Structure Solving from Filtered Diffraction Data of LTA Zeolite and FEACAC

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Abstract

This study investigates the optimal threshold for filtering reflections in diffraction data from LTA zeolite and FEACAC samples to determine when further filtering becomes counterproductive. Using the KinDyn software, reflections with the worst fit due to dynamical effects were systematically removed, and the solvability of the structure was assessed using the removed data. The analysis reveals that for LTA zeolite, particularly for LTA 1, critical reflections are lost after filtering to a target completeness of 97%, with thicker samples like LTA 4 experiencing this threshold at 93.8%. SCXRD data for LTA indicated that structure solvability is achievable even at 98.8% completeness. For FEACAC samples, the critical point is reached at 91.8% completeness for merged data and 83.8% for Experiment 3. These findings highlight the delicate balance required in filtering and underscore the need for a cautious approach in crystallographic analysis to preserve valuable structural information.

Introduction

Our goal is to identify the point at which filtering removes reflections that are still useful for structure determination, thus establishing when further filtering becomes counterproductive. The study therefore investigates at which point the filtering removes reflections of such quality that they can still be used for structure solution. The key focus is to identify the threshold where reflections contain useful data and where further filtering is not justified. The analysis covers LTA zeolite samples with four different thicknesses and SCXRD data, along with FEACAC samples from three different experiments and their merged data. The metrics evaluated include the completeness of the removed and remaining data, the percentage of data removed during filtering, and the corresponding unrefined R1 values at the point where structure solving becomes possible.

Methodology

In this study, we used the KinDyn software to filter the reflections that fit worst to the model due to dynamical effects. The filtering process systematically removed these reflections step by step. After each filtering step, we attempted to solve the structure using the removed data to investigate whether the removed data still contained valuable information about the structure. If the removed data could still provide an accurate structure solution, it indicated that those reflections were useful. Once we identified the point where the structure could no longer be reliably solved with the removed data, we considered further filtering unjustified, as it would discard essential structural information.

LTA Zeolite Results Summary

For LTA 1, the point where we are removing useful reflections occurs already after filtering to a target completeness of 97%. This point is lower for the thicker samples, down to 93.8% for LTA 4. For the SCXRD data, the structure for the removed data is solvable already at 98.8% completeness. After this point, further filtering is not justified given that we can solve the structure for the removed data (i.e., it is valuable).

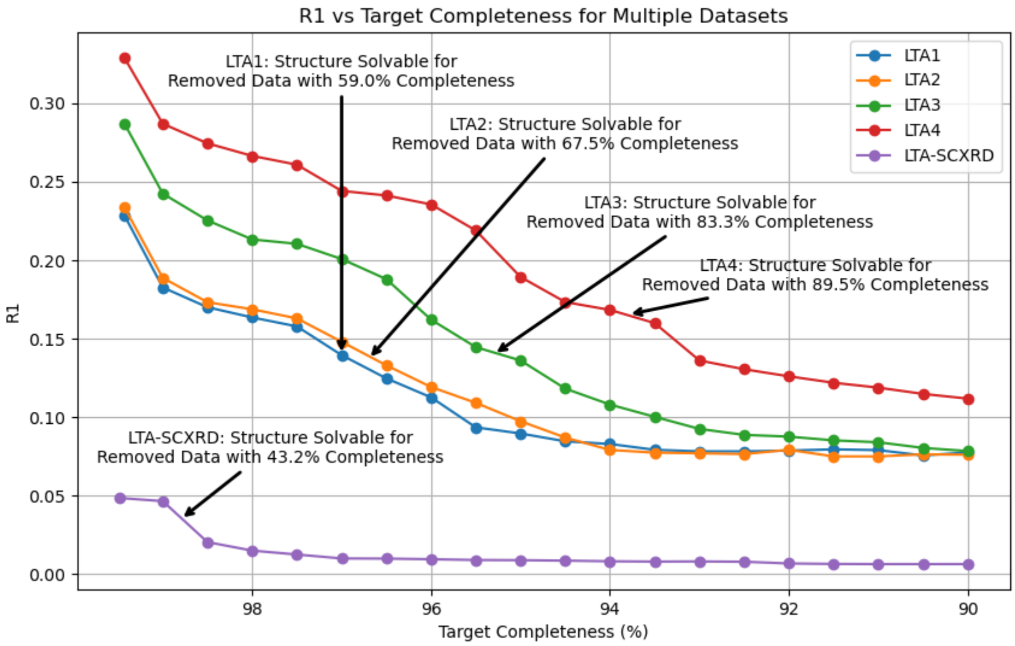

Figure 1: The graph shows the refinement R1 value plotted against the filtered target completeness for LTA samples with thicknesses 1 through 4. The inserted marks indicate the point at which the removed data becomes sufficient to solve the structure, leading to an accurate structure solution.

To enhance clarity, the findings for the LTA zeolite (including SCXRD data) are presented in two tables: one summarizing the characteristics of the removed data and another for the remaining data.

Removed Data for LTA Zeolite

Sample	**Removed Data Completeness (%)**	**Unrefined R1 Value (Removed Data) (%)**
LTA1	59.1	48
LTA2	67.5	45
LTA3	83.3	45
LTA4	89.5	44
SCXRD LTA	43.2	14.2

Table 1: Characteristics of removed data for LTA zeolite samples at the threshold where structure solving becomes possible.

Remaining Data for LTA Zeolite

Sample	**Remaining Data Completeness (%)**	**Unrefined R1 Value (Remaining Data) (%)**
LTA1	97	14
LTA2	96.7	14
LTA3	95.3	14
LTA4	93.8	15
SCXRD LTA	98.8	5.4

Table 2: Characteristics of remaining data for LTA zeolite samples after filtering.

Sample	**Total Data Remaining (%)**
LTA1	89
LTA2	87
LTA3	78
LTA4	72
SCXRD LTA	96

Table 3: Total data remaining after filtering for LTA zeolite samples.

FEACAC Results Summary

For FEACAC, the point at which useful reflections are removed occurs after filtering to a target completeness of approximately 91.8% for the merged data, and 83.8% for Experiment 3. Beyond this point, further filtering is not justified as the structure can be solved for the removed data, indicating that these reflections are valuable.

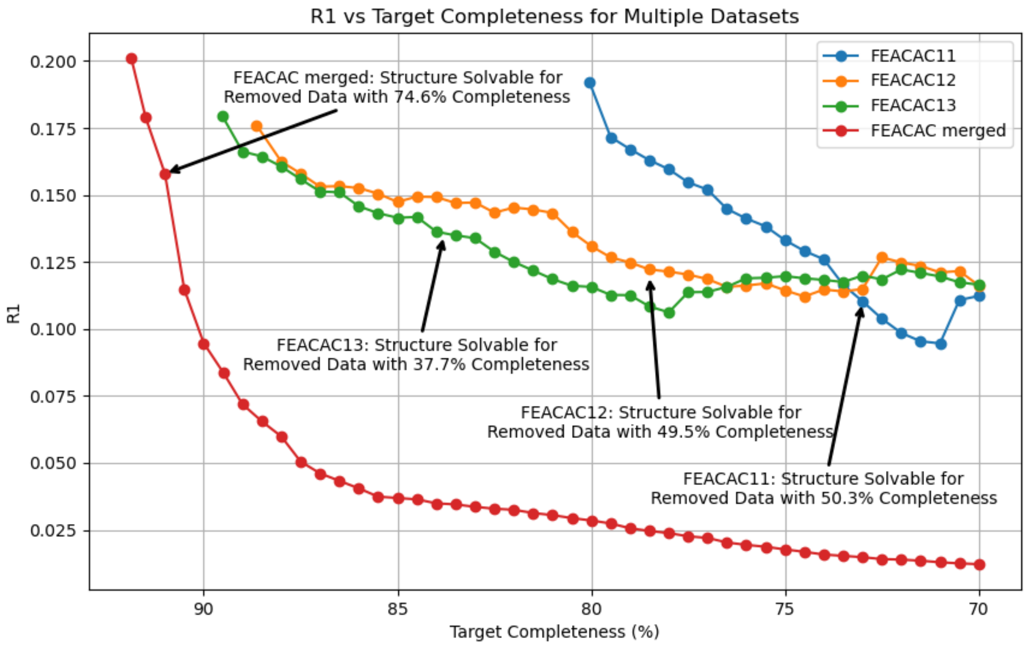

Figure 2: The graph presents the refinement R1 value as a function of filtered target completeness for the FEACAC samples, including individual experiments and the merged data. The inserted marks highlight the critical points where the removed data is capable of providing a reliable structure solution.

Similarly, the findings for the FEACAC samples are presented in two tables: one summarizing the characteristics of the removed data and another for the remaining data.

Removed Data for FEACAC

Sample	**Removed Data Completeness (%)**	**Unrefined R1 Value (Removed Data) (%)**
Experiment 1	50.3	38
Experiment 2	49.5	33
Experiment 3	37.7	29
Merged Data	72.2	42

Table 4: Characteristics of removed data for FEACAC samples at the threshold where structure solving becomes possible.

Remaining Data for FEACAC

Sample	**Remaining Data Completeness (%)**	**Unrefined R1 Value (Remaining Data) (%)**
Experiment 1	73	37
Experiment 2	78.5	36
Experiment 3	83.8	32
Merged Data	91.8	21

Table 5: Characteristics of remaining data for FEACAC samples after filtering.

Sample	**Total Data Remaining (%)**
Experiment 1	70
Experiment 2	72
Experiment 3	81
Merged Data	89

Table 6: Total data remaining after filtering for FEACAC samples.

Analysis Summary

The analysis revealed that for LTA zeolite samples, the point at which useful reflections are being removed occurs after filtering to a target completeness of 97% for LTA 1, with this threshold decreasing to 93.8% for the thicker sample, LTA 4. In the case of SCXRD data for LTA, the structure could still be solved with a high accuracy at 98.8% completeness, indicating that even minimal filtering could potentially remove valuable data.

For FEACAC samples, similar trends were observed, where the structure remained solvable with a target completeness of 91.8% for the merged data and 83.8% for Experiment 3. These results highlight the delicate balance required in filtering, as overly aggressive filtering risks discarding reflections that are crucial for accurate structure determination.

These findings underscore the importance of carefully selecting filtering thresholds in crystallographic studies, particularly when dealing with complex structures or varying sample thicknesses. The results suggest that overly aggressive filtering can lead to the loss of critical reflections necessary for accurate structure determination.

Conclusion

The results of this study suggest that while filtering can improve the fit to the model by removing reflections that poorly fit due to dynamical effects, there is a critical point beyond which further filtering is not beneficial and can be detrimental. This point varies depending on the sample thickness and the type of data used. For LTA zeolite samples, reflections removed after filtering to around 97% completeness are still valuable, and further filtering can lead to the loss of crucial structural information. The SCXRD data, which showed solvability even at 98.8% completeness, underscores the importance of careful filtering. Similarly, for FEACAC samples, filtering beyond the identified threshold risks losing data that is essential for an accurate structure solution. These findings emphasize the need for a cautious approach to filtering in structural analysis, ensuring that essential data is not discarded prematurely.

Our study highlights the necessity of a balanced approach to filtering in crystallography, where the removal of reflections must be carefully managed to preserve crucial structural information.

Further Remarks

Attempts were made to conduct a similar study for STW zeolite across four different thicknesses. However, due to the complexity of the molecule, structure solvability proved to be generally difficult. As a result, we have chosen to omit the few results that were achieved. In some cases, the structure solutions were not even correctly found from the raw, unfiltered data unless additional refinement steps were performed beforehand. This highlights the inherent challenges associated with analyzing more complex zeolites like STW, where standard filtering and structure-solving approaches may require further refinement or adaptation.