Synthesis and Characterization of Double-Cross Linked PVPMS Aerogels

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Introduction

Aerogels have been rising to the surface as a material of interest for many practical applications. Aerogels are classified as "dried gels with a very high relative pore volume".[1] The practical applications of aerogels include thermal insulators, electrical conductors, sensors, as well as optical applications and more.[2–5] Aerogels are typically created through supercritical drying, in which the liquid in a sol-gel is turned into gas without destroying the structure of the gel. However this process requires high pressures and temperatures and the solvents used are highly flammable and unsafe at these conditions.[6] The purpose of this research experiment is to create a undergraduate student laboratory for CHEM411 Materials and Synthesis of Characterization in which the students will create polyvinylpolymethylsiloxane (PVPMS) aerogels using a new method that has been created using ambient pressure drying rather than supercritical drying.[7,8] The method of ambient pressure drying is safer for an undergraduate laboratory and has significantly less risks than the method of supercritical drying. The proposed student laboratory would include both the synthesis and characterization of the PVPMS aerogels, which are both topics that fall under the American Chemical Society's guidelines for topics in Macromolecular, Supramolecular, and Nanoscale (MSN) systems.[9]

References

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Methods and Results

Step One: Radical polymerization of vinylmethyldimethyoxysilane (VMDMS) with di-*tert* butyl peroxide DTBP by mixing the two together in a hydro-thermal reactor (seen below) and placing in a temperature-controlled oven. An alternate setup using an oil bath (seen to the right) was also used but was unsuccessful.

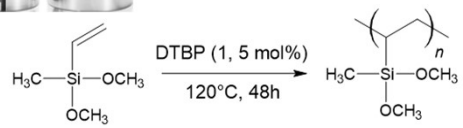


Figure 1: ^{1}H NMR Spectra of VMDMS Monomer

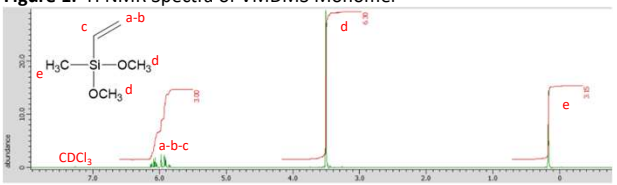
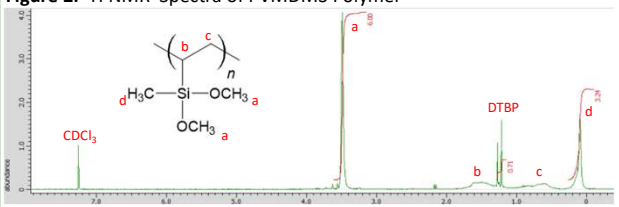
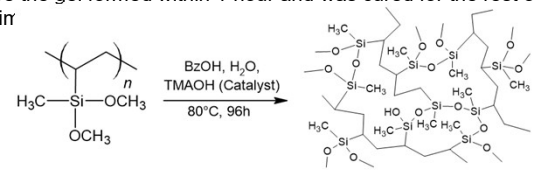


Figure 2: ^{1}H NMR Spectra of PVMDMS Polymer



As can be seen by the disappearance of the peaks around 6.0 ppm and the appearance of the broad peaks around 1.5 ppm and 0.7 ppm, the polymer was successfully synthesized. There is a strong doublet peak at1.2 ppm that can be attributed to the presence of DTBP end groups.

Step Two: Hydrolytic polycondensation of poly-VMDMS with TMAOH as catalyst with BzOH and H_2O. The solution was mixed and placed in a polypropylene container and placed in the oven where the gel formed within 1 hour and was cured for the rest of the tim



Methods

Step Three: Three solvent exchanges using isopropyl alcohol (IPA). Each solvent exchange took place overnight at 60°C.

Step Four: Ambient Pressure Drying (APD) for 2-5 days followed by drying at 80°C for 4 hours in the temperature-controlled oven, resulting in the final PVPMS aerogel.

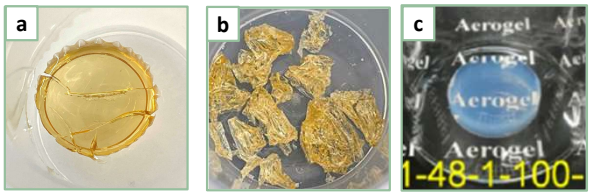
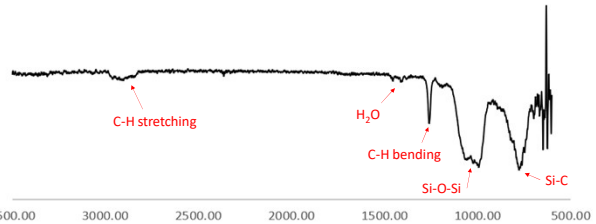


Figure 3: a) PVPMS aerogel created in this experiment, b) A second PVPMS aerogel created in this experiment that broke after drying, c) The expected look and color of the PVPMS aerogel

Figure 4: IR Spectrum of PVPMS Aerogel (b)



The IR spectrum collected from the PVPMS aerogel seen in Figure 3b displays the correct peaks that are to be expected. However, compared to previous research the C-H stretching peak is not as strong or sharp as is to be expected. The Si-O-Si peak is also broader than is to be expected.

Conclusion

This experiment was successful in synthesizing the PVMDMS polymer as well as the PVPMS hydrogel, but drying the hydrogel into its aerogel form was unsuccessful. Further research that can be done includes determining the source of error in the polymerization as well as continuing to attempt other APD drying techniques in order to successfully create the PVPMS aerogel and create an undergraduate student laboratory based upon the successful method.

Acknowledgments

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