

Novel Poly(orthosilicate)s Based on Linear Aliphatic Diols: Synthesis, Characterization, and Swelling Properties

Koksal Karadag, Hayal Bulbul Sonmez

Department of Chemistry, Gebze Institute of Technology, 41400 Gebze, Kocaeli, Turkey

Correspondence to: H. B. Sonmez (E-mail: hayalsonmez@gyte.edu.tr)

ABSTRACT: Condensation reactions between tetraethyl orthosilicate and linear aliphatic diol monomers at moderately high temperatures in proper stoichiometric ratios gave crosslinked poly(orthosilicate)s. The synthesized crosslinked polymers were found to be insoluble in common organic solvents, such as tetrahydrofuran, dichloromethane, benzene, and acetone, but did show swelling abilities in these solvents. All of the crosslinked polymers had moderate thermal stability and good, regenerable solvent uptake abilities. The synthesized polymers have been characterized by Fourier transform infrared spectroscopy solid-state ^{13}C - and ^{29}Si -NMR, thermogravimetric analysis, differential scanning calorimetry methods, and elemental analysis. In this report, the synthesis characterization, and swelling properties of crosslinked poly(orthosilicate)s for environmental removal and recovery of organic solvents are presented. The effect of the length of the aliphatic diol monomers on the properties of the synthesized polymers was also examined. © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 129: 2121–2127, 2013

KEYWORDS: swelling; gels; crosslinking

Received 4 September 2012; accepted 11 December 2012; published online 13 January 2013

DOI: 10.1002/app.38932

INTRODUCTION

Polymer gels consist of an elastic crosslinked network. Although they look like and possess all the other normal characteristics of solids, they can absorb solvents and swell to dimensions much larger than their dry sizes. Because of this property, they are referred to as “soft matter.”¹ Gels are capable of undergoing extensive deformation, and this property contrasts with most industrial materials, such as metals, ceramics, and plastics, which are dry and hard. Owing to these properties, polymer gels have important applications in both industry and scientific research, and have been used in items such as diapers, water retainers, drug delivery systems, ion-exchange resins, absorbent materials, and separation agents.^{2–9}

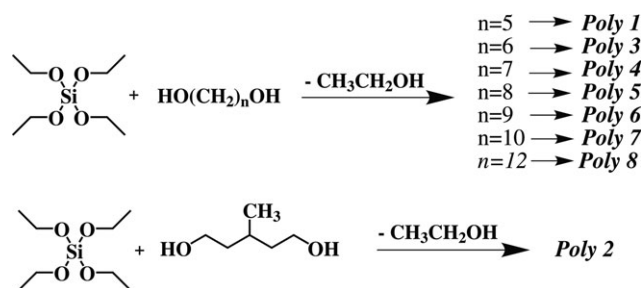
There are a variety of ways to classify gels, for example, they can be classified as natural or synthetic in accordance with origin; chemical or physical gels (hydrogen bonding, ionic interactions, Van der Waals interactions, and physical entanglements), in accordance with their crosslinkage; and hydrogel or organogel, in accordance with the swelling media.¹⁰ Polymers that absorb large amounts of water are known as hydrogels and polymers that absorb organic solvents or oils are known as organogels. The volumetric capacity of hydrogels can be altered by external stimuli such as pH, light, temperature, ionic strength, chemical reactions, and solvent.^{1,10–23} Because of these

excellent properties, hydrogels have been extensively studied and used in many domestic applications.^{24–27}

In contrast to hydrogels, organogels have been studied by fewer researchers. Organogels differ from hydrogels according to the liquid medium that can be absorbed by the polymer network. An organogel can absorb organic solvents or oils because its structure consists of hydrophobic polymer chains. Oil or organic solvent absorption is the process by which an organic molecule penetrates a network, causing it to expand or swell, which is affected by three factors: rubber elasticity, affinity to the solution, and crosslinking density.²⁸

Various materials are used to clean organic contaminants, such as dispersants, solidifiers, booms, skimmers, and natural and synthetic absorbents.^{29–39} Among these materials, the quickest and most effective are absorbents.⁴⁰ An effective absorbent should possess the following features: hydrophobicity and oleophilicity, high absorption capacity, a fast absorption rate, retention over time, reusability, and good absorption selectivity. Many polymers have been used to clean water contaminated with oil or other organic substances.^{40,41} Especially notable among them are aromatic polymers and alkyl acrylate polymers, which have attracted increasing attention from researchers.^{40–46}

Recently, we synthesized crosslinked poly(orthocarbonate)s as organic solvent absorbents.^{47,48} All of the synthesized polymers



Scheme 1. Polymerization reactions of linear aliphatic diol monomers with tetraethyl orthosilicate.

were thermally stable and had good, rapid sorption features for organic solvents such as dichloromethane (DCM), tetrahydrofuran (THF), dichloroethane, acetone, and a dry-cleaning fluid blend. Using the same methodology we previously reported, we have synthesized crosslinked poly(orthosilicate)s with tetraethyl orthosilicate (TEOS) and hydroxyl monomers (based on cyclohexanediol⁴⁹ or cyclohexanedimethanol^{50,51}). The obtained polymers are thermally stable and have very fast and efficient organic solvent absorption properties. To examine the saturation time of the all synthesized polymers, swelling kinetic measurements were conducted, and these results indicate that the all polymers reached their 85–90% swelling capacity within 20 min and saturated after 60 min. In this study, the novel crosslinked poly(orthosilicate)s were characterized, and their swelling features were examined, revealing a dependence on the lengths of the aliphatic diol monomers. The usage of these crosslinked poly(orthosilicate)s as organic absorbents was also investigated.

EXPERIMENTAL

Materials

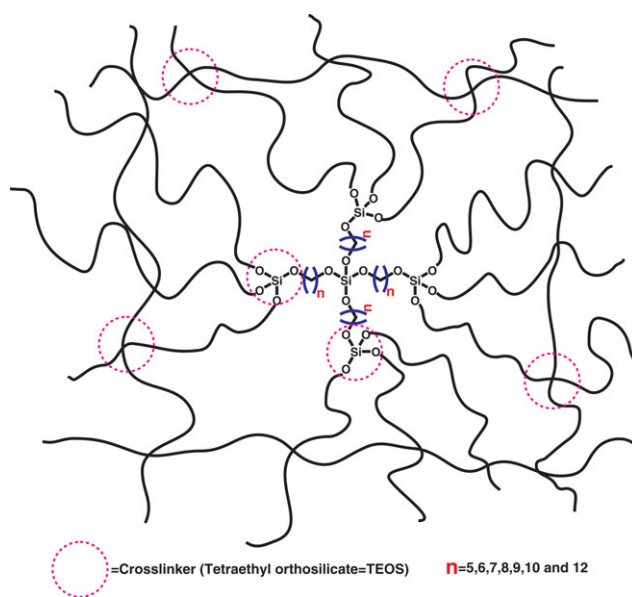
3-Methyl-1,5-pentanediol was purchased from Fluka. All other chemicals were purchased from Sigma-Aldrich and used without further purification.

Characterization

Fourier transform infrared spectroscopy (FTIR) spectra were recorded on a Perkin-Elmer Spectrum 100 FTIR spectrophotometer with an attenuated total reflectance objective.¹³C and ²⁹Si solid-state nuclear magnetic resonance (NMR) spectra were recorded on a 500-MHz Varian Inova spectrometer (Varian, Palo Alto, CA) in a cross polarization magic angle spinning (CPMAS) probe at 75.476 MHz. Thermogravimetric analysis (TGA) was performed under an argon atmosphere at 10°C min⁻¹ using a Mettler Toledo model TGA/SDTA 851 (Mettler Toledo, Greifensee, Switzerland). Differential scanning calorimetry (DSC) was performed with a Mettler TA Instrument DSC 822 at a heating rate of 10°C min⁻¹ under argon atmosphere.

Polymer Synthesis

Different crosslinked polymers were synthesized using TEOS and linear aliphatic diol monomers as explained below (Scheme 1). In this study, eight kinds of aliphatic diol monomers, 1,5-pentanediol, 3-methyl-1,5-pentanediol, 1,6-hexanediol, 1,7-heptanediol, 1,8-octanediol, 1,9-nonanediol, 1,10-decanediol, and 1,12-dodecanediol were used.



Scheme 2. Proposed polymer structure. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Synthesis of Poly 1. 1,5-Pentanediol (0.63 g, 6.04 mmol) and TEOS (0.67 mL, 3.02 mmol) were allowed to react for 6 days in a Pyrex (75 mL) pressure vessel placed in an oil bath at 160°C. The resulting product was washed with water, ethanol, and ether and vacuum dried to produce 0.41 g of a colorless, glassy, and transparent crosslinked polymer.

FTIR: 2937, 2874, 1074, 828 cm⁻¹. ¹³C-CPMAS-NMR: 64.28, 33.11, 23.05 ppm. ²⁹Si-CPMAS-NMR: -99.94, -80.87 ppm. Anal. Calcd. for C₁₀H₂₀O₄Si: C, 51.69%; H, 8.67%. Found: C, 47.70%; H, 8.52%.

Synthesis of Poly 2. 3-Methyl-1,5-pentanediol (0.97 g, 8.2 mmol) and TEOS (0.91 mL, 4.1 mmol) were polymerized in a pressure vessel at 160°C. After 3 days, the polymerization was stopped, and a transparent, glassy crosslinked polymer was obtained. The resulting product was washed with water, ethanol, and ether and vacuum dried to produce 0.86 g of polymer.

FTIR: 3500, 2928, 2881, 1071, 846 cm⁻¹. ¹³C-CPMAS-NMR: 62.30, 40.40, 26.90, 20.27. ²⁹Si-CPMAS-NMR: -100.52, -80.93 ppm. Anal. Calcd. for C₁₂H₂₄O₄Si: C, 55.42%; H, 9.28%. Found: C, 53.03%; H, 9.42%.

Synthesis of Poly 3. 1,6-Hexanediol (1.11 g, 9.46 mmol) and TEOS (1.05 mL, 4.73 mmol) allowed to react at 160°C for 3 days in a pressure vessel produced a colorless, transparent, crosslinked polymer. After being washed with water, ethanol, and ether, the polymer was dried under vacuum (1.11 g).

FTIR: 3400, 2940, 2877, 1089, 960, 852 cm⁻¹. ¹³C-CPMAS-NMR: 55.06, 23.85, 16.98 ppm. ²⁹Si-CPMAS-NMR: -93 ppm. Anal. Calcd. for C₁₂H₂₄O₄Si: C, 55.42%; H, 9.28%. Found: C, 44.58%; H, 8.67%.

Synthesis of Poly 4. 1,7-Heptanediol (0.75 g, 5.66 mmol) and TEOS (0.63 mL, 2.83 mmol) was kept in a pressure vessel at 160°C for 2 days to give very light yellow, glassy, and

transparent crosslinked polymer. After resulting polymer was washed with water, ethanol, and ether, it dried under vacuum to give 0.53 g polymeric gel.

FTIR: 2930, 2857, 1075, 849 cm^{-1} . ^{13}C -CPMAS-NMR: 63.64, 32.84, 29.65, 26.35 ppm. ^{29}Si -CPMAS-NMR: -95.39 , -88.07 , -81.31 ppm. Anal. Calcd. for $\text{C}_{14}\text{H}_{28}\text{O}_4\text{Si}$: C, 58.13%; H, 10.02%. Found: C, 56.58 %; H, 9.78%.

Synthesis of Poly 5. 1,8-Octanediol (0.64 g, 4.36 mmol) and TEOS (0.48 mL, 2.18 mmol) were placed in a pressure vessel at 160°C for 5 days to create colorless, glassy, and transparent crosslinked polymer. The resulting product was washed with water, ethanol, and ether and vacuum dried to produce 0.58 g of polymer.

FTIR: 3400, 2943, 2856, 1084, 852 cm^{-1} . ^{13}C -CPMAS-NMR: 64.09, 33.29, 30.43, 26.71 ppm. ^{29}Si -CPMAS-NMR: -88.69 , -85.47 ppm. Anal. Calcd. for $\text{C}_{16}\text{H}_{32}\text{O}_4\text{Si}$: C, 60.71%; H, 10.19%. Found: C, 58.89 %; H, 10.47%.

Synthesis of Poly 6. 1,9-Nonanediol (0.75 g, 4.68 mmol) and TEOS (0.52 mL, 2.34 mmol) were polymerized in a pressure vessel at 160°C . After 3 days, the polymerization was stopped and colorless, glassy, and transparent crosslinked polymer was obtained. It was washed with water, ethanol, and ether, after being dried under vacuum, gives 0.54 g of polymer.

FTIR: 3400, 2925, 2855, 1086, 856 cm^{-1} . ^{13}C -CPMAS-NMR: 63.41, 32.58, 29.87, 26.10 ppm. ^{29}Si CPMAS NMR: -101.07 , -88.28 , -81.24 ppm. Anal. Calcd. for $\text{C}_{18}\text{H}_{36}\text{O}_4\text{Si}$: C, 62.74%; H, 10.53%. Found: C, 62.10 %; H, 10.76%.

Synthesis of Poly 7. The reaction of 1,10 decanediol (0.55 g, 3.16 mmol) and TEOS (0.35 mL, 1.58 mmol) at 160°C for 5 days in a pressure vessel gave colorless, transparent crosslinked polymer. After the resulted polymer was washed with water, ethanol, and ether, and after being under vacuum, it afforded 0.37 g of polymer.

FTIR: 2924, 2853, 1084, 963, 842 cm^{-1} . ^{13}C -CPMAS-NMR: 63.95, 33.14, 30.54, 26.71 ppm. ^{29}Si -CPMAS-NMR: -84.65 , -78.17 ppm. Anal. Calcd. for $\text{C}_{20}\text{H}_{40}\text{O}_4\text{Si}$: C, 64.46%; H, 10.82%. Found: C, 61.45 %; H, 10.66%.

Synthesis of Poly 8. 1,12-Dodecanediol (0.69 g, 3.42 mmol) and TEOS (0.38 mL, 1.71 mmol) were polymerized in a pressure vessel at 160°C . After 3 days, the polymerization was stopped, and a colorless, glassy crosslinked polymer was obtained. The resulting product was washed with water, ethanol, and ether and vacuum dried to produce 0.55 g of polymer.

FTIR: 2922, 2852, 1089, 845 cm^{-1} . ^{13}C -CPMAS-NMR: 63.83, 32.85, 30.38, 26.44 ppm. ^{29}Si -CPMAS-NMR: -88.17 , -81.40 ppm. Anal. Calcd. for $\text{C}_{24}\text{H}_{48}\text{O}_4\text{Si}$: C, 67.23%; H, 11.28%. Found: C, 65.24%; H, 11.63%.

Techniques

Soluble Fractions. A weighed quantity of crosslinked polymer was put in a solvent, and the soluble fraction (SF) was extracted for long time (48 or 72 h) using THF. After extraction, swelled polymers were dried under vacuum at 45°C . The SF was calculated according to following equation:⁵²

$$\text{SF (\%)} = \frac{(W_0 - W)}{W_0} \times 100 \quad (1)$$

where W_0 and W are the weights of the polymers before and after extraction, respectively.

Swelling Test

The bags prepared from filter paper were used to determine swelling properties of the crosslinked polymers.⁵³ First, the bags were immersed in the solvent and blotted quickly with an absorbent paper; this was followed by addition of a dried polymer sample of known weight into the bags. The filled bags were immersed in the solvent. All swelling experiments were conducted at room temperature. When equilibrium was reached after 24 h, the bags were removed; their surface were dried gently by blotting to remove excess solvent and weighed in a stoppered weighing bottle. Solvent uptake percentages were calculated using the following formula:

$$\text{Solvent uptake (\%)} = \frac{(W_s - W_d)}{W_d} \times 100 \quad (2)$$

where W_d and W_s represent the weight of dry and swollen cross-linked polymer samples, respectively.⁵⁴

Swelling Kinetics

The swelling kinetics of solvent absorption was studied by repeating the previous measurements at different time intervals using DCM as a solvent.

Desorption Kinetics

To investigate the reusability of the polymers, the DCM retention of polymers in air was determined by weighing the swollen polymer in air as a function time.

RESULTS AND DISCUSSION

Synthesis and Characterization of Crosslinked Poly(orthosilicate)s

The reactions between TEOS and linear aliphatic diols to obtain crosslinked poly(orthosilicate)s are summarized in Scheme 1. The crosslinked polymers were prepared by condensation of monomers in a solvent-free medium at moderately high temperatures in one step without using a catalyst. In our previous work, TEOS was polymerized with cycloaliphatic diol monomers to prepare crosslinked polymers. In the present study, we have extended our work to the synthesis of polymers with high solvent absorption capacities. The diols used as hydroxyl functional monomers were 1,5-pentanediol for **Poly 1**, 3-methyl-1,5-pentanediol for **Poly 2**, 1,6-hexanediol for **Poly 3**, 1,7-heptanediol for **Poly 4**, 1,8-octanediol for **Poly 5**, 1,9-nonanediol for **Poly 6**, 1,10-decanediol for **Poly 7**, and 1,12-dodecanediol for **Poly 8**. These diols were used to investigate the effect of hydrophobicity on the properties of the synthesized polymers by varying the length of the alkyl groups on the polymers. The proposed polymer structure was given in Scheme 2. While all synthesized polymers were insoluble in common organic solvents, they showed swelling ability in these solvents and can be used to remove organic solvents from the environment.

The structures of the prepared polymers were confirmed by elemental analysis, FTIR, solid-state CPMAS ^{13}C - and ^{29}Si -NMR,

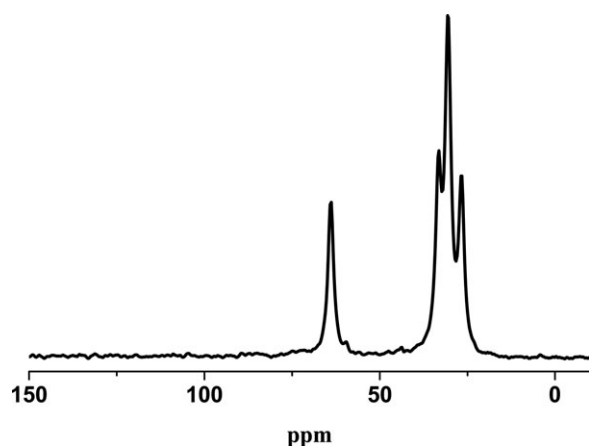


Figure 1. The CPMAS solid-state ^{13}C -NMR spectrum of Poly 7.

and thermal analysis. The elemental analyses were consistent with the theoretical values. The strong stretching vibrations detected at about 1084 and 842 cm^{-1} indicated the presence of $\text{Si}-\text{O}-\text{C}$ and $\text{Si}-\text{O}-\text{CH}$ functions, respectively. Stretching vibrations of the aliphatic $\text{C}-\text{H}$ bond were observed at about 2924 and 2853 cm^{-1} . The IR spectra also showed a broad absorption peak between 3400 and 3500 cm^{-1} , which is the characteristic stretching vibration of hydroxyl groups, and this is indicative of the desired structure containing hydroxyl functionalities as the end groups (especially for Poly 2, Poly 3, Poly 5, and Poly 6).

The CPMAS ^{13}C -NMR spectrum of Poly 7 is presented in Figure 1 as a representative example. The resonance at 63.95 ppm proves the presence of $\text{CH}_2-\text{O}-\text{Si}$ carbons. The signals at 33.14 and 30.54 ppm confirm the presence of $\text{CH}_2-\text{CH}_2-\text{O}-\text{Si}$ (aliphatic linear carbons) and the peak at around 26.71 ppm is from the methyl group that appears as an end group.

The CPMAS solid-state ^{29}Si -NMR spectrum of Poly 7 is shown in Figure 2. In the solid-state ^{29}Si -NMR spectrum, the signals at -78.17 and -84.65 ppm ($-\text{CH}-\text{O}-\text{Si}$) provide strong evidence for the incorporation of Si into the polymer structure. In addition

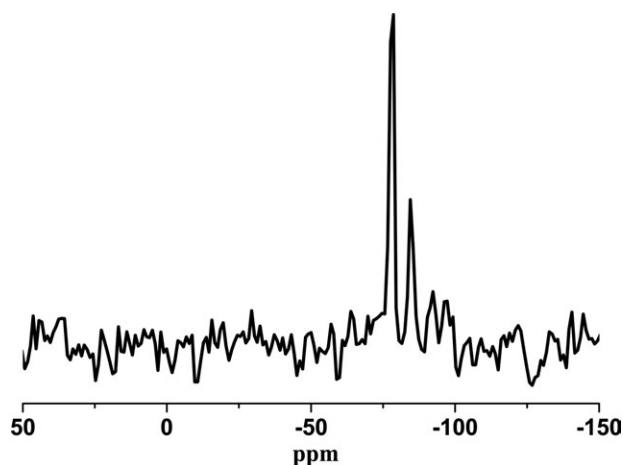


Figure 2. The CPMAS solid-state ^{29}Si -NMR spectrum of Poly 7.

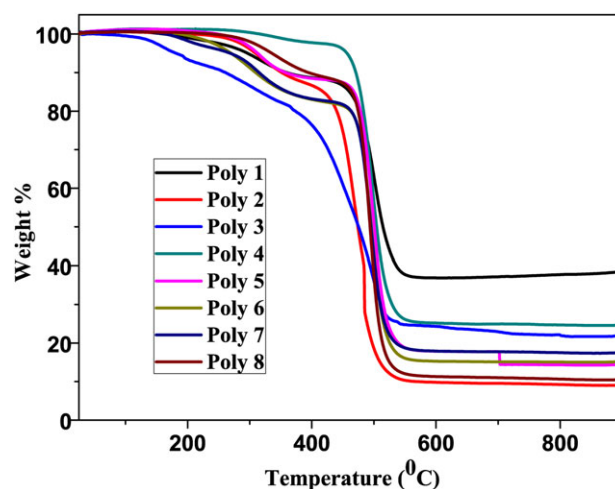


Figure 3. TGA thermograms of crosslinked poly(orthosilicate)s. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

tion to these peaks, there was a peak at around -100 ppm for Poly 1, Poly 2, Poly 3, Poly 4, and Poly 6, which is indicative of $-\text{SiO}_4$ groups.

The thermal stability of the polymers was evaluated by TGA and DSC under nitrogen. In the thermal analysis of all of the polymers, neither glass transition temperature (T_g) nor melting point (T_m) could be observed. The TGA thermograms (Figure 3), indicate that all of the crosslinked poly(orthosilicate)s based on linear aliphatic diols are thermally stable at temperatures lower than 300°C , except for Poly 3, which is thermally less stable than the others. The order of thermal stability of the polymers was Poly 4 (C_7) > Poly 8 (C_{12}) > Poly 5 (C_8) > Poly 2 (C_6) > Poly 1 (C_5) > Poly 7 (C_{10}) > Poly 6 (C_9) > Poly 3 (C_6) at 300°C . The silicon content of the polymers was determined by thermogravimetric analysis to be 38.5% for Poly 1, 9.1% for Poly 2, 22.0% for Poly 3, 24.6% for Poly 4, 14.4% for Poly 5, 15.1% Poly 6, 17.5% for Poly 7, and 10.5% for Poly 8.

Swelling Properties of Crosslinked Poly(orthosilicate)s

All polymers were insoluble in THF, DCM, acetone, benzene, and other common organic solvents, but they did display swelling properties in these solvents. Some polymer chains were not attached to the polymer network and could be extracted from the gel fraction. To eliminate or minimize these fractions, the likely SFs in the crosslinked poly(orthosilicate)s were isolated by means of extraction. To execute this, a known amount of dry crosslinked polymer was added to a solvent and the SF was extracted into THF for 48 or 72 h at room temperature. After extraction, the swelled polymers were dried under vacuum at 45°C . The values of the SFs of the crosslinked polymers were found to be 2.5% for Poly 1, 1.5% for Poly 2, 5.2% for Poly 3, 0.7% Poly 4, 6% for Poly 5, 8.5% for Poly 6, 11.1% for Poly 7, and 1.2% for Poly 8.

The synthesized crosslinked poly(orthosilicate)s were found to have good solvent absorption properties in organic solvents. In this study, the swelling abilities of the polymers were observed

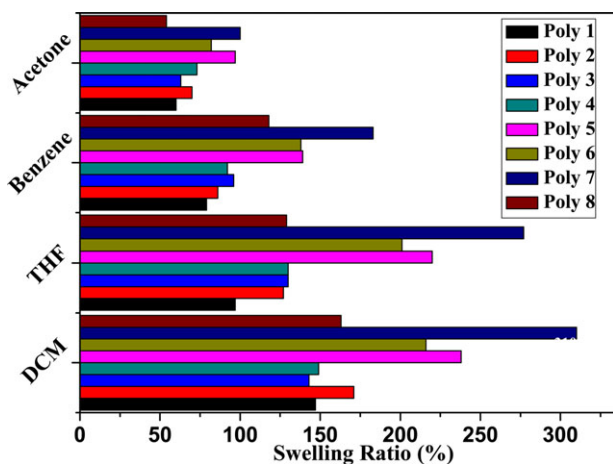


Figure 4. Swelling values of crosslinked poly(orthosilicate)s. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

in four common solvents, THF, benzene, DCM, and acetone. To research the maximum sorption capacity of the polymers, after extraction of the SF, a known amount of polymer sample was placed in a filter paper bag and then immersed in a solvent. The swelling rate was monitored for each solvent over 24 h, and the samples were removed from the solvent and weighed in a stoppered bottle. All swelling experiments were conducted at room temperature. This procedure was repeated at least four times for each polymer to confirm the replicability of the values. The results of the absorption capacity studies of the prepared crosslinked poly(orthosilicate)s in different organic liquids are shown in Figure 4.

Among the synthesized polymers, **Poly 7**, which was synthesized from the condensation of TEOS and 1,10-decanediol, showed the best organic solvent absorbency, whereas **Poly 1** showed the lowest solvent absorbency. For **Poly 7**, the highest solvent absorbency of ~ 310% was obtained in DCM, which was the most absorbable solvent for all of the synthesized polymers. The solvent absorption capacity of the synthesized polymers was in the order of **Poly 7** (C_{10}) > **Poly 5** (C_8) > **Poly 6** (C_9) > **Poly**

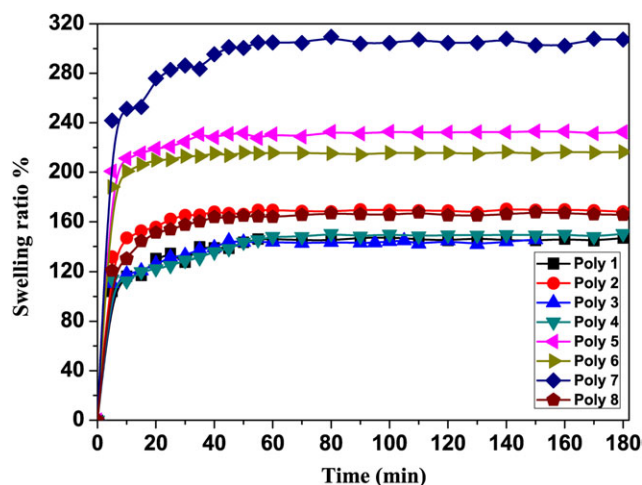


Figure 5. Swelling kinetics of the crosslinked poly(orthosilicate)s in CH_2Cl_2 (DCM) at room temperature. Each point presented in the figure is an average of at least four different measurements. There is a maximum 5% deviation from each point. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

8 (C_{12}) > **Poly 4** (C_7) ≥ **Poly 3** (C_6) ≈ **Poly 1** (C_5). The swelling capacity values of the polymers were found to be associated irregularly with the number of carbons in the linear aliphatic diol monomers. As the length of the aliphatic diol monomer increases, the voids in the polymeric network increase so that more solvent molecules can diffuse into the voids. When the number of carbon atoms exceeds 10, the extension of the linear group no longer increases the solvent absorption. Beyond this point, the extension of the alkyl group causes chain entanglement, and the solvent–polymer interaction is decreased; thus, the solvent absorbency of the polymer is reduced.

In comparison with **Poly 2**, which was based on 3-methyl-1,5-pentanediol, **Poly 1**, which was based on 1,5 pentanediol, had a higher swelling capacity. Inserting the 3-methyl group into the polymer structure resulted in more hydrophobic character in the crosslinked polymer structure of **Poly 2**, thereby increasing the absorption capacity.

Table I. Comparison of Swelling Properties of Polymers

Polymer	Solvent	Swelling %	References
Butyl rubber	Toluene	223	55
PDMS/PVA	Dichloromethane	120	56
Amino-polysiloxanes	Hexane	360	57
Poly(dimethylsiloxane) sponge	Toluene	400	58
Poly(stearylmethacrylate-co-DVB)	Toluene	360	54
Crosslinked poly(stearyl methacrylate-co-cinnamoyloxyethyl methacrylate)	Toluene	610	59
Polycarbonate	Dichloromethane	148	60
Polyether-based polyurethanes	Benzene	181	61
Crosslinked poly(orthocarbonate)s	Dichloromethane	320,314	47,48
Crosslinked poly(orthosilicate)s (based on cyclohexanediol)	Dichloromethane	120	49
Crosslinked poly(orthosilicate)s (based on cyclohexanedimethanol)	Dichloromethane	200	50,51
Crosslinked poly(orthosilicate)s (based on linear aliphatic diol)	Dichloromethane	310	This work

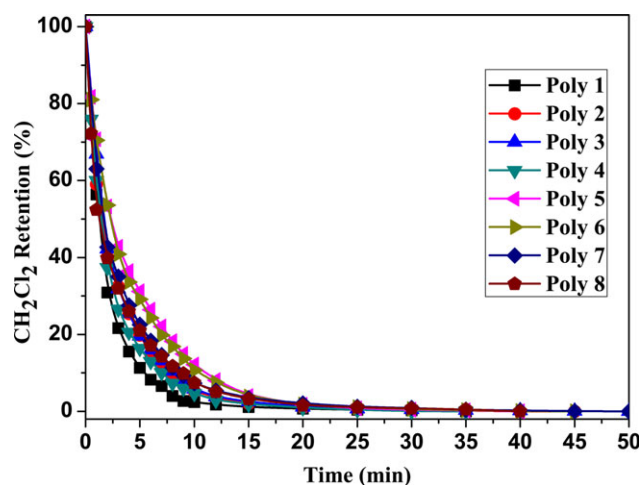


Figure 6. CH₂Cl₂ (DCM) retention of crosslinked poly(orthosilicate)s. Each point presented in the figure is an average of at least four different measurements. There is a maximum 5% deviation from each point. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The swelling kinetics of the polymers in DCM, which was the most absorbable solvent, is shown in Figure 5. To understand the saturation time of the crosslinked poly(orthosilicate)s, the swelling percentages of the polymers were examined with respect to time. All of the synthesized polymers reached ~80–90% swelling in 15 min, reached equilibrium 45–60 min later, and then remained constant. These results show that all of the synthesized polymers have very fast solvent uptake capabilities. Comparison of swelling capacities of synthesized linear aliphatic diol based crosslinked poly(orthosilicate)s with other polymers from literature is given in Table I. It can be seen from the table that the synthesized polymers have good and competitive swelling properties.

The retention times of the polymers for DCM were obtained by determining the weight loss of the swollen polymers in air and it was found that almost all absorbed DCM was released within 20–25 min. These results show that all of the sorbents have and nearly all of the absorbed DCM could be released from the sorbents very quickly (Figure 6).

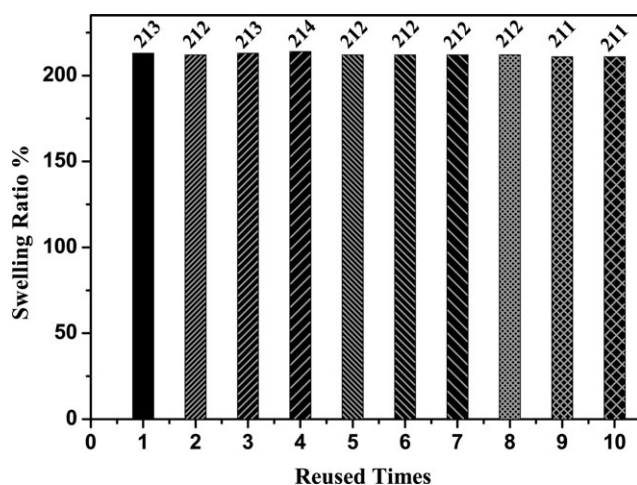


Figure 7. Reusability of Poly 6 in DCM.

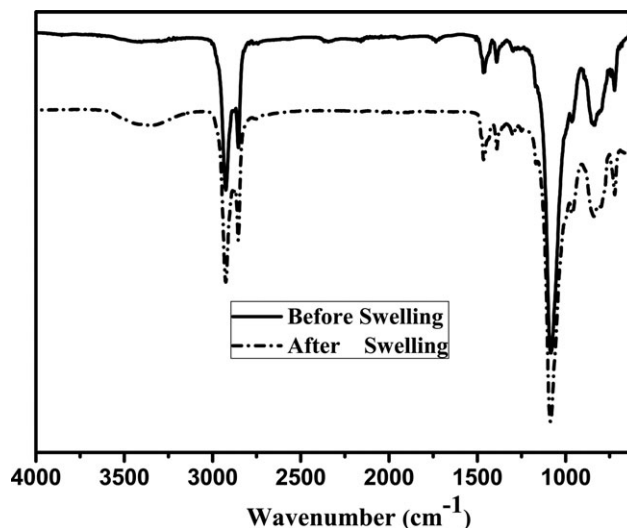


Figure 8. The FTIR spectrum of Poly 7 before and after swelling measurements.

The reusability of polymers without any capacity loss is very important, especially for practical applications. For this purpose, the reusability of the crosslinked poly(orthosilicate)s synthesized from linear aliphatic diols was investigated by measuring their solvent absorption and desorption properties. Despite enduring repeated absorption–desorption tests, the capacities of the polymers did not change. In Figure 7, the reusability results for Poly 6 are given as a representative example. According to the figure, this polymer absorbed DCM at the same percentages for at least 10 uses. After the polymers were regenerated several times, examination by FTIR was performed to determine whether any structural changes resulting from further condensation reactions of unreacted groups or hydrolysis of Si–O–C bonds had occurred (Figure 8). The FTIR spectra of the polymers clearly showed that the synthesized crosslinked poly(orthosilicate)s can be used several times without loss of capacity.

CONCLUSIONS

In conclusion, we have reported the synthesis and characterization of novel crosslinked poly(orthosilicate)s based on linear aliphatic diols. The influence of the diol monomers on the organic solvent absorption capacity of the synthesized crosslinked polymers was examined for sorbent applications. All of the synthesized polymers were thermally stable and possessed rapid and high-capacity organic solvent uptake abilities. These results obviously show that these polymeric gels can be used as organic solvent absorbents.

ACKNOWLEDGMENTS

The authors thank the Scientific and Technological Research Council of Turkey–TUBITAK for the support of this work through Grant TBAG/109T515 and 106T616.

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