METHODOLOGICAL CONSIDERATIONS IN INTERPRETING THE SODIUM CONTENT OF NaY ZEOLITE FROM SEM-EDS DATA

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Abstract. An additional computational analysis of previously obtained elemental composition data for hydrothermally synthesized NaY zeolite was performed. The Na/Al atomic ratio, normalization of SEM-EDS mass fractions, and limitations of directly comparing sodium content with an idealized anhydrous FAU model were examined. The values of 3.77 and 4.80 mmol/g were shown to refer to different mass bases and therefore cannot be interpreted as a quantitative measure of cation deficiency without additional information.

Keywords: NaY zeolite; SEM-EDS; Na/Al; mass fraction; cation balance; normalization; hydration; elemental analysis.

INTRODUCTION. Scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS) is used to investigate the morphology and local elemental composition of zeolite materials. In the sodium form of FAU-type zeolite Y, the Na and Al contents are particularly important because the negative

framework charge associated with tetrahedral aluminum is compensated by extra-framework cations.

However, SEM-EDS results are subject to methodological limitations. The analysis characterizes a specific region of the sample, and quantitative values depend on spectrum acquisition conditions, data processing, and reporting procedures. Furthermore, the mass of the analyzed material may include water and other components absent from the idealized anhydrous formula. Comparing experimental mass fractions with theoretical stoichiometric quantities therefore requires a consistent mass basis.

The aim of this study was to compare the Na/Al atomic ratio calculated from previously obtained SEM-EDS data for NaY with an idealized charge-compensation model and to demonstrate the limitations of directly comparing sodium contents expressed in mmol/g.

SOURCE DATA AND METHODOLOGY. Previously published SEM-EDS results for a hydrothermally synthesized NaY sample were analyzed. The reported mass fractions were O: 48.80±1.13%; Na: 8.66±0.59%; Al: 11.01±0.69%; and Si: 24.14±1.10%. The corresponding atomic fractions were 57.44, 7.10, 7.69, and 16.19 %, respectively. No new measurements were conducted in the present work.

The Na/Al ratio was determined from the elemental mass fractions and molar masses using $Na/Al=(w_{Na}/M_{Na})/(w_{Al}/M_{Al})$. Molar masses of $M_{Na}=22.990$ g/mol and $M_{Al}=26.982$ g/mol were used. The Na and Al atomic fractions directly reported by EDS were also used as an internal arithmetic check.

The sodium mass fraction was converted into an amount of substance per unit mass of the reported basis according to $n_{Na}=(w_{Na}/100)/M_{Na}\times1000$. The resulting value was compared with the theoretical value of 4.80 mmol/g calculated for an idealized anhydrous FAU cell with Si/Al≈2.10. Particular attention was paid to the difference between the two mass bases.

RESULTS AND DISCUSSION. The original mass fractions gave Na/Al=(8.66/22.990)/(11.01/26.982)=0.923. Recalculation from the reported atomic fractions gave the same result, 7.10/7.69=0.923. This agreement confirms the arithmetic consistency of the report, but both values originate from the same measurement and therefore do not constitute independent experimental confirmation.

In the idealized sodium form of FAU zeolite, each tetrahedral AlO_4^- center corresponds to one negative charge balanced by a monovalent cation. Thus, Na/Al=1 is expected for a model in which charge compensation is complete and provided exclusively by Na^+. The observed ratio of 0.923 is close to this reference value but does not by itself prove complete sodium exchange or establish the distribution of Na^+ ions among crystallographic sites.

A sodium mass fraction of 8.66 % corresponds to (8.66/22.990)×10=3.77 mmol/g. The theoretical Na^+ content of the idealized anhydrous formula $Na_{61,94}Al_{61,94}Si_{130,06}O_{384}$ is 4.80 mmol/g. Although the former is numerically lower than the latter, their difference cannot automatically be interpreted as a sodium-cation deficiency.

The reason is that the calculations use different mass bases. The value of 3.77 mmol/g is derived directly from the mass fraction reported by EDS, whereas 4.80 mmol/g refers to the mass of an idealized anhydrous structure. Without information on water content, the normalization of EDS results, and the overall chemical composition of the sample, a direct quantitative comparison is not justified. In particular, the difference cannot be presented as a demonstrated percentage of unoccupied cation sites.

The sum of the reported O, Na, Al, and Si mass fractions is 48.80+8.66+11.01+24.14=92.61%. The remaining 7.39% cannot be assigned unambiguously to a specific chemical component: the discrepancy may reflect the reporting procedure as well as the composition of the analyzed region. Assigning it to water or to a particular impurity without additional analysis would be unjustified.

For a more defensible preliminary interpretation, the Na/Al atomic ratio is preferable because it compares the elements linked to charge compensation in the idealized model. Nevertheless, this ratio provides no information about the distribution of Na^+ among crystallographic sites I, I′, II, and III. Cation locations in FAU are known to depend on the composition and state of the material [1, 2].

Further methods are required to test these interpretations. The total Na, Al, and Si contents could be quantified by ICP-OES or ICP-MS after appropriate sample preparation, while the water content should be assessed by thermogravimetric analysis. Measurements of ion-exchange capacity and structural investigations of cation positions address different questions and cannot be replaced by a single EDS spectrum.

Accordingly, SEM-EDS data are informative for preliminary assessment of atomic ratios, but their interpretation requires clear separation of directly measured quantities, arithmetic conversions, and parameters derived from an idealized model.

A further distinction is necessary between the deviation of Na/Al from unity and the difference between sodium contents expressed in mmol/g. The former compares dimensionless quantities calculated from the same EDS dataset. The latter requires both values to be referred to a comparable sample mass. Without such harmonization, even an arithmetically correct difference has no unambiguous physicochemical meaning. For this reason, the present study emphasizes atomic ratios rather than calculating a nominal percentage of Na^+ deficiency.

For further work on this sample, the complete original EDS report should be provided, including details of normalization, analyzed regions, and acquisition conditions. Additional measurements at several locations could characterize local compositional heterogeneity; however, no such measurements are claimed in the present study. Comparison with independent bulk chemical analysis would help establish how representative the local SEM-EDS estimate is of the overall material

composition. These procedures are important for making a defensible transition from elemental composition to ion-exchange behavior.

CONCLUSIONS

Reanalysis of SEM-EDS data for hydrothermally synthesized NaY yielded Na/Al=0.923, close to the idealized value of 1.00. The reported sodium mass fraction of 8.66 % corresponds to 3.77 mmol/g on the EDS reporting basis, whereas 4.80 mmol/g was calculated for the anhydrous FAU model. The difference between these mass bases precludes interpreting the discrepancy as direct evidence of cation deficiency. The reported mass fractions sum to 92.61%, further highlighting the need to clarify normalization and sample condition. A methodologically cautious approach based on atomic ratios and independent analytical measurements is proposed for evaluating the cation balance.

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