The Effect of Acid Concentration on the Synthesis of Hydrophobic Silica Based Aerogels

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Abstract

Aerogel has a meaning of an air gel that made of a gel solution [1]. Aerogels are transparent and highly porous materials which known with low density. Since the 1930's, researchers have been focused on the synthesis methods and applications of aerogels [2]. In recent years, silica aerogels have been widely used in optics, microelectronic, electrical engineering, acoustics, heat insulation, capture of micro particles, pharmaceutical, chemistry, and biology [1-3].

In this study, the effect of the acid concentration on the synthesis of hydrophobic silica based aerogel was investigated. Tetraethyl orthosilicate (TEOS) and chlorotrimethylsilane (TMCS) were used in the synthesis as a silica source and silylation reagent, respectively. Different concentrations (0.5 M, 1 M, and 1.5 M) of acid solutions were prepared in order to see the effect of acid concentration in the synthesis of silica based hydrophobic aerogel. The synthesized samples were characterized by Fourier transform infrared spectroscopy (FTIR) and contact angle analysis. The obtained results show that the optimum acid concentration was found as 0.5 M.

Keywords-component; Silica based aerogel, acid concentration, FT-IR, contact angle.

1 Introduction

Aerogels are nanoporous lightweight materials that discovered in 1930s by Kistler in California. The most important feature of aerogel is the liquid component within the pores of the solid is replaced by air. [4]. Silicon, aluminum, titanium, carbon, several group metal oxides, metals (copper and gold), organic polymers, biological polymer can be used as a raw material in synthesis of aerogels. Depending on the raw materials, aerogel has different properties [5-7]. The surface of aerogel consists of millions of tiny holes, aerogel resembles a sponge. Aerogel is composed of 99.8% air regarding that they are good insulators. It has an insulating ability 39 times more than the most advantaged fiberglass insulation material. They also called as "frozen smoke" or "blue smoke" because of the appearance of smoke [7-9]. Density of aerogels ranges from 0.003 to 0.35 g/cm^3. Shrinkage begins slowly at 500 °C regarding the increase of temperature. Melting point is 1200 °C. Sound velocity through the medium is 100 m/sec [9].

Aerogels have gained much importance in recent years and has been used in any different areas. In commercial applications of aerogels are thermal insulation, sound insulation, in the form super capacitors and catalytic promoters [10]. On the other hand monolithic silica aerogels are extensively used in energy physics as Cherenkov radiation detector [11]. Other applications of aerogels are; adsorbent, thickening agent, laser target, opacification additives and in pharmaceutical industry as drug delivery system [12-15].

Hydrophobicity property occupies an important place in the aerogel applications. It is particularly preferred to use hydrophobic and super-hydrophobic material in some special applications.

This study is supported by a grant (2014-07-04-KAP02) from Scientific Research Projects Committee of Yıldız Technical University.

In this study pH effect of the acid concentration on the synthesis of hydrophobic silica based aerogel was investigated. Different concentrations of acid solution were used to see the effect of pH to hydrophobicity of aerogels.

2 Materials and Methods

In the present study, TEOS was used as a pure silica source that supplied from Merck (≥99% purity). The chemicals used in the synthesis of silica based aerogel were hydrochloric acid (Merck 37%), ethanol (Sigma Aldrich 25%), and ammonia solution (Merck 25%).

Characterizations of the samples were performed by FT-IR analysis and contact angle measurement. Infrared data were obtained by FTIR spectrometer (Perkin Elmer, Spectrum One) with Attenuated Total Reflection (ATR) technique in the range of 4000-650 cm^{-1} (Fig. 1).

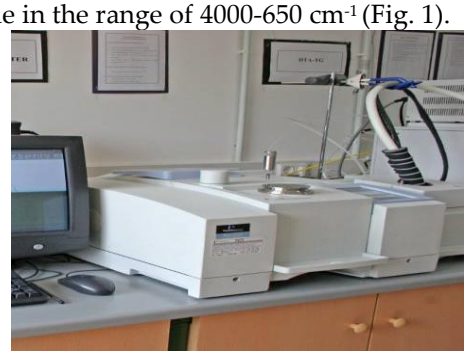


Figure 1. Image of the FT-IR device

Figure 2. Image of the optical microscobe

The contact angle measurement was done using optical microscope (Veho 100) to quantify the degree of hydrophobicity (Fig. 2).

2.1 Synthesis of silica based aerogel

Synthesis scheme of silica based aerogel were given in Fig 3. In the first step of study, TEOS, TMCS, ethanol, distillated water and acid catalyst (HCl) mixed with molar ratios 1: 0.36: 3.1: 1.2:0.0007 in a beaker. In order to determine the effect of acid concentration to hydrophobicity with different concentrations (0.5 M, 1 M, and 1.5 M) HCl solution were used. Prepared mixture was treated to one hour reflux process. After reflux process the mixture that containing ethanol, ammonia solution, and distillated water was added to the mixture. Ammonia was used as a base catalyst to accelerate the gelation. After processing the mixture was allowed to aging for 24 hours at room temperature. At the end of the aging, the gel formed and then it was dried for 4 hours at 50°C eluting with hexane.

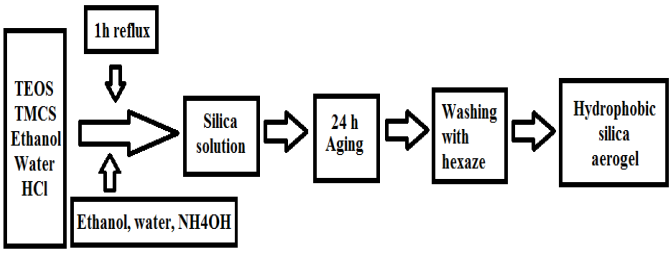


Figure 3. Synthesis scheme of silica aerogel

3 Results and Discussion

The obtained FTIR absorption bands were interpreted by referring to previous publications (Table 1) and Fig. 4 presents the infrared transmittance spectra of the hydrophobic aerogels.

Table 1. Vibration frequencies (cm^{-1}) in FTIR spectra of the hydrophobic aerogels

Type of vibration	Vibration frequencies (cm-1)			
	0.5M	1M	1.5M	References [16,17]
C–H symmetric stretching	2960	2960	2960	2900
Si–C stretching	1252	1252	1252	1260
Si–O–Si symmetric stretching	1046	1050	1047	1100
Si–C stretching (silicon-bonded alkyl groups C-H bending)	836	836	835	850
Si–O–Si symmetric stretching	754	753	753	800

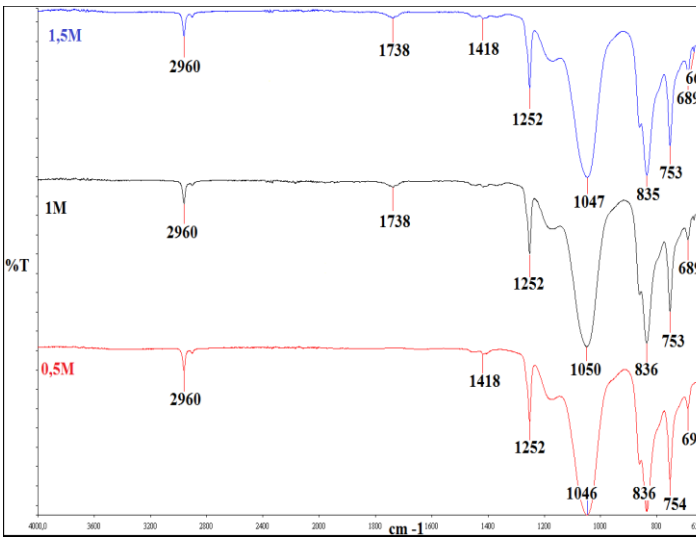
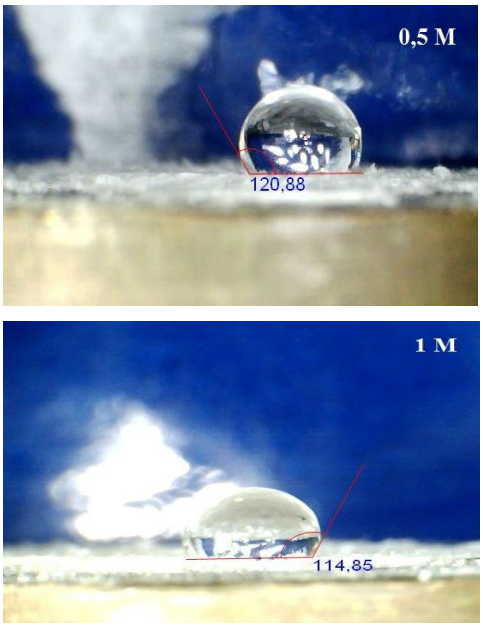


Figure 4. FT-IR spectrum of silica aerogels that synthesized under different acid concentrations

It is observed that all synthesis for three concentrations recorded with success. According to the obtained FT-IR results it was found that synthesized samples are consistent with the literature.

Hydrophobicity is an expression of a shape of any water droplet from a great surface. A surface is called hydrophobic if the contact angle, related to surface tension or surface free energy, is >90° [18]. Fig. 5 shows a photograph of a water drop placed on the surface of hydrophobic aerogels surface. The drop maintained spheroid shape with contact angles of 120.88°, 114.85°, and 110.40°, respectively.



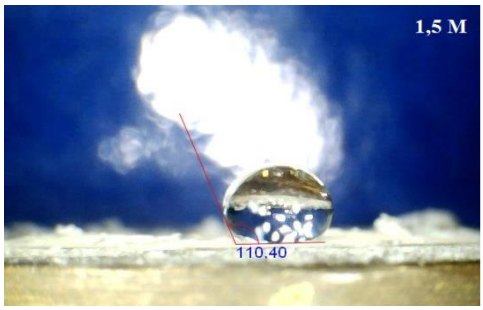


Figure 5. Contact angles of silica aerogels that synthesized under different acid concentrations

According to the results of contact angle measurement showed that the product that best hydrophobicity expressed in molar concentration 0.5.

4 Conclusion

The synthesis of silica based aerogel in different acid concentrations to examine the effect of acid concentration on hydrophobicity was performed successfully. The chemical bond structure of obtained product was determined by FT-IR spectroscopy. The contact angles measurement of the surface properties of the synthesized samples were determined. According to the FT-IR analysis results the bands that placed in 2960, 1418, 1252, and 836 cm^{-1} were providing hydrophobic agent successfully connected to the silica structure. It was seen that FT-IR spectra of the obtained samples were similar when compared to each other. The sample synthesized using of 0.5 M acid had the more hydrophobic character than the other samples.

5 References

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