Coating of powder particles by a continuous method of reaction by using V-star chemical reactor

Ibragim M Bamatov¹, Evgeniy V Rumyantsev² and Dzhabrail M Bamatov³

¹ FGBOU VO "Chechen State University", 32, Sheripova str., Grozny, 364024, Russia
² Ivanovo State Polytechnic University, 21, Sheremetjevsky ave., Ivanovo, Russia
³ University of Surrey Institute of Physics and Engineering in Medicine, Surrey, Great Britain

E-mail: Ibragim-1991@mail.ru, naturer@yandex.ru, Bamatov1993@mail.ru

Abstract. The aim of this project is the application of microparticles to a particle of powder which leads to increase the stability of the detergent. Moreover, the idea of the project is to develop a process for coating a powder in a reactor with a continuous flow of reactants through the cooling crystallization, in other words, to change the method of chemical flow process reaction. In this project, the coating process will be illustrated on sodium aluminosilicate (particle core), which will confirm the concept of the powder coating. Moreover, the ultimate goal of this project is to ensure the stabilization of the powder particles (sodium aluminosilicate) with an inorganic coating mixture, where coating material contains a mixture of sodium sulfate and sodium carbonate dissolved in water. In addition, the sub-goal of this project is to investigate the importance of the size of the layer of coated material that needs to be optimized to achieve a desired performance.

1. Introduction

The coating process of sodium aluminosilicate (core) is known to be in a batch reactor with sodium carbonate or sodium sulphate (coat). This process takes time to load chemicals, mixing the particles, emptying, cleaning and etc. The process in a batch reactor occurs at ratio of 1:2 (core to coat), within high conversion rate between the processing particles. Therefore, in this report, the research will be based on demonstrating the possibility of the coating process through a cooling crystallisation by using chemical materials: sodium aluminosilicate as a core particle and sodium carbonate or sodium sulphate as a coat and minimisation of coat particles to make ratio 1:1 (core to coat).

Sodium aluminosilicate (also known as sodium silicoaluminate) is a chemical compound in the form of powder and it is white crystalline powder with a chemical consistence of aluminium, silica, sodium and oxygen [3]. Mostly, it is used as a food additive (salt). In some of the processes the sodium aluminosilicate is used primarily as an anticaking agent that prevents bleaching in solids. In household products it can be found in air fresheners, cleaning detergents and sometimes sodium aluminosilicate is used as a water softener.

Washing powder or laundry detergent is a cleaning agent that is used for the cleaning laundry process and generally refers to a chemical mixture compound that includes sodium carbonate (sodium salt), which has the bleaching effect of soap. In recent studies, the described technique shows that the technical achievements of the coating process are the main discussion of the powder industry.
Detergents are composed of many elements that each play important roles in the laundry process:

- **Surfactants** – (known as surface active agents) are substances that minimises the surface tension of solvent (water), providing the ability to remove oil (dirt). Moreover, the surfactants break up dirt and keep it in the water solution to prevent re-deposition of the oil into the surface from which it just been detached from.
- **Oxidizing agents** (known as bleaching agents) act to lighten (bleach) the cleaning surface.
- **Enzymes** – are proteins that increases the speed of chemical reaction (catalysts). Certain catalyst has been used in detergents since 1960s to efficiently remove different types of dirt. Enzymes increase the power of the detergents at removing the dirt at lower temperature in washing.
- **Water softening agents** removes certain ions from water, making water softer. This process helps for detergents to bind with dirt. Chelators are a specific type of agent that deactivate the metallic ions in water, which makes water softer.
- **Polymers** – are used in detergents as antiredeposition agents. Moreover, they are also used in detergent structure to ensure compatibility with other components.

Mostly, the research will be based on experimental work, where the complex structure product will be tested under the microscope and SEM (surface area of the coated material). 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 and more transportable. The coating itself plays a protective role of the particle (core) and has the ability of decorative finishes, as powder coating is available in almost 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 [6]. The detailed theoretical study for powder coating have not been studied intensively, so all prediction difficulties in the type of zeolite can cause problems in the preparation of high quality core coating.

However, in the latest studies on zeolite coating (batch process) shows that one of the most important aspect in particle coating is the thickness of the layer that must be minimised as much as possible due to better control of the core over a layer. Depending on the pore structure of sodium aluminosilicate the crystal orientation on the surface is also significantly important. On the other hand, for some applications, the thicker layer can be preferable for optimal performance.

The average thickness layer of zeolite (sodium aluminosilicate in this case) varies ranging from 50-100 μm [5]. The research document that was published in 5 January 1998 shows one more way of coating process, coating of zeolite (sodium aluminosilicate) with Glass Fibers at temperature 110 °C. “Nichias Fibers 5100” was used as the starting material with powder. The results showed that the material has coated and a layer size of 20 μm [7].

![Figure 1. The mixing ability of the reactors Coflore VS Plug Flow [9].](image-url)
In 2010 [4] introduced the new coating process technology for dull and semi-matte powder coating. In generally, the coating process is known to be in a batch reactor, but the idea of this project is to transfer the process into a continuous reactor. To have a crystallisation process in a continuous reactor the process must be very well controlled, so the reactor that has high control proceeds is required for the transferring.

Therefore, a Coflore reactor will be illustrated as it has ability to control the process very well due to its stages.

Figure 2 illustrates that the flow reaction in a V-star is much more efficient than the reaction in a continuous tubular reactor.

Due to the presence of stages, V-star has the ability to control the temperature at each stage, thereby regulating (increasing or decreasing) the rate of a chemical reaction, which accordingly leads to an improvement in the conversion of the starting materials into product. Besides, an advantage of the V-star in front of other reactors is when accident occurs, the emergency stage can be turned off and the reaction can be resumed after the problem has been fixed.

The crystallisation process will occur due to the "cooling crystallisation" (temperature drop) in the last two stages.

The zeolite (sodium aluminosilicate) coating will be tested using various process conditions: rotation speed, temperatures, reagent flow rates, etc.

Experimental work (IBM1) will be conducted to demonstrate deposition of the layer (coat) over the powder (core) in a continuous reactor and the results will illustrate the concept of the possibility of the coating process over the core.

«V-star is a 6-stage reactor whose are parallelly located on a horizontal (left-right) moving platform. Each stage (pipe) on both sides has interchangeable tips, that have 2, 3 or more holes (inter-exit), which makes possible to make a connection between stages within each other by using hoses, i.e. At the end of stage 1, a hose is attached that goes to the beginning of stage 2, the end of stage 2 to the beginning of stage 3, etc. The sixth stage is the last stage and, accordingly, at this stage the process of the chemical reaction is completed. Also, a heat exchanger was installed at stages 5 and 6 for cooling (crystallisation) of the final product» [2].

2. Methodology

In a first tank the mixture of 100 g of sodium sulphate and 100 g of sodium carbonate were dissolved in 4 litres of deionised water. The solids have partially dissolved in deionised water at room temperature (24°C), however within further stirring and heating up, the solids have dissolved completely at 34°C (clearly dissolved). In a 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 further increasing the temperature up to 40 °C.

The moving speed of the V-star reactor platform was chosen to be at 2 (15 cm / s), the process of feeding the starting materials to the reactor was carried out using peristaltic pumps, with a speed set at 1.50 ml / s.

Three different temperature measurements at different stages of the reaction were measured. The V-star reactor has the ability to control the temperature, so there are 6 temperature control zones in the reactor, but in this case only 3 measurements are required, which were installed at stages: 2 (beginning of the stage), 4 (end of the stage) and 6 (end of the stage).

Raw materials were delivered (pumped) to the reactor at a temperature of 40 ° C, where the temperature in the reaction system was cooled down at each stage and, accordingly, the process of crystallisation of the final product began, where at the subsequent stage (stage 6) - white crystals in aqueous solution were obtained and filtered using vacuum filter.

3. Results

Experimentally obtained data of temperature change and experimental "features" are listed below (table 1):
Temperature at various stages of the IB1 reaction.
Temperature of solutions (zero stage) - 40 °C.

Table 1. Change of the temperature at each stage.

| Number of stage | Temperature °C. |
|-----------------|-----------------|
| 0               | 40.0            |
| 2               | 30.0            |
| 4               | 18.8            |
| 6               | 8.3             |

Based on the data obtained from experiment IB 1, the graph was illustrated (figure 1), where the temperature drops were recorded.

![Change of the temperature at each stage](image)

Figure 2. Temperature indicators of the experimental work of IBM1.

From the graphic data (figure 2), the fact of temperature drops steadily (angular coefficient is 5) and represents a straight line in the drawing, in other words at each stage, the temperature drops can be determined, and in this case from 40 °C to 8.3 °C, at 6 stages. It is can be estimated that there is a possibility that the temperature can be reduced even lower at the final stage, while reducing the feed rate of the reactants, thereby increasing the time of the technical process.

The yield of the final product amounted to 153.17 g

Product Percent = (Actual Revenue) / (Theoretical Income) =>

In order to develop a theoretical yield, solid masses dissolved in water must be subtracted from the total mass, therefore it is necessary to use solubility graphs. The solubility of sodium sulfate and sodium carbonate at 8.3 °C is approximately 8 g / g of water, so this mass will need to be subtracted from the total mass of the core and the shell particles.

Consequently, the yield percent will be; (153.17) / (200-8) * 100 ≈ 80%.

The loss of the product is due to the fact that the temperature at the exit (stage 6) of the product is high for 100% crystallization and, accordingly, some of it is still in dissolved form. Moreover, the loss of the actual product is due to the inaccurate process of vacuum filtration of the product.

The reason for the filtration was the disposal of water from the final product (the complex structure of sodium aluminosilicate with sodium carbonate / sodium sulfate)

The obtained sample was dried in an oven at atmospheric pressure (1.01 * 105 Pa) at 45 °C for 24 hours.

The analysis of the obtained product (sodium aluminosilicate complex with sodium sulfate and sodium carbonate) was tested under an optical microscope at a 100-times magnification with a resolution of 0.9.
A 100-times increase gives a real picture of a 1000-fold increased in the material under study. Therefore, the results shown in figure 2 must be multiplied by 100 to get an actual scale (size) of the material in microns.

Figure 3. Image from a microscope (complex structure of sodium aluminosilicate with sodium carbonate / sodium sulfate) at 100x magnification and 0.9 resolution.

From the figure 3, the size of the obtained material with a coating can be defined as 206 μm (2.06 per μm in figure 3), however, there are materials with larger sizes and this is estimated due to differences in the form of the coated material. The thickness of the applied layer can be defined as 20 μm (figure 3), mathematically, the actual thickness of the layer can be calculated using the Abbe equation (Equation 1 and 2)

\[ D = \frac{\lambda}{2NA} \]

where:

\( NA \) = numerical aperture
\( \lambda = \text{number of wavelength} - \text{visible light (400 μm)} \)

\( D \) = diffraction limit in μm

The numerical aperture can be defined by using the following equation (Equation 2)

\[ NA = n \times \sin\theta \]

where:

\( n \) = is the refraction index of the medium in which the objective is working, (as the analysing material was dry powder, the refraction index will be 1).
\( \theta \) = is the half angle of the maximum cone of light that can exit and enter the microscope objective. \( \theta \) can be calculated as 100 \* \( x_a \)

where 10 is the magnification multiple factor.
\[ x_A = 0.9 \text{ (given on microscope objective)} \]

Consequently, the numeric aperture will be:

\[ NA = 10 \times \sin 90 \]
\[ NA = 10 \times 1 \]
\[ NA = 10 \]

Therefore, the diffraction limit will be:

\[ D = \frac{400 \mu m}{2 \times 10} = 20 \mu m \]

The diffraction limit was calculated to be as 20 \( \mu m \) which is the same value (20 \( \mu m \)) that was determined by computer in the microscope image (figure 3).

4. Conclusion
To conclude, the fact must be stated that the data calculated (layer size) by computer and through formulas are equal to 20 \( \mu m \), which correspond to the data obtained on January 5, 1998, where the process of the reaction proceeded by a batch flow reactor. From this article it can be determined that the process of the chemical process has been changed and a new, modeled chemical reactor V-star) has succeeded in changing the technology from batch to continuous flow.

References
[1] Bamatov I M 2017 Development of the chemical reactor V-STAR for continuous flow reactions Journal of the Chechen State University 6(2) 205-7
[2] Carrera A L 2013 What is Sodium Aluminosilicate? Available from: http://www.livestrong.com/article/272868-what-is-sodium-aluminosilicate/
[3] Dennis D 2010 New technology for dull and semi-matte powder coatings (America: Novel Polyester Resin technology)
[4] Erdem-Şenatalar A, Tatlı M and Ürğen M 1999 Preparation of Zeolite Coatings by Direct Heating of the Substrates Microporous and Mesoporous Materials 32 331
[5] Fristad W E 2000 Epoxy Coatings for Automotive Corrosion Protection SAE International pp 617-24
[6] Okada H S 1998 In-Situ Coating of Zeolite Na-A on Al2O3-SiO2 Glass Fibers Journal of Porous Materials 5 163-8
[7] Leininger S 2007 PCT Patent No. 056746
[8] Sung S 2007 Laundry Detergents (New-York: Wiley)
[9] Willis W P 1960 Patent No. 3053419
[10] Xiang J D 2013 Structure and properties of Sodium Aluminosilicate Glasses from Molecular Dynamics Simulations J Chem Phys 139 4-7