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MECHANICAL PROPERTIES OF SILICA-CELLULOSE AEROGELS

RÄNI- JA TSELLULOOSI AEROGEELIDE MEHAANILISED OMADUSED

BACHELOR THESIS

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Antud uurimistöö eesmärgiks oli uurida lisatud tselluloosi mõju räni aerogeelide mehaanilistele omadustele. Eesmärgi saavutamiseks valmistati aerogeelid erinevatel tingimustel erineva tselluloosi sisaldusega. Mehaanilisi omadusi hinnati mõõdetud elastsuse moduli põhjal.

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List of abbreviations

TMOS - Tetramethoxysilane
MTMS - Methyltrimethoxysilane
MeOH - Methanol
NH_4OH - Ammonium Hydroxide solution
NFC - Nanofibrillated Cellulose
SCD - Supercritical Drying

ABSTRACT

Aerogels are interesting class of the materials which are nanoporous solids formed by replacing liquid in a gel with gas. This material is porous, light but strong enough. Due to their properties aerogels have found the application in different fields such as for example, as insulators in homebuilding as well as space applications. The aerogels are good absorbents of both oil and water.

Mechanical properties of aerogels depend not only on the components of the aerogel but also on the drying method of the prepared aerogels. By adding the various fillers it is possible to change the properties of the aerogels.

The focus of the present study was to investigate the mechanical properties of the silica aerogels with added cellulose in different concentrations. Two groups of the prepared aerogels, with different composition without and with adding cellulose, were studied. The influence of the drying method of the aerogels on the mechanical properties was also investigated. The mechanical properties of the aerogel materials were estimated by elasticity modulus. It was found that the elasticity of the aerogels depend on the cellulose concentration in the sample. The drying method of the aerogels samples also influences the mechanical properties. The optimum concentration of the added cellulose cellulose has been found to get the most elastic aerogels.

INTRODUCTION

The aerogel is light nanostructured porous material, where gas replace the liquid phase. Samuel Stephens Kistler invented this material in 1930s by using the sol-gel synthesis method and drying process of the gel was performed under critical conditions for obtaining the desired structure of aerogel sample. Due to porous structure, aerogels have unique properties for that kind of materials. They are heat-proof, sound-proof and have low density; they are strong enough to keep the form but have low elasticity and are very fragile. Cellulose as natural polymer is used to modify silica-aerogels to make them more stable and plastic. Today silica-cellulose aerogels are used for making new kind of batteries which will be light and with the high capacity, also for the building materials and clothes and at the same time they help recycle the wasted paper. Up to very little literature data have been found how the cellulose concentration influence on the mechanical properties of the silica-cellulose aerogels. The aim of this study is to investigate the effect of cellulose concentration on the mechanical properties of aerogel. The study is focused on reviewing the topical literature of aerogel types, properties, applications and methods of preparation. Experimental part is included material preparation with different cellulose concentration before mechanical testing and the measuring the mechanical properties.

LITERATURE REVIEW

1. AEROGELS

Aerogel (from lat.aer-air and gelatus frozen) is one of the new and insufficiently exploredporous material classes. This material structure is formed by nanoporous solids and liquid, which is replaced in a gel with gas [1]. The most common propotion of air and solid materials in aerogels is 99.8% of the air and 0.2% of solid material [2]. Aerogel does not have a designated material with set chemical formula, but the term is used to group all the material with a certain geometric structure [3].

Samuel Stephens Kistler was the first scientist, who investigated the first aerogel in 1930 [2]. He used sodium silicate for low-cost aerogel production and invented the general material for commercial synthesis of silica aerogel by using supercritical drying (SCD) method [4]. He spent large part of his life studying aerogels properties and uses.In 1980´s the first carbon aerogel was presented also by Samuel Stephens Kistler [2].

In 1985, Stanislaus Teichner was revived sol-aerogel technology by producing silica aerogel for the storage of rocket fuels at Universite Claud Bernard [4]. Development of a practical form of aerogel was pursued by NASA in 1992 [1].

The aerogels are separated in three groups: inorganic, inorganic-organic and organic [5]. There are most common types of inorganic aerogels: silica, metal oxides and carbon aerogels [6]. Aerogels can be modified widely with different fillers, for example carbon, iron oxide, organic polymers, semiconductor nanostructures gold and copper [2].

Because of aerogels have such unique properties like extremely low density and low thermal conductivity. Due to these properties, aerogels are used in space, electronic components and architecture [3].

They have found the largest use in practical and experimental applications comparing to other aerogel types because of the properties, which are studied more thoroughly [6]. For example,they are used in housing, refrigerators, skylights, windows, also in clothing, apparel and blankets [7].

1.1 SILICA AEROGELS

Silica aerogel is the most studied aerogel material. Silica aerogel is principally specially prepared silica or silicon dioxide [8]. The silica aerogel was the first aerogel, which was created by Samuel Stephens Kistler in the 1930s and had little development for next decades.

Silica aerogel is described as a porous material with high specific surface area, high porosity, low density, low dielectric constant and excellent heat insulation properties [4]. The structure of silica aerogel is a nanostructured solid network, which is formed in a liquid reaction medium as a result of a sol-gel polymerization process.

Silica aerogel is chemically inert and non- harmful to the human. Due to these properties, this type of aerogels can be used in pharmaceutical industry and agriculture [9].

Most of literature data refer to the application of aerogels in insulate electronics on the MARS rovers and to using them to collect space-dust and comet particles returned to EARTH for analysis [10].

Silica aerogel had 15 the place in the Guinness Book of World Records, till 2011, for being the lowest density solid and the best insulator. The melting point of silica aerogel is 1200 ºC. Also it is so strong, that can support thousands of times its own weight [11].

Nevertheless, despite the fact that the aerogel was invented almost hundred years ago, the scope of its application is currently limited. First of all, this is due to a very high price. The cost of the initial materials for aerogel is about $ 1000 per cubic centimetre, and this is not including serious time costs. For today the aerogel is much more expensive than gold. The second drawback is an excessively low plasticity, aerogels are very fragile. They will withstand pressure, but do not blow [12].

1.2 SILICA- CELLULOSE AEROGELS

Silica-cellulose aerogels can be considered as class of modified aerogels. The first mention of silica-cellulose aerogels was by Chibing Tan in 2001 [5].

Silica-cellulose aerogel is usually based on nanofibrillated cellulose (NFC). NFC aquatic dispersion contains disintegration of pulped wood fiber cell walls. NFC preparing includes mechanical disintegration of wood pulp, which contains mostly cellulose.

The difference between silica aerogels and silica-cellulose aerogels is in their structure. The comparison of molecular structures of silica aerogels and silica-cellulose aerogels is presented in Figure 1.

Inorganic aerogels are usually very fragile; therefore hybrid aerogels have been developed to improve aerogel elastic properties by adding organic polymers [13].

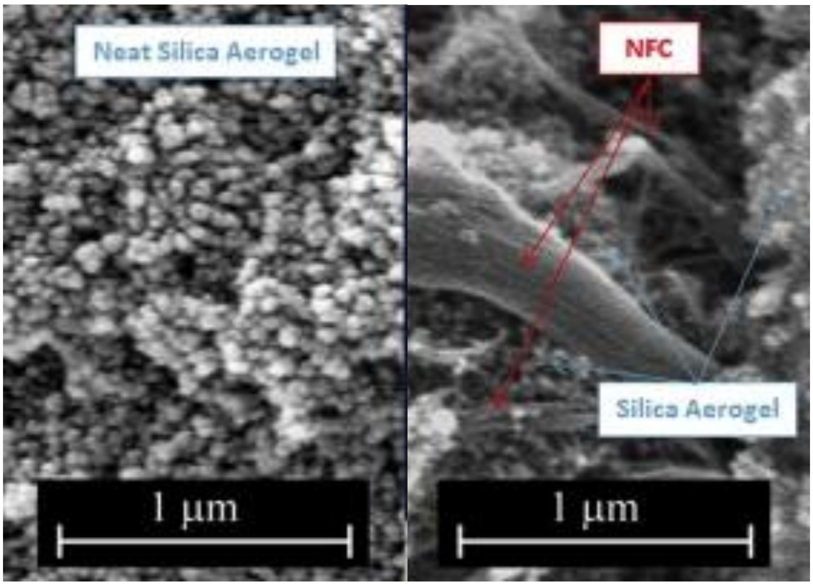


Figure 1.Silica and silica cellulose aerogels and their molecular structure [14].

The cellulose aerogel is composed of cellulose fibrils, which are typically less than 10 nm wide. The cellulose aerogel, which is approximately 0.35 mm thick, was fairly transparent, as a result demonstrating its homogeneous nanoporosity [15].

A new composite aerogel demonstrates an interesting combination of useful properties: mechanical stability, flexibility and low thermal conductivity, translucency and compatibilitywith biological materials [16].

Due to the improvement in connection with NFC, thechange in composite of aerogels was observed. Tensile strength of these aerogels was 25-40% higher than the tensile strenght of aerogels with pure silica; despite they were of equal densities [14].

Scientists from the National University of Singapore (NUS) used waste paper to create an aerogel. NUS technology, however, is much more environmentally friendly cleaner, using less energy, releasing less toxic emissions and requiring less hazardous chemicals - plus it uses material that otherwise can go to landfill.

The exact method of production is property, but it is known that it is associated with the transformation of cellulose harvested from paper waste into an aerogel. The process takes three days and leads to the biological decomposition of the material, which is non-toxic, flexible, mechanically strong and oil-absorbent.

The cellulose aerogel which coated with MTMS becomes very hydrophobic (water repellent). This means that if it were placed in an oil spill, it could absorb 90 times its dry weight of crude oil, without filling with water. Then it can be wrung out like a sponge, which allows you to recover more than 99 percent of the absorbed oil. The material can also find application in the warming of walls in buildings. In addition to preserving the heat contained in the structure, it will resist the accumulation of moisture, add strength to the walls and take up less space than traditional materials such as fiberglass. It can also be used as a form of protective packaging. Alloyed with metallic nanoparticles and compressed without air to become flat, the aerogel can be further converted into a mechanically strong thin magnetic film. The high-porous aerogel which is not coated with trimethoxy-methylsilane absorbs water and other liquids, which allows its use in products such as diapers or sanitary napkins [17].

1.3 PROPERTIES OF AEROGELS

The structure of any substance gives certain properties to the material and the uniqueness of aerogels lies in its properties.

This kind of material is breathable and fireproof and it absorbs both oil and water. Aerogels are suitable for use as great electrical conductors and they are also one of the best insulators today [18].

Aerogel is amazingly strong, considering its weight.They are translucent in appearance, the touch is reminiscent of a light but firm foam, because they have high porosity (~99%). Aerogels also have high strength (can withstand a load of 2000 times of their own weight), but at the same time their density reaches only 0.3-0.03 g / cm^3 (many times lighter than fluff).

Aerogels, especially quartz ones, are good heat insulators, because of low thermal conductivity (~0.01 W/m.K) and low dielectric constant (~1.0–2.0).

They are very hygroscopic, effective absorbers of sunlight and absorb toxic heavy metals, etc. Their sound velocity is low - 100 m/s, that is suitable for using aerogels as soundproof material [7]. For comparison, the dielectric constant of water is 80,4 and sound velocity is 1480 m/s [19].

1.4 METHODS OF PREPARATION OF AEROGELS

The main process to make aerogels is extracting the liquid component of a gel through supercritical drying. The method of obtaining an aerogel is based on the removal of liquid from the gel at the temperature and pressure above the critical one. In this case, the liquid passes into the vapour directly through the pores of the material, which excludes pore compression due to surface tension forces. In order to reduce the operating temperature and pressure, water in the hydrogel is pre-substituted with ethyl or methyl alcohol [20].

Sol-gel process is the transition from a liquid “sol” into a solid “gel” phase. The sol-gel process is divided into the following steps like forming a solution, gelation, aging, drying and densification [7].

The synthesis of silica aerogels has 3 stages of the process such as gel preparation, aging of the gel and drying. Schematically the process of the syntesis is demonstrated in Figure 2.

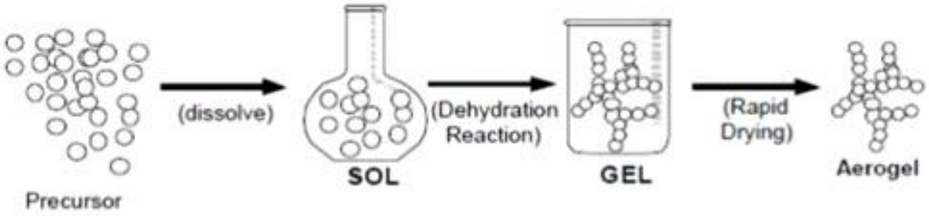


Figure 2. Schematic representation of sol-gel process of synthesis of aerogels [21].

GEL PRERARATION

Gelation is the process where solution is converted into 3D solid network. The gel is a semisolid which is rich in liquid. It is interesting that the liquid does not allow destroying a solid network and a solid network does not allow the fluid to flow out. The gelation point is determined by a sharp increase in viscosity and an elastic reaction to stress. To obtain aerogels, it is most convenient to cause gelling by changing the pH of the reaction solution. The mechanical state of the gel strongly depends on the number of cross-links in the network [7].

AGING OF THE GEL

After gel formation, the Si-O-Si network is formed by the hydrolysis and condensation reactions. The term "aging" refers to the strengthening of the gel network. This may include further condensation, dissolution and precipitation of sol particles or phase transformations in the solid or liquid phases. This leads to a porous solid in which the solvent is captured. This aging process strengthens the gel, so shrinkage during the drying phase is minimized [7].

DRYING OF THE GEL

In this step the liquid from the gel pores should be released. When the liquid begins to evaporate from the gel, concave menisci are created by the surface tension in the pores of the gel. Finally, surface tension causes the collapse of the gel body. To prevent build-up of surface tensionthe gel is dried in an autoclave under supercritical conditions as shown in Figure 3. When the temperature and pressure in the autoclave increase above the critical point (for methanol the critical temperature and the critical pressure values are 243 ºC and 7.9 MPa, respectively), liquid turns into a "supercritical" fluid in where each molecule can move freely and surface tension ceases. After the vapours should be slowly released from the autoclave until the pressure in the autoclave reaches atmospheric pressure. This method of drying alkogels is called "supercritical drying" [7].

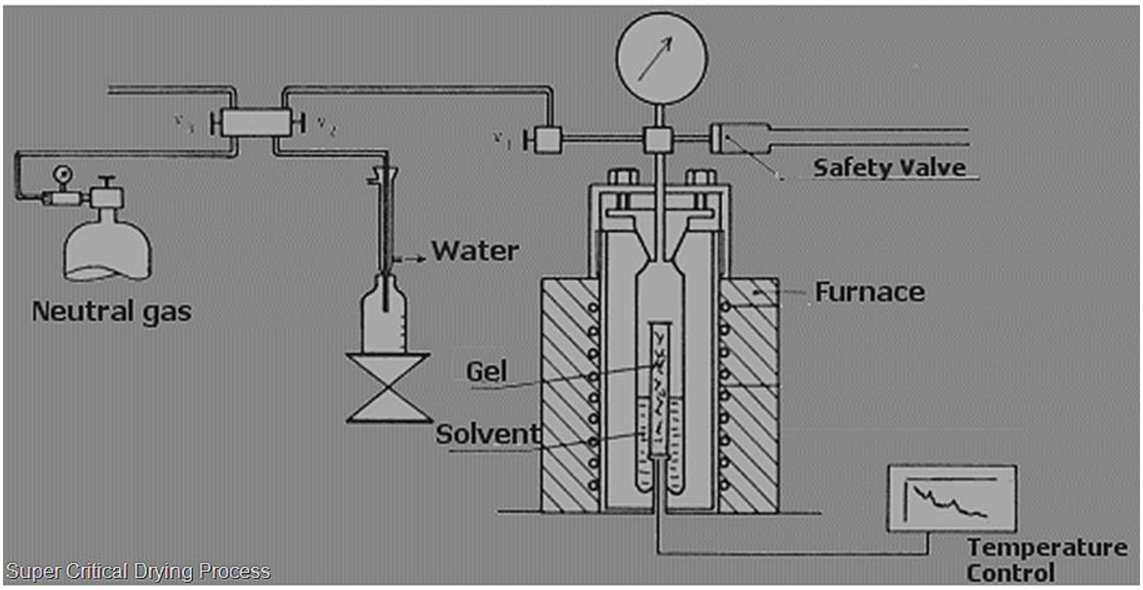


Figure 3. Schematic representation of supercritical drying autoclave [22].

These three general steps are involved in all methods of aerogel production. There are also additional procedures, which influence the changes in final product structure [4].

SILICA-CELLULOSE AEROGEL PREPARATION

Process of silica-cellulose aerogel preparation is the same as silica aerogel preparation. First of all, it is necessary to get a gel solution. The main solution includes: NCF mass (cellulose), methanol (MeOH), tetraethyl orthosilicate (TMOS) and the last one, which should be added, is ammonium hydroxide solution (NH_4OH). All components should be mixed and stayed at night. Figure 4 shows schematically the process of gelation.

The cellulose hydrogel is a transparent material where water content is 92% and porosity is about 95%. The sol-gel process is catalysed by ammonia where SiO_2 precipitates on the cellulose network (Figure 4 (b)). The composite is converted into aerogel by drying with supercritical CO2 to maintain the porous structure (Figure 4 (c)) resulting in a flexible and translucent cellulose-silica silica aerogel [23].

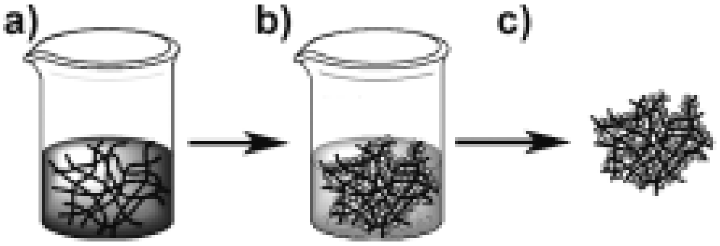


Figure 4. Silica-cellulose aerogel preparation.a) Nanoporous cellulose gel with interconnected nanofibrillar network. b) Silica formation by hydrolysis and condensation (sol–gel process) c) composite aerogel [23].

1.5 APPLICATION OF AEROGELS

Characteristics of aerogels give them a large advantage in applications for gas or liquid permeation and adsorption, optical applications, carriers for catalysis, drug release or thermal and acoustic insulation.

There are nine main fields of industry in which aerogels have found their application, among them thermal and noise isolation technologies, electronics, chemistry, medicine, biology, pharmaceutics, environmental protection, the production of sensors and high-tech instruments, energy, aerospace, space exploration, consumer goods and military technology [24].

Aerogels are used in construction and in industry as heat-insulating and heat-retaining materials for the thermal insulation of steel pipelines, various equipment with high and low temperature processes, buildings and other objects. It can withstand temperatures of up to 650 ° C, and a layer of 2.5 cm thick enough to protect the human hand from direct exposure to the soldering lamp [25].

Aerogel allows significantly improve the thermal insulation characteristics of modern insulating glass units. Especially for frame construction, the material Spaceloft is offered. It is an "alloy" of fiberglass and aerogel 5 and 10 mm thick, with a record heat conductivity of 0.015 W / mK. It is not difficult to calculate that 10 mm of such insulation will replace 42 mm of standard mineral wool.

Also aerogel proved to be a very effective tool for studying the properties of superfluid helium. The injection of super-fluid helium into the pores of the aerogel led to a significant change in its properties.

They are used in microelectronics. Aerogels have the lowest dielectric constants, and their use, for example, as insulating layers in multi-layer printed circuit boards, can significantly improve the performance of electronics.

Aerogel is used to record cosmic dust and small high-speed particles of all kinds of origin. In a collision with a solid such particles melt or even evaporate, the aerogel provides a sufficiently smooth decrease in the velocity of the particles, and, being a transparent material, makes it possible to observe their tracks.

Due to the large total pore area of the aerogel, it is possible to fabricate highly efficient filters on its basis for various purposes, for example, water purification. Today it is used for cleaning sea water from oil spills.

In the meantime, aerogels can be found in a range of products, for example wetsuits, firefighter suits, skylights, windows, rockets, paints, cosmetics and also innuclear weapons. The aerogels are perspective as highly efficient filters useful in the production of sports equipment to strengthen the design. There are also shoes and sleeping bags with thermal pads from aerogel [26].

EXPEREMENTAL PART

2. MATERIALS AND METHODS

2.1 MATERIALS

Two groups of aerogels materials were investigated in the present study. The aerogels of the first group materials were prepared with different content and with various cellulose concentrations. Table 1 presents the materials used in the present study. The aerogels of the second group were prepared with the same content of the main solution, but with different cellulose concentration of 1%, 5%, 10%, 15%, 20% and 25%. The main solution of aerogels contains Tetramethoxysilane (TMOS), Methyltrimethoxysilane (MTMS), Methanol (MeOH) and Ammonia solution (NH_4OH). All aerogels were prepared in the laboratory by using the sol-gel method [7].

Sample Name	TMOS	MTMS	MeOH	H_2O	NH_4OH	Cellulose %	Density, g/cm3
KE2(HF)	1	0	12	4	$3,6*10^{-3}$	0	0,119
KE4	1	0,75	12	5	$5,8*10^{-3}$	0	0,167
KE6	0,4375	1,3125	12	5	$5,8*10^{-3}$	0	0,253
KE7	0,875	0,875	12	5	$5,8*10^{-3}$	0	0,274
KE8	1,325	0,4375	12	5	$5,8*10^{-3}$	0	0,174
KE9	0	1	3,5	4	$3,5*10^{-1}$	0	0,230
KE10	0	1	3,5	4	$1,7*10^{-1}$	0	0,249
KE12	1	0	3,65	0	$4,4*10^{-2}$	1,6	0,221
KE15	1	0	7,29	0	$5,4*10^{-3}$	10,7	0,163
KE16(air)	1	0	12,76	0	$7,38*10^{-3}$	10	0,143
KE17(FD)	1	0	12,76	0	$7,38*10^{-3}$	4,1	0,127
KE18	1	0	12,76	0	$7,38*10^{-3}$	6,9	0,111
KE19	1	0	12,76	0	$4,03*10^{-3}$	10	0,103
KE20	1	0	12,76	0	$1,34*10^{-3}$	10	0,093
KE21	1	0	25,52	0	$7,38*10^{-3}$	10	
KE22	1	0	17,01	0	$7,38*10^{-3}$	10	
KE23	1	0	25,52	0	$7,38*10^{-3}$	15,6	

Table 1. List of firts type of aerogels and their content.

For silica-cellulose aerogel preparation chemicals, Tetramethoxysilane (TMOS), Methanol (MeOH) and Ammonia solution (NH_4OH) were used. The Ammonia solution was of concentration - 1M. For getting aerogels with different concentration of cellulose nanofibrillated cellulose mass was obtained from VTT Technical Research Centre of Finland, with a dry material content of 3.5% cellulose.

2.2 METHODS

2.2.1 THE PREPARATION OF AEROGEL SOLUTION AND SAMPLES

The aerogels used in this study were prepared by using standard procedure [27]. In shortly, the process of the aerogels can be divided into 3 steps. Firstly, the aerogels solutions were prepared by using one solvent, methanol, TMOS as regent and NH4OH was catalyst.

There was also small amount of water for aerogel forming. The properties of the solvents are presented in Table 2.

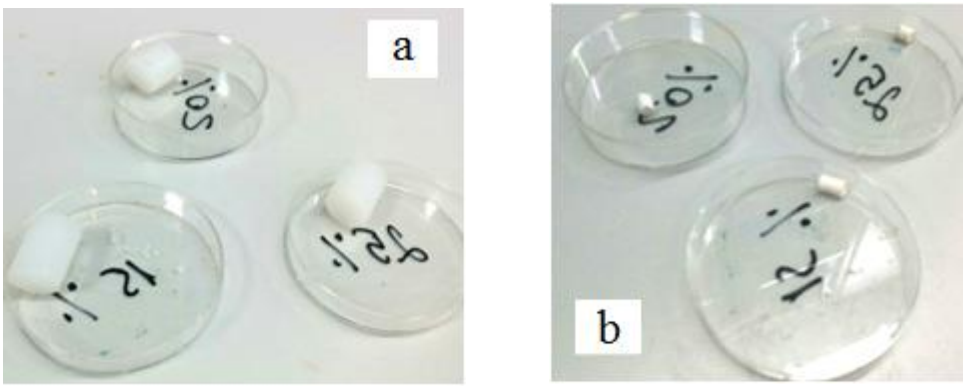
The mixtures of solvents with different volume ratios were prepared by adding various concentration(W/V) of the cellulose mass. The process was performed in the taste-tube. After solutions preparation the aerogel solutions were kept for 24 h for to achieve the gelation.

Name of chemical	MW ,g/mol	Assay , %	Boiling point, °C	Vapor density (vs air), g/cm3	Vapor pressure (20 °C),mmHg
TMOS	152,22	≥ 98,0	121-122	5,25	173.24
MeOH	32,04	≥ 99,9	64,7 (lit.)	1,11	97,68
NH_4OH-solution 1M -	35,05	25-27	38	-	-

Table 2. Chemicals which were used in present study.

Secondly, after achieved gelation the post-process procedure was applied. The aerogels were dried by two different methods [28], under supercritical drying conditions and in the air, respectively. For the supercritical drying methanol was used to fill the pores of the aerogels. The supercritical drying was performed in autoclave at 45 ºC and under 100-122 bar pressure. The time of supercritical drying was 6 hours.

In the air drying conditions the samples were kept in the open air for 24 h. The changes of samples with cellulose concentration of 15%,20% and 25%, occurred after drying in the air are showed in Picture 1(a, b).



Picture 1.(a) Samples 15, 20 and 25 berofe air-drying (b) Samples 15, 20 and 25 after air-drying

When the samples were dried in the air the density of the prepared samples was calculated by using following equation 1(Archimedes et al. around 250 B.C.).

$$\rho = \frac{m}{V} = \frac{m}{\pi r^3 h} \text{ , g/cm}^3 \quad \text{Eq.1}$$

where ρ is the density, m is the mass, and V is the volume of aerogel sample.

2.2.2 MECHANICAL TESTING

The mechanical properties of the prepared aerogels were measured by using the tensile machine Instron 5866. Measurements show the elasticity, which is the property of solid materials, where the material resists its original shape and size after the forces deforming them have been removed [29]. The measurements were performed at 500 N load with a constant strain rate of 4 mm/min.

Before the measurements of the mechanical properties the samples of the aerogels were prepared by cutting the pieces of the same size with equal height and diameter. Air-dried aerogels were stitched to the desired size.

From the got results of the tensile stress and strain the Young's modulus was calculated to estimate the influence of cellulose on the mechanical properties of aerogel. By using equation 2 (Thomas Young , after the 19th-century).

$$\mathrm{E} = \frac{\mathrm{F/A}}{\Delta \mathrm{L/L0}} = \frac{\mathrm{F}/\pi \mathrm{r}^2}{\Delta \mathrm{L/L0}} \text{ , N/mm}^2 \quad \text{Eq. 2}$$

where E is the Young's modulus (modulus of elasticity), F is the force exerted on an object under tension, A is the actual cross-sectional area through which the force is applied, ΔL is the amount by which the length of the object changes and L0 is the original length of the objec

3. RESULTS AND DISCUSSION

3.1 MECHANICAL PROPERTIES OF AEROGELS CONTAINING NO CELLULOSE

In this study, after mechanical test aerogels were divided into 3 parts in order to compare the results.The first group of aerogels were prepared without adding nanofibrillated cellulose. The mechanical properties of this type of the aerogels were estimated by Young's modulus. The results are presented in Table 3. From Table 3 it can be seen that the composition of the aerogels affect drastically the elasticity of the material. The higher the content of the MTMS, the stiffer the aerogel. But at the same time when the content of TMOS reagent increases with increasing MTMS the elasticity of the aerogel decreases. The results in Table 3 show that the content of the aerogels preparation is the critical, the changing of the one of the component amount may significantly influences the mechanical properties.

Sample Name	TMOS	MTMS	MeOH	H_20	NH_4OH	Young´s modulus, kPa
KE4	1	0.75	12	5	$5.80 * 10^{-3}$	17.75
KE6	0.44	1.31	12	5	$5.80*10^{-3}$	19.11
KE7	0.87	0.87	12	5	$5.80*10^{-3}$	10.28
KE9	0	1	3.5	4	$0,04*10^{-3}$	17.17

Table 3.The influence of the chemical composition of the aerogel samples on the elasticity.

3.2 MECHANICAL PROPERTIES OF AEROGELS WITH ADDING CELLULOSE

The second group of aerogels was prepared with different concentration of cellulose to investigate the effect of adding cellulose on the mechanical properties of the aerogels. The results are presented in Tabe 4. As it is seen from the presented results the mechanical properties of the aerogel affected not only by the cellulose concentration but also by the initial composition of the aerogels. 10% of cellulose of the samples KE16 and KE15 demonstrate the highest Young´s modulus. When the ratio of the MeOH and NH_4OH in the composition changes the elasticity of the aerogels decreases despite the same amount of added cellulose,10%.

Sample name	TMOS	MTMS	MeOH	H_20	NH_4OH	Cellulose,%	Young´s modulus , kPa
KE17	1	0	12.76	0	$7.38*10^{-3}$	4.10	22.54
KE16	1	0	12.76	0	$7.38*10^{-3}$	10	239.10
KE19	1	0	12.76	0	$4.03*10^{-3}$	10	19.58
KE20	1	0	12.76	0	$1.34*10^{-3}$	10	28.55
KE21	1	0	25.52	0	$7.38*10^{-3}$	10	5.67
KE22	1	0	17.01	0	$7.38*10^{-3}$	10	3.38
KE15	1	0	7.29	0	$5.40*10^{-3}$	10.70	135.58
KE23	1	0	25.52	0	$7.38*10^{-3}$	15.60	3.10

Table 4. The influence of adding cellulose on the mechanical properties of the aerogels.

3.3 THE INFLUENCE OF THE DRYING METHOD ON THE MECHANICAL PROPERIES OF THE AEROGELS

The silica-aerogels with the same composition [Tetramethyl orthosilicate (TMOS), Methanol (MeOH) and Ammonia solution (NH_4OH)- 1M] but with differentcellulose concentration, ,were dried by using two different methods, in the open air and under supercritical conditions. The influence of the drying method on the elasticity of the aerogels with different cellulose concentration was estimated by measuring Young´s modulus. The results are presented in Figure 5 and 6. Figure 5 demonstrates the mechanical properties of the aerogels dried under supercritical conditions.Figure 6 demonstrates the mechanical properties of the aerogels dried in the open air conditions.

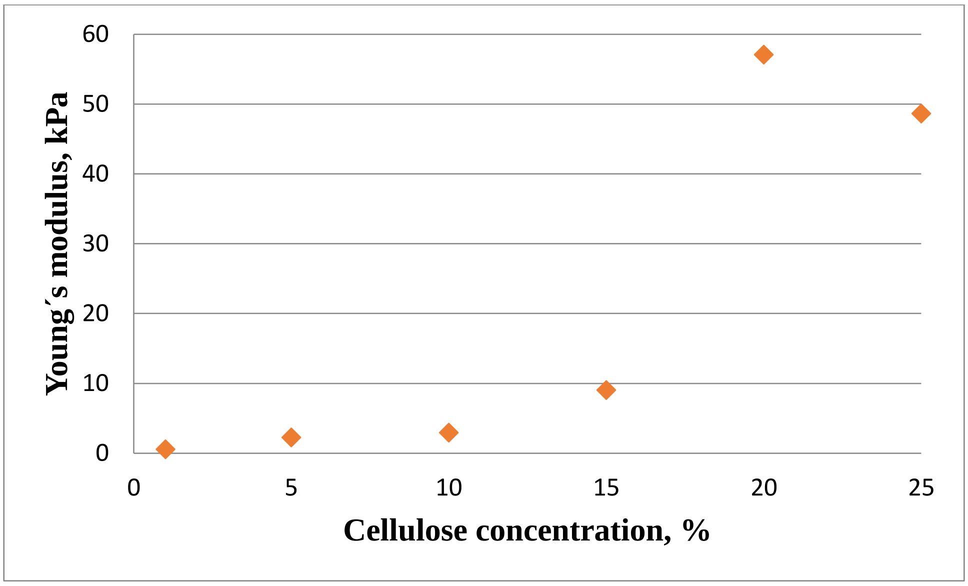


Figure 5. The influence of the cellulose concentration of the aerogels dried under supercritical conditions on the elasticity.

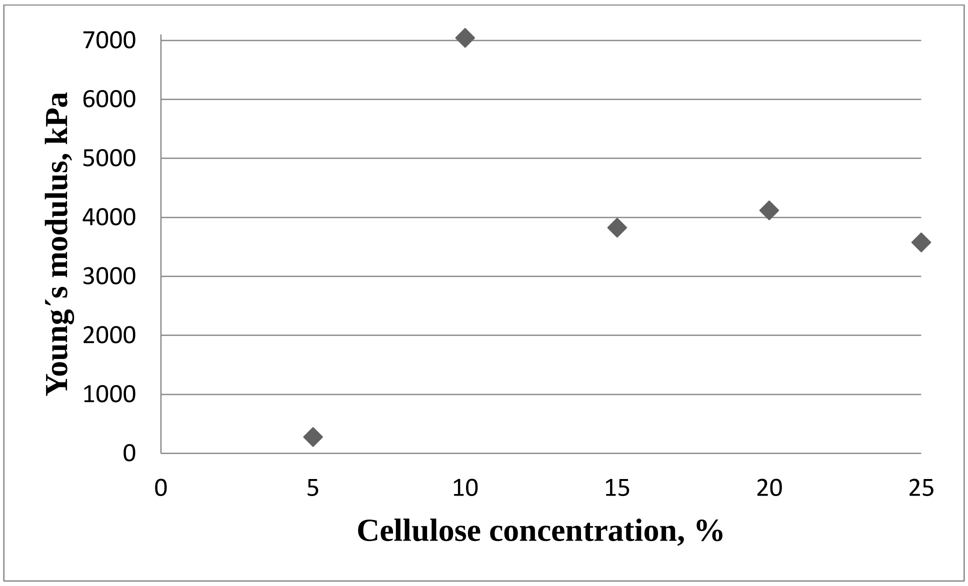


Figure 6. The influence of the cellulose concentration of the aerogels dried in open air condition on the elasticity.

Comparing two figures, 5 and 6, it is seen that the elasticity modulus of the aerogel dried in the open air (Figure 6) is higher about 100 times than the elasticity modulus of the aerogels dried under supercritical conditions (Figure 5).

From Figure 5 it can be observed the elasticity increasing with increasing cellulose concentration in aerogel samples. The most significant increase of the elasticity occurs at 15%. At concentration 20-25% the strongest aerogel has been produced.

The aerogels dried in the open air (Figure 6) demonstrate the same tendency that the aerogels dried in the open air, with increasing cellulose concentration the elasticity increasing. But here more significant increase of the elasticitycan be observed already at 10% added cellulose. Later, The increase in cellulose concentration 15% to 25% does not affect a lot the elasticity. At this range of the concentrations the elasticity was measured around 4000 kPa. From Figure 6 it is also seen the maximum optimal concentration of added cellulose when the aerogel is the most elastic. This is 10% respectively when the elasticity measured to be 7042 kPa. This change may be caused because of drying in the open air, methyl evaporates and the pores are poured, as well as in contact with air, cellulose loses its elasticity and when the concentration of cellulose is more than 10% the structure of aerogel is not strong enough.

It can be assumed that the evaporation of methanol and a higher concentration of cellulose the aerogel structure becomes more fragile, because there are no clearly located structural pores and the cellulose becomes drier and begins to predominate in aerogel composition.

4. CONCLUSIONS

The focus of the present study was to investigate the influence of adding cellulose to aerogel composition on the mechanical properties of silica-cellulose aerogels. The mechanical properties were estimated based on elastic modulus of material.

During the investigations it has been found experimentally that the mechanical properties of silica-cellulose aerogels are affected not only by adding cellulose of different concentration but also by the drying method applied to the materials in preparation process.

The results have shown that the adding cellulose into the composition of aerogels improves the elasticity of aerogels. With increasing the concentration of cellulose the elasticity increases.

Despite the adding cellulose the mechanical properties of the aerogels drastically depend on the drying methods. The aerogels dried in the open air conditions are 100 times more elastic than the aerogels dried under supercritical conditions.

The maximum optimum concentration of cellulose in the aerogels dried in the open air has been found to be 10%. At this concentration the highest elastic behavior has been fixed.

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7. ATTACHMENT

Aerogel samples dried under supercritical conditions

Aerogels pressure test in graphs.

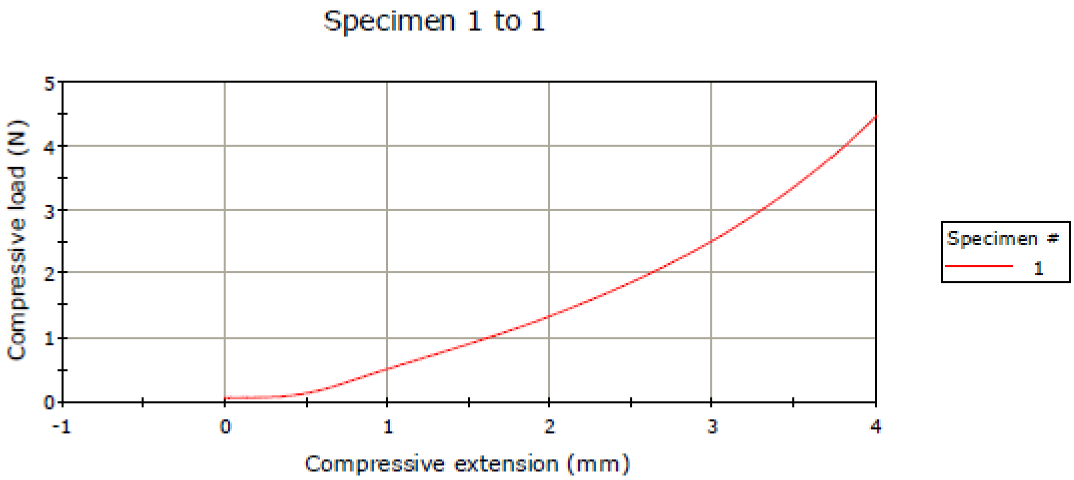

Figure. 7 1% silica-cellulose aerogel.

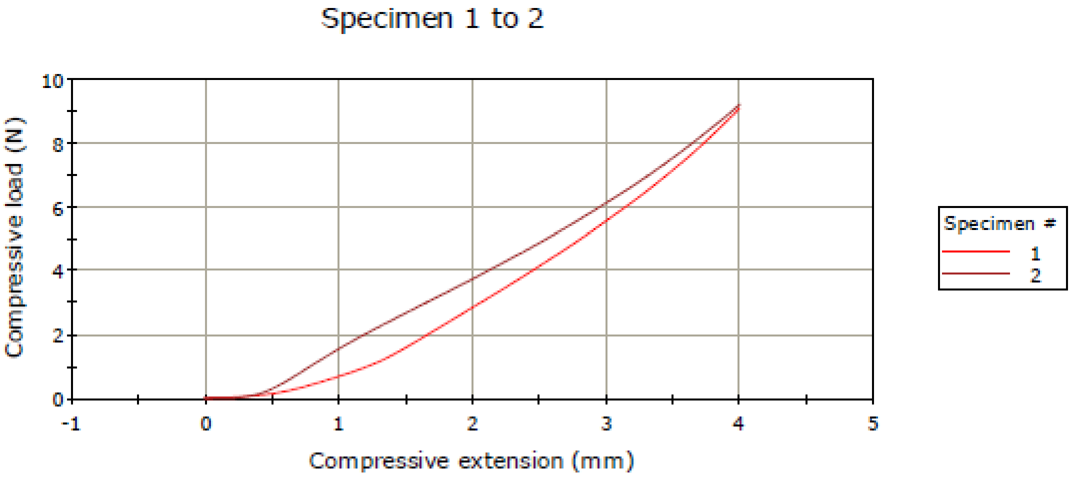

Figure. 8 5% silica-cellulose aerogel.

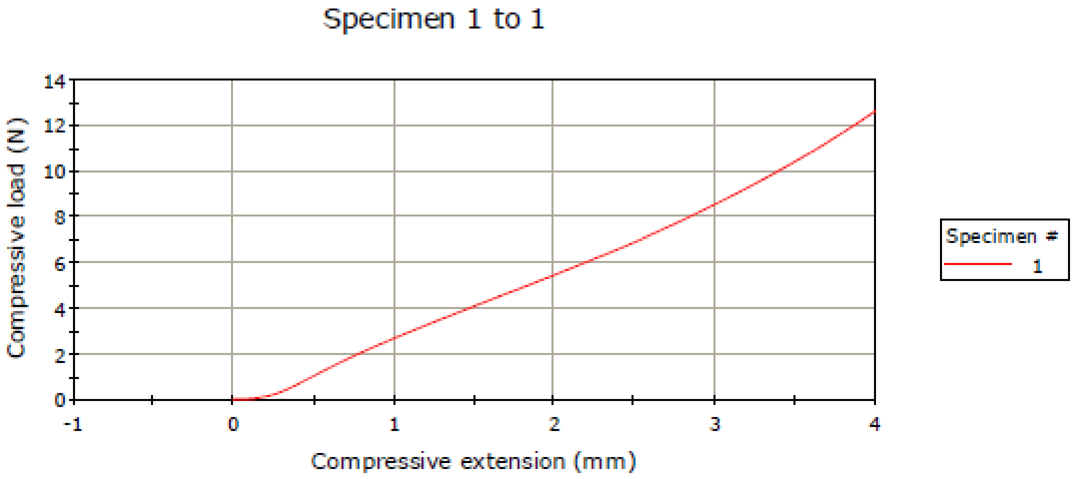

Figure. 9 10% silica-cellulose aerogel.

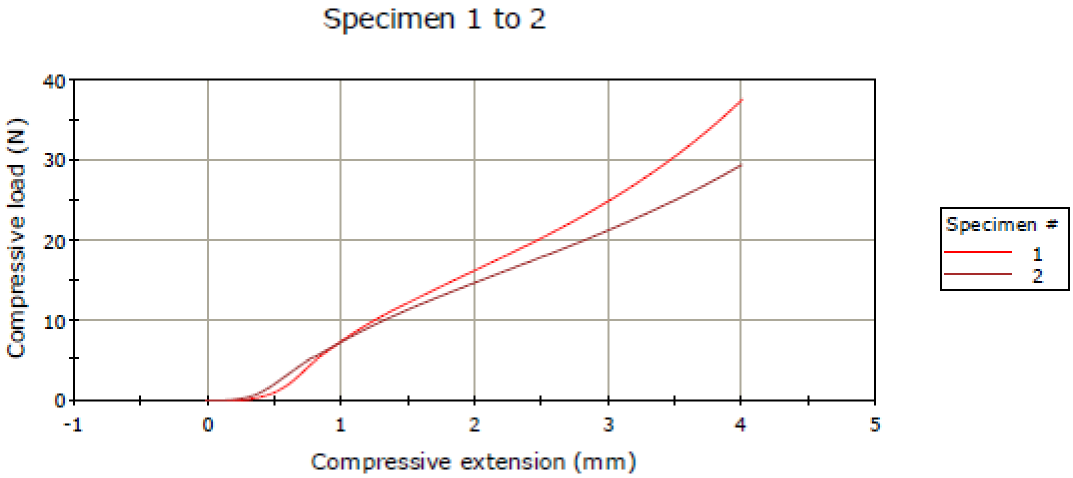

Figure. 10 15% silica-cellulose aerogel.

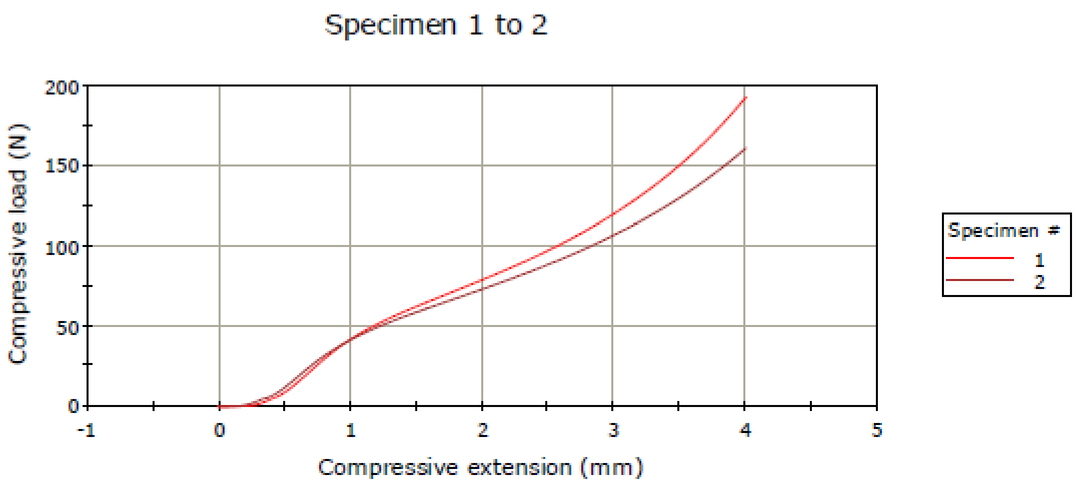

Figure. 11 20% silica-cellulose aerogel.

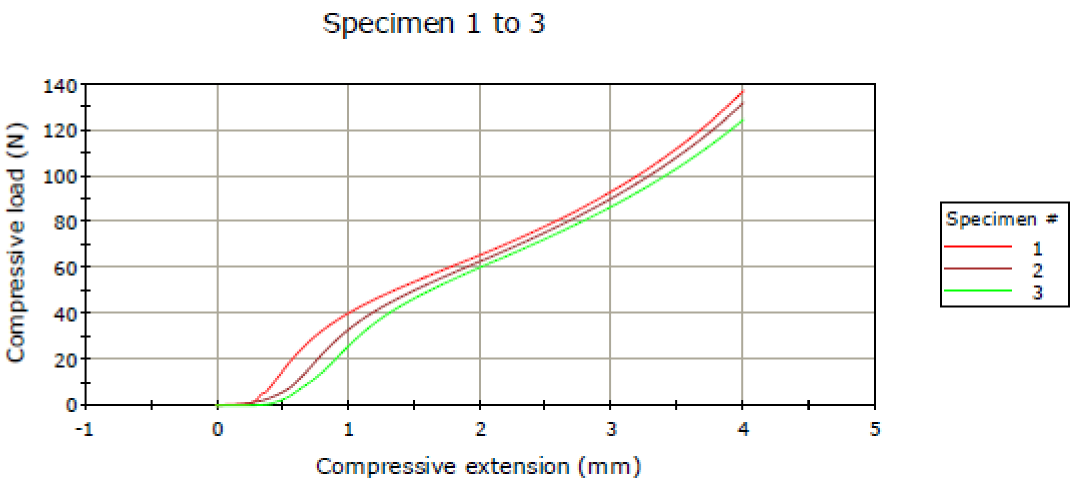

Figure. 12 25% silica-cellulose aerogel.

8. ATTACHMENT

Aerogel samples in the open air drying conditions.

Aerogels pressure test in graphs.

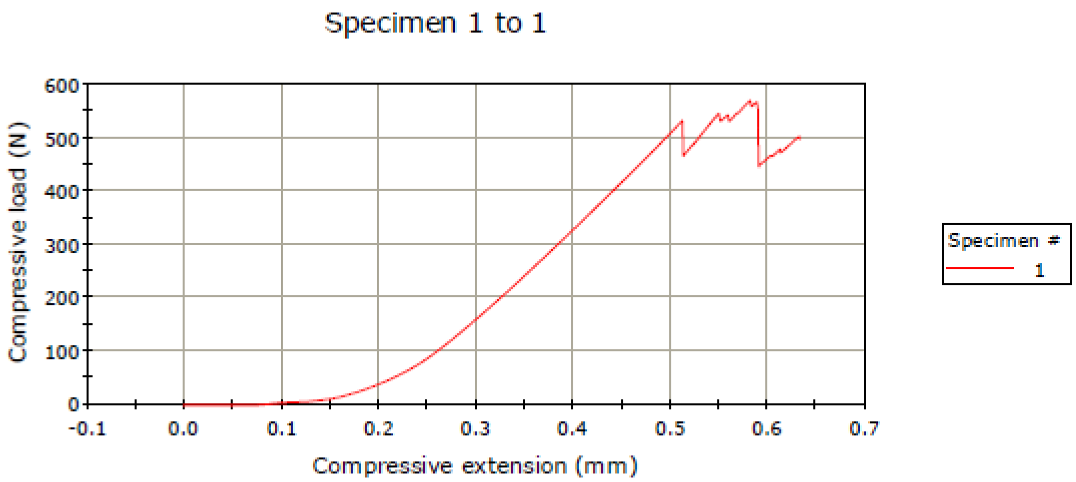

Figure. 13 5% silica-cellulose aerogel.

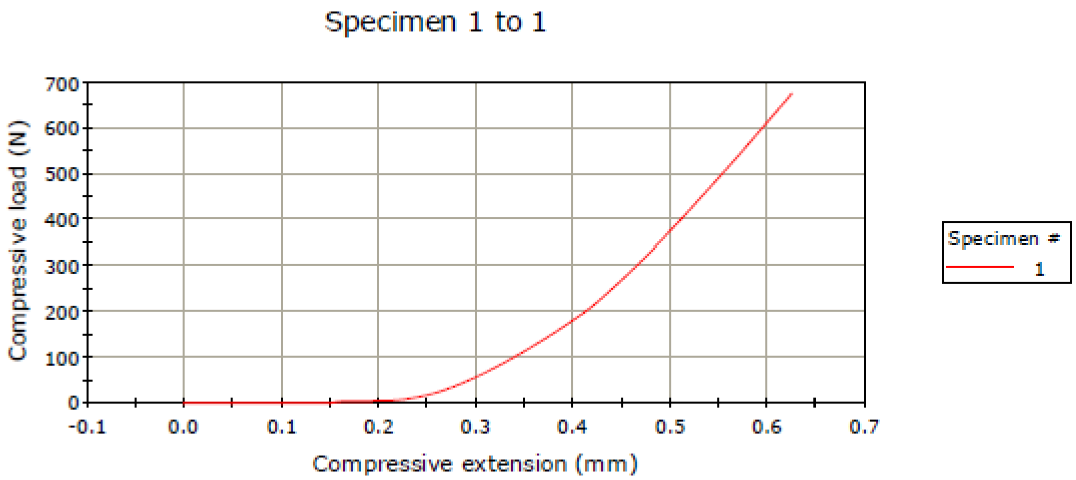

Figure. 14 10% silica-cellulose aerogel.

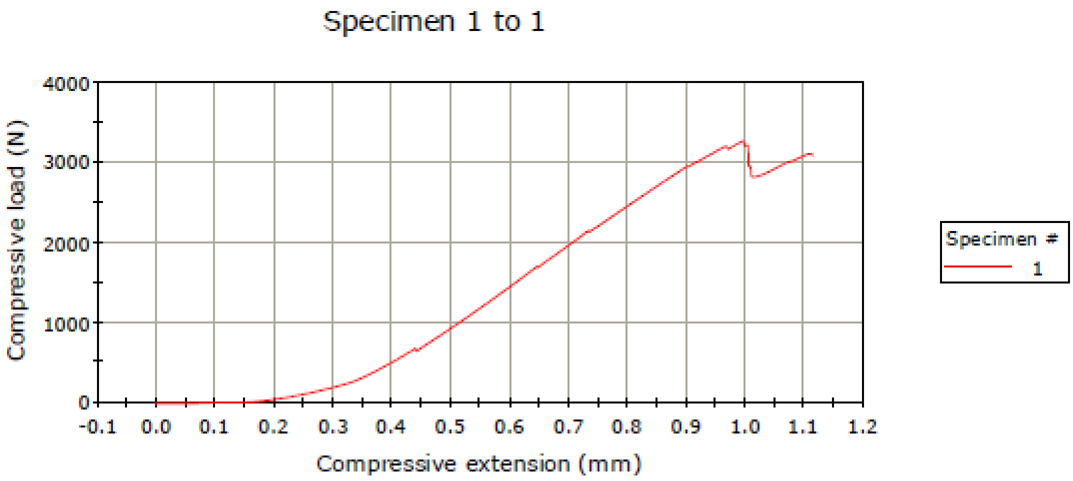

Figure. 15 15% silica-cellulose aerogel.

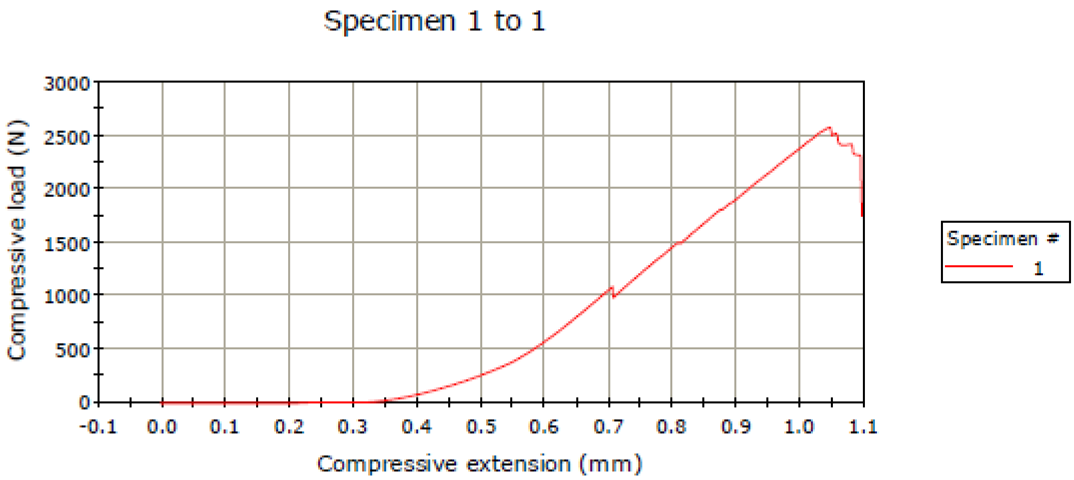

Figure. 14 20% silica-cellulose aerogel.

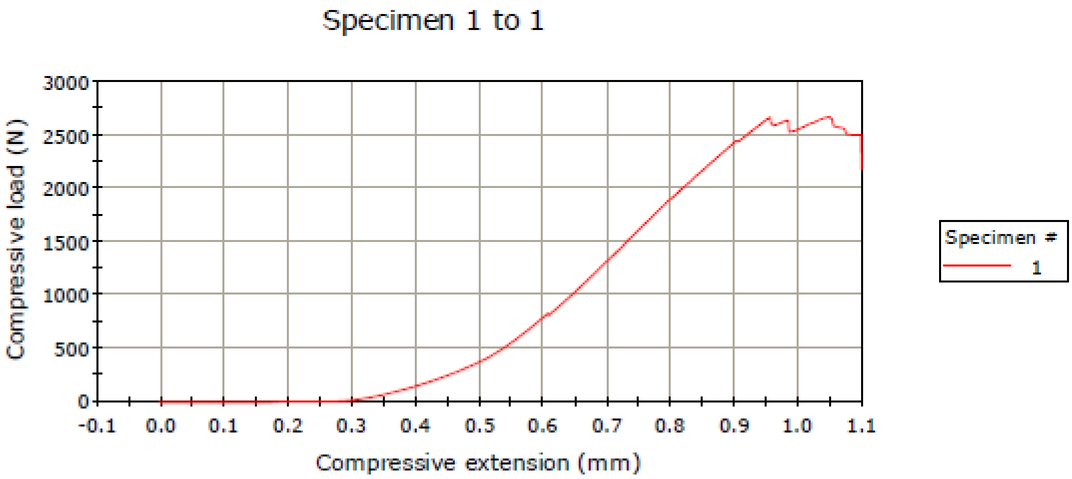

Figure. 16 25% silica-cellulose aerogel.

SUMMARY

The aerogels are unique and investigated class of materials. They have organic and inorganic types of composition, fortoday the main type of aerogels is silica aerogel. The aerogels have many fields for application, from clothes to space filters. Aerogels have very uniqie properties such aslow density, fireproof, soundproof, environmentally friendly, enduring.The main problem of the aerogel´s propertiesislow elasticity.To improve the properties of aerogels, a new type of aerogel was discovered not so long time ago. The name of the new aerogel is silica-cellulose aerogel where in aerogel composition were added nanofibrillatedcellulose (NFC). Due to this component aerogel got a better elasticity, but the dependence of aerogel properties on cellulose concentration has not been studied yet.

The aim of the study was to find out how NFC influence the mechanical properties of aerogels. The aerogels with different cellulose concentration and prepared in different conditions have been researched.

Before testing, there was the aerogel samples preparation. Some of them had different main solution composition, some had the different cellulose concentration. Before this study, there was hypothesis, that if aerogels have a higher concentration of cellulose, then the mechanical properties will be better. The results that were obtained in this study disproved this hypothesis. Based on the results, the mechanical properties of aerogel depend not only on the concentration of cellulose, but also on the drying method of this kind of aerogel. The elasticity property of aerogel is improved by adding NFC to 20% rectilinearly and only with supercritical drying. This concentration can be considered optimal. When aerogels were dried in the open air with different cellulose concentration, the results were quite differentthan with SCD metod. The optimal concentration of the aerogels,which were dried in open air conditions,was 10%. The aerogel with this concentration has the highest result in the Young´s modulus test. It means that aerogel mechanical properties is possible to improve by adding cellulose and by using different drying methods. In this study it has been found the optimal concentrations of cellulose for getting aerogels with the best indicators of mechanical properties.

KOKKUVÕTE

Aerogeelid on unikaalsed ja vähe uuritud materjalide klass. Neil on orgaanilised ja anorgaanilised kompositsiooni tüübid. Tänaseks kõige tuntumad aerogeelid on ränidioksiidi aerogeelid. Aerogeele kasutatakse paljudes valdkondades nagu tekstiilivaldkond, kosmoses filtritena jne. Aerogeelid omavad unikaalseid omadusi nagu madal tihedus, tulekindlus, helikindlus, keskkonnasõbralikus, vastupidavus. Kuid peamiseks probleemiks on aerogeelide väike elastsus. Aerogeelide omaduste parandamiseks leiutati uut aerogeeli tüüpi, ränidioksiidi-tselluloosi aerogeeli, kus aerogeeli koostisesse lisati nanofibrillaalset tselluloosi (NFC). Tänu sellele aerogeeli elastsus parenes, kuid aerogeelide omaduste sõltuvust tselluloosi kontsentratsioonist ei ole uuritud veel.

Antud uurimistöö eesmärgiks oli selgitada välja, kuidas NFC mõjubaerogeelide mehhaanilistele omadustele. Aerogeelide proovid olid erineva tselluloosi kontsentratsiooniga. Uuriti aerogeelide mehaaniliste omaduste sõltuvust tselluloosi kontsentratsioonist.

Uurimistöö jaoks valmistati aerogeelid erineva tselluloosi kontsentratsiooniga kasutades aerogeelide valmistamis metoodikaid. On olemas hüpotees, et aerogeelid kõrgema tselluloosi kontsentratsiooniga onparemate mehaaniliste omadustega. Saadud tulemused aga näitasid, et aerogeeli mehaanilised omadused olenevad mitte ainult tselluloosi kontsentratsioonist, vaid ka kuivatamise meetodist. Aerogeeli elastsust saab parandada lisades NFC kuni 20% ning kasutades ülekriitilist kuivatamist aerogeelide valmistamise protsessis. Kasutades kuivatamist õhu käes optimaalseks kontsentratsiooniks oli leitud 10% lisatud tselluloosi. Selle kontsentratsiooni juures oli saadud materjal kõige kõrgema Young'i elastsusmooduliga katsete käigus.

Antud töö katselised tulemused näitasid, et aerogeelide mehaanilised omadused sõltuvad mitte ainult lisatud tselluloosi kontsentratsioonist vaid ka kuivatamismeetodid.