In recent years, silica aerogels have attracted increasingly more attention due to their extraordinary properties and their existing and potential applications in wide variety technological areas. Silica aerogel is a nanostructured material with high specific surface area, high porosity, low density, low dielectric constant and excellent heat insulation properties. Recently many research works have been devoted to ambient pressure drying which makes the production commercial and industrial. The silica source powder (perlite) was dissolved by boiling in the sodium hydroxide aqueous solution for 2h. Most of the perlite powder was dissolved in NaOH solution. The solution was filtered to remove the undissolved residues. Surface modification and drying are the most critical steps in sol-gel production. The bonds in the structure are strengthened and the material strength is increased by surface modification with TEOS (tetraethylorthosilicate). The aging process was supported by treatment of gels with solutions prepared using ethanol and TEOS chemicals. Solvent exchange with n-heptane was performed before drying in atmospheric pressure and finally the silica aerogel powders were dried at 90°C for 2 day at atmospheric pressure. Microstructure and morphology of the silica aerogel powder was observed by field emission scanning electron microscopy (FE-SEM) and SEM-EDS. The phases of silica aerogel powders were analyzed using XRD (X-ray Diffraction) technique. The weight loss of the aerogel powders were studied thermo gravimetric and differential thermal analysis (TG-DTA) in N\textsubscript{2} atmosphere at a heating rate of 5 \(^\circ\)/min from room temperature to 1000 \(^\circ\). In the present study, perlite, which is a cheap silica source with plenty of reserves in our country, was used for the first time in the production of silica aerogel powder.

1. Introduction

Silica aerogels have received significant attention in many fields such as catalysis, adsorption, thermal insulation and drug delivery system because of their unique properties of large surface area, large pore volume, low bulk density and low thermal conductivity [1]. Aerogels derived from sol–gel oxides such as silica have become technologically and scientifically popular because of their extremely low density (0.06 g/cm\(^3\)), high optical transmission (>90%) in the visible range, very low refractive index (1.01–1.1), large surface area (>1000m\(^2\)/g) and very low thermal conductivity (0.02 W/mK) and their interesting optical, dielectric, thermal, acoustic applications and low dielectric constant materials for MOS devices. Even though aerogels have ultra fine particle and pore sizes, extremely large surface area and a microstructural composition of interconnected colloidal particles and have the atomic density as low as a gas 1026/m\(^3\), pure silica aerogels are hydrophilic and became wet with atmospheric moisture and water. The aerogels get deteriorated with time due to the adsorption of water molecules from the humid surroundings because they posses on their surface, polar OH groups that can take part in hydrogen bonding with H\(_2\)O. The replacement of H from the Si–OH groups by the hydrolytically stable Si–R groups through the oxygen bond prevents the adsorption of water and hence results in the hydrophobic aerogels [2-8]. In 1864, Graham showed that water in silica gel could readily be replaced by organic liquids. This fact led Kistler to explore replacing the liquid in a wet gel with gas by extracting the liquid from the gel at a high temperature and high pressure. He produced aerogels in an autoclave, which was an expensive process. Since the drying takes place at a high temperature and pressure, it is a very difficult and unreliable method. Furthermore, the Kistler method was very tedious and time-consuming. In 1968 a team of researchers headed by Teichner developed a method for producing silica aerogels within one day using (albeit costly) silicon alkoxide precursors. For commercial production, however, it is desirable to produce the silica aerogels using a low-cost precursor such as sodium silicate and to dry the wet gels at ambient pressure [9]. Traditionally, silica aerogels were usually synthesized by supercritical drying process so as to obtain porous structure. Supercritical drying process can avoid capillary stress and associated drying shrinkage. However, supercritical drying process is so costly and risky that real practicality and commercialization are difficult. Thus, in recent years, synthesizing silica
aerogels by an ambient pressure drying technique has been attracting considerable attention [1]. In order to obtain highly porous aerogel structure, elimination of capillary stress during ambient pressure drying is very important [3]. In order to further reduce the cost of fabrication and realize the commercial production of silica aerogels, using more cheap silica sources, synthesizing silica aerogels via ambient pressure drying at a reasonable cost is very necessary [1-8]. The conventional method of silica aerogel preparation is sol-gel process using organic silicon monomer, such as tetramethyloctethosilicate (TMOS), tetraethylorthosilicate (TEOS) or polyethoxysiloxane (PEDS) as precursors. However, such organic precursors are so expensive that aerogel production in an industrial scale is not economically viable [1,4]. In the present study, we report upon our efforts to synthesize silica aerogel through ambient pressure drying using perlite as the silica source.

2. Experimental

Sieved perlite powder (Table 1.) was mixed with 250 ml 1 mol.l⁻¹ NaOH aqueous solution. The mixture was refluxed for 2 h. Most of the perlite was dissolved in NaOH solution. The solution was filtered to remove the undissolved residues. The sodium silicate solution was neutralized using 250 ml 1 mol.l⁻¹ HCl solution to form silica gel at pH 7. The prepared gel was aged at room temperature for 24 h under sealed condition. The aged gel was washed using de-ionized water to remove Na and Cl ions. The extent of Na removal was measured by analyzing Na-content of dried gel using EDS. Subsequently, the silica gel was soaked in a solution of 20 vol% H₂O/ethanol for 24 h at 50°C followed by aging with ethanol at same condition. The ethanol treated gel was aged in a solution of 70 vol% TEOS/ethanol for 24 h at 70°C. Finally, modified gels were aged for another 24 h inside n-heptane at room temperature before air drying. The gel was dried in 48 h interval at 90°C with partially covered condition. Fourier transform infrared spectroscopy (FTIR, Spectrum RX-1, Perkin Elmer) was employed to investigate the chemical bonding state of surface modifying agent with aerogels.

3. Results and Discussion

3.1. Morphology of the silica aerogels

In the preparation process, it was found that the prepared silica aerogel powder products by sol-gel method were effected using HCl catalysis. Fig. 1 shows the SEM photographs of perlite raw material (Fig.1a.) and produced silica aerogels powder (Fig.1b-d). It can be seen that the SEM images provide more information about the particle morphology of silica aerogels. While the perlite powders have a sticky shape; produced silica airgel powders consist of spherical agglomerate powders. The produced silica aerogel powder was observed by field emission scanning electron microscope (FESEM) (Fig. 2) because the pore size is not clear due to the limits of SEM resolution. The silica aerogel exhibited a pearl necklace morphology and a three-dimensional network which was constructed by nanometer-sized silica particles.

Table 1. Chemical composition of perlite

<table>
<thead>
<tr>
<th>Bileşenler</th>
<th>Miktar (a.q.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>74</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>14.33</td>
</tr>
<tr>
<td>Na₂O</td>
<td>4.97</td>
</tr>
<tr>
<td>K₂O</td>
<td>4.95</td>
</tr>
<tr>
<td>CaO</td>
<td>0.50</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>0.97</td>
</tr>
<tr>
<td>MgO</td>
<td>0.28</td>
</tr>
</tbody>
</table>

(a) (b) (c) (d)

Fig. 1. SEM photographs of a. perlite raw material b-d. produced silica aerogels powder

(a) (b)

Fig. 2. FESEM photographs of silica aerogel powders
3.2. FTIR analysis

Fig. 4 shows the FTIR spectra of the silica aerogel powders. The peaks at 3435 and 1630 cm\(^{-1}\) correspond to the O–H absorption band, which is caused by physically adsorbed water. The absorption peaks near 1091 and 463 cm\(^{-1}\) are due to Si–O–Si vibrations, which will appear in any silica products. It is obvious that apart from Si–O–Si and O–H absorption peaks, the absorption peaks at 2963, 1256 and 846 cm\(^{-1}\). The residual Si–OH groups are the main source of hydrophilicity of the aerogels. It can be seen from the figure that with the increase of alkyl groups in the SA, the intensity of the C–H absorption peaks increase, clearly indicating the replacement of surface H from the Si–OH by the nonhydrolyzable Si–R3 groups resulting higher hydrophobicity [2].

![Fig. 4. FTIR spectra of a. Perlite and b. SiO\(_2\) aerogel powder](image)

3.3. XRD analysis

Fig. 5 shows XRD profiles of the silica aerogel and perlite powder. Both perlite powder and silica aerogel powder have amorphous structure.

![Fig. 5 XRD profiles of the a. silica aerogel b. Perlite powder](image)

<table>
<thead>
<tr>
<th>Element</th>
<th>Bileşim</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>52.915</td>
</tr>
<tr>
<td>Na</td>
<td>1.795</td>
</tr>
<tr>
<td>Al</td>
<td>2.147</td>
</tr>
<tr>
<td>Si</td>
<td>43.143</td>
</tr>
<tr>
<td>Toplam</td>
<td>100.000</td>
</tr>
</tbody>
</table>

![Fig. 3. SEM-EDS analyses of silica aerogel powders](image)

Generally, hydrophilic silica aerogel consists of surface OH groups. If the OH groups are modified to O–Si–(CH\(_3\))\(_3\), then the atmospheric pressure method can be used to get the hydrophobic aerogels, otherwise while drying the unsilylated alcogels, the OH groups can condense and hence the gel shrinks leading to xerogels. In the drying process, evaporation of hexane from the silica network starts as soon as the wet gels were kept for drying. The decrease in relative volume of the wet gels with temperature up to 100°C is because of vaporization of the hexane from the gel network. When the hexane completely evaporates from the gels, the gels shrink to their lowest volumes. Further increase of temperature to 150 °C, because of the volume shrinkage of the gels, when the CH\(_3\) groups of the surface modified gels come nearer, there will not be any condensation among the end CH\(_3\) groups, instead there is a repulsive force acts between the organic groups, therefore the gels start to increase in volume, “spring back”. Therefore, the surface modification is the key and important process in the preparation of ambient pressure dried silica aerogels [2].

![Table 2. Some properties of silica aerogels](image)
4. Conclusions

Silica aerogels could be synthesized by using perlite as raw materials via ambient pressure drying. A kind of sodium silicate solution could be obtained by hydrothermal reaction of perlite with sodium hydroxide solution. The weight ratio of sodium hydroxide to perlite, concentration of sodium hydroxide solution and reaction time have great effects on the modulus of the obtained sodium silicate solution.

From our research on the synthesis of silica aerogels via ambient drying by using perlite, the following conclusions can be drawn:

1. Silica aerogels powder was successfully prepared from perlite powder via ambient drying. The obtained silica aerogels powders showed properties of 0.497 g/cm³ density and meso size particles.
2. The surface modification and strengthening of wet gel was obtained by aging it in TEOS/ethanol solution. Low surface tension liquid n-heptane was used to suppress capillary stresses and associated shrinkage during ambient pressure drying of the gel.
3. The process of aerogel production from perlite by ambient pressure drying method is very important from the industrial point of view and it will significantly widen the commercial exploitation of the silica aerogel.

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References