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Determining the Accessibility of Zeolite L Channels Using the Color Change of Thionine Dyes

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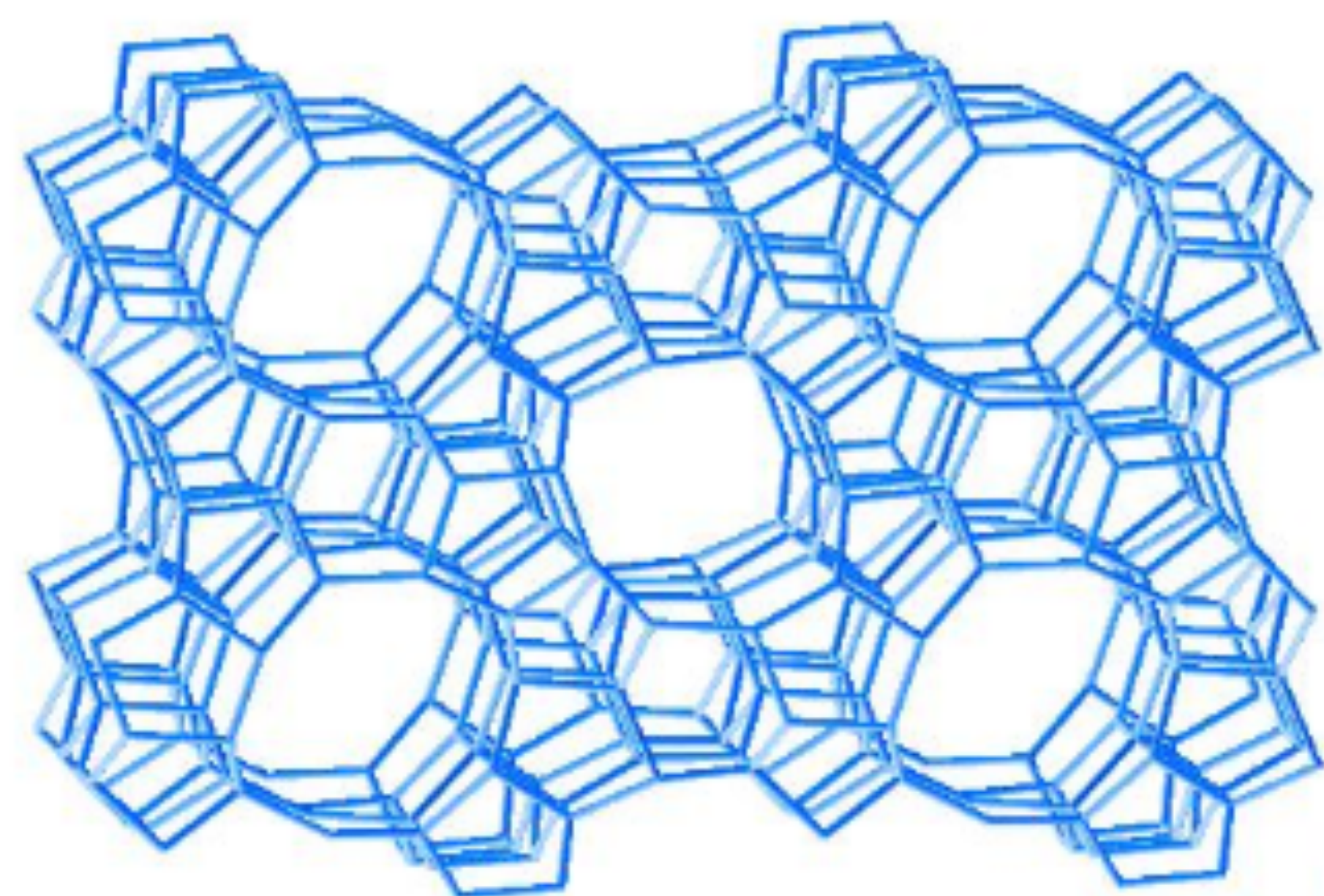
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Abstract

Host-guest materials are created when a guest dye molecule enters the channels within the zeolite host, which can be useful to understand how molecules act in a constrained environment. In the study of Zeolite L, it was previously unclear if Brooker's merocyanine was able to go into the channels of Zeolite L. The purpose of this research was to determine if thionine dye could be inserted into the zeolite crystal channels and how much dye would be adsorbed. Thionine was chosen as a guest dye to compare synthesized zeolite with known literature behavior. Zeolite L was synthesized in the lab, and the crystal structure was confirmed with XRD. Then, a zeolite suspension was added to a 2.5×10^{-5} M aqueous thionine solution and heated to boiling until the solution turned from purple to blue. The color change indicated that the dye molecules entered the channels of the zeolite, which lead to three conclusions. First, the synthesized Zeolite L channels could incorporate dye molecules, which followed the literature example. This project also proved to be a faster way of confirming that the crystal was correct versus examining the crystal in the XRD. Lastly, the amount of dye that entered the zeolite channels was measured and compared to previous studies.

Introduction

- Host-guest chemistry is composed of materials where two or more molecules are held together by structural relationships.
- One application of host-guest chemistry is drug delivery. Other studies have shown that delivering a drug using a host-guest delivery system would allow the drug to be specifically targeted to a tissue area with controlled release rates.
- A zeolite is a solid with a relatively open, three-dimensional crystal structure built with tube-like channels inside of it. Zeolites can act as hosts for supramolecular organization of molecules, ions, complexes, and clusters.
- Zeolites can act as the host when a guest dye molecule enters the channels and interacts with the zeolite. Studying this interaction allows for the understanding of how molecules behave in a constrained environment.



Zeolite L Channel Structure³

- In previous experiments in our lab, it was unclear whether Brooker's merocyanine (another guest dye molecule) was able to go into the channels of Zeolite L. Research suggested that Brooker's merocyanine was on the surface of the zeolite rather than inside the zeolite channels.
- Previous research by Calzaferri et al. used a thionine dye color test to prove the dye was inserted into the zeolite. We followed their procedure to confirm that our crystals were able to insert the dye inside of the channels, since there had been previous problems with Brooker's merocyanine in zeolite L. In addition, the thionine color test could be a faster way to examine whether we had synthesized the correct crystal, rather than using the XRD every time we synthesized crystals.

Methods

- Zeolite crystals were synthesized following the zeolite Linde type L recipe and scaled down by a factor of 10. After synthesizing, the gelled crystal solution was put in a pressurized vessel and heated at 175°C for 72 hours. Crystals were rinsed with water and dried in an oven for 24 hours at 100 °C.
- After heating, XRD was used to confirm the synthesis of zeolite L and checked for the formation of other zeolites.
- For the color test, a thionine solution and a zeolite suspension was made. The zeolite suspension consisted of 0.2 g zeolite powder into a vial with 10mL of EZ pure water which was then mixed well. 4 mL of 2.5×10^{-5} M of thionine mixed with 2 mL of the zeolite suspension and heated until boiling. The solution color changed from purple to blue once the vial had started to boil. After boiling for 1 minute, the vial cooled, and the blue color remained. Then, the reference thionine and zeolite-thionine sample were measured in the UV/Vis spectrometer to quantify the amount of dye adsorbed to the zeolite.

Results

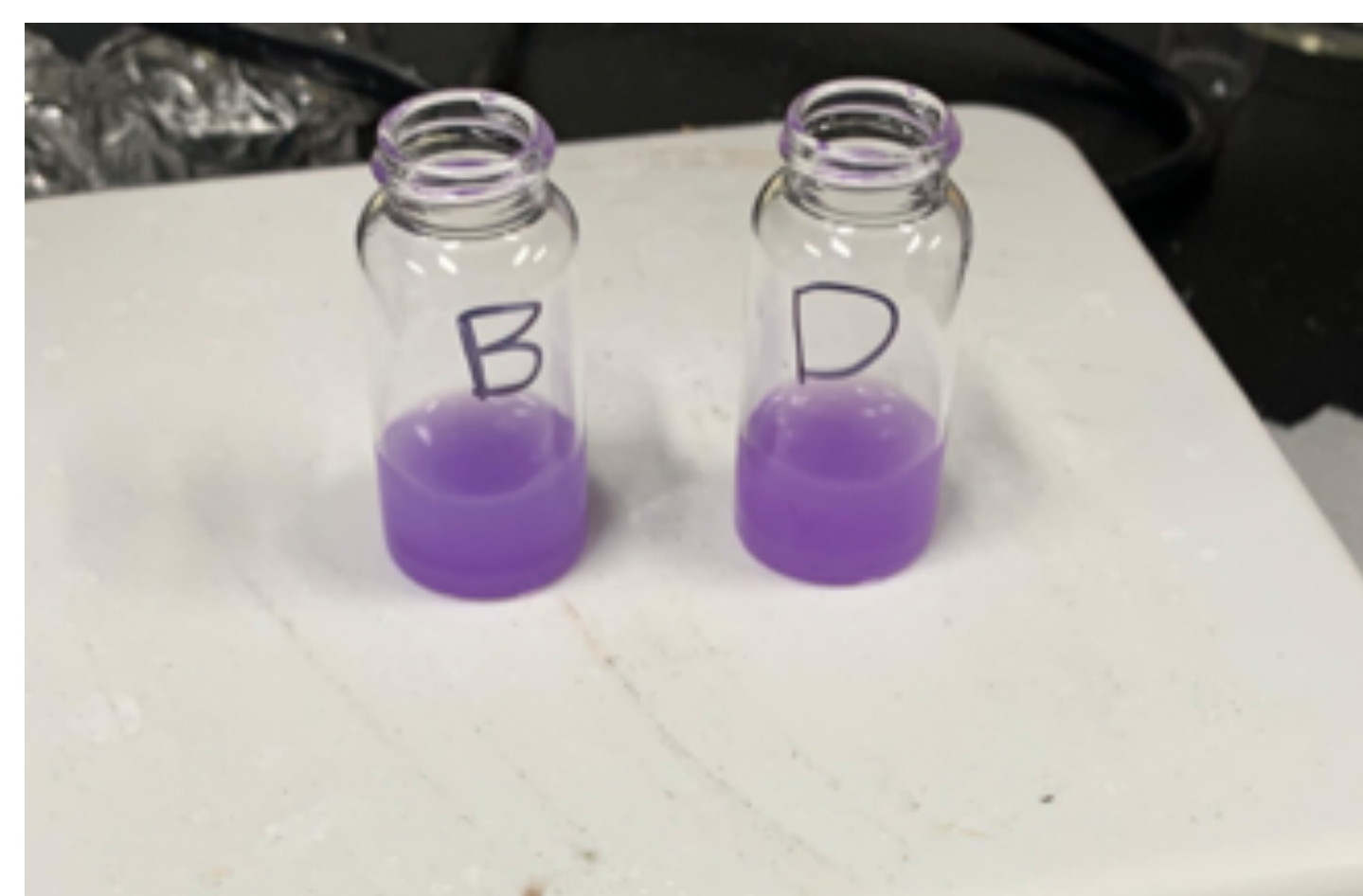


Figure 1. Zeolite-thionine suspension before boiling and color change.



Figure 2. Zeolite-thionine suspension after boiling and cooling.



Figure 3. Bad batch of zeolite in a Zeolite-thionine suspension after boiling and cooling.

- Figure 1 shows the zeolite-thionine suspension before boiling, while Figure 2 shows the color change after boiling and cooling.
- Figure 3 is an example of a batch of zeolite that was not synthesized correctly and did not change colors.
- With an incorrect synthesis, the suspension remains purple and does not turn to blue after cooling.
- Dye absorption was calculated in molecules of dye per grams of zeolite in the following concentrations:

Table 1. Dye adsorption data.

Dye Solution Concentration (M)	Dye molecules/g of zeolite
2.99×10^{-4} M	1.7×10^{19}
3.0×10^{-3} M	2.0×10^{20}

- The values in Table 1 show that we have not yet reached maximum thionine loading yet. However, the magnitude of these numbers are similar to the previous study of Brooker's merocyanine.

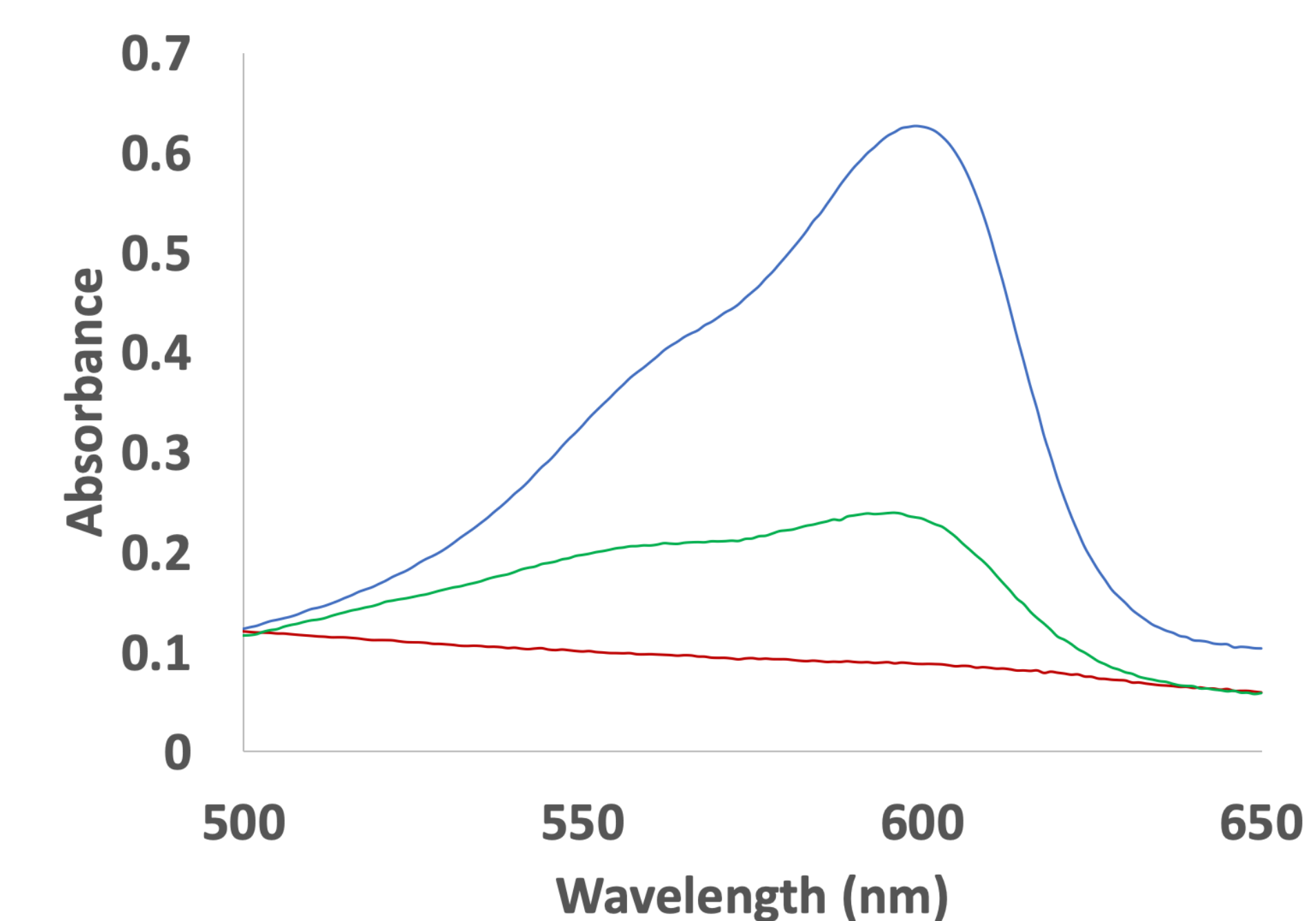


Figure 4. The UV/Vis data of 1.19×10^{-5} M of thionine reference in blue, 3.0×10^{-3} M thionine-zeolite sample in green, and 1.19×10^{-5} M thionine-zeolite sample in red.

- The UV/Vis data of 1.19×10^{-5} M thionine solution is shown in blue, and the solution decreased in concentration when the zeolite was added (in red). A higher concentration of 3.0×10^{-3} M thionine with zeolite added (in green) was used to quantify the amount of dye adsorbed
- This was done with different data sets since we had instrument difficulty in measuring both the thionine reference and the thionine-zeolite solution at the same concentration.

Conclusion

- The results of this research shows that the thionine color test proved our zeolite crystal channels were accessible to dye molecules.
- We also proved that the color test could be used to confirm the correct zeolite was synthesized rather than using XRD.
- The color change corresponds to thionine aggregates immediately forming at the outer surface of the zeolite crystals. When heating a thionine-zeolite suspension to boiling, molecules have enough energy to break up the aggregates and enter the channels, resulting in a color change that remains when cooled. This indicates that the dye molecules have entered the channels of the zeolite.²
- This test is effective in confirming correct synthesis of zeolite since a color change could not occur if a different crystal structure was synthesized.

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References

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