) batch sintering in stationary furnaces for the post-treatment of agglomerated products, (ii) continuous sintering, mostly in tunnel kilns for ceramics.

The sintering of agglomerated materials is an effective, simple and economical processing method. Consequently, the two-step sintering is a promising method for obtaining high-density bodies and smaller grain sizes. The sintering parameters, including the temperature, heating or cooling rate, sintering time and atmosphere type, were determined to obtain the best characteristic
of the final body [Löh et al. 2016]. Microwave sintering has achieved greater acceptance against conventional sintering methods, because of: much lower energy consumption, diffusion process intensification, sintering time reduction, and better properties of bodies [Oghbaei and Mirzaee 2010].

The post-treatment sintering is widely used for the agglomerates that grew during tumbling/pelleting in the presence of a binder and must be cured to achieve a permanent state. This process has been widely studied, for example in the iron and steelmaking industry. Achieving an adequate sintered product depends on the adequate raw materials supply and the previous stage the iron ore granulation [Fernández-González et al. 2017].

Sintering of the rubber granules made from used tires can be a source of ‘environmentally friendlier’ composites. A wide range of these composite materials are characterized by quite good mechanical and functional properties [Sienkiewicz et al. 2017]. Fathi and Kharaziha [2009] investigated compacted bioceramics which were produced at $T_1$ temperature ranging from 1100 to 1300 °C and a 6-min holding time as well as with $T_2$ temperature at 750 and 850 °C for 2–15 h. The heating rate was 10 °C·min⁻¹, and the cooling rate between $T_1$ and $T_2$ was 50 °C·min⁻¹. The best results were obtained with $T_1$ at 1300 °C, $T_2$ at 750 °C, and a 15-h holding time. The obtained bodies were characterized by relative density of approximately 98.5%.

The sintering process is an alternative method to fabricate glass-ceramics from the incinerator residues – mixing bottom ash and fly ash [Arslan and Baykal 2006]. This process consists of the shaping a powder by cold-compacting, followed by a high temperature heat treatment to sinter the compact for the fabrication of ceramics [Dinh Hieu et al. 2012]. Various processes of sintering can be adapted for the manufacturing of artificial aggregates from industrial wastes, usually fly ash and bottom ash. Sintered lightweight aggregates are appropriate for structural applications [Huang et al. 2007, Zhang et al. 2011].

Borowski [2011] produced an artificial aggregates in the process of sintering the previously briquetted fly ashes, in an amount of about 50 wt%, with the addition of silica dust (40 wt%) and milled glass waste (10 wt%) (Fig. 7).

The addition of glass dust is beneficial, because it lowers the temperature of thermal synthesis. After mixing all ingredients, briquettes were made, which were then heated in a chamber oven at 1100 °C for 1.5 hours. The obtained products proved to be durable and resistant to the environmental conditions corresponding to the materials proposed for foundations of paved road surfaces [del Valle-Zermeño 2013].

**CONCLUSIONS**

The numerous examples were shown, that agglomeration processes were an effective way to prevent the dusting of particulate materials and was excellent for the utilization of many types of industrial waste. The conglomerates produced were fully utilized for waste reduction in material management. The effect of agglomeration was also benefiting to reduce the consumption of natural resources and fossil fuels aa well as eliminate waste and pollutant emissions into the environment.

In the case of solidification of waste from the thermal transformation in fluidized bed furnaces, the neutralization of harmful substances contained was immobilised. Briquetting of raw and utilized minerals was carried with use of binders, which was affecting with high strength of bodies. The extrusion technique was used in transforming of bio-wastes into agglomerates that might be an alternative fuel. And last but not least, the high-temperature sintering for agglomerate post-treatment was widely used to produce an artificial aggregate.

**Fig. 7.** View of the surface of sintered artificial aggregate (Phot. G. Borowski)
REFERENCES


