Influence of the SiO₂/Al₂O₃ molar ratio on the specific properties of NaA zeolite

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Abstract

The aim of this research was to determine the influence of the SiO₂/Al₂O₃ molar ratio on the specific commercial properties of NaA zeolite subtypes as final market products (4A, 4A-AG and 4A-MS) under the real production and process conditions. The value of the SiO₂/Al₂O₃ molar ratio, so-called silicate module, was set as independently variable and the effect on the physical and chemical properties of each of the subtypes of NaA zeolites was examined. The paper investigates how the SiO₂/Al₂O₃ molar ratio affects specific properties of NaA powders, namely the ion exchange capacity, oil adsorption capacity and water adsorption capacity. Some previous theoretical and experimental studies have shown that the molar ratio plays a crucial role in the formation of these very similar but for final application different subtypes of NaA zeolite. The experimental part of this work was performed and tested in real production conditions, which can be considered as an advantage in relevance to the obtained results. Various analytical and instrumental testing methods were used for the analysis of the obtained powders, including SEM, XRD and PSD analyses.

Keywords Adsorption · Ion exchange capacity (IEC) · Silicate module · Zeolite

Introduction

Zeolite 4A (Na₁₂(AlO₂)₁₂•nH₂O), (Golbad et al. 2017) belongs at the group inorganic silicate materials, called tectosilicates (Baerlocher et al. 2007) characterized by a three-dimensional structure built of [TO₄] tetrahedrons where T, presents a silicon (Si), aluminium (Al), phosphorus (P) or rarely, some other atom depending on the type of material.

In the factory Alumina Ltd., three commercial types of NaA zeolite (4A, 4A-AG and 4A-MS) are produced. Zeolites 4A and 4A-AG are used as one of the basic components in the detergent industry as well as PVC stabilizers, as the ion exchange capacity (IEC) of 4A zeolite (Scott, Auerbach 2003) which represents the ability of 4A zeolite to perform substitution between Ca⁺ and Na⁺ ions. Zeolite 4A-AG is a zeolite with a slightly more porous structure and its main characteristic is oil adsorption capacity (OAC). The 4A-AG zeolite (AG-adsorption grade) was named after the high porous structure that indicates a higher specific surface area (SSA). Zeolite 4A-MS is used as an adsorbent, molecular sieves or stabilizer in various industries, and its main characteristic is water adsorption capacity (WAC).

According to the World zeolite market research, 72% of zeolite consumption is in the area of detergents, mostly NaA and Type P, (Flanigen 2010), 13% are used in the area of catalysts, 7% have applications as adsorbents. This market distribution emphasizes the importance of type 4A zeolites research (Janković 2014) and justifies the need for testing their physical and chemical properties.

In general, zeolite (Georgiev 2009) can be presented by the following expression:

\[
M_{x/n} \left( \frac{Al_2O_3}_x \cdot \frac{SiO_2}_y \right) \cdot mH_2O
\]  

(1)

where are \(M\)—alkaline or earth-alkaline positive ion, \(n\)—the valence of the positive ion, \(m\)—the number of water molecules per elemental cell unit, and \(x, y\)—present the number of tetrahedrons per elemental cell unit.

Zeolites can be presented as hydrated aluminium silicates of alkaline and earth-alkaline elements whose crystal...
structure is composed of \([\text{SiO}_4]^{4-}\) and \([\text{AlO}_4]^{5-}\) tetrahedra (Rozhkovskaya 2021) interconnected by oxygen atoms. NaA zeolite has a skeleton formed by connecting \(\beta\)-cages into a three-dimensional structure LTA (Loiola et al. 2012) (Xu et al. 2007) via double four-membered rings (D4R). As a type of micro-porous materials, zeolites have the following characteristics:

- A three-dimensional, regular, very well-defined and open network structure, with a series of channels, cavities, and very fine pores of molecular dimensions;
- High thermal stability;
- The possibility of active sites modelling (Corma 2003).

The process of zeolite production is based on the hydrothermal synthesis from the sodium-aluminate and sodium silicate solutions, as sources of the \(\text{SiO}_2\), \(\text{Al}_2\text{O}_3\) and \(\text{Na}_2\text{O}\). Synthetic zeolites are obtained through the process of nucleation (Subotić 2008) and spontaneous but controlled growth (Mohd Nazir et al. 2020) of crystals from a synthetic solution or from the so-called hydrogel, formed during the synthesis, resulting in micrometre-sized particles.

As mentioned, the application of zeolites in the process of mass production of detergents is based on their ion exchange and adsorption properties. For this application, zeolites need to have such a granulometric composition suitable for the smaller mean diameter of the particle size distribution (PSD) to avoid retention of the product, for example on the textile fibres.

In accordance with these requirements, it is necessary to create conditions for controlled crystallization (da Costa-Serra et al. 2020) of zeolite particles during the real production process to obtain specific quantitative and qualitative zeolite specifications (Table 1), including relevant silicate module \((M_{\text{Si}})\), i.e. \(\text{SiO}_2/\text{Al}_2\text{O}_3\) molar ratio (Zhang et al. 2013).

To reach the required zeolite specifications, certain steps in the dosing of the reactants during the synthesis and production must be followed (Fig. 1).

### Table 1: Chemical properties of NaA zeolite produced in “Alumina” Ltd

| Chemical properties of NaA zeolite | Typical content | Limits |
|----------------------------------|----------------|-------|
| Water content, 1 h 800 °C [%]    | 19             | 18–21 |
| \(\text{Al}_2\text{O}_3\) [%]    | 36             | 34–37 |
| \(\text{Na}_2\text{O}\) [%]     | 22             | 21–23 |
| \(\text{SiO}_2\) [%]            | 42             | 41–43 |
| Iron (Fe) ppm                   | 120            | 150 max |
| Colloidal iron ppm              | 30             | 40 max |

Zeolite synthesis is a multistep process involving different stages, such as dosing of reactants, crystallization, filtration, drying of the final product and storage (Fig. 1). The filtrate formed in the production process need to be concentrated up to a satisfactory level in order to be reused as such and molar composition of the mixture is important (Jain and Rimer 2020) and this step is done at evaporation unit of the plant.

### Experimental

#### Materials, methods and synthesis of zeolite

The process of zeolite production based on the hydrothermal synthesis use relevant sources of the basic components- \(\text{SiO}_2\), \(\text{Al}_2\text{O}_3\) and \(\text{Na}_2\text{O}\). The two main raw materials for synthesis of zeolite in Alumina Ltd. factory are Bayer liquor (sodium aluminate solution) and water glass (sodium silicate solution). The concentrations of input components in zeolite synthesis are: \(C_{(\text{Aluminate solution})}^\text{Na}_2\text{O} \approx 160 \text{ g/l} \), \(C_{(\text{Aluminate solution})}^\text{Al}_2\text{O}_3 \approx 170 \text{ g/l} \); \(C_{(\text{Filtrate})}^\text{Na}_2\text{O} \approx 40–50 \text{ g/l} \); \(C_{(\text{Water glass})}^\text{SiO}_2 \approx 380 \text{ g/l} \).

Zeolite synthesis is conducted in real process conditions but fixed temperature range \(t_1 = 76–88 \text{ °C} \), as it is known that
the temperature range affects (Menad 2016) the structure of the particles by defining their porosity. Also, synthesis of zeolites was performed during a continuous zeolite production process. Bayer liquor and water glass were synthesized and the silicate module \( \text{SiO}_2/\text{Al}_2\text{O}_3 \) was monitored in order to examine its effect on the specific properties of all three subtypes of NaA zeolite (Bayati et al. 2008). The Bayer liquor and the water glass are mixed into the reactor in a certain stoichiometric ratio. After dosing of input reactants, a sample of the synthesis slurry is taken and filtered in order to be analysed. Synthesis of 4A and 4A-AG zeolites is performed in 3 h., and for 4A-MS zeolite the synthesis of the reactants is performed in 6 h. After the completion of dosing the reactants, but before crystallization has started, the synthesis slurry is analysed. When it comes to 4A and 4A-AG zeolites, the control check of the concentration \( \text{Al}_2\text{O}_3 \) is performed. However, since the synthesis of 4A-MS lasts 6 h., an additional analysis of \( \text{Al}_2\text{O}_3 \) concentration at the certain

| Table 2 Basic properties of 4A, 4A-AG and 4A-MS subtypes of produced zeolites |
|---------------------------------------------------------------|
| Subtype of zeolite | Synthesis | \( d_{50} \) [\( \mu \text{m} \)] | Crystallinity [%] | Molar ratio \( \frac{\text{SiO}_2}{\text{Al}_2\text{O}_3}, M_{SS} \) | IEC [mg CaO/g] | OAC [ml/g] | WAC [%] |
|-------------------|----------|----------------|----------------|---------------------------------|-------------|-----------|---------|
| 4A                | S-1      | 5.50           | 97.2           | 1.80                            | 160         | 0.50      | 25.2    |
|                   | S-2      | 5.40           | 96.8           | 1.79                            | 163         | 0.47      | 24.0    |
|                   | S-3      | 5.38           | 97.3           | 1.85                            | 162         | 0.47      | 24.9    |
|                   | S-4      | 5.42           | 98.1           | 1.86                            | 164         | 0.45      | 24.0    |
|                   | S-5      | 4.60           | 97.6           | 1.81                            | 165         | 0.44      | 25.8    |
|                   | #AVRG    | 5.41           | 97.45          | 1.83                            | 163,50      | 0.46      | 24.00   |
|                   | #STDEV   | 0.371          | 0.484          | 0.0311                          | 1.923       | 0.023     | 0.782   |
| 4A-AG             | S-AG-1   | 5.12           | 96.2           | 1.91                            | 164         | 0.56      | 24.2    |
|                   | S-AG-2   | 5.30           | 94.7           | 1.94                            | 163         | 0.58      | 24.0    |
|                   | S-AG-3   | 5.80           | 94.0           | 1.89                            | 165         | 0.59      | 24.1    |
|                   | S-AG-4   | 6.12           | 94.8           | 2.01                            | 159         | 0.58      | 25.8    |
|                   | S-AG-5   | 5.90           | 98.0           | 2.04                            | 156         | 0.55      | 23.8    |
|                   | #AVRG    | 5.85           | 96.00          | 1.97                            | 160,50      | 0.57      | 23.95   |
|                   | #STDEV   | 0.421          | 1.589          | 0.064                           | 3.781       | 0.016     | 0.807   |
| 4A-MS             | S-MS-1   | 4.34           | 95.0           | 2.12                            | 158         | 0.35      | 25.5    |
|                   | S-MS-2   | 4.28           | 96.2           | 2.16                            | 156         | 0.34      | 25.7    |
|                   | S-MS-3   | 5.00           | 93.0           | 2.04                            | 159         | 0.35      | 26.6    |
|                   | S-MS-4   | 5.20           | 98.0           | 1.99                            | 157         | 0.35      | 25.3    |
|                   | S-MS-5   | 4.90           | 97.3           | 2.02                            | 157         | 0.37      | 26.8    |
|                   | #AVRG    | 4.74           | 97.10          | 2.08                            | 156,50      | 0.35      | 25.50   |
|                   | #STDEV   | 0.411          | 1.979          | 0.071                           | 1.140       | 0.011     | 0.676   |

Fig. 2 XRD crystallinity analysis of zeolites and referent CRM standard
time of the crystallization is done. For the purposes of this research, relevant analytical and instrumental methods of analysis are used. Standard analytical methods were used to determine content of Al$_2$O$_3$, SiO$_2$, ion exchange capacity, oil and water adsorption capacity at Alumina Ltd. Central Laboratory.

**Content of Al$_2$O$_3$**

Sample the aluminium solution is well homogenized, then added 5 ml into a normal 250 ml dish and added to the distilled water and mix well. From this solution, is taken 50 ml in a 500 ml bowl and added concentrated acetic acid (CH$_3$COOH). Then, is added 10 drops of ammonium acetate and 12 drops of PAN indicator and heated up and 8 drops of Cu-complexion. Titrated at 0.1 M K-III at boiling temperature, until constant yellow colour. Titration is performed until the boiling of colours returns.

**Content of SiO$_2$**

Mass of 1 g zeolite, is heated in a porcelain bowl with 40cm$^3$ concentrated HCl to wet salts. Soaked with 10 cm$^3$ concentrated HCl, added warm distilled water up to 2/3 volume of bowl and 14 cm$^3$ 1% gelatin and heated to soluble salts. The insoluble sediment SiO$_2$ is quantitatively transferred to a filter paper-white ribbon and washed with warm water to a negative reaction to chloride. In a pre-heated platinum pot, is burned the sediment on filter paper and heated for 1 h per 1000 °C and measured $g_1$. The rest is placed in a pot with a few drops H$_2$SO$_4$ and carefully add 10 cm$^3$ HF. Gently is heated to dry, then heated to 1000 °C 15 min., cooled and measured $g_2$. The difference between $g_1$ and $g_2$ relative to moisture content, is the content of SiO$_2$.

**Ion exchange capacity (IEC)**

Mass of 0.24–0.25 g zeolite is weighed, then transferred to a 250 ml beaker containing a magnetic stirring magnet and added 200 cm$^3$ of CaCl$_3$ solution. After stirring for 10 min., the solution was filtered off with suction through a B-3 filter. From this solution, 50 cm$^3$ of solution is taken and the concentration of Ca$^{2+}$ is determined by complexometric titration with 0.02 M ZnCl$_2$. In a 50 cm$^3$ sample, 10 cm$^3$ of pH = 10 buffer from a 25 cm$^3$ burette of 0.02 M EDTA solution was added. Titration is performed until a barely noticeable change in colour to red.

Fig. 3 Particle size distribution of 4A a, 4A-AG b and 4A-MS c zeolite (Cilas Liquid 1090)
Oil adsorption capacity (OAC)

Mass of 1 g of the zeolite sample and titrated with Dibutyl phthalate-DBP (IUPAC Dibutyl benzene-1,2-dicarboxylate). The titration is considered complete when all the zeolite powder is adsorbed by the solvent. The value of the solvent consumed (ml/g) is then read.

Water adsorption capacity (WAC)

A sample of 5 g of zeolite was weighed, the zeolite sample was annealed in a ceramic pot for 2 h. at 550 °C. After 2 h., the sample was removed and cooled in a desiccator without silica gel for 30 min. After cooling, the sample was quickly poured into a glass bottle and weighed. The sample is placed in the air chamber, in which the vegetables with the sample without the lid are placed. After 24 h., the value of adsorbed moisture W (%) is read.

The instrumental methods of analysis used in this research are Scanning electron microscopy (SEM), X-ray diffraction (XRD) and Particle size distribution analysis (PSD). Scanning electron microscopy (SEM) magnifies a surface area of the sample by using a high energy beam of electrons, the sample is vacuumed to ensure that the electron beam remains focused and does not interact with the particles in the air. SEM analysis was performed on a device “Mira 3XMU” Tescan at the University of Belgrade, Faculty of Technology and Metallurgy, Serbia. The degree of crystallinity (%) of the samples was determined by XRD analysis using the reference sample method. X-ray diffraction (XRD) and Particle size distribution (PSD) analyses were performed in the Central Laboratory of the Alumina Ltd., using “Endeavor D8” by Bruker, with a CoKα tube, λ = 1.78897 Å. The program package processes the spectral image area of the samples, i.e. determine the crystallinity degrees (%) of a sample relative to the zeolite standard CRM 8852 sample, i.e. highly crystalline NaA zeolite sample as reference (Liu et al. 2013). Particle size distribution analysis is performed on a “Cilas 1090 Liquid” device.

Results and discussion

For the purpose of this research five representative samples of each subtype were selected out of fifteen syntheses of zeolite production for review of the obtained results (Table 2). Also, these zeolite samples were subject of XRD analysis in order to investigate their crystal structure and crystallinity (Fig. 2).

XRD diffractogram peaks matching (Fig. 2) show that the molar ratio SiO₂/Al₂O₃ has no effect on the
crystallinity of any of the zeolite samples (typical representatives of 4A, 4A-AG and 4A-MS). Crystallinity degrees, examinations were conducted within the same conditions (Table 2). The crystallinity degrees of samples have values of Cryst ≥ 95% which is a satisfactory property level of final 4A products.

The size distribution of particles from the selected samples is tested on “Cilas 1090 Liquid” and results show more or less a regular distribution. The mean particle diameter (d50) ranges from 4.34 to 6.12 µm (Table 2). This indicates that there is no deviation in particle size distribution of the samples of zeolite which are representative samples of 4A, 4A-AG and 4A-MS zeolite.

Figure 3a illustrates, the diameter is less than 10 µm for 96.57% of the analysed samples of zeolite particles, or only 3.43% of particles in the sample are above 10 µm in diameter. Median particle diameter (d50) has an approximate value of 5.5 µm. In the case of 4A-AG zeolite, 99.88% of the analysed samples particles are less than 20 µm in diameter (Fig. 3b). Based on this, almost all the particles have a diameter of ≤ 20 µm. For 98.95% of analysed 4A-MS zeolite sample (Fig. 3c), particle size is less than 10 µm. This demonstrates that only 1.05% of particles are above 10 µm in diameter. Median particle diameter (d50) has an 4.74 µm. However, the particle size distribution analysed on the “Mira 3XMU” Tescan, shows more significant difference between these three zeolite samples particle size. SEM scanning also provides a more detailed, visual 3D presentation of zeolite particles (Figs. 4, 5 and 6).

SEM scans show the diameters of zeolite particles as well as surface structures and the shape of particles. The scans further demonstrate that the particles diameter of the detergent zeolites 4A (Fig. 4) and 4A-AG (Fig. 5) is slightly lower than the particles of zeolite for molecular sieves 4A-MS (Fig. 6). The average diameter (d50) according to the SEM analysis in 4A and 4A-AG is 1.8–2.10 µm while in the case of 4A-MS the diameter is about 3.3–4.0 µm.

Additionally, for detergent grade zeolites, especially 4A-AG, there is a higher particles porosity (Fig. 5), the particles are much smaller and have more specific surface contacts.

The influence of the molar ratio of SiO2/Al2O3 change confirms that molar ratio has influence on some specific properties of these NaA zeolite subtypes. Figure 7 illustrates the last five batches demonstrating that 4A-MS zeolite can be formed with lower molar ratios without undermining its qualitative properties.

But more specifically, Fig. 8 illustrates correlation between SiO2/Al2O3 molar ratio and IEC, WAC and OAC of NaA subtypes of zeolites. The value of ion exchange capacity (IEC) as the basic characteristic of 4A (Fig. 8a) and 4A-AG (Fig. 8b) zeolites ranges from 160–165 mg CaO/g. Besides IEC, oil adsorption capacity (OAC) is determined for 4A-AG zeolite; this parameter demonstrates the basic difference between 4A-AG and 4A zeolites.

For 4A-MS zeolite (Fig. 8c), the basic characteristic is water adsorption capacity (WAC). The value of WAC is around 25.3–26.8%, which is an extremely high level of water adsorption. The results show that the oil adsorption capacity for 4A-AG zeolite ranges from 0.55 to 0.59 ml/l, which is noticeably higher compared to the other two subtypes (OAC is somewhat lower for 4A, and significantly lower for 4A-MS, 0.35 ml/l).

However, individually observed, WAC has a satisfactory value only for 4A-MS zeolites. The oil adsorption capacity (OAC) has a satisfactory value only for 4A-AG zeolite, while
the ion exchange capacity (IEC) has the highest values for 4A zeolite, although these values do not differ much from 4A-AG zeolite. This is not surprising, since both subtypes of zeolites belong to detergent zeolites, with 4A-AG zeolite having a more porous structure and being used as an adsorbent.

**Conclusions**

This research work was performed during real process conditions and emphasizes the relevance of the obtained results and the practical approach.
Based on the results it can be concluded that optimal SiO₂/Al₂O₃ molar ratio (M₅₃) for commercial grade properties of final products of synthetic zeolite is 1.8 < M₅₃ < 1.9; 1.9 < M₅₃ < 2; 2 < M₅₃ < 2.12, for 4A zeolite, 4A-AG zeolite and 4A-MS zeolite, respectively. At these molar ratio values, commercial zeolites have appropriate and targeted specific quality properties WAC, IEC or OAC. For 4A-MS zeolite, it is the water adsorption capacity (WAC) with range from 25.3 to 26.8%, while for 4A detergent grade it is the ion exchange capacity (IEC) with range from 160 to 165 g/g. In the case of 4A-AG the main property is oil adsorption capacity (OAC) with range from 0.55 to 0.59 ml/l. On the other hand, oil adsorption capacity (OAC) is largest with SiO₂/Al₂O₃ module values ranging from 1.9 to 2.0. This implies that a lower silicate module (M₅₃) as well as higher temperature ranges of crystallization results in an irregular surface structure and a more porous zeolite particle (4A-AG).

Unlike 4A-MS, the other two subtypes of zeolites have a slightly more irregular surface, especially the 4A-AG zeolite, obtained under conditions bordering on the transition to another form of zeolite. Its porous surface allows higher adsorption potential of this type of zeolite, which is the reason why it is used also as a catalyst carrier.

It can be concluded, although it is not the only, that the molar ratio is a decisive factor in determining the specific properties of each of these researched subtypes of NaA zeolites.

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