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Effect of Annealing Temperature on Physical Properties of Xerogel Structures Prepared by Cryogenic Compression Technique

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Abstract

In this work, xerogel materials were synthesized by the cryogenic compression technique and the effects of annealing temperature in the range of 100-1000°C on their physical properties were introduced and studied. Results showed that the porosity continuously decreases from its maximum at 85% to its minimum at <15% with increasing temperature due to the merging, sintering, and collapse of porous structure. An inverse relationship between porosity and relative hardness was observed as the hardness at 1000°C increased by 8 times than its value at 100°C. These results present an important understanding for the relationship between heat treatment (annealing) and xerogel characteristics, which assists in the preparation of xerogel materials for specific applications such as insulation or catalysis.

Keywords: Xerogel structures; Cryogenic compression technique; Annealing effect; Mechanical properties

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1. Introduction

The most recent decades witnessed an increasing development in the field of advanced materials in order to satisfy the increasing needs in various sectors such as energy, environment, medicine, and electronics. Among these materials, xerogels has been explored as a promising category of dried gel materials those combine unique characteristics making them very interested by the researchers and engineers [1]. Xerogel is a form of gel materials that is dried by conventional methods (usually under atmospheric pressure) to induce a shrink in the gel network while maintaining partially the porous structure [3]. Despite the xerogels show lower porosity than aerogels, they show easy preparation, relative mechanical stability, and low production cost, which make it an attractive choice for many industrial and research applications [4,5].

Xerogel is a solid gel material that can be produced by drying the wet gel (alcosol or hydrogel) under atmospheric pressure and room temperature (or slightly higher temperatures) as the solvent is gradually evaporated. This drying process leads to drastic shrink in the size of the gel network due to the capillary forces, therefore, a solid structure containing micropores and mesopores is finally formed with specific surface area ranging in 100-1000 m^2/g [6-8]. The xerogels mainly differ from aerogels dried under critical conditions to avoid the collapse of the porous structure, while they differ from the conventional organic gels in their porous internal structure [9].

It is necessary to explain the differences between xerogels and aerogels and hydrogels. Hydrogel is a polymeric network containing large amounts of water, while xerogel is the dry form after removing water or solvent [10-12]. Aerogel is produced by supercritical drying, which prevents the collapse of the porous structure. Accordingly, the aerogel keeps its original size and very high porosity (>90%), whereas xerogel reasonably shrinks (70-90% of the original size) with relatively lower porosity (30-50%). However, xerogel shows relatively better mechanical strength than the brittle aerogel. Moreover, xerogel can be easily prepared without sophisticated instruments or harsh conditions [13-15].

Mechanical properties of xerogels can be considered the most important challenges to use xerogels as they have directly effect on material's strength and its applicability. In its pure form, xerogel (especially silica xerogel) is a solid but relatively brittle material as it can be fractured under low tensile or bending stresses. This brittle behavior is attributed to the nature of covalent bonds among material's molecules [16]. The Young's modulus of silica xerogel is between 0.1 and 10 GPa depending on the density and porosity. Xerogels can hold reasonable compressive stresses between 5 and 100 MPa, which allows using them in applications not including high tensile or bending stresses [17]. The compressive strength can be enhanced by increasing the density (i.e., reducing porosity) or by adding reinforcing materials. In organic or hybrid xerogels, specific elastic properties can be produced as some categories can hold distortions of 10-20% before fracture, while inorganic xerogel is usually lower elastic [18]. The mechanical properties of xerogels can be modified throughout several methods [19]. Organic polymers can be added to form organic-inorganic hybrids. Reinforcing fibers (silica, carbon, or glass fibers) can be added. Fine control of drying process can reduce the internal stresses. Post-preparation heat treatments (partial sintering) can increase the bonding among particles [20].

Due to their unique characteristics, xerogels are widely used in various applications. The large surface area and porosity of xerogels are very useful to adsorb pollutants from water and air. Chemically-modified silica xerogel is used to adsorb heavy metals (such as lead, mercury and cadmium), organic pigments, and drug pollutants [21]. They also are used to treat the petroleum leakages due to their ability to adsorb hydrocarbons. Xerogels are used as catalyst support due to their large surface area and thermal stability [22]. Metallic nanoparticles (such as platinum, palladium, and gold) can be maintained on the xerogel surface to operate as a high-efficient catalyst in oxidation, reduction, and polymerization reactions [23]. Xerogel itself can also be used as an inhomogeneous catalyst after the incorporation of catalytic-active groups. Despite that the xerogel is lower insulating than the aerogel, it still good insulating material with low production cost [24]. Xerogel can be as an insulating layer in buildings (as plates or coatings) and industrial applications such as transmission lines and storing tanks [25]. Some types of xerogels have acoustic insulating properties due to the porous structure [26]. In biomedical applications, xerogel is used as a base for drug delivery systems as the drug can be loaded inside the pores and then controllably released. Polymer xerogel, such as chitosan and gelatin, can be used in tissue engineering as scaffolds to support cell growth and damaged tissue regeneration [27]. Xerogels are employed in promising applications such as supercapacitors and batteries as they are used as electrodes or separators due to their porosity and good ionic conductivity [28]. They are also used in the fabrication of gas sensors as they change their electrical behavior upon adsorbing specific gases. Xerogels are included in the structures of paintings and functional coatings that make the surfaces anti-reflecting, scratch resistant, and easily cleaned (self-cleaning) [29]. Xerogel coatings are also used to protect metallic surfaces from corrosion. In agriculture, xerogels are used to enhance the ability of soil to keep water and nutrients, especially in dry environments, due to their ability to absorb and slowly release waters. They are also used in seed shielding for protection and enhanced implantation [30].

In this work, xerogel was synthesized by cryogenic compression technique and the effects of thermal annealing on porosity and relative hardness of the synthesized xerogels were determined and analyzed. The synthesized xerogels showed easy production, low production cost and reliable performance.

2. Experimental Part

The cryogenic compression technique is one of the innovative method to prepare xerogels. This technique combines the deep freezing and controllable compression to produce porous materials with unique characteristics. This technique conceptually differs from conventional drying methods under atmospheric pressure as well as supercritical drying as it depends on maintaining the gel structure at frozen state before removing the solvent. This limits the collapse of the pores and enhances the mechanical properties of the final product.

The main idea of this technique is based on the attempt to avoid the effects of capillary forces those may destroy the porous structure during drying process throughout the conversion of solvent into solid state and then remove it by sublimation under controlled pressure conditions. This technique has been developed and employed to produce eco-friendly highly-efficient porous materials. This technique is based on a simple but effective physical principle. When the wet gel (hydrogel or alcosol) is frozen, the solvent molecules form ice crystals inside the polymeric network. The size and distribution of these crystals can be controlled via the cooling rate and applied pressure during freezing process. Beyond the completion of freezing, the frozen solvent is removed by sublimation under low compressive pressure leaving voids to reasonably keep the original structure. Figure (1) shows a flow chart of the experimental part of this work, while figure (2) shows a photograph of the synthesized xerogel samples.

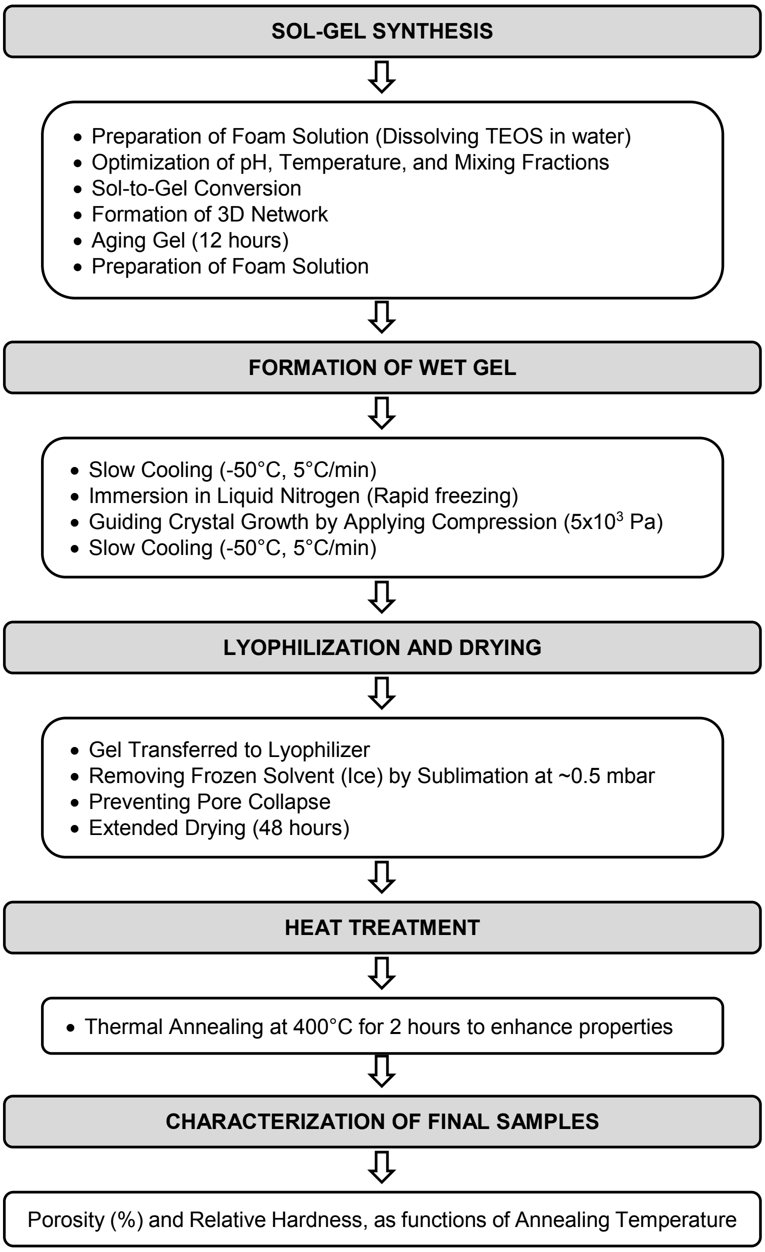

Fig. (1) Flow chart of the experimental part of this work

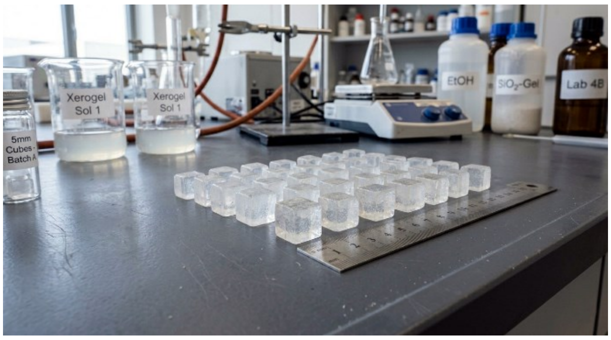

Fig. (2) Photograph of the synthesized Xerogel samples

3. Results and Discussion

Figure (2) shows the relationship between the annealing temperature and room-temperature porosity of the synthesized xerogel sample in the temperature range from 100 to 1000°C. In general behavior, the porosity uniformly decreases with annealing temperature from 85% at low temperatures (<100°C) to reach 15% at 900-1000°C. This behavior is expected and indicating the effect of heat treatment on the porous structure of the synthesized xerogels. The gradual decrease in porosity is attributed to several physical and chemical mechanisms occurring during the annealing process. The most effective four mechanisms are explained herein. In the first mechanism, at low annealing temperatures (<300°C), the unreactive surface functional groups such OH^- starts to separate and hence the xerogel particles converge causing the pores to slightly shrink and hence the porosity to limitedly reduce (from 85% to 75%). In the second mechanism, at temperatures between 300 and 600°C, the material enters the primary sintering stage as the nanoparticles forming the pore walls are merged at the contact points due to the diffusion of atoms. This phenomenon causes much more apparent decrease in porosity (from 75% to 40%) with increase in material's density and decrease in average pore diameter. In the third mechanism, at temperatures of 600-800°C, a gradual collapse of mesopores and micropores occurs leading to sharp decrease in porosity (20-30%). This stage represents a reasonable loss of the original porous structure maintained by cryogenic compression technique. According to the fourth mechanism, at 900-1000°C, the sintering is approximately completed and the material converts into glassy or dense crystalline structure with very low porosity (~15%), which is close to the theoretical density of the solid material.

In summary, figure (2) clearly shows that increasing annealing temperature leads to gradual then increasing decrease in xerogel porosity as a result of the particles incorporation, sintering, and porous structure collapse. These data present a guidance to select the appropriate heat treatment conditions according to the aimed application as a balance is required between high porosity and thermal stability.

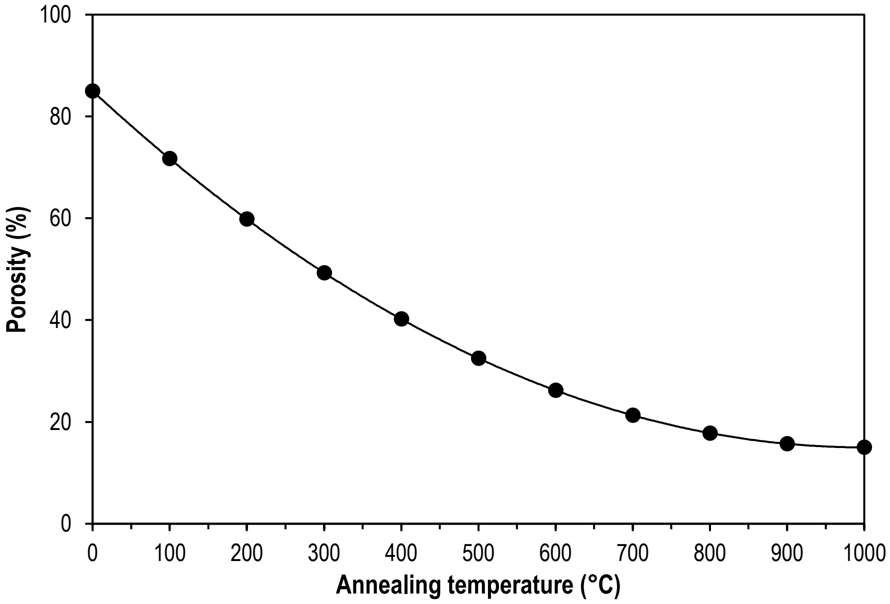

Fig. (2) Variation of xerogel percentage porosity with annealing temperature

Figure (3) shows the variation of relative hardness of xerogel samples synthesized in this work with the annealing temperature in the range 100-1000°C. This chart can be divided into three distinct thermal regions, each obeys a physical mechanism responsible of increasing hardness. In the first region (100-300°C), the increase in hardness is relatively small. In this range of temperatures, some fundamental processes occur as follows. The evaporation of the residual solvent and water adsorbed inside pores is mostly possible. As well, the separation of the surface functional groups (such as OH^-) starts with slight increase in the bonding between particles without complete merge. Here, xerogel still highly porous, therefore, the hardness is low and the material is brittle. The second region (300-700°C) represents the most dynamic stage as sintering process reasonably starts as the particles are merged at the contact points that increase the number of bonds among these particles. Also, the average particle size increases, the specific surface area decreases, and then the porosity reasonably decreases. The collapse of micropores and mesopores leads to increase the density of the sample and hence its resistance to the mechanical distortion, which explains the increased hardness. The rate of increasing hardness gets faster beyond 500°C. In the third region (700-1000°C), the material reaches an advanced stage of sintering as the pores completely disappeared (porosity <15%). The hardness sharply increases to reach its maximum at 1000°C, which is 8 times the hardness at 100°C.

By comparing figures (2) and (3), an apparent reciprocal semi-linear relationship is observed between porosity and hardness. At high porosity, the hardness is at the minimum because the thin walls between pores provide a low resistance to distortion. At medium porosity, the hardness is at the medium too as the number of contact points among the particles is increased. At low porosity, the hardness is at the maximum as the material is approximately solid without internal voids. Such relationship is expected in porous materials as the porosity is the main factor that determines the mechanical properties and it can be mathematically described by Gibson-Ashby model of foams, in which the hardness is directly proportional to the cubic relative density (or reciprocally to the porosity with specific exponent).

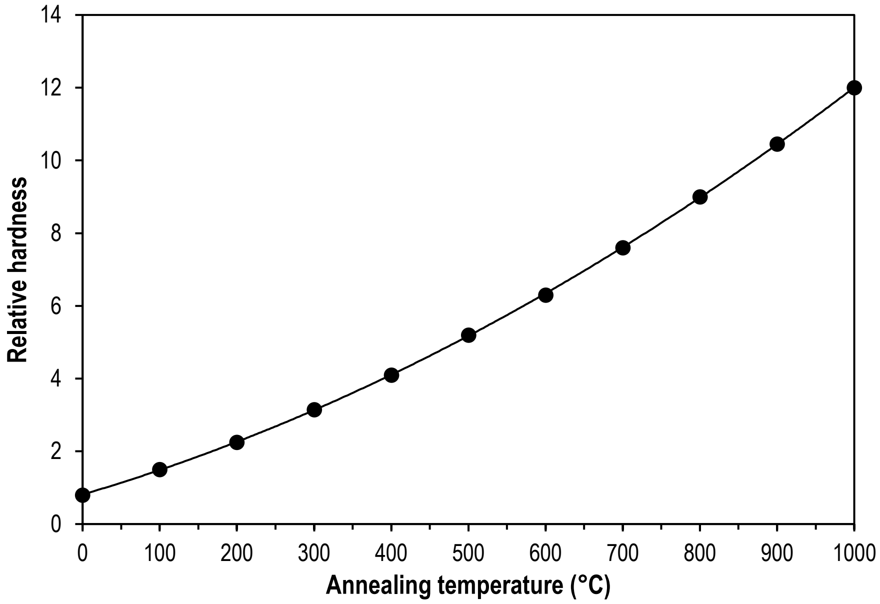

Fig. (3) Variation of the relative hardness of xerogel with annealing temperature

4. Conclusion

In concluding remarks, increasing heat treatment temperature leads to gradual decrease in xerogel porosity from 85% to 15% as a result of sintering process and particles merge. An inverse relationship between porosity and relative hardness is concluded as the relative hardness has increased 8 times as the annealing temperature was increased from 100°C to 1000°C. The characteristics of the synthesized xerogels can be controlled by selecting the appropriate annealing temperature for the desired application.

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