

Conclusion

Within sol-gel reactions knowledge about the degree of condensation is essential to create products with designed properties. With the combination of ¹H-NMR and ATR-IR it is possible to online measure the progress of hydrolysis and condensation.

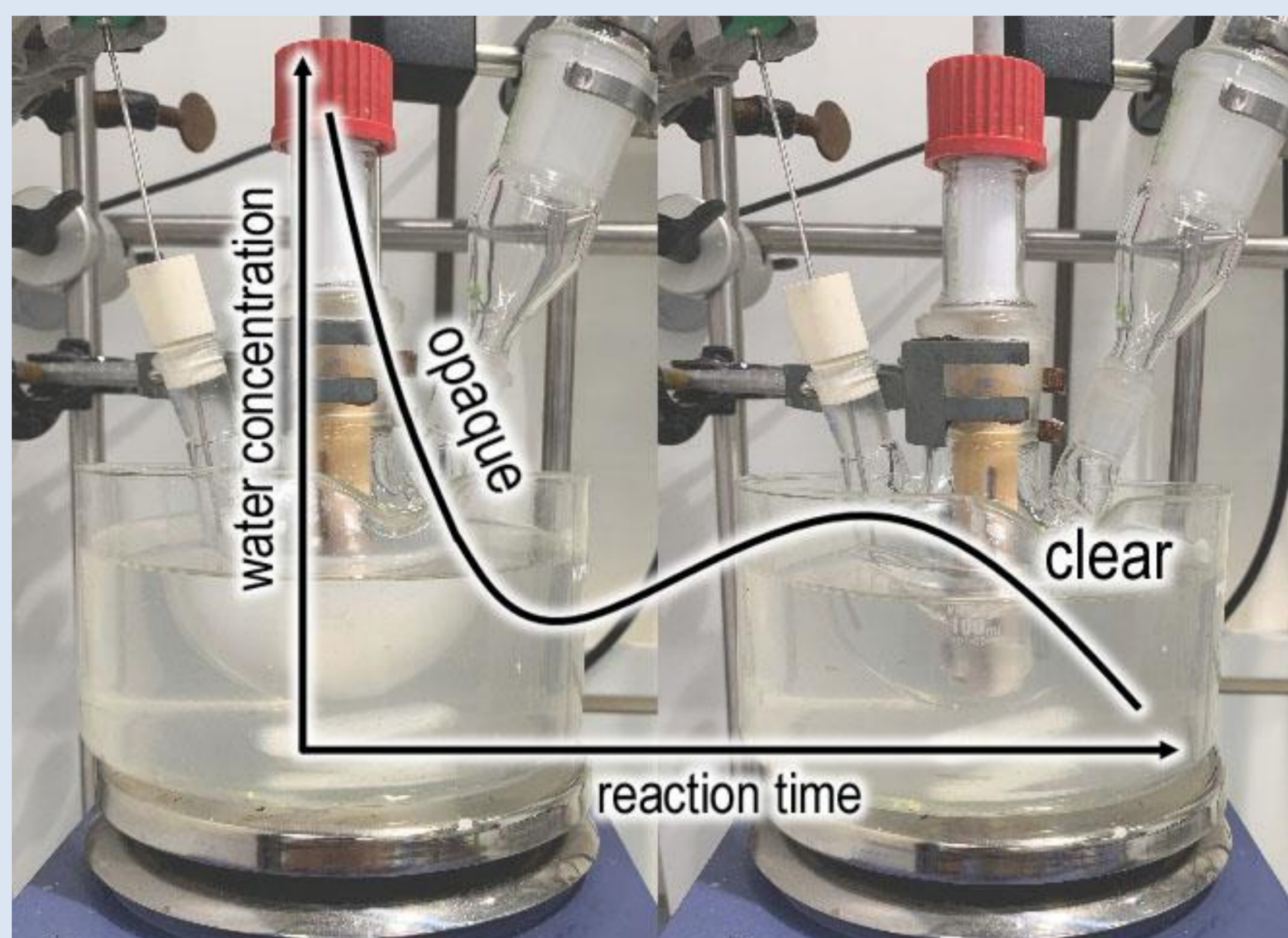


Figure 1: Schematic progress of hydrolysis and condensation for the sol-gel reaction. Opaque reaction solution turns clear after sufficient hydrolysis followed by controlled condensation.

Introduction

Coatings for specialty applications require flexibility, high temperature-, and abrasion resistance. One approach for the production of such coatings is the sol-gel process based on hydrolysis and condensation. Figure 2 shows the principle reactions for tetraethoxysilane (TEOS). These two steps are not separable from each other, however with tailored conditions (temperature, pH-value, amount of water, catalyst) it is possible to control the reaction towards hydrolysis or condensation. Herein, the progress measurements of hydrolysis and condensation with ATR-IR and ¹H-NMR are shown.

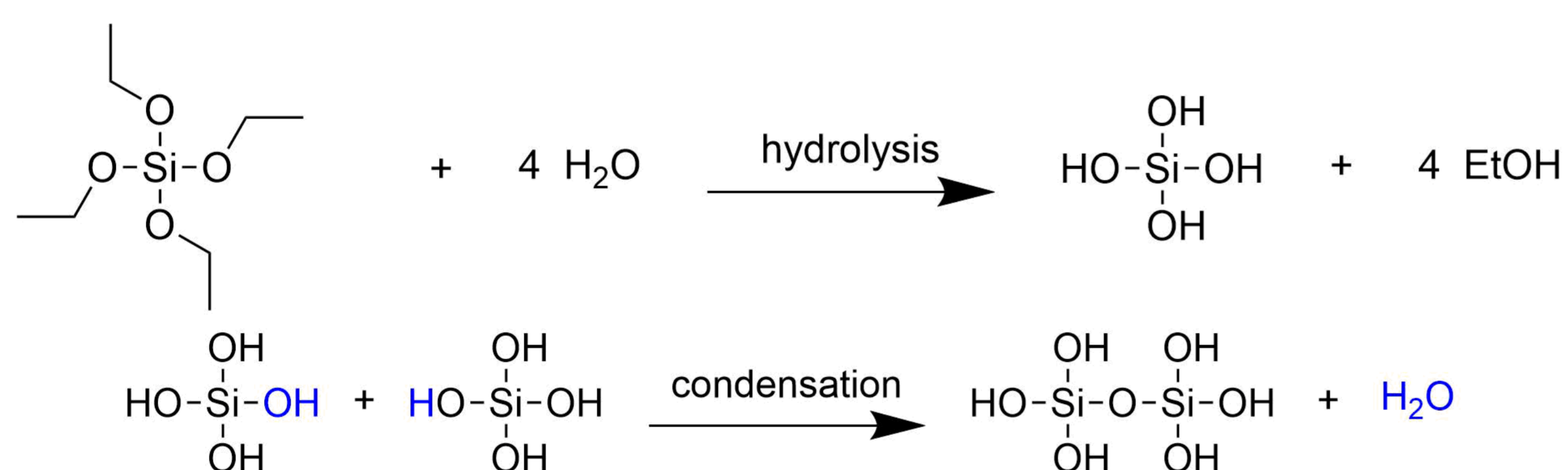


Figure 2: Hydrolysis and condensation demonstrated on the example of tetraethoxysilane (TEOS).

Results and Discussion

Hydrolysis can be favored with a reaction temperature of 60 °C and succinic acid as catalyst. During hydrolysis water is consumed and the solution turns from opaque to clear, which can be seen in Figure 1. Thereby the water concentration decreases. In the next step the solution is heated up to 150 °C to evaporate water and alcohols. In this step condensation increases which releases water.

Figure 3 shows ATR-IR spectra of the silane mixture (red), the reaction solution after turning clear (blue) and the final product (green). In the highlighted image section at about 1100 cm⁻¹ a decrease in concentration of ethoxy groups (representing educts) is observed and attributed to hydrolysis. In the region from 3000 – 3600 cm⁻¹ the concentration of all OH-groups (water as well as silanols and alcohols) can be estimated.

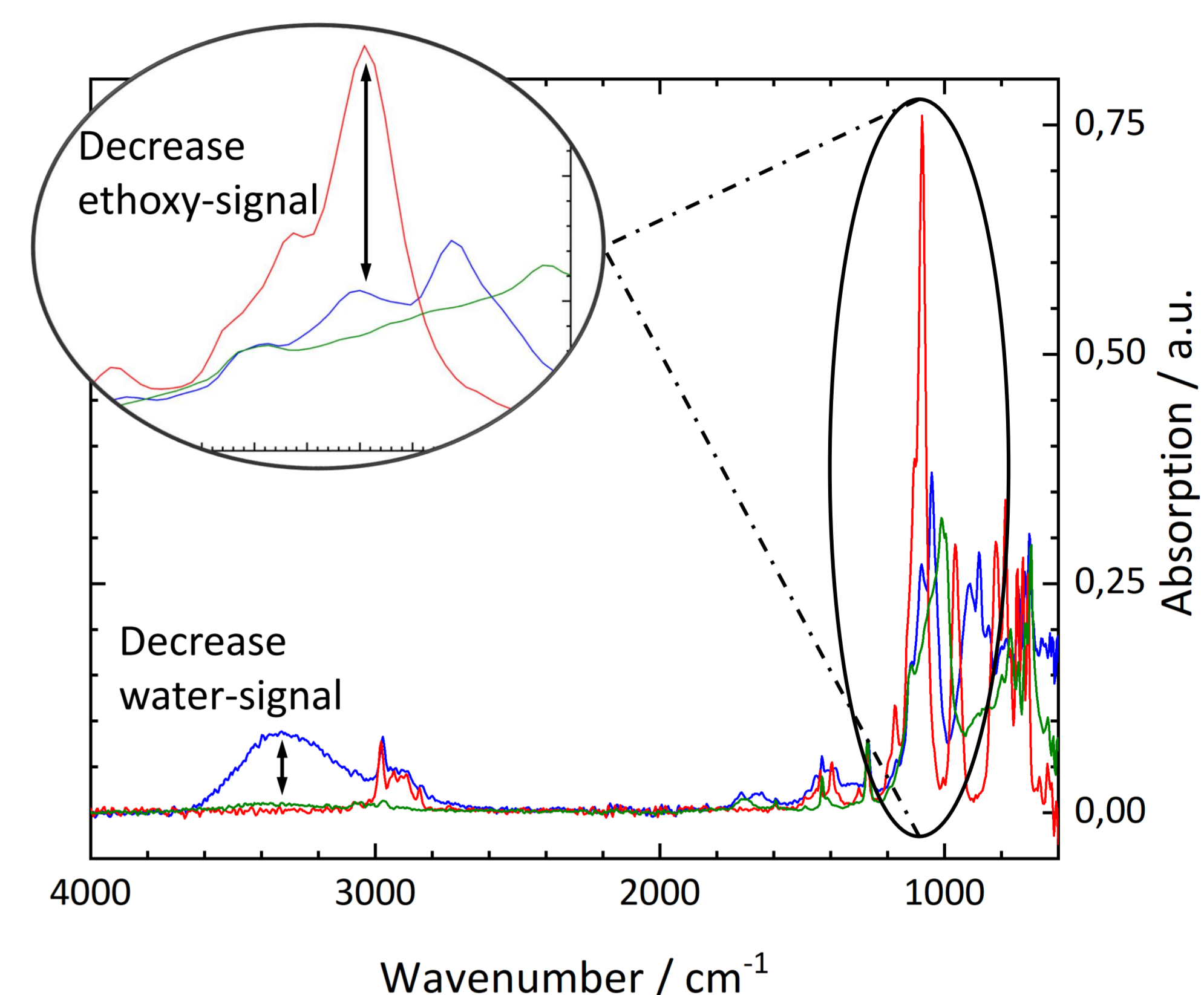


Figure 3: ATR-IR measurements of the silane mixture (red), the reaction solution after turning clear (blue) and the final product (green).

Figure 4 represents the progress measurement with ¹H-NMR, where the educt concentrations of water, ethoxy, and methoxy groups decrease over time. At first alkoxy-groups are hydrolyzed and the silanol concentration increases. The condensation reaction cannot be completely stopped and by forming the sol-gel products it leads to a decrease of silanol groups.

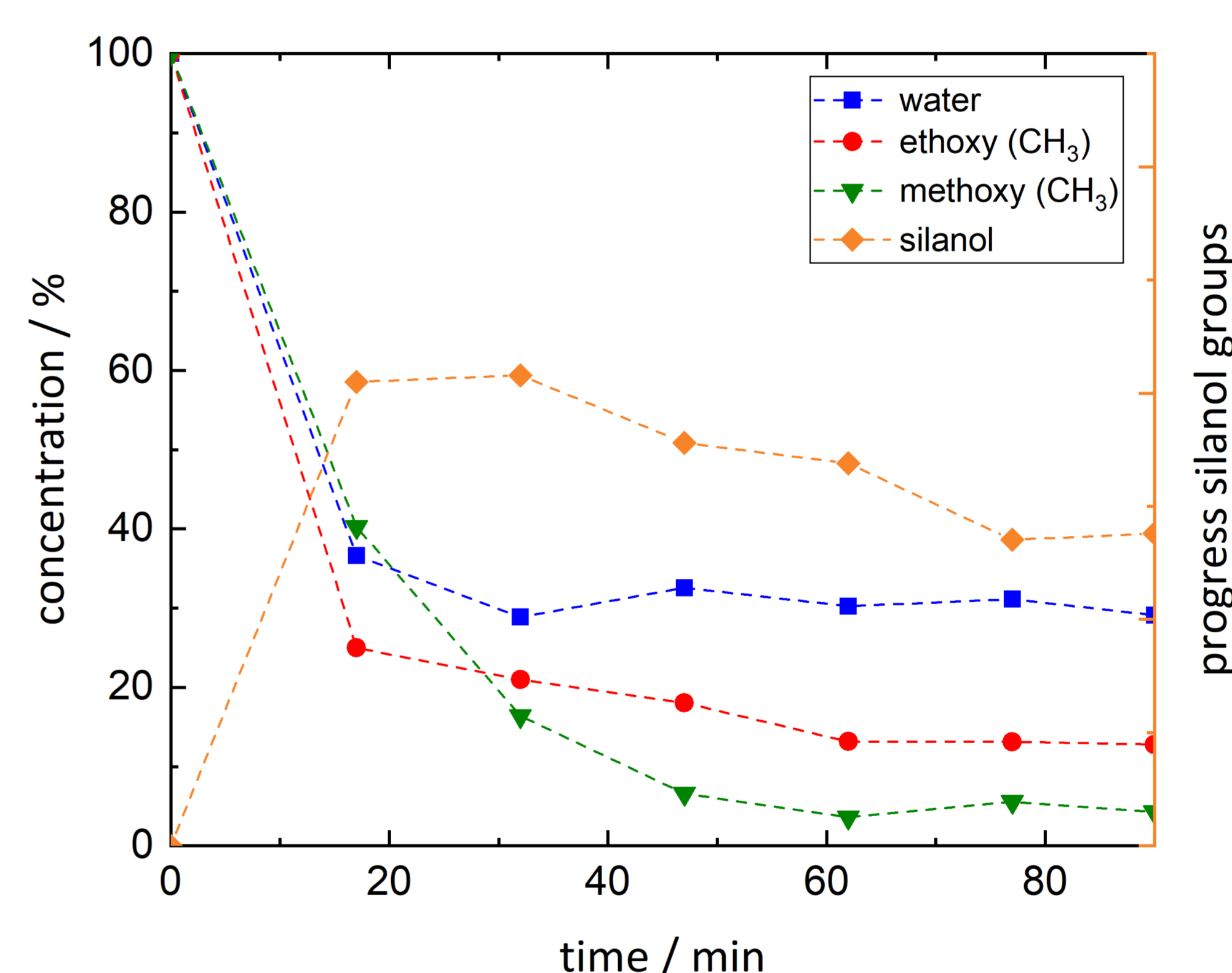


Figure 4: Progress measurement with ¹H-NMR of educt- and product concentrations.

About the author

Thomas Kisling is currently working on his PhD thesis in the field of coatings based on sol-gel reactions. The focus lies on controlling the structure-properties relationship in sol-gel products.

