Process for the preparation of the low sodium salt Suprasel OneGrain®

E. Altena, G. J. Stokkers, A. Zwijnenburg, AkzoNobel

Process for the preparation of the low sodium salt Suprasel OneGrain®

E. Altena, G.J. Stokkers, A. Zwijnenburg, AkzoNobel

Salt production

Keywords: milling, compaction, edible salt

Abstract

Sodium chloride is used in foods for its particular taste and its taste-enhancing properties. However, there may be a need to reduce human sodium intake as a too high sodium intake is thought to be related to a number of health problems. Suprasel OneGrain® is an answer to this need. In its grains part of the sodium chloride is replaced by other mineral salts like potassium chloride, while a flavor is added to mask the potential unpleasant taste and enhance the saltiness.

As particle density, particle shape and particle size of the ingredients differ to a greater or lesser extent, the preparation of a simple blend will face segregation issues. That is why Suprasel OneGrain® was developed. Its preparation process is described in this paper.

It provides a product in which all ingredients are homogeneously mixed in the individual grains. The preparation is a classic example of a compaction process followed by crushing the compacts and sieving the crushed particles. Special attention is paid to the particle size of the starting materials. They must be small enough, at least 3 times smaller than the size of the final salt product, to satisfy the need for grains with a comparable composition.

Suprasel OneGrain® is currently successfully applied in bakery and meat products. It provides a unique combination of a low sodium salt and an excellent taste. A further broadening of the application area is being pursued.

1 Introduction

Among other reasons, sodium chloride is used in foods for its particular taste and its taste-enhancing properties. There is a market need for low sodium salt, as a too high sodium intake is thought to be related to a number of health problems. Therefore, in a number of salt products part of the sodium chloride is being replaced with other mineral salts, like potassium chloride. Potassium chloride, however, is characterized by a more metallic and bitter taste than sodium chloride, which makes it less preferred for human consumption. Alternatively, actual sodium intake can be lowered via products, sometimes partly based on sodium chloride, that generate a strong salt taste sensation and so ensure that less of the product needs to be consumed for a similar taste and taste-enhancing effect[1].

In order to mask the unpleasant taste of sodium chloride-replacing materials like potassium chloride, further additives, so-called masking agents, can be applied. In addition, it is known to add taste enhancers to sodium chloride based salt products to enhance the sodium chloride taste effect[2].

2 The need for a "OneGrain"

The additives added to sodium chloride-based products can have a smaller particle size than sodium chloride and potassium chloride raw materials, especially where they concern organic additives. For example, yeast based additives have a particle size which is significantly below 100 microns, while sodium chloride and potassium chloride as industrially available generally have a particle size of a few hundred microns.

As an illustration of the particle size effect, two experiments were conducted with different particle sizes of the ingredients. In the first experiment, the ingredients used were 69 wt% NaCl, 26 wt% KCl and 5 wt% yeast extract Maxarite™ Delite ex DSM Food Specialties BV. The formulation was made using commercially available NaCl (mass median diameter, d_{50} = 375 µm) and KCl (d_{50} = 296 µm). These components were charged together with an unmilled yeast extract having a d_{50} of 86 µm and mixed in 1.5 kg batches in a 2-litre Nauta mixer for at least 10 minutes at 19 rpm. From this mixture 50 g tablets (40 mm diameter, ca. 20 mm height) were made on a Herzog tablet press using 1.0 t/cm^2 pressure (which corresponds to a pressure of 100 MPa). The resulting tablets were broken diametrically and milled on a Frewitt sieving mill using a 6 mm, 3 mm, and finally a 1 mm screen. Particles of the fractions 90 - 200 µm and 200 - 710 µm were analyzed for component distribution.

In Figure 1 a picture is shown of the 200 to 710 µm fraction, using SEM-EDX analysis (scanning electron microscope energy dispersive analysis of X-Rays), to determine Na, K, Cl elemental and organic material distribution. As can be seen in Figure 1, the yeast particles appear to be primarily present on the outer surface of sodium chloride and potassium chloride particles. Moreover, it can be seen that the particles to a very large extent consist of the primary particles that were used as starting material. In other words, crushing has taken place primarily via the original particle surfaces, thereby freeing particles of the original individual components, i.e. NaCl and KCl and yeast extract. Because of this last observation, it is expected that the yeast extract particles are located solely on the surfaces of the original KCl and NaCl starting material particles.

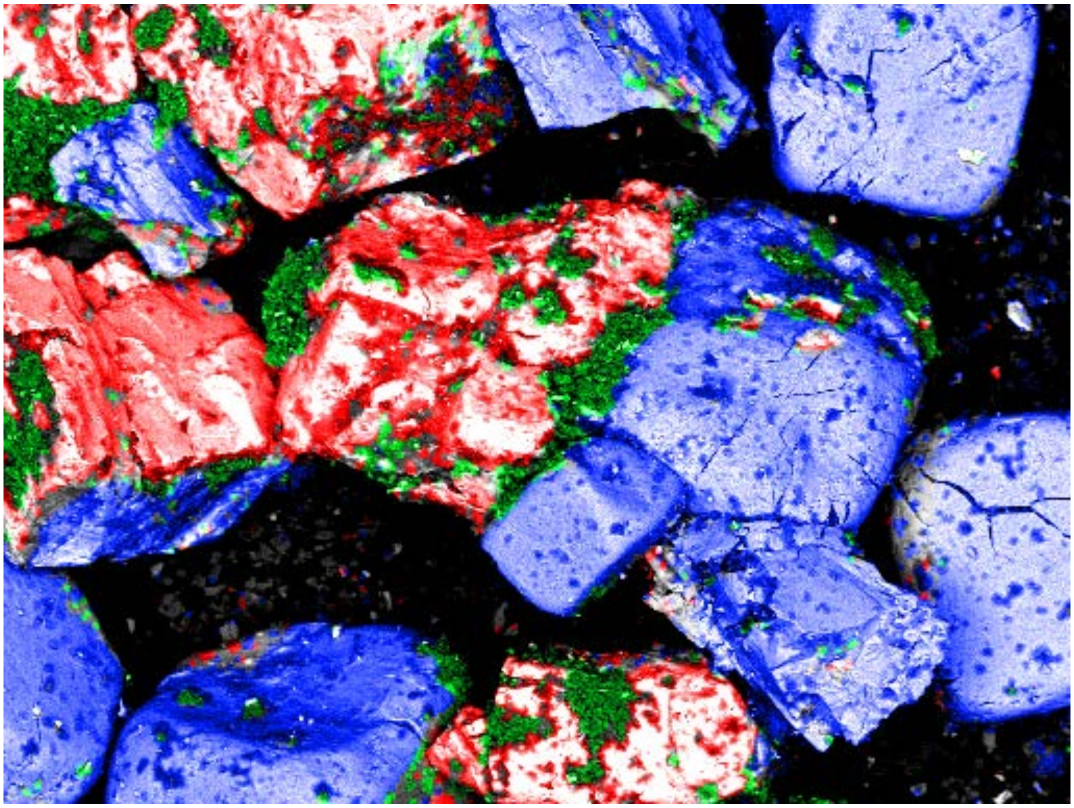

Figure 1: SEM-EDX of compacted NaCl/KCl/yeast mixture with different particle size. The sample shows mainly separate NaCl (blue) and KCl (red) particles containing clumps of yeast extract (green) on their surface.

In the following section, the effect of smaller particle size starting material is shown. For this second experiment 70 wt% NaCl, 26 wt% KCl, and 4 wt% yeast extract Maxarite™ Delite were used. The NaCl and KCl were milled using the Alpine 160 UPZ pin mill operated at 5,700 rpm. The milled NaCl (d_{50} = 69 µm) and KCl (d_{50} = 58 µm) were charged together with unmilled Maxarite™ Delite ex DSM Food Specialties BV (d_{50} = 58 µm) in 1.5 kg batches to a 2-litre Nautamixer and mixed for at least 10 minutes at 19 rpm. The mixed powder was collected in a bin from which the Herzog tablet press was manually fed with 50 g portions. The applied pressure ranged from 0.5 t/cm^2 to 1.0 t/cm^2 (which corresponds to a pressure of 50 to 100 MPa). The majority of the tablets were compacted at 1.0 t/cm^2 pressure. The dimensions of most of the tablets were 40 mm diameter and ca. 20 mm height. The resulting tablets were broken diametrically.

After preliminary breaking, further crushing of the tablets was done in 3 steps:
1. Merz toothed (pyramids) roller crusher with a diameter of 200 mm, roll distance 8.0 mm, roll speed 295 rpm (both rolls).
2. Merz smooth roller crusher with a diameter of 200 mm, roll distance 3.0 mm, roll speed 195 and 300 rpm. This means that the crusher is operated by friction.
3. Merz smooth roller crusher with a diameter of 200 mm, roll distance 1.0 mm, roll speed 195 and 300 rpm.

The oversized fraction after the final crushing step appeared to be large. Therefore, the product was crushed once more on the Merz smooth roller crusher, now operated at a 0.8 mm roll distance. The crushed product was sieved on the Mogensen Piccolo equipped with a 200 µm and a 710µm screen. In Figure 2 a picture of the 200 to 710 µm fraction of the sieved product is shown. The same SEM-EDX analysis as in the previous experiment was used for the individual compounds visualization.

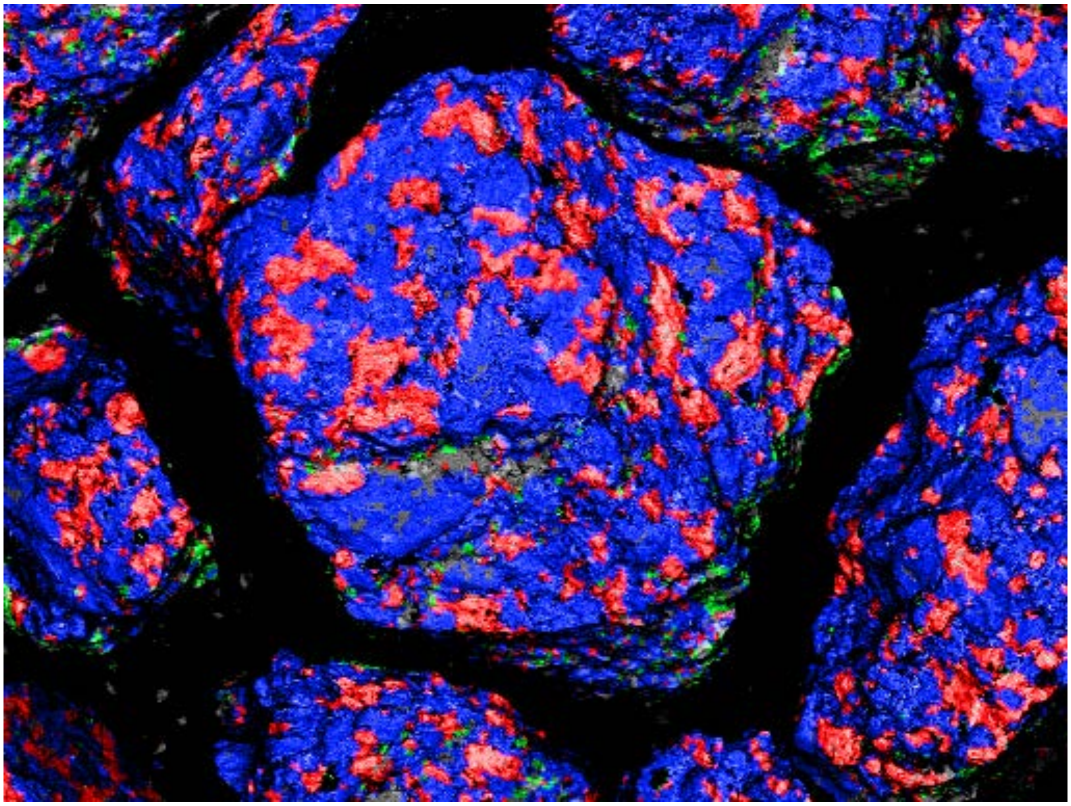

Figure 2: SEM-EDX of compacted NaCl/KCl/yeast mixture with similar particle size of salts and yeast prior to compaction. The sample shows NaCl (blue), KCl (red), and yeast extract (green) well mixed.

The distribution of the individual substances within the particles has been analyzed by embedding the particles in a resin and carefully removing the upper layers of the particles. The result is given in Figure 3.

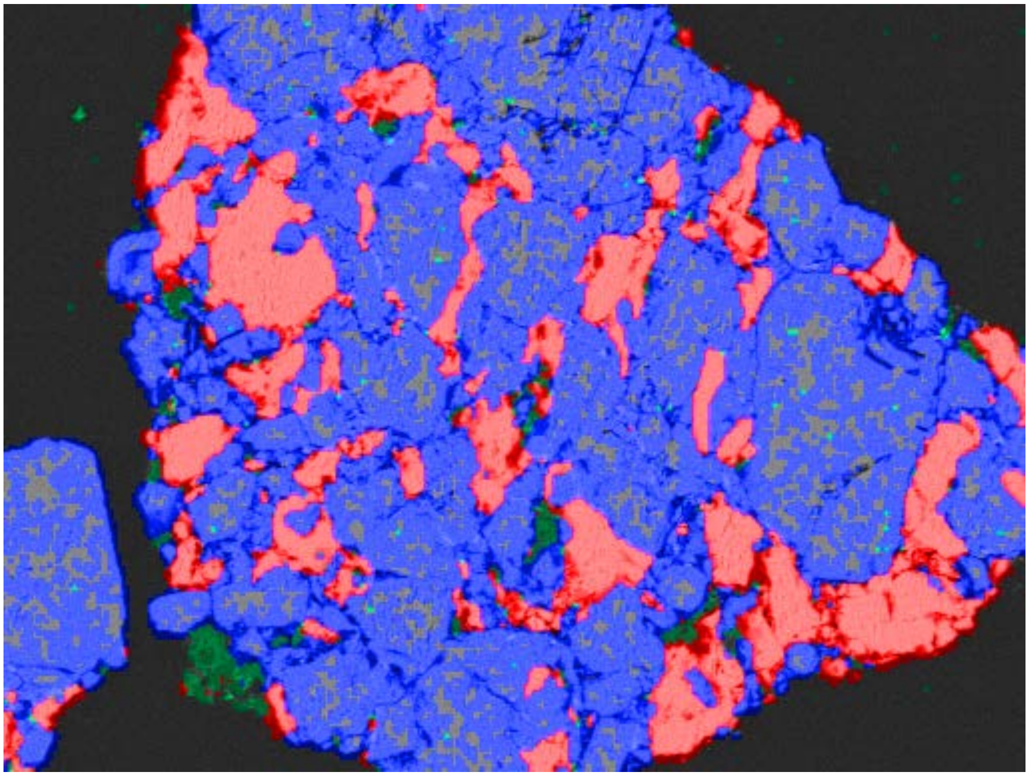

Figure 3: SEM-EDX of compacted NaCl/KCl/yeast mixture with similar particle size of salts and yeast prior to compaction (cross section).

As can be seen in Figures 2 and 3, the product consists of particles that each have the individual components present. It is possible to identify the milled starting materials in the particle and it is clear that the components are evenly and homogeneously distributed in the particles, contrary to the sample of Figure 1 in which the yeast additive is preferentially present at the surface.

From a processing point of view it is better to avoid the smaller particles being located for the major part on the outer surface of the end product. For example, a number of additives are more hygroscopic than sodium chloride and potassium chloride, which results in the salt product showing a more hygroscopic behavior when the additive particles are located on the outer surface than when they are entrapped and homogeneously mixed through the salt product[2].

3 Milling and compaction production process

As discussed in the introduction, the application of small-particle size additives and the need for prevention of demixing, would mean that small NaCl particles are desirable for low sodium salt products. The minimum particle size prior to compaction has been analyzed.

Suprasel Microzo® (AkzoNobel) NaCl salt was milled in an Alpine 160 UPZ pin mill. The pulley that drives the rotor was selected to achieve a rotational speed that would provide a particle size after milling close to 10 µm. The salt was continuously fed to the mill via a vibrating trough.

After milling three batches (10 kg each) were available for further processing:
1. Suprasel Microzo® as such
2. milled Microzo®, use of the Ø100 mm pulley (rotor at 11400 rpm)
3. milled Microzo®, use of the Ø80 mm pulley (rotor at 14250 rpm)

From these products, the particle size distribution was determined on the Sympatec Helos-KF by laser light diffraction.

Subsequently the products were compacted at the Bepex CS 25 roll compactor. The compacted product was sieved on a 710 µm screen to remove fines. Both fractions were weighed. The compact density was determined using the mercury submerging method. From the compact density the porosity of the compacts could be calculated using the NaCl true density of 2165 kg/m^3.

Where the Microzo® as such could be processed very well, processing was already more difficult in the 2nd test. The applied force did not reach the desired level, although the compacts were still sufficiently strong. An even lower d_{50} in the 3rd test gave troublesome compaction as a result of decreased flowability of the powder. A lot of uncompacted material passed the rolls and the compacts that were made were weak and looked more like fragments of the cigars that are usually made via the roll compactor.

The results (fines and compact density) are given in Figure 3 as a function of the particle size (d_{50}) of the material after milling, before compacting.

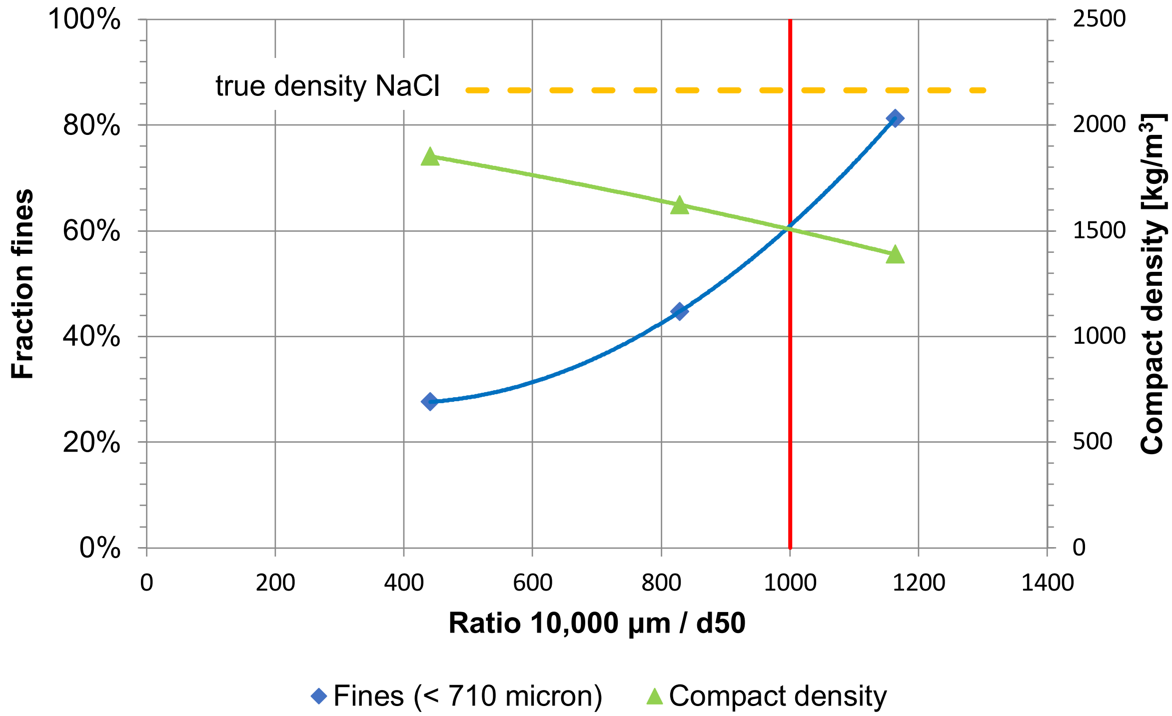

Figure 4: Compact density and fines percentage as function of ratio 10 mm / d_{50}. D_{50} of the material after milling of the starting materials.

With decreasing particle size after milling, there is an increased fraction of fines after compaction (resulting in a lower product yield). Also, compact density decreases with a decreasing particle size of the milled product. A maximum typical particle size after compaction is 10,000 µm (10 mm), this implies the minimum particle size of the milled NaCl is 10 µm in order to achieve reasonable fines production and compacted density of the final product.

4 Conclusion

In this contribution, an improved process that is more (energy) efficient and that results in a low sodium salt product has been presented. Salt additives are homogeneously mixed with the sodium chloride, and optionally sodium chloride-replacing material, and included in the individual grains. The product is commercialized by AkzoNobel under the brand name Suprasel OneGrain® and in the meantime has found many applications, amongst others in bread and meat products.

It has been shown that milling/compaction is a possible route to this product and that the particle size before compaction is of critical importance. If two materials of different size are mixed, demixing will occur upon transport and storage. Agglomeration is a way to avoid such demixing. However, after compacting and crushing to the desired particle size, the smaller particles will end up on the outer surface of the particles, resulting in loss of the additive.

The particle size of the starting materials must be small enough, at least 3 times smaller than the size of the final salt product, in order to make sure the individual grains obtained after compaction and milling contain all ingredients homogeneously mixed.

5 References

[1] E. Frerot, F. Benzi, Aminoacid derivatives of dicarboxylic acids as flavor ingredients, WO2004075663 A1, 2004, Assigned to Firmenich SA.

[2] G.J. Stokkers, E. Altena, Process to prepare a low-sodium salt product, product obtainable thereby and the use thereof, WO 2010124905 A1, 2010, Assigned to Akzo Nobel Chemicals International B.V.