

# *Coating of Sodium Aluminosilicate with Sodium Sulphate and Sodium Carbonate in V-Star Reactor*

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**Abstract** — The idea of the coating process of powder is to increase the stability and any damage resistances of the detergent. Therefore, the idea of project is to develop the coating process of powder in a continuous flow reactor through the cooling crystallisation. In this project, the coating process will be based on sodium aluminosilicate, which will confirm the concept of powder coating. Moreover, the ultimate goal of this project is to provide stabilising of powder particles (sodium aluminosilicate) with a solid inorganic coated material, wherein the coating material comprises a mixture of sodium sulphate or sodium carbonate with water.

**Keywords**—Powder coating, cooling crystallisation, layer size, continuous flow, V-star reactor

## I. INTRODUCTION

### 1.1 Importance of coating

The idea of the coating is to create a barrier against ambient moisture, to improve the storage stability of washing powder and decreases the possibility of microbial growth within the particle and it will make the surface of the particle more resistible to scratches.

Washing powder has ability to clean the surface area of an item where it is mostly referred to compounds that have sodium salt, which allows having a bleaching effect on soap.

In the most recent studies, the described methodology illustrates that the technical advances of the coating process are the main discussion for the powder coating industry. Consequently, the publication in 2010 (Dennis, 2010) introduced the new coating process technology for dull and semi-matte powder coating. The process represents about 15% of the total market of industrial finishing [10]. Nowadays, many types of powders are used in a wide range of products, such as detergents, cleaners, medicine, food additives and lots of other products. The other techniques based on the choice of cross-linking agents are possible, but not beneficial, as the usage of special matting agents is very expensive.

The coating process of sodium aluminosilicate (core) is the known process that occurs in a batch reactor) with sodium carbonate or sodium sulphate (coat), where the process takes time to load chemicals, mixing the particles, emptying, cleaning, etc. The process in a batch reactor occurs at a ratio of 1:2 (core to coat), which increases the conversion between the processing particles. Therefore, in this report, the research will

be based on illustration of the possibility of the coating process for cooling crystallisation using chemical compounds: sodium aluminosilicate as a core particle and sodium carbonate or sodium sulphate as a coat and minimisation of coat particles to make a ratio of 1:1.

For the coating of sodium aluminosilicate, the size of the surrounded layer (thickness) must be considerate, from the literature research the authors found that the thickness of the layer should vary within the range of 20  $\mu\text{m}$  – 106  $\mu\text{m}$  [7, 12]. The main role in success of the process belongs to a V - star that has very good control process ability. The coating itself plays a protective role for the particle (core) and has the ability of decorative finishes, as powder coating is available almost in the limitless range of colours and textures. The coated products are usually more resistant to corrosion as a result of chemical impact or any other weather condition [9]. The layer on the core provides a barrier against ambient moisture, improves the storage stability of zeolite, and decreases the possibility of microbial growth within the particle. In addition, the particle coating reduces the risk of wear issues (scratches, corrosion and so on) [6, 8]. A known procedure shows that the main component of the coating process material is a mixture of sodium carbonate or sodium sulphate in water, where the mixture should represent at least 60% of the coating material weight [14].

### 1.2 V - star reactor

A V - star reactor (VS) is a multi-stage flow reactor that can deliver very good mixing and plug flow over a wide variety of operating conditions where the design of the reactor employed a patented mixing technique [10]. Free moving agitators within each reactor stage (10 stages in overall) in the reactor promote mixing, when the reactor body is subjected to lateral shaking (at controlled speed). The advantage of the shaking is in generating intense mixing without the need for rotating tubes.

The flow in the VS does not run in the same way as the flow in a plug, and it is due to having series of mixing stages. However, enough mixing stages can deliver very good mixing which can be as good as in the lug flow (Figure 1).

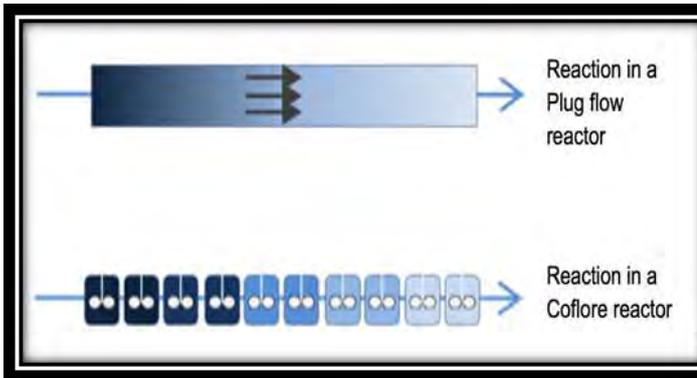


Fig. 1. The mixing ability of reactors V - star VS Plug Flow [15].

Using Figure 1, the effect of free moving agitators in flow mixing can be estimated, as the flow in a V - star reactor gets in the same conditions as that in a plug flow (shown by the change of colour). Moreover, the V - star reactor produces similar flow conditions to plug the flow reactor – similarity in conversion and yield that can be achieved by the new-modelled VS reactor.

## II. METHODS AND MATERIALS

### 2.1 Setting up the V - star reactor

The reactor is available in different sizes, different volumes, but it has the exact number of stages (tubes). Ten tubes with the same diameter are placed on the reactor, and each tube has an inlet and an exit that can be used to inject the material (feed and thermometer) and eject the product, so it can be done at any stage. Each tube is connected with smaller diameter tubes, and they are flexible to avoid any cracking. At every stage, a (tube) T-junction distributor can be added, from where the feed, thermometer, smaller tube and so on can be added [10]. The control of the V - star reactor can be done by using the control

panel, such as temperature determination at each stage, switch on/off, vibration of the reactor (at different speed rates) and some other control functions of the reactor. [10]

### 2.2 Experimental work

In a V - star reactor, different positions for the feed are available. So from the first tube (1-st stage) sodium sulphate or sodium carbonate, dissolved in water, will be fed to the reactor (reactor) at 40 °C, and from the inlet of stage 2 a slurry of sodium aluminosilicate in water will be added to the solution at 40 °C as well. Thus, the temperature of the mixture will be cooled down through the reactor to 5 °C (by each stage) and collected at the end of tube number 6 (stage 6). The product will be then separated from water by filtration. Table 1 represents the experimental work where the experiments are labelled as Ibragim Bamatov 1 – 7 (IB 1 to IB 5), and “Y” states for a particular component were presented in the crystallisation mixture. In the box labelled as IB 2 (IB 7), there were states of the complex structure product of IB 2 that was used as a core material (instead of sodium aluminosilicate) and in the empty box, there were states for “no particle component used in the coating process”.

Referring to the experimental table (Table 2.1), the it can be stated that IB1 and IB7 are represented as a complex structure where sodium sulphate and sodium carbonate are presented (the differences in conditions are given bellow in relative parts). IB 2 – IB 6 are illustrated as single component complex structures.

The experimental method for IB 1 – IB 7 is described below (IB 1 – IB 7). The complex structure of aluminosilicate and sodium sulphate (product from IB 2) was chosen as initial material, because it shows very good results under the microscope within the layer size and particles that have been coated.

TABLE I. EXPERIMENTAL ILLUSTRATION

Experiment number	Shaking Speed (rpm)	Na-Aluminosilicate	Na-carbonate	Na-sulphate
IB 1	2	Y	Y	Y
IB 2	2	Y		Y
IB 3	2	Y	Y	
IB 4	3	Y		Y
IB 5	3	Y	Y	
IB 6	2	Y	Y	Y
IB 7	2	IB2		Y

Only IB 2 and IB 3 will be illustrated as the entire experimental work is quite similar.

**IB 2.** In the first tank, 100 g of sodium sulphate was dissolved in 4 litres of deionised water. The solids were partially dissolved in deionised water at room temperature (24°C); however, during subsequent stirring and heating up, the solids dissolved completely at 34°C (fully dissolved). In the second tank, 100 g of sodium aluminosilicate was stirred with 4 litres of deionised water; however the solid particles did not

dissolve in water even with the further increase in the temperature (colour was milky). Both solutions were heated up to 40 °C to ensure if any particles in a mixture of sodium sulphate and sodium carbonate undissolved. For the coating process of aluminosilicate with sodium carbonate and sodium sulphate, 3 different temperature measurements at different stages of the reactor were taken: stage 2 - inlet, 4 - outlet and 6

– outlet, flow rate - 1.54 ml/s, shaking speed of the reactor - 2 rpm.

**IB 3.** The second experiment is similar to IB 2 and the only difference is instead of using 100 g of sodium sulphate, 100 g sodium carbonate was dissolved in 4 litres of water. The process conditions were the same as in IB 2. The flow rate - 1.54 ml/s, the shaking speed is 2 rpm.

### III. RESULTS

The total volume of the solution for the mixture (sodium aluminosilicate, sodium carbonate and sodium sulphate in presence of water) needs to be calculated. Therefore:

$$\text{Total mass of solution (g)} = \text{mass of solute} + \text{mass of solvent} \quad (1)$$

However, the mass of the solute (sodium aluminosilicate, sodium sulphate, sodium carbonate) was used in grams (100 g), so the mass of the solute in grams must be divided by the density of the solute to work out the total volume of the solute in ml. The mass of the solute (water, density is 1 g/cm<sup>3</sup>) was used with 4 litres (4000 ml).

There was the following assumption: the volume of the solid and the dissolved solute is the same.

The example is shown for sodium carbonate. The density of sodium carbonate is 2.54 g/cm<sup>3</sup> (density of sodium aluminosilicate is 0.48 g/cm<sup>3</sup>). Consequently, the density needs to be divided by the mass of the solute that was used (100 gr).

$$\text{Total volume of solute} = \frac{100}{2.54} = 39.4 \text{ cm}^3$$

The total volume of the solution can be calculated using (Eq.1); therefore, the total volume of the solution (sodium carbonate in water) will be:

$$\text{Total volume of the solution} = 39.4 + 4000 = 4039.4 \text{ ml}$$

The same method was used to calculate the total volume of sodium sulphate and sodium aluminosilicate. The summarising values are given in Table 3.1.

The mixture of solid (double component solutions) for IB 1 and IB 6 was found using the following method.

**For IB 1.** The volume of 100 g of solids (sodium carbonate and sodium sulphate):

$$\begin{aligned} \text{The total volume of the solution} &= 39.4 + 4000 + 37.6 \\ &= 4077 \text{ g} \end{aligned}$$

All the values are represented by using the same method as it was demonstrated in the example.

TABLE II. TOTAL VOLUME OF THE SOLUTION IN THE CRYSTALLISATION PROCESS.

<i>Chemical compound (in water)</i>	<i>Total volume of solution (ml)</i>
<b>Sodium aluminosilicate</b>	4208.3 (4 litres of water were used)
<b>Sodium carbonate</b>	4039.4
<b>Sodium sulphate</b>	4037.6
<b>Sodium sulphate and Sodium carbonate / IB 1</b>	4077
<b>Sodium sulphate and sodium carbonate / IB 6</b>	3037.9 (3 litres of water were used)

Referring to the calculated volume of solutions in the table (Table 3), the higher is density for the solid particle, the higher will be the total volume of solution. The error in weightings (experimentally) of solute and solvent is less than > 1%, which can be ignored.

Therefore, by adding volumes of the solutes for each experiment from IB 1 – IB 7, the overall volume that was processed through the reactor can be calculated by the following method:

For example, for IB 3.

For IB 3, the solution of sodium aluminosilicate in the 2-nd tank and sodium carbonate in the 1-st tank were used.

The total volume processed through the reactor = the 1-st tank volume of the solution + the 2-nd tank volume of the solution

$$\text{Total volume processed} = 4039.4 + 4208.3 = 8247.7 \text{ ml}$$

Referring to Table 3.2, the density of the coated structure product (IB 2) that was used in IB 7 can be found at a known flow rate and time.

The actual experimental time is 35.2 minutes, so a known flow rate, the total volume of the solutions can be calculated using the following procedure (Eq. 2):

$$\text{Total volume of the solutions} = \text{Time (s)} * \text{flow rate} \left( \frac{\text{ml}}{\text{s}} \right) \text{ (Eq. 2)}$$

Consequently,

$$\begin{aligned} \text{Total volume of the solutions} &= 35.2 * 60 * 1.54 \\ &= 3252.48 \text{ ml} \end{aligned}$$

$$\begin{aligned} \text{Total mass of the solvent} &= 3252.48 - 3000 \text{ (mass of solvent)} \\ &= 252.48 \text{ ml} \end{aligned}$$

Therefore, the density of the coated material (complex structure of sodium aluminosilicate and sodium sulphate IB 2) can be calculated:

$$\begin{aligned} \text{Density of coated sodium aluminosilicate} &= \frac{\text{mass of coating}}{\text{volume of coating}} = \frac{252.48}{100} \\ &= 2.52 \text{ g/cm}^3 \end{aligned}$$

TABLE III. THE CRYSTALLISATION PROCESS TIME FOR THE COATING PROCESS

<i>Experiment number</i>	<i>Process flow rate, ml/s</i>	<i>Theoretical time for the process crystallisation, min</i>	<i>Experimental time for the process crystallisation, min</i>
<b>IB 1</b>	1.54	44.8	46.2
<b>IB 2</b>	1.54	44.6	46.8
<b>IB 3</b>	1.54	44.6	45.7
<b>IB 4</b>	1.54	25.1	25.7
<b>IB 5</b>	1.54	25.1	26.7
<b>IB 6</b>	1.54	33.8	35.0
<b>IB 7</b>	1.54		35.2

Referring to the calculated density of the coated sodium, aluminosilicate (complex structure of sodium aluminosilicate and sodium sulphate) has bigger density (2.52 g/cm<sup>3</sup>) where the initial density of the sodium aluminosilicate was 0.48 cm<sup>3</sup>. Therefore, it can be stated that the particle (sodium aluminosilicate) was coated by sodium sulphate.

**2.3 SEM and EDX analysis.**

Scanning Electron Microscope (SEM) (Figures 2.3.1 – 2.3.2) and Energy – Dispersive X-ray spectroscopy (EDX) (Figures 2.3.3 – 2.3.4) were carried out for the complex structure of IB 2 and IB 3. Figures 2.3.1 – 2.3.2 (“LEMAS”) show the analysing testing of the specimens. “2.0kV” represents the energy that was put into the sample to read the images, 20.00k represents the number of times that the view of the specimen increased. SE(U) is the short name of the SEM machine that was used to analyse the specimens; 2.00 μm represents the scale of the image.

**IB 2.** By comparing this cubic structure with the literature SEM data of the sodium aluminosilicate (Figure 2.3.5), it can be stated that the cubic structure in Figure 2.3.1 is a core. By studying the surface area of the core, it was found that the core (sodium aluminosilicate) did not coated well and the coating process of the zeolite was not fully successfully. However, some aggregates can be noticed in Figure 2.3.1; therefore, to determine the composition of the complex structure of IB 2 and IB 3, an EDX analysis was processed (Figures 2.3.3 – 2.3.4). By using computer software ImageJ, the size for each of the partly coated particles (core) of IB 2 was recorded; therefore, the average size of the coated particle in Figure 2.3.1 was measured to be 1.263 μm. Thickness of the layer was measured and equaled to 0.192 μm. The complex structure of sodium aluminosilicate and sodium sulphate is at a speed rate of 2 rpm, flow rate - 1.54 ml/s)

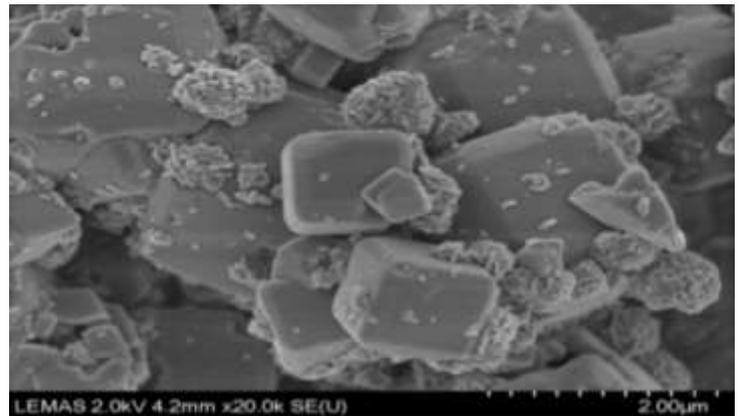


Fig. 2. SEM image of IB 2

**IB 3.** Referring to Figure 2.3.2, the coated particles (sodium carbonate) sticking on the sodium aluminosilicate can be determined, but the core particle is partly coated. If one compares the complex product of IB 3 to IB 2, it can be determined that sodium carbonate particles are coated much better than in sodium sulphate even if the coating is partly. Similarly, as in the analysing process of the IB 2 complex structure product, the average size of the complex structure of IB 3 also was measured by using the same software (ImageJ), where the average sizes were 1.43 μm and 0.213 for the core and coat, respectively. The complex structure of sodium aluminosilicate and sodium carbonate at a speed rate of 2 rpm, flow rate - 1.54 ml/s).

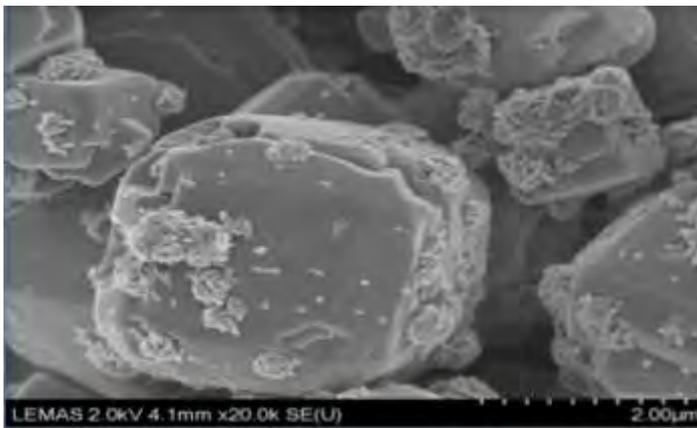


Fig. 3. SEM image of IB

2.4 EDX analysis.

**IB 2.** From the composition data given in Figure 2.3.3, it was found that sulphur is presented in the process structure of

IB 2, so that indicates that some of the part of the core was coated. However, the presence of sulphur represented as a very small number (1 wt %) and can be explained, as in EDX the X-ray heats only a part of the sample. It can not read an actual mass of the elements presented in the specimen; moreover the layer is not spread all around the core. So an X-ray could read from the place, where the particle was not coated. The most weight percentage in the complex product of IB 2 was recorded for oxygen (51.02%). The composition of aluminium, silicon and sodium was detected to be almost similar to each other: 15.07%, 14.05% and 14.49%, respectively. Iridium presented in the EDX graph (Figure 4.9) comes from the coating process of specimens that was carried out in the sample preparation part. The presence of carbon (3.48%) can be expected in the elementary composition of the complex product of IB 2, as it comes from the sticker that was stick to aluminium stub in the sample preparation process.

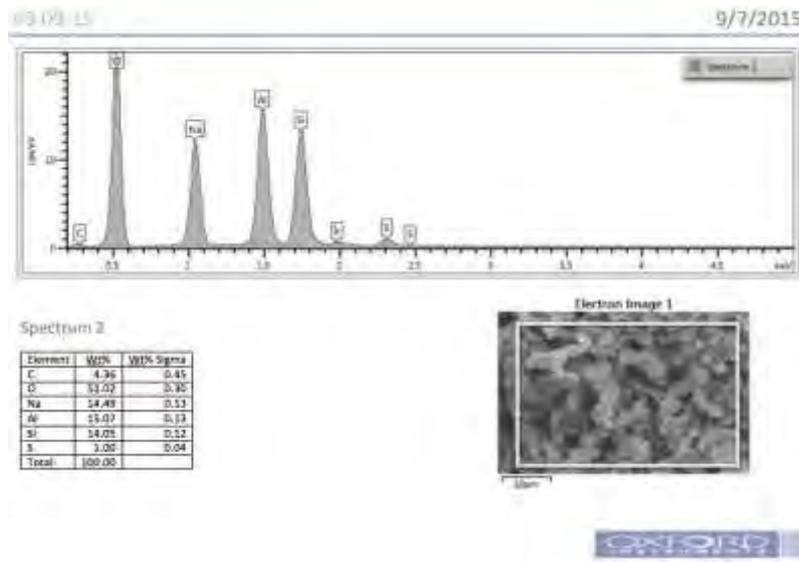


Fig. 4. Elementary analysis of IB 2.

**IB 3.** The amount of oxygen (Figure 4.11) is greatest in the elementary composition by weight percent – 52.31 %, where the contents of aluminium, silicon and sodium are represented as 15.12%, 13.85% and 14.47%, respectively. The presence of

carbon was measured as 4.50%, which is higher than in the elementary composition of IB 2 (3.48%). Therefore, it can be stated that the coating process of sodium aluminosilicate with sodium carbonate was considered partly.

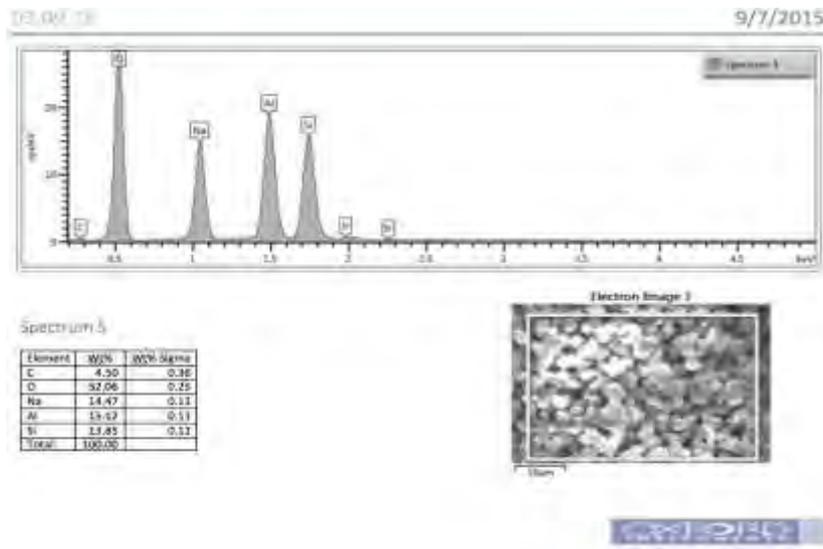


Fig. 5. Elementary analysis of IB 3.

#### IV. CONCLUSION

This project shows a possibility of the coating process in a continuous plug flow with a cooling crystallisation. Initial coating powder must be sodium percarbonate (washing powder) that must have higher washing power and better stability storage, as it is subjected to moisturising. The reason of using sodium aluminosilicate (insoluble in water) in this illustrative project was due to the high solubility of the sodium percarbonate in water. In other words, this project was an illustrational work of possibility of the coating process in a V - star reactor that could have a perfect control process. From the ESM analysis (Figures 2.3.1 and 2.3.2), the study of the surface area shows that the sodium aluminosilicate was coated only partly. From the EDX figures (Figures 2.3.3 and 2.3.4), the presence of the coat materials for a particular experiment can be detected: e.g. for IB 3 where sodium sulphate was used as a coat material, the presence of coat material (sodium sulphate) in EDX can be detected, even at low percentage. This project illustrates that the coating process, increases the density of the core particle, as it is shown (Results and Discussion) for the IB 2 complex structure, where the initial density of sodium aluminosilicate was  $0.48 \text{ g/cm}^3$ . But after the coating process, the density of the complex product was calculated within 10% of error -  $2.52 \text{ g/cm}^3$ . For more accurate determination of density, the hydrometer or pycnometer can be used.

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