

# Granulation of sodium silicates by pressing method

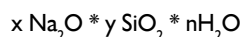
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## Introduction

Silicates are salts of weak silicic acid and base. On an industrial scale, they are mainly derived from sodium and potassium silicates. Their manufacture involves either of the two methods: classic and direct. The classic method consists in the manufacture of the so-called sodium or potassium glaze by means of fusing quartz sand and sodium or potassium carbonate at high temperatures. The composition of solid silicate may be controlled through the selection of the content ratio of respective reagents. The glaze, upon its dissolution in water at a temperature of over 150°C and the module correction, yields aqueous silicate solutions, the so-called sodium water glass with a defined molar ratio of  $\text{SiO}_2/\text{Na}_2\text{O}$ . On the other hand, the direct method yields sodium silicates as solutions as a result of a hydrothermal reaction of quartz sand with sodium hydroxide under augmented pressure [1]. Potassium glass is manufactured in a similar manner. Water-soluble solid sodium or potassium silicates are obtained by means of evaporation, e.g., in a spray dryer.

Synthetic silicates constitute permanent compositions or aqueous solutions of  $\text{SiO}_2$  silicate anions oligomers combined with metal cations, predominantly sodium. Soluble sodium silicates usually form stable aqueous solutions that do not, however, have an unequivocal composition, taking chemistry into account. They evince the capacity to form stable coupled structures that are the complexes of particular sodium silicates and silicic acid. Their composition varies and changes within the range from 4 moles of  $\text{SiO}_2$  per 1 mol of  $\text{Na}_2\text{O}$  to 1 mol of  $\text{SiO}_2$  per 4 moles of  $\text{Na}_2\text{O}$ . The general chemical formula of sodium silicates may be represented thus:



Nota bene, the values of x and y vary from 1 to 4 [1, 2].

The main parameter allowing to differentiate between particular, for instance, sodium silicates, is the silicate module that equals the quotient of the molar content of silicon dioxide to sodium oxide (y:x) in the system analysed. It is sufficient to use the module for the purpose of the characterising sodium silicates. However, precise specification of silicates that permits the determination of the form they occur in (anhydrous, hydrated, aqueous solution) entails the provision of one of the interdependent physical parameters apart from the module: concentration, density or viscosity [3, 4].

The materials under investigation are readily applied in many industry branches for various purposes by virtue of their non-toxicity and eco-friendliness coupled with unique physicochemical properties. Their application is determined by the silicate module as well as their form. In the past, sodium silicate was universally used in the detergent industry as the basic soap component. Nowadays, aqueous solutions of sodium silicates serve to manufacture cleaning agents, fire-resistant and ceramic materials. They are also deployed in the paper and chipboard industry as glues and bleaching agents, in the production of moulding sands in metallurgy and foundry engineering and in water conditioning. They serve as a luting agents for building mortars. Solid silicates are applied for the manufacture of detergents as well as cleaning-detergent agents, detergent-disinfecting agents and ingredients for the products of household chemistry. They are deployed in the manufacture

of a number of composite materials as well as detergents, paints and glues [5÷8]. Solid sodium silicates may also be used as active fillers of washing agents.

Sodium silicates belong to mass products with a broad scope of application, the manufacture of which requires only a basic resource base: silicon dioxide ( $\text{SiO}_2$ ) as quartz sand as well as sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) [9]. They are eco-friendly. The above mentioned properties along with the growing consumption of silicates determine their potential and increasing economic significance.

In the industrial applications of solid sodium silicates the grain size and bulk density, that affect the application of the above mentioned, are of great significance. For sodium silicates derived by spray drying the properties specified may be altered to a limited extent. A broader scope of alteration is possible in granulation technique. The application of granulated silicates reduces their dust content, which with their high alkalinity is a factor that greatly contributes to lowering the hazard related to human exposure. A larger grain size also has an effect on the speed of the dissolution of silicates in water baths, while granulates with particular granulometric distribution and bulk density in detergent mixtures are less susceptible to segregation and tend to form mixtures with a uniform composition.

Sodium silicates are available both in dust and granulated forms on global markets. The characteristics of marketed granulated sodium silicates is provided in Table I

Table I

Characteristics of granular sodium silicates

Britesil®	H 20	H 265 HP	H 265 LC
Module	2.0	2.65	2.65
Bulk density, kg/m <sup>3</sup>	825	725	500
Mean size, μm	700	700	450
Particle size distribution, μm			
> 16,000	≤ 1	≤ 1	≤ 1
< 250	≤ 5	≤ 10	≤ 10

In the literature available, mainly patent-related, a number of methods of obtaining sodium silicates with a particular granulometric distribution is specified. The universally deployed spray drying, in relation to the run-time, yields particle size distribution < 250μm and bulk density exceeding 500 kg/m<sup>3</sup> or >250μm and bulk density exceeding < 200 kg/m<sup>3</sup> [10]. Both particle size distribution and bulk density depend on silicate module and bound water content. Another method of granulation involves the application of a drum or multiple blade granulator, allowing to obtain granules with the size of 0.2- 2.0 mm [11,12]. The sodium silicate granulation methods presented above yield products within a limited scope. A method that provides greater possibilities is pressure granulation. It permits to obtain a broader range of granules with respect to the required particle size distribution and bulk density.

Powder granulation by means of pressing (compacting), or densification through the application of external force, consists in the formation of the substance compact structure by means of stable, cohesive bonds between the particles subjected to pressing. The briquette obtained by way of densification (board, strip) is crushed and filtered in order to isolate a particular fraction that constitutes the finished product. The mechanism of agglomeration is rather complex and depends on the pressing force value. The basic process of granulation thus realised, comprises the following steps: component dosing and mixing (sometimes preliminary densification), pressing, crushing, sieving. Undersized material is recharged to the mixing facility and oversized material is subjected to crushing. The pressing method has the advantage of being suitable for almost all materials in the absence of particle shape requirements [13].

The main parameter to affect the density and final thickness of the compact is the unit compression force. The determination of relationships between the distributions of compression force in the compression zone has been the subject of numerous theoretical and experimental works. In practice, however, each material and type of compacting device requires an empirical validation of such correlations. During powder material densification under matrix press, extruder or roll press there occurs a gradual change from the material bulk density  $\rho_0$  to homogeneous structure density  $\rho_w$ , with the material taking on the form of tablet, fibre, board or tape, etc. During granulation by pressing method the structure is deformed at the outset of densification process. This involves the mutual dislocation of particles, alteration of their array and the destruction of the weakest particles. At this time solely elastic strain is operating. When pressure increases, the structure becomes more compact with the particles dislocating (setting) that causes their partial destruction, change in form and internal porosity as well as an increase in the number of contact sites between particles and the size of intermolecular bonds. Due to the effect of the Van der Waals forces and electrostatic forces the particles merge. At the final stage of compaction, the entire system is compressed due to elastic or elastic-plastic factors. The chaotically located particles come into contact more often and a novel structure of the material becomes more stable. The board undergoes elastic expansion upon exiting from the compression zone, which results in a minimal decline in density and strength of the compacted product. The material may undergo fusion due to high pressure and increased temperature in the deformation zone, resulting in the crystallisation of molten membranes, adding to the strength of intermolecular bonds. The finished granulated product is derived by way of crushing the compacted material and isolating the desired fraction. The final density of the compacted material  $\rho_w$  determines the bulk density of the marketed fraction [14].

Pressure granulation is performed by means of roll compactors. The selection of a suitable device is determined by the properties of the material to be pressed. Below there is a description of silicate granulation installations that deploy pressing method as proposed by the three leading global manufacturers.

For the purpose of silicate granulation Hosokawa Bepex GmbH [15] recommends an installation comprising the following equipment and devices: starting material tank, intermediate container, two-shaft feeder, roll compactor, hammer mill, vibration screen, roll mill for oversized material crushing. In addition, the installation is fitted with a number of feeders/transporters conveying sodium silicate between operations as well as interoperation intermediate containers. The main elements feature a roll compactor and a hammer mill. The roll compactor (compactor® MS 36) is equipped with two rolls, each with the length of 120 mm and the diameter of 300 mm that may compact sodium silicate even under the pressure of 360 kN. The resulting "briquettes" of sodium silicate are then ground by

means of hammer mill rotating at the speed of 950 rpm. The ground material is sieved through vibration screens with the desired eyelet size. The isolated oversized grain upon grinding in the roll crusher is transferred onto the screens, while undersized material is redirected to the compactor.

The basic device of the installation offered by Powtec Maschinen und Engineering GmbH [16] is a roll compactor type RC 170. Sodium silicate is fed through a feed tank and feeding screw and pressed by means of two rolls with the length of 65 mm and a diameter of 170 mm under the compression force of 1.4-17.8 kN/cm of the roll running length. Sodium silicate, once compacted in the compactor, is then ground in a rotor mill integrated with the compactor. The mill comprises two rotors, each fitted with 6 the so-called „fingers” crushing the compacted silicate. Behind the second rotor there is a sieve that passes grain with a maximum size specified by the customer. The compactor-granulator system is controlled via a control panel, optionally equipped with a control screen as well as an interface allowing for the recording of 100 different settings of the compactor and granulator operation. Additional compactor features include undersized material separator along with a system for undersized material pneumatic transportation to a compactor feed tank. A grain classifier that isolates different fractions of the granulate in accordance with the customer's preferences is also available.

Freund Vector Corporation [17] offers a single-unit roll compactor TFC-1230 of a „combine” type that enables compacting, granulation, filtering through a screen as well as pneumatic transportation of silicate within a single device. Sodium silicate is conveyed to the compactor through a feed tank and feeding screw. The compactor is equipped with two rolls with the length of 120 mm and a diameter of 300 mm that may compact sodium silicate with a maximal compression force of 29 tons. For the purpose of granulation, a blade mill integrated with the compactor, fitted with 16 blades rotating with the velocity of 3600 rpm is recommended. This system of compactor-granulator may optionally feature a screen for undersized material, product or oversized material, integrated with the entire device with a system for the pneumatic transportation of respective fractions to:

- compactor feed tank (oversized and undersized material) via intermediate container
- material tank (product).

This type of installation also includes a control panel that facilitates control and the management of the compacting and crushing process.

It is of note that both the offer by Powtec and Freund Vector Corporation have the another advantage, apart from being fitted with a control panel: convenient size. The compact structure of the installation does not require large cubature of facilities.

The solid sodium silicates manufactured in Poland are derived by means of drying in a spray dryer. The products of the sole domestic manufacturer (Chemical Works – Zakłady Chemiczne - „Rudniki”) are characterized by grain size of mainly below 250 $\mu$ m and bulk density ranging from 100 to 700 kg/m<sup>3</sup>. In view of the growing demand for larger-grain silicates, the Inorganic Chemistry Division in Gliwice of the Fertilizer Research Institute preliminary test concerning sodium silicates granulation by pressing method were carried out. The tests were divided in two stages: the first involved laboratory tests, while the second – tests on industrial devices. The article herein reports on the first stage of tests. The laboratory tests conducted aimed at the determination of the compliance of analysed silicates to compaction as well as the establishment of compaction pressure value with respect to the module and initial bulk density [18]. The test were carried out with a view to selecting an industrial device that could yield sodium silicate granulates (of different modules) with bulk density within the range of 700-825 g/l and the mean grain size of 700 $\mu$ m.

## Laboratory tests

For the tests of granulation by pressing of solid sodium silicates, three types of solid silicates manufactured in the Chemical Works „Rudniki” divergent in terms of module, bulk weigh as well as particle size distribution (Tab. 2) were used. Products with the names 200 N 10; 265 N 50 as well as 340 N 60 were used to this end.

Table 2

Characteristics of studied sodium silicates

Sg.	Sample description	SiO <sub>2</sub> (%)	Na <sub>2</sub> O (%)	Molar module ±0,1	Bulk density ± 30 (g/l)	Loss on calcination at 6000C (%)
1	Sodium disilicate sample I	56.9	29	2.00	104.2	15
2	Sodium silicate sample II	59.6	22.6	2.72	533.8	17.8
3	Sodium silicate sample III	63.2	18.9	3.45	605.5	16.8

The sodium silicates used in the tests are the products of spray drying. Microscopic tests indicate that the grains are sphere-shaped as shown in the Figures below (Phot. 1).

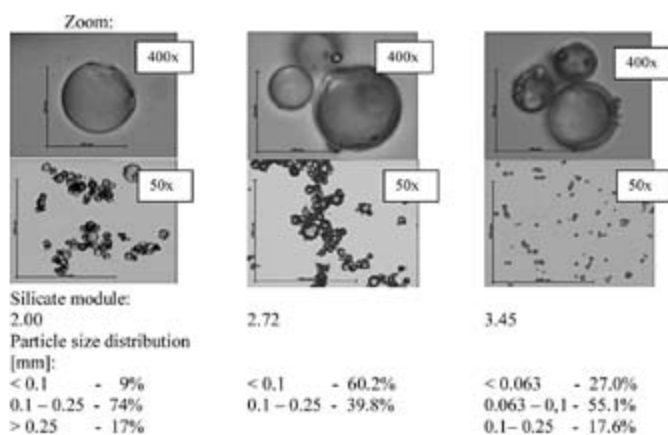


Photo 1. Microphotography of sodium silicates with modules: 2,0; 2,72; 3,45

Granulometric analysis of analysed silicates as well as micro-photography of the grains manifests considerable variation with respect to particle size distribution. The highest content of coarse fractions of > 0,1 mm occurs in the sodium silicate with a module of 2.0. As micro-photography indicates, this is the effect of a high conglomerate content consequent upon a higher hygroscopic properties and higher viscosity conducive to agglomerate formation. Agglomerates affect bulk density that approximately equals 100 kg/m<sup>3</sup>. Much finer grains were contained in two remaining silicates, i.e., that with a module of 2.72 and 3.45 as well as a higher bulk density. They amounted to 533.8 and 605.5 kg/m<sup>3</sup> respectively.

Laboratory tests of granulation by pressing method were conducted under a matrix press manufactured by AWPMA with a variable compression force, maximally of 10 t and a maximal compression speed of 20mm/min. The samples of the material characterized by particular mass were placed in a mould and compacted under the pressure ranging between 10-240 MPa (Phot.2).

During the densification of sodium silicates, the bulk density of the material gradually changed due to compression. At an adequately high compression, the material takes on the shape of a stable tablet. The compacts obtained were weighed as well as their height was measured in order to calculate the density of the material upon compaction, the so-called homogeneous density. This is the ratio of the tablet mass to

its volume and it is a variable dependent on the compression applied. The results obtained are a mean of at least five values. The stable tablets were fractured and shredded. The granulate obtained shows various shapes. They are filtered on a sieve in order to isolate a fraction with the required particle size distribution. In the fraction isolated the granulometric composition as well as bulk density were determined. The preparation of such a batch involved the compaction of a much larger amount of the material.

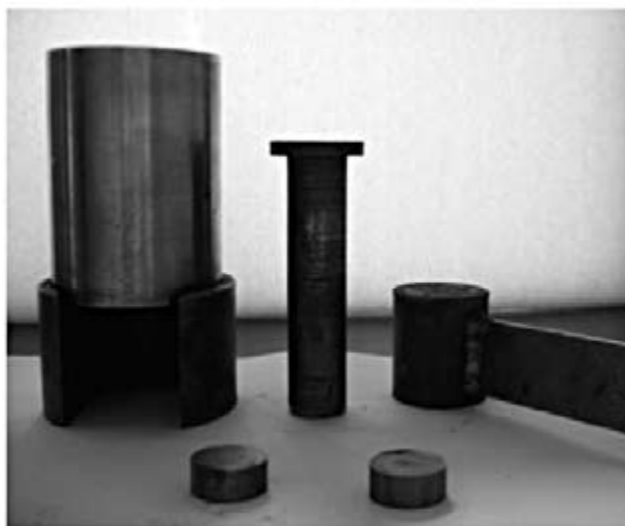


Photo 2. Matrix press and form of the compression

In order to establish the compaction pressure at which the structure obtained has the compactness allowing for the formation of a stable granulate, the material analysed was exposed to the forces between 1 t and 10 tons. The pressure of compaction was determined as the quotient of the compression force applied and the area applied. Throughout the densification of sodium silicates, a gradual change in the bulk density of material in natural state occurs under compression ( $\epsilon_s$ ) to the density  $\epsilon_w$  – the so-called homogeneous structure density. The variations in the density of the compacted structure are demonstrated in Figure 1.

Sodium silicate with a module of 2.0 formed a compact structure at a pressure beginning from 20 MPa, below which the tablets undergo crushing. The structure density increased up to a pressure of approximately 100 MPa reaching the value of 1,200 kg/m<sup>3</sup>. The increase in pressure of up to 240 MPa did not significantly affect density. The density of the remainder of silicates was much slower to raise.

A compact structure was obtained at a higher pressure – 40 MPa. Hence, density grew at a much lower rate. The density of a compact structure, comparable with the density of sodium silicate with a module of 2.0, was obtained not until the pressure of 240 MPa was achieved.

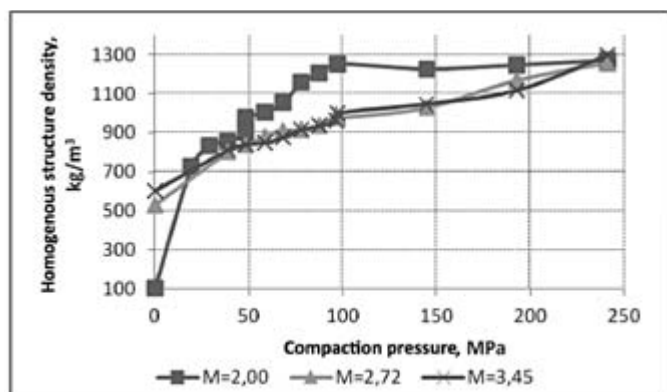


Fig. 1. Effect of compaction pressure on the density of sodium silicate structure

The measure of the outcome of compaction is the degree of densification ( $z$ ) determining the relationship between bulk density in the natural state ( $\zeta_0$ ) and the homogeneous structure density ( $\zeta_w$ ) with the form:

$$z = \zeta_w / \zeta_0$$

By determining the impact of compaction pressure on the densification degree of material it may be established at what pressure a compact structure may be achieved, that is to say, what pressure does not affect the inner arrangement of a homogenous structure. The effect of the compaction pressure applied on the densification degree for the analysed silicates is shown in Figure 2.

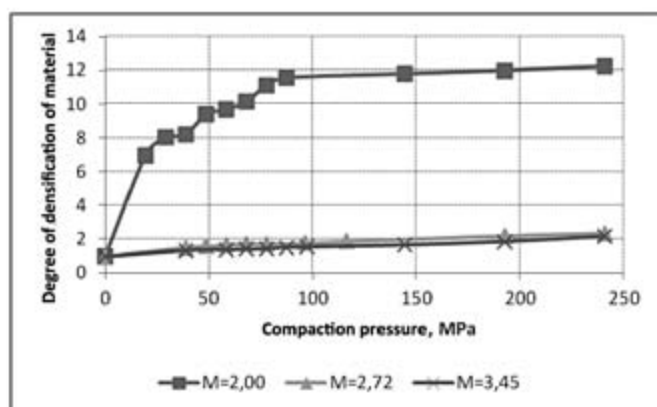


Fig. 2. Effect of compaction pressure on the degree of densification of sodium silicate

Compaction augmented the degree of densification of analysed silicates with respect to all pressure values applied. In case of a sodium silicate with a module of 2.0 a 12-fold rise in the densification degree may be seen at a compaction pressure of as much as 97.2 MPa. Under higher compaction pressure, the degree of densification underwent only a slight change. After crushing the tablets acquired at a pressure of 97.2 MPa, a granulate with the required strength was obtained. Hence, for silicates with a module of 2.0 it may be assumed that this is the optimal compaction pressure.

For the analysed sodium silicates with a higher silicate module, the degree of densification underwent change to a decisively lesser extent by contrast to silicate with a module of 2.0. However, the change in pressure showed an ascending tendency across the entire pressure range. Tablets with the required strength and a compact structure were obtained within the range of pressure of 150 MPa. At the compaction pressure of 144.5 MPa the densification degree of sodium silicate with a module of 2.72 amounted to 2.0, a slightly lower – 1.65 was obtained for the module of 3.45. Further compression increase within the range of 150 – 250 MPa caused a densification

degree increase by a mere 0.36 for the module of 2.72 and 0.5 for the module of 3.45. The data supplied indicate that for the sodium silicates with a module within the range of 2.72 – 3.45 it may be assumed that the pressure of 150 MPa is optimal and sufficient for acquiring compressed material possessing an adequately compact structure that warrant the required particle size distribution and strength of granulate upon crushing.

Granulation by press compaction features a fraction of material that fails to comply with the standards concerning granulometric distribution. It is undersized material remaining after the removal of the target product. In order to minimize loss, the said material is usually fed into the compactor after blending with the starting material. Thus recovered material may undesirably affect the granulation process. Hence, it was verified during tests how the addition of the compacted material affects the starting material subjected to compaction (simulation of material recovery in an industrial process). Shredded undersized material in the respective amounts of 10, 20, 30, and 50 % was added to the starting material and, subsequently, compacted under the optimal pressure determined as detailed above. The tests were limited to two modules: 2.0 and 2.72. The graph below (Fig. 3) depicts the effect of undersized material on the homogeneous structure density of silicates.

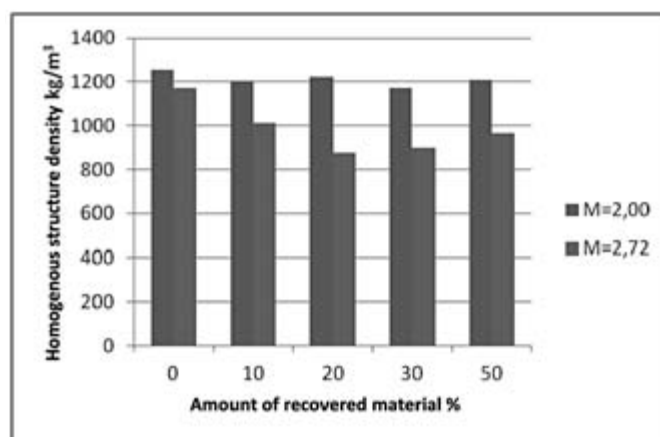


Fig. 3. Effect of amount of recycled fines on the density of uniform structure

Drawing from the results obtained, a reuse of compacted and fragmented particles of sodium silicate with a module of 2.0 fails to considerably affect homogeneous structure density, even at a recovered material content reaching 50%. For a silicate with a higher module, the addition of recovered material induces the decrease in the homogeneous structure density of up to approximately 20%. Such a marked decrease has a negative effect on the strength of the obtained tablets or the products of the crushing thereof. In this case, the maximal recovered material amount reaches 10%. The application of a higher content of recovered material results in an excessive decrease of the density of the compacted material.

The bulk density as well as granulometric distribution of the granulates of sodium silicates with a module of 2.0 and 2.72 obtained after crushing compacts were also determined. The results obtained have been detailed in Table 3.

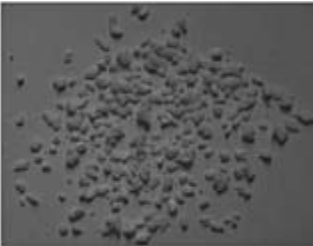
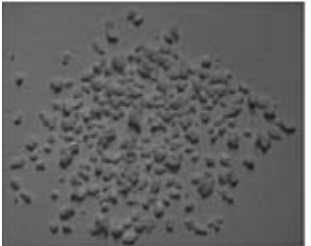
The laboratory tests conducted have allowed to obtain granulates with the bulk densities within the range of 0.62-0.68 g/cm<sup>3</sup> and the mean grain size of 0.8 - 0.25mm. The said parameters with respect to the sodium silicates analysed tended to be similar despite the considerable variations in the bulk densities of the materials applied. In the tests performed, the target bulk density of silicates, i.e. above 700 kg/m<sup>3</sup> was not reached. A rise in bulk density may be obtained through conditioning by way of moulding the granules to a more oval shape.

The application of pressing method for the purpose of the granulation of sodium silicates with silicate modules from 2.0 to 3.45

allows to obtain granulates with any grain size and the required bulk density, which allows the manufacturers of silicates to customise their products to the preferences of their clients. The results of the laboratory tests provide the grounds for the preliminary selection of the devices for industrial installation.

Table 3

## Characteristic of the obtained granules

Granulate – bulk density 0.6215 g/cm <sup>3</sup> Module – 2.00	Granulate – bulk density 0.6810 g/cm <sup>3</sup> Module – 2.72
	
> 2mm – 5.23%	> 2mm – 7.8%
2-1.6mm – 18.45%	2- 1.6mm – 11.7%
1.6-0.8 mm – 23.08%	1.6-0.8 mm – 35.4%
0.8-0.25 mm – 29.09%	0.8-0.25 mm – 13.7%
0.25-0.2mm – 11.83%	0.25-0.2mm – 9.6%
<0.2mm – 11.6%	<0.2mm – 21.8%

## Conclusions

The laboratory tests of sodium silicate granulation achieved by means of press compaction permitted to draw the following conclusions:

1. Sodium silicates with modules varying from 2.0 to 3.45 form compact structures under pressure. Within the pressure range of 10 ÷ 240 MPa all alkali silicates under investigation show an increase in the degree of densification.
2. The structure density for sodium silicate with a module of 2.0 and a low initial bulk density rose to the pressure of approximately 100 MPa, reaching the value of 1,200 kg/m<sup>3</sup>. The increase in pressure of up to 240 MPa has not significantly affected the density.
3. For sodium silicates with higher bulk densities and modules of 2.72-3.45, augmenting press compaction pressure resulted in a significantly slower rise in the degree of densification. The density of a compact structure, comparable with the density of sodium silicate with a module of 2.0, was not achieved until the setting of pressure to the value of 240 MPa.
4. The optimal compaction pressure with respect to sodium silicates with a module of 2 and a low bulk density - 100 kg/m<sup>3</sup> is 100 MPa, for silicates with a higher module and densities between 500-600 kg/m<sup>3</sup>, it amounts to approximately 150 MPa.
5. Upon simulation, no significant impact of recovered undersized material has been detected for the raw material subjected to compaction for sodium silicate with a module of 2.0, even at the recovered material amount reaching 50%. For silicates with a higher module, the addition of recovered material may lead to a drop in the density of homogeneous structure of up to approximately 20%. Undersized recovered material that is not bound to significantly decrease the structure density may reach a maximum of 10%.
6. The bulk densities of granules obtained in the laboratory tests show a slightly lower size as opposed to the standard. This parameter may be amended either on the industrial installation or through the application of the process of conditioning.
7. The results obtained allow to match industrial devices for sodium disilicate granulation as the main product with an simultaneous option of the manufacture of sodium silicates with different module and bulk densities.

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