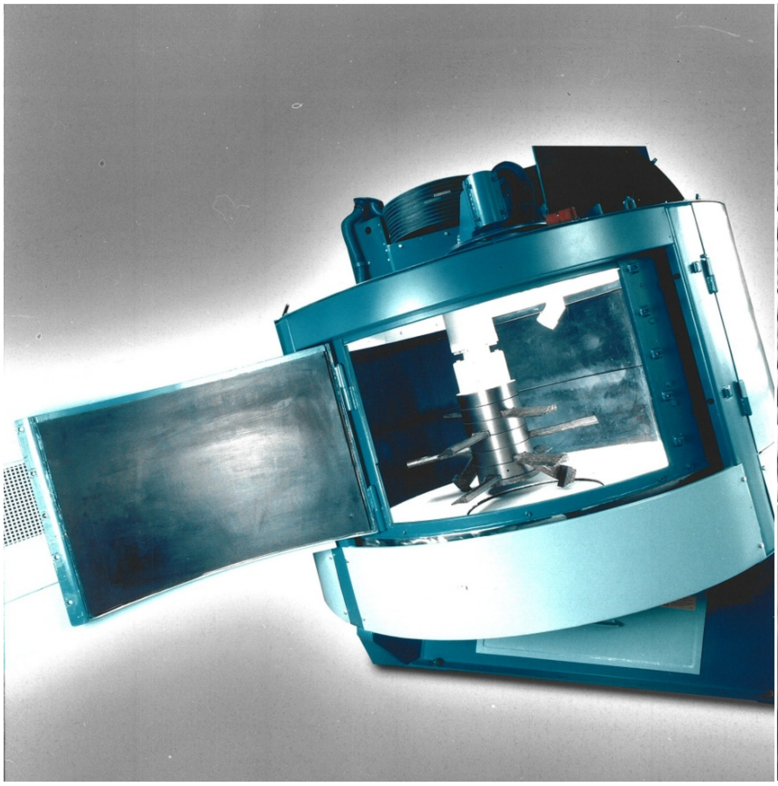
EIRICH MACHINES
EIRICH GROUP

2012

Granulation



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Preface

This small assembling should give you only a basement of granulation to understand what granules are, how granules can be done and which roll plays the Eirich-Mixer.

Details or general information about other systems are to find in books specially based in process engineering and chemical engineering.

This paper should give a general idea which kind of physics is behind the granulation.

Contents

Definition

Granulation or also called agglomeration means an accumulation of very fine particles to bigger units in a strong matrix.

You find agglomerates or granules everywhere not only in the industry, also at home, e.g. medicine tablet that you take, washing powder for the laundry, dish washer tablets, coal briquettes for BBQ, fertilizer for plants, cat litter….

All these daily items are produced in different ways but they all follow the physical laws of the granulation.

http://de.wikipedia.org/wiki/Tablette

http://de.wikipedia.org/wiki/Katzenstreu#Katzenstreu

http://www.oekokraft.com/kokoskohle.html

http://www.esmeyer-shop.de/Hygiene-Einweg-Bad/Reinigen/Spuelen-maschinell/Somat-Tabs-10-Reiniger-fuer-Geschirrspueler-Inhalt-44-Tabs-ACHTUNG-Nachfolger-zu-Somat9.html

http://www.learn2grow.com/gardeningguides/houseplants/caremaintenance/TheNeedToFeed.aspx

http://blogs.reading.ac.uk/tropical-biodiversity/2012/03/substrate-notes-seramis/

Why granulate?

Granulation has a lot of benefits. If you compare the flowability of fine powders like flour or starch to sugar crystals, then it is clear that sugar flows much better. The reason for this property is that the sugar is much coarser. And the interparticulare forces couldn't work in that "big structure" anymore.

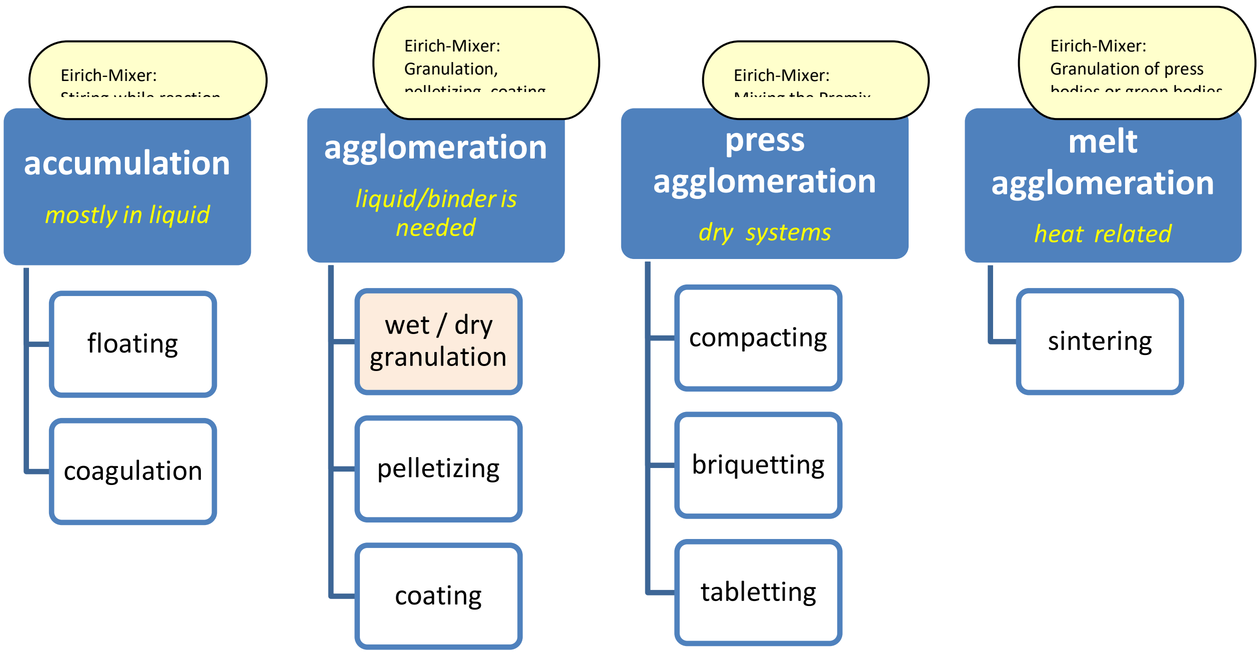
Also with a bigger size you avoid dust while you work with the product. This could be important e.g. for farmers when they bring out the fertilizer onto the field. Due to the coarse form the granules can spread with the farmer's machine in the right range that is desired. If it would be a powder there wouldn't be a trajectory and the wind would bring the fertilizer perhaps to another field. So the competitor farmer had a more or less unwanted gift.

A granule also has the behavior to hold together different components in a matrix so that it provided a certain ratio between different components all the time.

There are many more reasons in the industry to granulate materials. Also the requirements for the granules are changing with the industries and the applications. For recycling products the size and the shape of the granules are not very important. On the other side for a high quality product like proppants, the size and roundness and further the produced yield is important.

4 different ways to produce granules

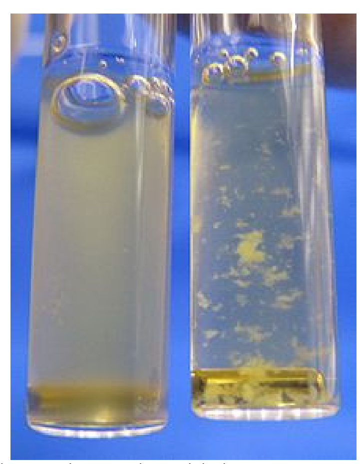
In all four main ways to produce granules an Eirich-Mixer can be used. If not for the granulation itself, the premix or the reaction can be done in the mixer.



Accumulation

This process is mostly used in a liquid. Like in the waste water treatment to flocculate the solid out of water. In most cases a chemical substance, called clarifying agent is needed to create flakes in the suspension and to separate the solid from the liquid. This process is also used in the food industry e. g. production of cheese and beer.

In some cases (only a few materials) an accumulation of dry solids is possible but only while energy input (e.g. rotor in a mixer can bring the particles closer together due to the compression effects).



http://upload.wikimedia.org/wikipedia/commons/thumb/c/c3/Thrombocyteaggregation.jpg/220px-Thrombocyteaggregation.jpg

Press agglomeration

The best examples for this category are tablets. A mix of dry powder is brought to a matrix and with a die the powder is compressed. This area isn’t only used in the pharmacy. Due to a special shaping of the press dies several shapes can be done for industrial applications.

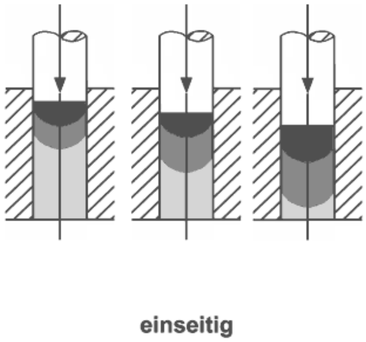


Figure 1: Tablet press with one moving die
(http://www.keramverband.de/brevier_dt/4/1/4_1_3.htm)

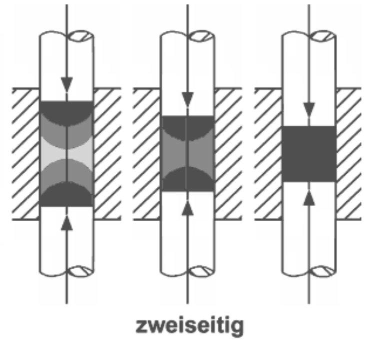


Figure 2: tablet press with two against moving dies
(http://www.keramverband.de/brevier_dt/4/1/4_1_3.htm)

The benefit of an isostatic pressing is that the compaction happens uniformly because the entire mold will be set under pressure. This kind of shaping is often used for high performance products.

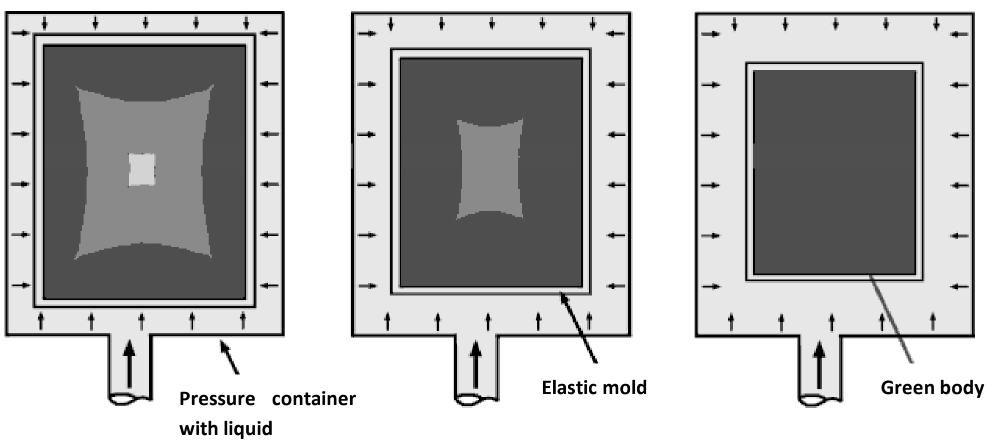


Figure 3: isostatic pressing process (http://www.keramverband.de/brevier_dt/4/1/4_1_3.htm)

This compaction can also be done with compaction rollers. Dry material is fed into a small gap between two contra-rotating rolls. The wideness of the gap influences the strength of the granules. Due to the shape of surface of the rollers the process is called compacting or briquetting.

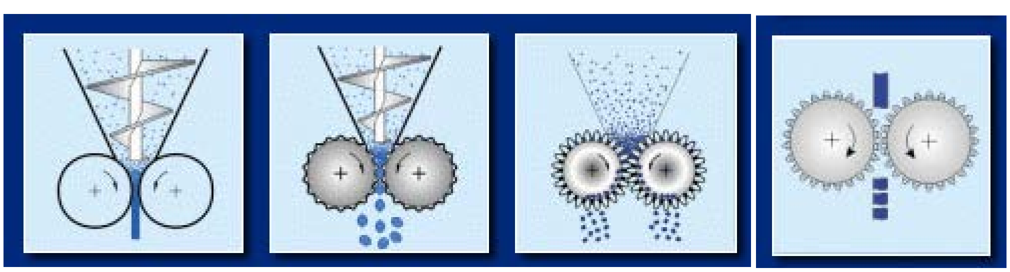


Figure 4: different ways to do a press compaction (http://www.bepexhosokawa.com/01compaction/prodpro/prodpro.htm)

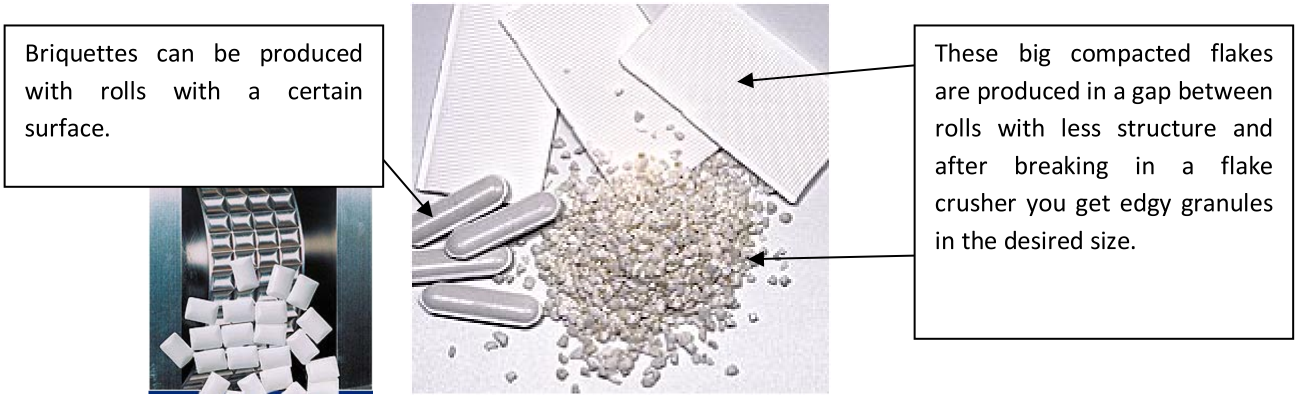


***Figure 5: exaples for press agglomerates** (www.bepexhosokawa.com)*

A further possibility to produce pellets is an edge mill. The material will be pressed due to the rotating rolls or scraper through a matrix.

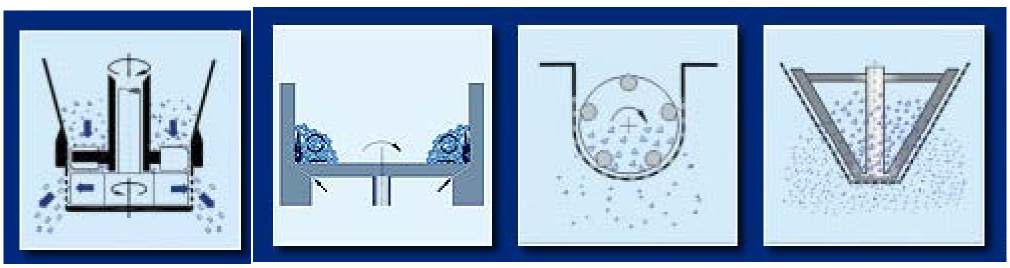


***Figure 6: different ways to do a press compaction** (http://www.bepexhosokawa.com/01compaction/prodpro/prodpro.htm)*

Figure 7: Kollergang
(http://www.umwelt-campus.de/ucb/typo3temp/pics/462ceb89ed.jpg)

Figure 8: granules from Kollergang
http://www.magic-cosplay.com/wp-content/uploads/2012/05/wood-pellet2.jpg

Melt agglomeration

The melt agglomeration can only be done with a high temperature over 1000 °C and is called sintering. While the sintering the different components melt and stick together into a solid matrix.

The sinter process is a very often used further processing for the granules out of the mixer. The granule itself can be the product (like proppants) or its working as a press body and will formed to a certain shape (e.g. for bottom tiles, technical ceramic).

To get a sinter product following steps have to be done:

- Mixing / Granulation
- Drying step (press moisture: still about 7 %)
- Shaping of the product
- Coking of Additive
- Sintering

Granulation

Due to the desired shape of the product, the granules size has to be very small. A tube with a wall thickness of 1 mm couldn't be filled with a press body of 1 mm. They have to be smaller than all gaps in the matrix will be.

Drying step

In case you use a spray dried granule a drying step isn't necessary.

The granules out of the mixer have certain moisture. This moisture has to be reduced to the press moisture.

Shaping

(look to Chapter: Press agglomeration)

Coking of additive

A lot of granules crumble down after the drying if they don't have a malleable composition inside or organic filler which hold the particles together until the sintering is stared. While the huge temperature in the kiln these organic filler disappears totally and leftover some pores. The size of the pores depends on the amount and the kind of additive.

The table below shows a overview of the most common organic binder which were used with granulation in the Eirich Mixer. The quantity has all the time to be adapted to the product; the numbers are only a starting point.

Table 1: Overview about common organic binders

Organic binder	*Usual quantity per batch / in a liquid*
Carboxymethyl cellulose (CMC)	1 – 5 % (liquid)
Polyvinyl alcohol (PVA)	1 – 5 % (liquid)
Starch	3 – 10 % (batch)
Molasses	3 – 10 % (liquid)
Lignosulfonates	3 – 10 % (liquid)

Sintering

The sintering happens while a huge temperature mostly in a rotary kiln. Due to the high temperature the single grains start to melt together. This melting happens in several steps till a strong matrix appears.

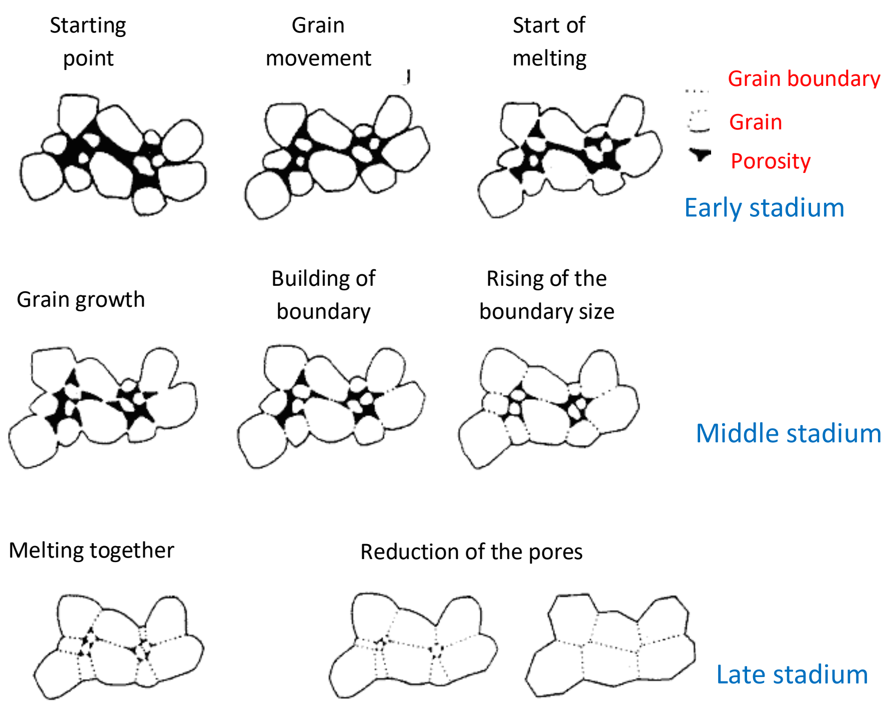


Figure 9: Stadiums in the kiln (http://www.keramverband.de/brevier_dt/4/1/4_1_4.htm)

Different materials need different temperatures to achieve a sintering effect. Below is a table of sinter temperatures of several ceramics.

Keramik	Sintertemperatur
Tonerdeporzellan	ca. 1.250 °C
Quarzporzellan	ca. 1.300 °C
Steatit	ca. 1.300 °C
Cordierit	1.250 - 1.350 °C
Aluminiumoxid	1.600 - 1.800 °C
rekristallisiertes Siliciumcarbid	2.300 - 2.500 °C
gesintertes Siliciumcarbid	ca. 1.900 °C
Siliciumnitrid	ca. 1.700 °C

Figure 10: sinter temperature for ceramic components

Agglomeration

An agglomeration or also called granulation can be done as a wet agglomeration or a dry agglomeration but it is to note that with a so called dry agglomeration also a certain amount of wetness in the system is needed. The difference between both systems is also the mechanical equipment. A wet granulation is done in a spray dryer (Figure 12). In this unit, a slurry of less solid content is sprayed against a hot air stream. Due to the temperature the water evaporates and the solid stick together to a solid matrix, that means a granule. The granule size in this case can be very small, less than 100 µm. The structure of these granules looks like a doughnut. This happens due the high content of water and also of binder. While the evaporation of water, the solid is layering to the outside of the granule structure and a hole in the center is leftover (Figure 13 with 1% binder). If the binder amount is too high, the evaporation pressure in the granule increased until it explodes and produces shell. This appears due to the overloaded binder. The possibility, that the binder seals the surface from granule while the drying, increases with the amount added

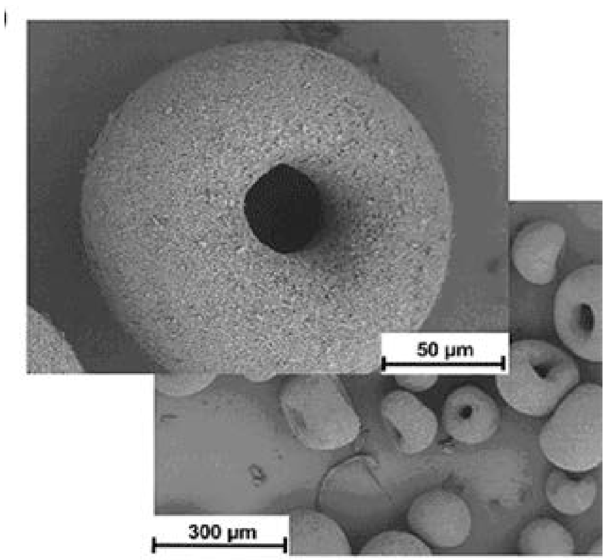


Figure 11: doughnut structure out of the spray dryer (http://mvwww.bci.uni-dortmund.de/monse/Untersuchung.htm)

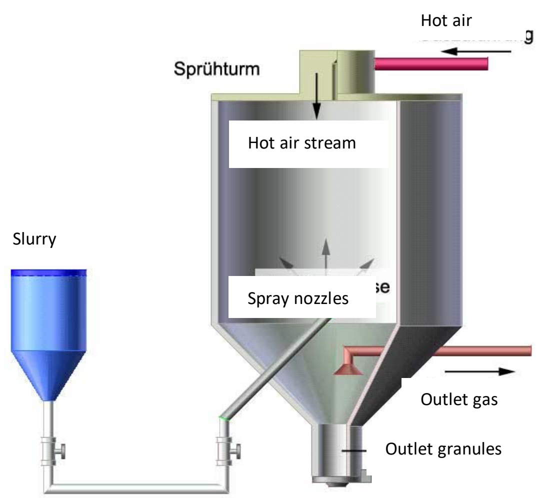


Figure 12: spray dryer (http://www.saar-hartmetall.de/pics/rohteile/spruehturm.jpg)

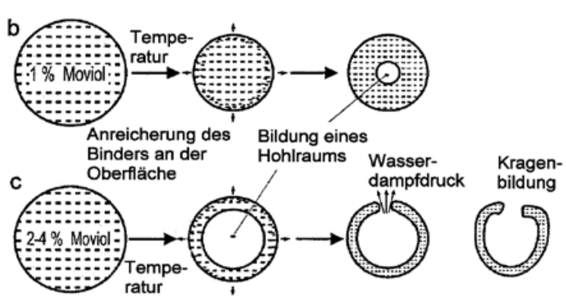


Figure 13: building mechanism of the doughnut structure

The dry granulation can be done in a mixer (Figure 20) or a fluidized bed dryer (Figure 16). The Granulation needs a certain ratio between water and the dry particles. In general, the particles should be to 70 % smaller than 100 µm. Also the system has to be moved to spread the water over the particle surface. Due to this spreading the surface gets sticky and the other particles can stick to it. Over time the granule size grows as long as enough water is available.

There are different kinds of granulation; is the original particle size very narrow (almost the same size) a granule with more porosity is doable. On the other side, if a very wide distribution of particles is available, the granules can be very dense. It could appear, that this range is too wide and the small granules couldn't stick to the surface of the big particle / granule.

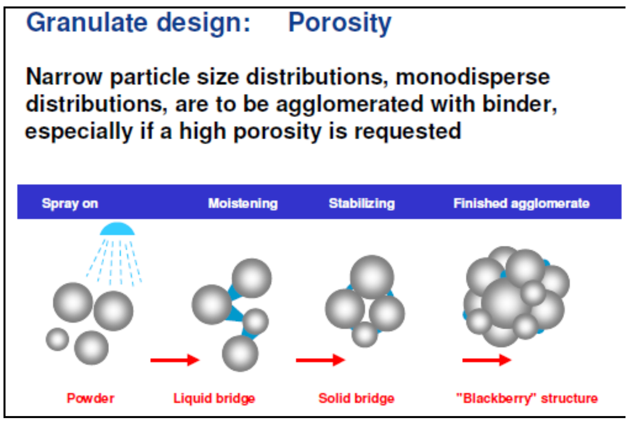


Figure 14: narrow PSD (Granulat-design ppt, Eirich, Markus Mueller)

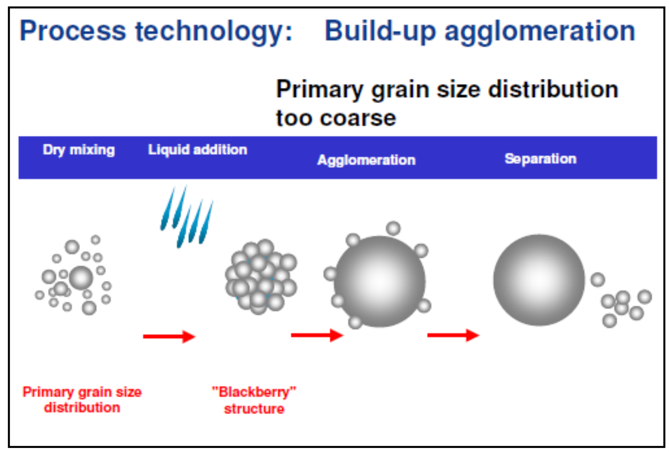


Figure 15: wide PSD (Granulat-design ppt, Eirich, Markus Mueller)

In the fluidized bed dryer material is moved due to the vibration and streaming air. From above liquid is sprayed to the surface of the particles and due the movement they stick together. The structure of these granules looks like blackberries (Figure 17).

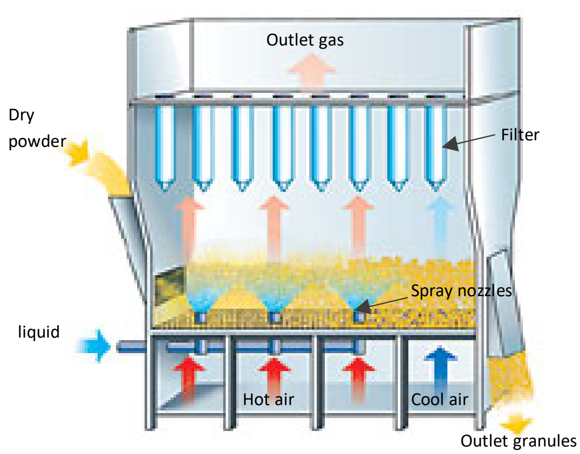


Figure 16: fluid bed dryer (http://www.eksprocess.at/images/glatt-gfg-bottom-spray.jpg)

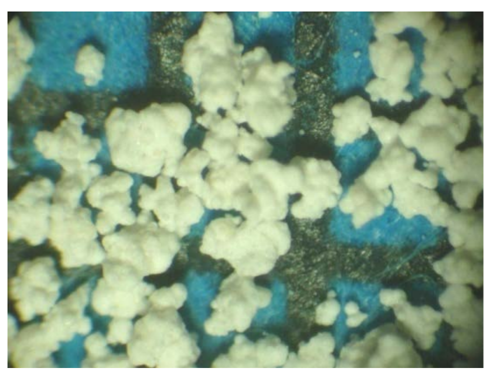


Figure 17: blackberry structur of granules

Due to the size (mostly several cm) the granules were called pellets. They can be produced in disk pelletizer and tumbling drum (wet preparation) or also in a edge mill but (dry preparation)

Tumbling drum

In the tumbling drum wet material or already a micro granule with a lot of available fines is brought into the rotating drum (Figure 18 position A). Due to the rolling effect and the right moisture of the material (granulation wetness like in the mixer), granules are built. If it is needed a liquid can be sprayed onto the granules while the process. In Position p in Figure 18 is the outlet of the granules.

Basic material:	**dry, fine-dispersed matter**
Granulating liquid:	**solution, suspension**
Process technology:	**build-up agglomeration**

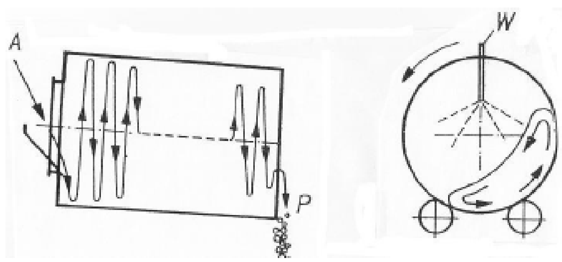


Figure 18: tumbling drum (granulat-design ppt, Eirich, Markus Mueller)

Disk pelletizer

The granulation principle is the same like in the tumbling drum only shape of the machine and its 4 working areas are different. In the lower area near the bottom the small, so called pellet nuclei are rotating. The higher the layer position, the bigger the granule size. In area 4 are rotating the desire size. In section 3 are too big granules and they circulate there.

The circulation area is called granulation kidney due to the shape of a kidney. This circulation is to generate onto the disk by adapting the rotation speed, moisture, feeding speed of premix or powder.

The separation effect can only work if the granules' surface is powdered. If the granules are too wet on the surface, a sticking of smaller granules or particles will still going on.

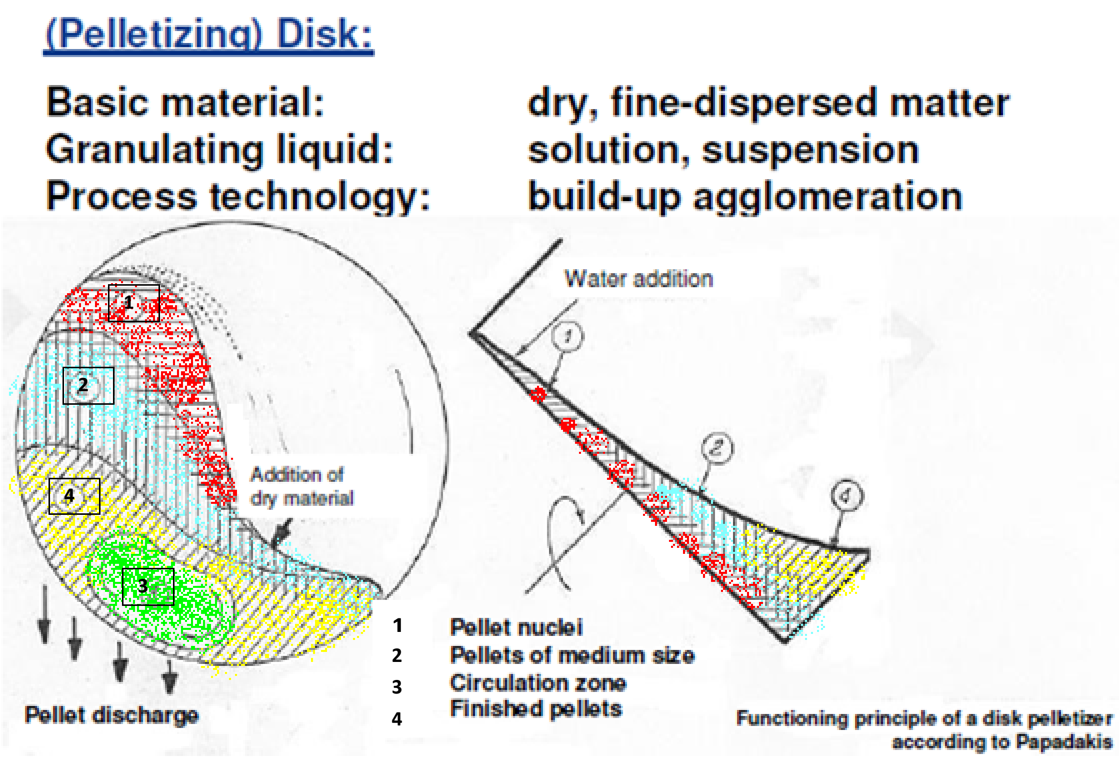


Figure 19: disk pelletizer (granulat-design ppt, Eirich, Markus Mueller)

Principle:

- The feed material is added from the top or the front to the disk
- The rotating movement of the disk carries the material up to the highest point from where it rolls on the disk bottom down to the lowest point.
- Nozzles above the disk spray liquid onto the material
- The rolling movement causes the forming of pellets

Parameters:

- Inclination angle of the disk
- Disk speed
- Ratio: disk height to disk diameter
- Manner of adding liquid
- Manner of adding solids

Mixer

With a mixer the granules consist of a full grain structure. Due to the permanent energy input with the rotor, the granules appear very dense and the shape can be create very round if it is needed.

The densification of the granules can be described like a creation of a snow ball. The snow crystals themselves appear very fluffy in the beginning. They maybe stick together with tight fitting forces. With pressing a certain amount of snow crystals together there appear solid bridges due to the melting of crystals, means water appears and recrystalized to a bigger unit -> snow ball.

Further a build up is seen with building a snow man. Due to the pressure some water is coming up to the surface and “dry” snow crystals are stick to the surface.

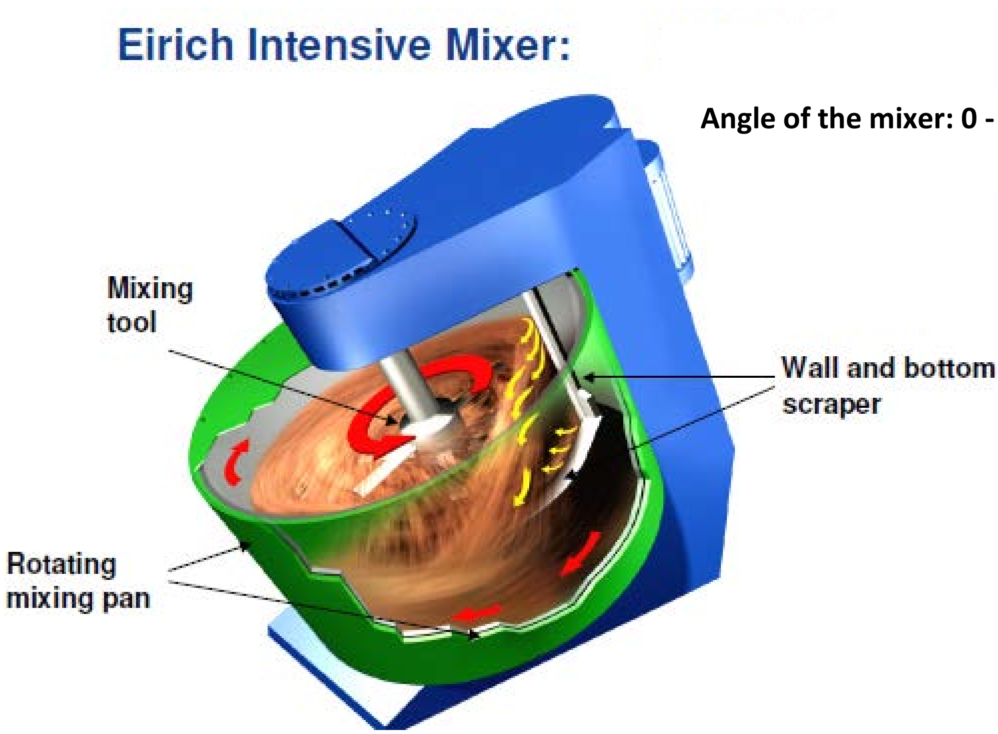


Figure 20: Eirich-Mixer (granulat-design ppt, Eirich, Markus Mueller)

The difference to other mixers the special system of the Eirich-Mixer helps to bring more energy in the system. The moving pan moves the material. For this reason the rotor can do his one job, to granulate, to mix, to dissolve, to kneed, to spread etc. In other systems the rotor has to do both jobs and therefore the results are worse. In addition the angle of 30° and the wall-bottom scraper are helping to move the material into the rotation rotor. This rotor is able to run in clockwise or counterclockwise direction which can increase the shear rate if it is needed.

The required particle size distribution (PSD) can be influenced by rotor speed, granulation time and also the moisture and /or binder system.

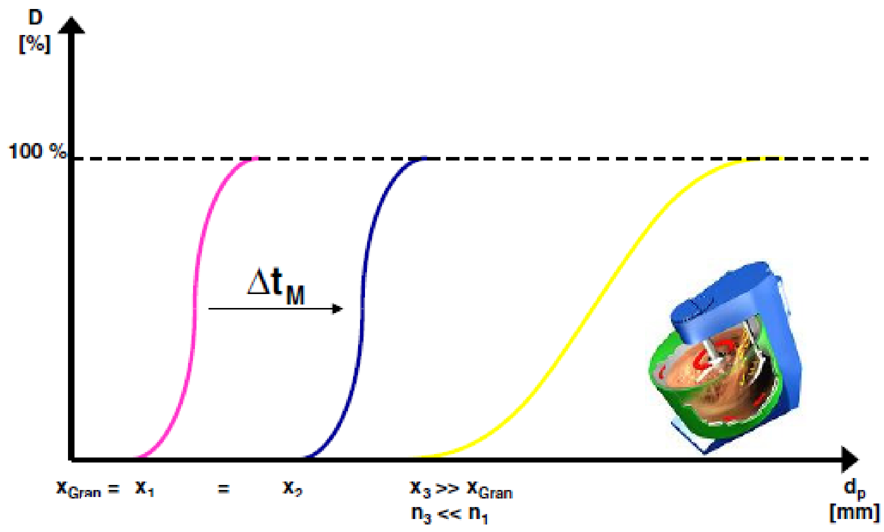


Figure 21: influence speed and time to PSD (granulat-design ppt, Eirich, Markus Mueller)

In Figure 21 it can be seen that a certain distribution can be stable and only over time all granules are growing in the same ratio (pink and blue line). Also bigger granules can be achieved with a slower speed, but in this case, the distribution will be wider (yellow line).

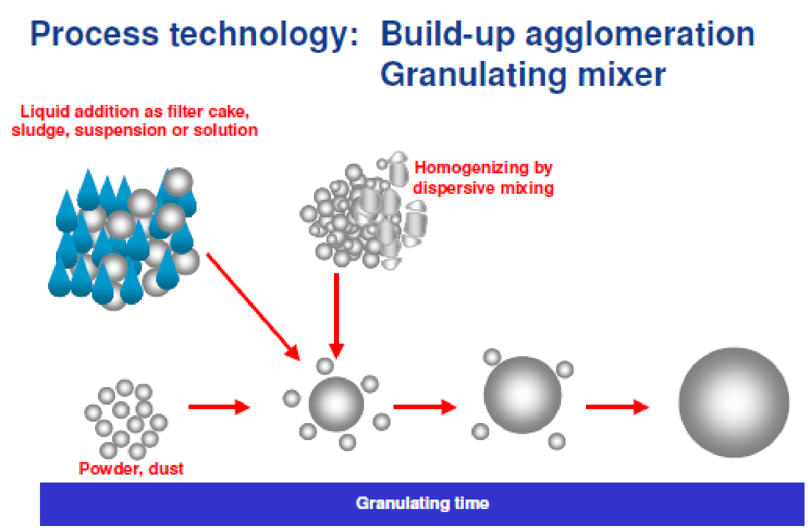


Figure 22: Granulation in Eirich Mixer

The granulation in the Eirich Mixer has the possibility to do different processes in one unit (Figure 22):

- a dry dispersive mixing of powder; lumpy material; 100 % dispersion of doping material
- due to a certain amount of water, a solution, a suspension, a sludge or a filter cake a granulation can be done
- with an additional adding of a dry powder, a coating of the granules is possible
- if more water is added a kneading of the material is possible
- and again, if a dry powder is added the granulation out of a paste is possible

Particle forces

To understand why this system is so flexible the granulation itself should be understood.

Satisfaction of the pores

To build a castle at the beach you need sand and water. If you put to less water to the sand you can't shape anything there are to less bonding forces (bridge building area). If you put a bit more water to it and you achieve a satisfaction of the pores of > 0.3 to 0.8 you will have the possibility to create shapes but there won't be very stable. Only over 0.8 satisfactions there are enough bonding forces which hold the castle stable. And if you put too much water in it (satisfaction about 1) you will create a mud.

I.	Bridge building area	$0 < S \geq 0{,}13$
II.	Transfer area	$0{,}3 < S \geq 0{,}8$
III.	Capillary area	$0{,}8 < S \leq 1$
IV.	Suspension area	$S \approx 1$

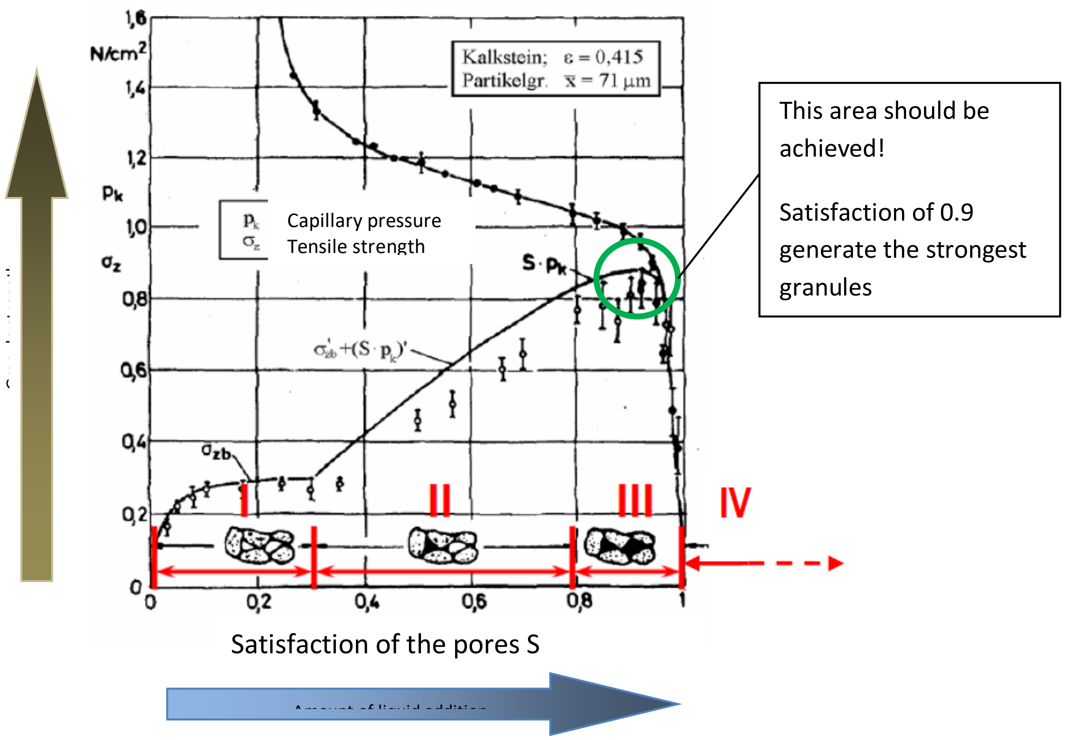


Figure 23: satisfaction of the pores against the appearing strength (Masterthesis C. Klein, Analyse von Mischwerkzeuggeometrien im Hinblick auf die Ausbeutemaximierung bei der Mikrogranulierung)

The bonding effects appear due to the adhesive forces, especially the Van-der-Waals forces. This forces appears only in very short lengths of a few Angstrom (1Å = 0.1 nm) and there are dipole – dipole interaction between molecules like in H_2O itself and to other materials as well (Figure 24 B). The strongest bonding is the sinter bridge. This strong matrix appears only after a high tempering of the product in a kiln. The crystallization between particles appears with certain humidity. It is often seen in a kitchen with sugar or salt if it isn't stored free from humid.

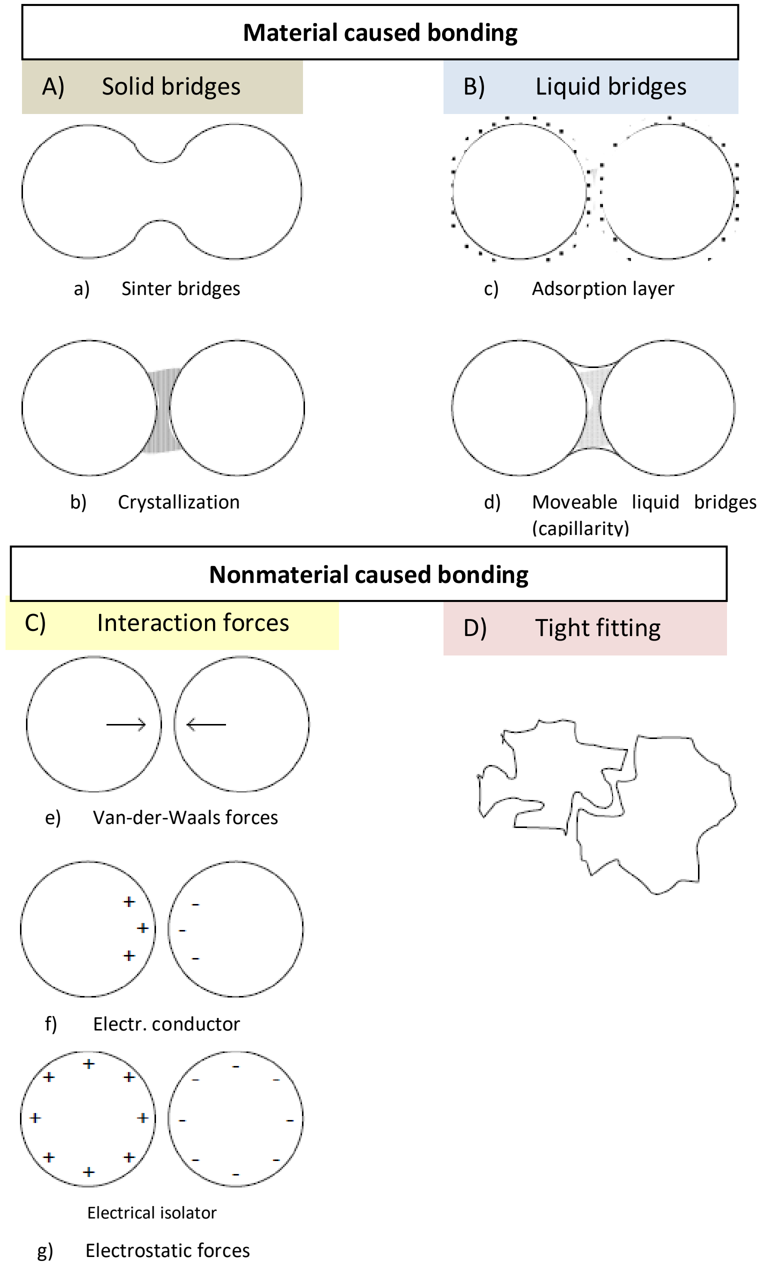


Figure 24: bonding effects between particles (Masterthesis C. Klein, Analyse von Mischwerkzeuggeometrien im Hinblick auf die Ausbeutemaximierung bei der Mikrogranulierung)

The nonmaterial forces are often seen in e.g. with flour. The easy breakable lumps in the flour package appeared due to the cohesive forces. That means the particles themselves have conduction forces.

The “tight fitting”-bonding can be seen in a bundle of paper clips in a jar. They can snag together due to their shape.

Wetting of the solid

This interaction in the granulation process appears because liquid is penetrating the surface of a solid and creates forces between these particles. If wetting of the solid isn’t working, and the liquid appears like a ball on the surface or in the other direction, if the solid it easily to penetrate it is spread out totally, means there is no angle anymore between liquid and solid (example S).

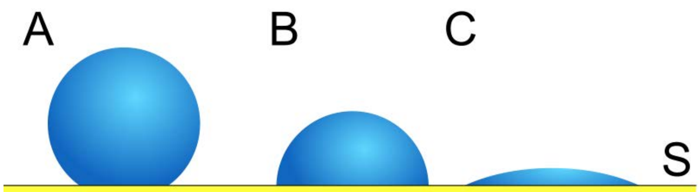


Figure 25: penetration water to a surface (http://de.wikipedia.org/wiki/Benetzung)

In case of water, if the solid “likes” water a penetration is possible (example C). It is called hydrophilic and the appearing angle between the solid and the water is very high. In case the two media doesn’t “like” each other it is called hydrophobic and a wetting isn’t possible (example A). The possibility if a liquid can wet a solid depends on the balance of adhesive and the cohesive forces to each other.

The adhesive force of the liquid itself, the so called surface tension is often seen in nature. Due to this force a water bug is able to walk over water or a drop can be built on a leaf.

Figure 26: water surface tension (http://en.wikipedia.org/wiki/Surface_tension)

The following pictures show the two different ways to wet the solid. In case of distribution and coalescence (Figure 27), liquid droplets are small and in generally the amount in comparison to the solid is less. After a mixing with the solid, the liquid is spread onto particles. This makes the surface sticky and the other particles stick to the surface. In the nuclei building step, the water in the gaps spread out and layer the surface again, so that the whole surface gets sticky again. This procedure is working as long as enough liquid is available.

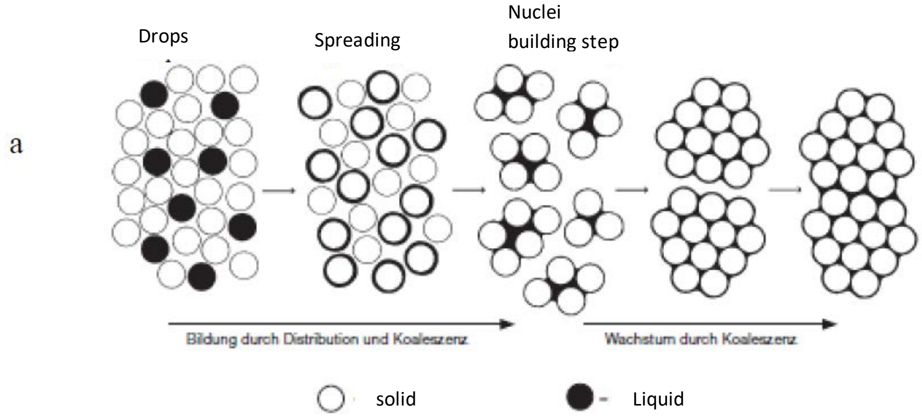


Figure 27: granulation with distribution and coalescence

In case of Figure 28 there is more liquid in the first steps and the structure is more like sludge (satisfaction of the pores ~ 1) but with increasing of the solid amount it turned into the coalescence step as before described.

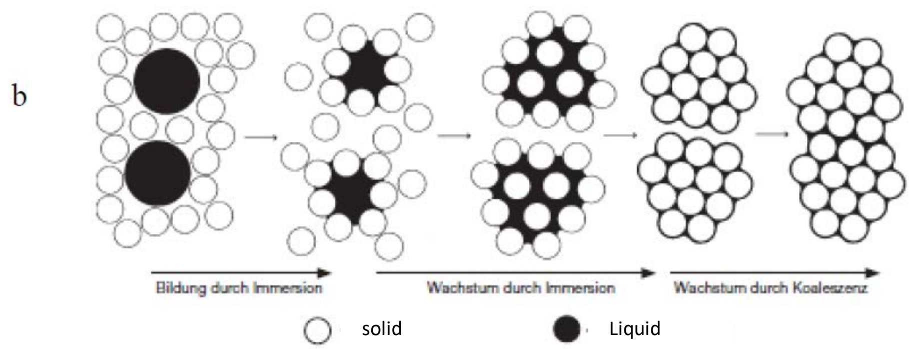


Figure 28: granulation with immersion and coalescence