

The effect of process variables on the properties of nanoporous silica aerogels: an approach to prepare silica aerogels from biosilica

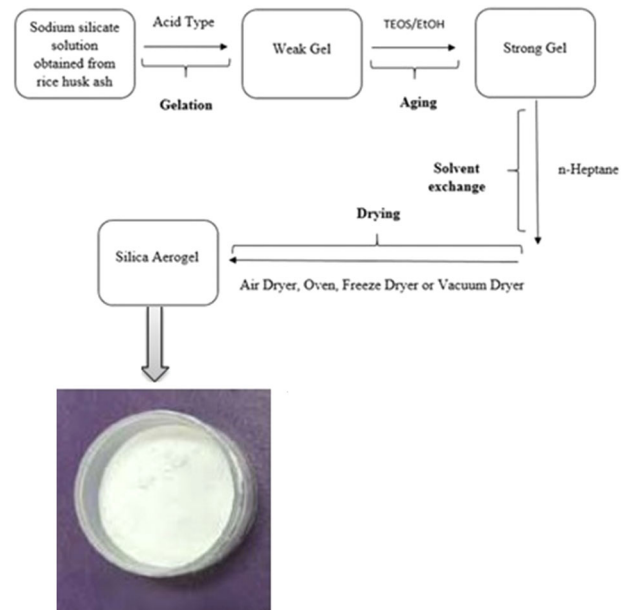
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Abstract Silica aerogel, a nanoporous material, was produced by using rice husk ash via sol–gel method. The aim of the study is to examine effects of the acid type (acetic, hydrochloric, nitric, oxalic and sulfuric acid), dryer type (air, freeze, oven and vacuum) and the addition of tetraethyl orthosilicate on the structural and physical properties of aerogels produced from rice husk ash. In addition, this is the first study investigating the effect of vacuum oven drying on the structure of rice husk based silica aerogel. Specific surface area and pore size of obtained silica aerogels have been analyzed by the N₂ adsorption and desorption measurements at 77 K via Brunauer–Emmett–Teller (BET) and Barrett–Joiner–Halenda (BJH) methods, respectively. Surface functional groups were determined with fourier transform infrared spectroscopy (FTIR). Surface morphology was examined with scanning electron microscopy (SEM). Moreover, density was calculated by tapping method. The results showed that all of the variables had remarkable effects on the final properties of the silica aerogel. The BET specific surface area of the silica aerogels increased with the addition of tetraethyl orthosilicate, while the tapping density decreased. The BET specific surface area and pore size of

silica aerogels varied between 140.7–322.5 m² g⁻¹, and 5.38–12.05 nm, respectively. Silica aerogel which was obtained by using oxalic acid, tetraethyl orthosilicate addition and air dryer had the highest BET specific surface area (322.5 m² g⁻¹).

Graphical Abstract



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Keywords Dryer type · Drying method · Rice husk ash · Silica aerogel · Sol–gel method · Tetraethyl orthosilicate

1 Introduction

Silica aerogels are nanostructured materials with extraordinary properties that have various practices in a wide variety of technological fields. Beyond the myriad applications of aerogels, thermal insulation especially in building construction and aerospace sectors is the most leading one [1, 2]. Silica aerogels have attracted increasingly more attention during last decades due to their high specific surface area (up to $1200\text{ m}^2\text{ g}^{-1}$), high porosity (up to 99.8%), low bulk density (down to $\sim 0.003\text{ g cm}^{-3}$), low refraction index (~ 1.05), ultra-low dielectric constant ($k = 1.0\text{--}2.0$), and low thermal conductivity ($0.005\text{--}0.1\text{ W (mK)}^{-1}$) [3, 4].

In conventional approaches, silica aerogel is synthesized by the sol–gel process, mostly followed by supercritical drying. The high cost of silica precursors is a predominant drawback for the commercial production of aerogels. In recent years, bamboo leaf [5], fly ash [6], oil shale ash [7], and water glass [8], have been utilized instead of pricey organoalkoxysilanes, such as tetramethoxysilane, and tetraethoxysilane (TEOS) [9]. Different types of agricultural wastes, such as wheat husk [9] and bagasse ash [10] have been also used as low-cost precursor to produce silica aerogels due to rich silica content ($\sim 22\text{ wt.}\%$) like rice husk [11, 12]. Rice husk is one of the most promising sources of silica which can solve the cost problem of aerogel production because of its potential surplus availability. Moreover, numerous researches have been conducted to prevent the high cost and risks of supercritical drying [6–10]. Ambient pressure, freeze and microwave drying are the other alternative methods.

Silica aerogel preparation can be divided into three main parts as gelation, ageing and drying of the gel. However, precursor, acid type, methylation agent, water/precursor ratio, pH of the medium, ageing time and drying method are among the number of variables that strongly influence the structure of the final product. These parameters affect the kinetics and mechanism of hydrolysis and condensation processes, and thus the three-dimensional porous structure of the aerogels.

In a previous work, Tang et al. [13] prepared silica aerogel using rice hull ash silica as silica precursor by sol–gel process and supercritical carbon dioxide drying. As a result of analysis, surface area and bulk density of silica aerogel were $597.7\text{ m}^2\text{ g}^{-1}$ and 38.0 kg m^{-3} , respectively [13]. However, using supercritical drying is expensive and risky. In another study, Li et al. [14] investigated the evaluation of rice hull ash silica for atmospheric-dried silica aerogel preparation. The sodium silicate solution neutralized with sulfuric acid and the aerogel obtained by addition of appropriate quantity of TEOS. The specific surface area, bulk density, pore size and porosity of silica

aerogels produced with this method were found to be $500\text{ m}^2\text{ g}^{-1}$, 0.33 g cm^{-3} , $5\text{--}60\text{ nm}$, and 87% , respectively [14].

The present study aimed to prepare and characterize silica aerogel from rice husk ash biosilica. In this paper, new process parameters for preparing silica aerogel was proposed. The effect of TEOS additive, acid types and drying conditions on properties of silica aerogel such as morphology, pore volume and surface area were discussed based on results provided by means of various techniques including BET, FTIR, SEM and EDS.

2 Materials and methods

2.1 Materials

Rice husk ash was obtained from a rice mill (Yetis Food Industry, Turkey). Acetic acid (CH_3COOH), hydrochloric acid (HCl), ethanol, nitric acid (HNO_3), n-heptane, oxalic acid ($\text{C}_2\text{H}_2\text{O}_4$), sulfuric acid (H_2SO_4), and tetraethoxysilane (TEOS) were purchased from Merck (Darmstadt, Germany).

2.2 Methods

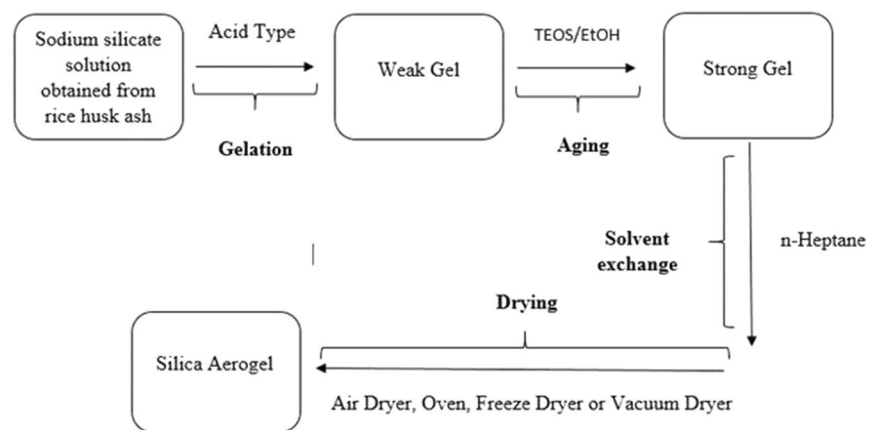
2.2.1 Sodium silicate preparation from rice husk ash

Sodium silicate as a precursor for aerogel preparation was obtained from rice husk ash using the method described previously [15, 16]. Firstly, as received rice husk ash was burned for 5 h at $600\text{ }^\circ\text{C}$ in an incineration furnace (Protherm, Turkey) followed by refluxing for 2 h in an acidic solution prepared with addition of 6 M HCl solution (pH 1). In order to obtain the acid leached ash the mixture was filtered and washed under vacuum and then dried overnight at room temperature. The acid leached ash was mixed with 1 M NaOH solution and then boiled for an hour to extract silica. Silica was obtained in the form of sodium silicate and was separated from the residual ash via vacuum filtration. The filtrate was collected subsequent to the washing step of the residual ash (400 mL distilled water).

2.2.2 Silica aerogel preparation

The silica aerogel preparation procedure used in this study is demonstrated in Fig. 1. In order to obtain silica aquagel, pH of the sodium silicate solution was decreased to 7 by the addition of acid (acetic, hydrochloric, nitric, oxalic or sulfuric acid). Formed silica aquagel was aged for 18 h at room temperature to obtain a stronger silica network. The aged gel was washed three times with distilled water to remove sodium salts formed during the condensation reaction.

Fig. 1 The flowchart of rice husk ash-based silica aerogel preparation



Water present in the aquagel was replaced with 80% (v/v) ethanol solution by ageing for 24 h at room temperature. In the case of TEOS added aerogel preparation, ageing was continued with 70% (v/v) TEOS in ethanol solution for 18 h at room temperature and 4 h at 70 °C. Subsequently, during solvent exchange step, 70% (v/v) TEOS–ethanol solution and 80% (v/v) ethanol solution present in the gels was replaced with n-heptane by washing for three times and the gel was aged for 24 h at room temperature. Then n-heptane was replaced with fresh n-heptane for two times. Following the centrifugation (5 min, 4800 rpm) obtained gels were dried using four different types of dryers, namely air (Elektromag, M 420 P), freeze (Telstar, Spain), oven (Binder, Germany) and vacuum dryers (Binder, Germany). The gels were dried in 6 h intervals at 50, 60, and 70 °C in air and oven dryer, in 6 h intervals at 40, 50 and 60 °C in a vacuum dryer and at −52 °C for 24 h in a freeze dryer.

2.2.3 Characterization of silica aerogels

Prepared silica aerogels were characterized by a specific surface area and pore size distribution analyzer (Micromeritics, TriStar II 3020) at 77 K to determine the related properties as surface area, pore size and mesopore volume. The BET method was used to estimate specific surface area of aerogels. Pore size and pore volume was calculated by BJH method. FT-IR (Shimadzu IR Prestige 21, Kyoto, Japan) instrument was used to approve the chemical structure of aerogels in the wave number range of 650–4000 cm^{-1} . The tapping density of aerogels was calculated according to the ratio of measured weight to volume. The porosity of aerogels was estimated according to the following equation [17]:

$$\text{Porosity}(\%) = \left(1 - \frac{q}{qs}\right) \times 100 \quad (1)$$

where qs is the true density of silica (2.19 g cm^{-3}) and q is the tapping density of silica aerogel.

3 Results and discussion

3.1 Dryer type effect on textural features of silica aerogels

The textural properties of the silica aerogels were measured to evaluate the effects of different drying methods. Table 1 shows the BET specific surface area (S_{BET}), BJH pore size and mesopore volume (V_{meso}) of the nanoporous silica aerogels determined from the N_2 gas adsorption/desorption analyzes. It can be seen that silica aerogels dried using air dryer had the largest specific surface area ($287.7 \text{ m}^2 \text{ g}^{-1}$) and pore size (11.19 nm). Based on the data, the minimum BET specific surface area ($213.1 \text{ m}^2 \text{ g}^{-1}$) occurred in the aerogel dried using freeze dryer, which can be explained by the fact that crystallization of the solvent in the pores lead to the deformation of gel network [3]. The BET specific surface area of oven dried silica aerogel was relatively small which strongly depends on the high drying shrinkage [18]. In contrast, the drying shrinkage occurring in air dryer due to capillary forces may be relatively low due to uniform heating of the silica gel. There was a high contacting efficiency between the aerogel and hot air which was due to the circulation of air provided by ventilation fan in air dryer. This gave rise to better solvent removal that was trapped in the pores and the drying shrinkage stopped earlier. Due to the fact that the air-dried aerogels were noticed to have larger specific surface area, pore size and pore volume than that of oven and freeze dried aerogels. The BET specific surface area of air dried and vacuum dried samples were similar. Besides the density and porosity features of these aerogels were different (Table 1), the air dried one had more desired properties due to its low density and high porosity values compared to vacuum dried aerogel. According to these results, air dryer seems to be the most suitable dryer to prepare silica aerogels. Additionally, air dryer renders a high drying performance as well as higher drying capacities as compared to vacuum and freeze dryers. Besides, air

Table 1 Effect of dryer type on the textural and physical properties of silica aerogels neutralized with nitric acid

Dryer type	S_{BET} ($\text{m}^2 \text{g}^{-1}$)	BJH Pore size ^a (nm)	V_{meso} ($\text{cm}^3 \text{g}^{-1}$)	Density (g cm^{-3})	Porosity (%)
Air	287.7	11.19	0.923	0.29	87
Freeze	213.1	8.11	0.345	0.30	86
Oven	234.0	7.33	0.333	0.18	91
Vacuum Oven	285.4	7.79	0.881	0.43	80

^a The pore size values were provided from the desorption portion of the N_2 adsorption–desorption isotherm

drying method is cheaper and simpler than vacuum and freeze drying. Keeping these in view, further experiments were performed using air dryer.

The tapping density values and porosities of silica aerogel samples dried using air, freeze, oven and vacuum dryer are also summarized in Table 1. The oven dried silica aerogel had less density than the other type dried silica aerogels. The tapping densities of the air and freeze-dried silica aerogels were so similar; however, the vacuum oven-dried aerogel tendered a higher density value. The porosity of silica aerogels obtained with different dryers was varied from 80 to 91%, which were higher than the values obtained by Tadjarodi et al. [19] for nanoporous silica aerogel prepared from rice husk ash. The pore size analysis of aerogels was achieved by Barret–Joyner–Halenda (BJH) equilibrium model which is applied to samples with pore sizes between ~ 1 –150 nm (mesopores and part of macroporous) [20]. The pores of all aerogels were found in the nanoporous domain (~ 5 –12 nm) which indicates that the particle sizes were small (Tables 1–3).

The FT-IR spectra of silica aerogels are provided in Figs. 2, 3 and 4. The peaks at around 3600 cm^{-1} and 1635 cm^{-1} are ascribed to water hydroxyl groups absorbed on the surface [21]. The spectrum exhibits the characteristic peaks concerning the presence of asymmetric and symmetric bending of the Si–O–Si bonds at around 1062 and 794 cm^{-1} , respectively. Similar peaks can be observed in both spectra in Figs. 3 and 4 for all prepared aerogels.

Figure 5. shows the SEM micrographs of silica aerogels dried using different dryers. As can be seen from SEM images, all aerogel types exhibit a porous network structure. The particle and pores were smaller in size. Particle size values of prepared aerogel samples were found to be lower than 100 nm. It is clear from Fig. 5 that the porosity of oven dried aerogel (Fig. 5d) was higher than the porosity of aerogel samples dried using other dryers. Besides, it can be stated that vacuum dried aerogel exhibited denser structure owing to its low porosity. These results are in good agreement with porosity and density results as presented in Table 1.

3.2 TEOS additive effect on physicochemical features of silica aerogels

The effect of TEOS addition on the various physicochemical properties of the silica aerogels is tabulated in Table 2.

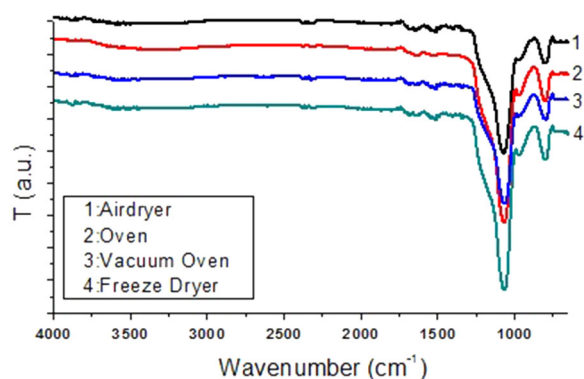


Fig. 2 FT-IR spectra of silica aerogels dried using (1) air, (2) oven, (3) vacuum oven, and (4) freeze dryers

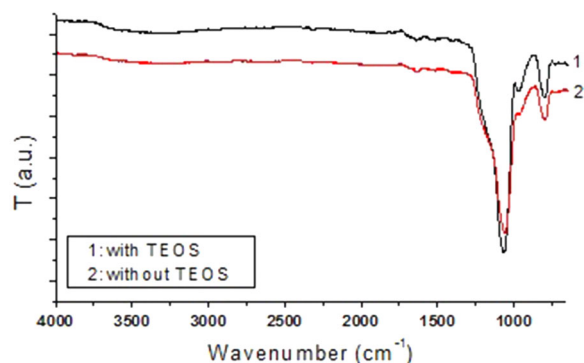


Fig. 3 FT-IR spectrum of oven dried silica aerogels (1) with TEOS addition, and (2) without TEOS

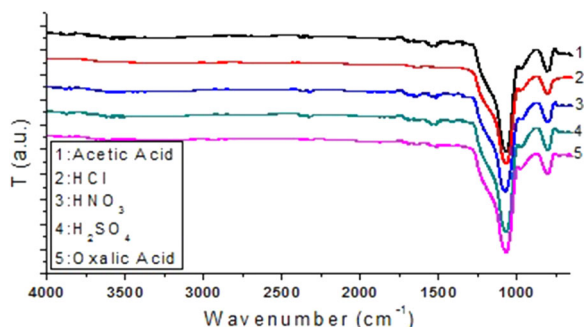


Fig. 4 FT-IR spectra of silica aerogels produced using (1) acetic, (2) hydrochloric, (3) nitric, (4) sulfuric, and (5) oxalic acids

Fig. 5 SEM image of aerogels dried using **a, b** air dryer **c, d** oven dryer **e, f** vacuum dryer **g, h** freeze dryer

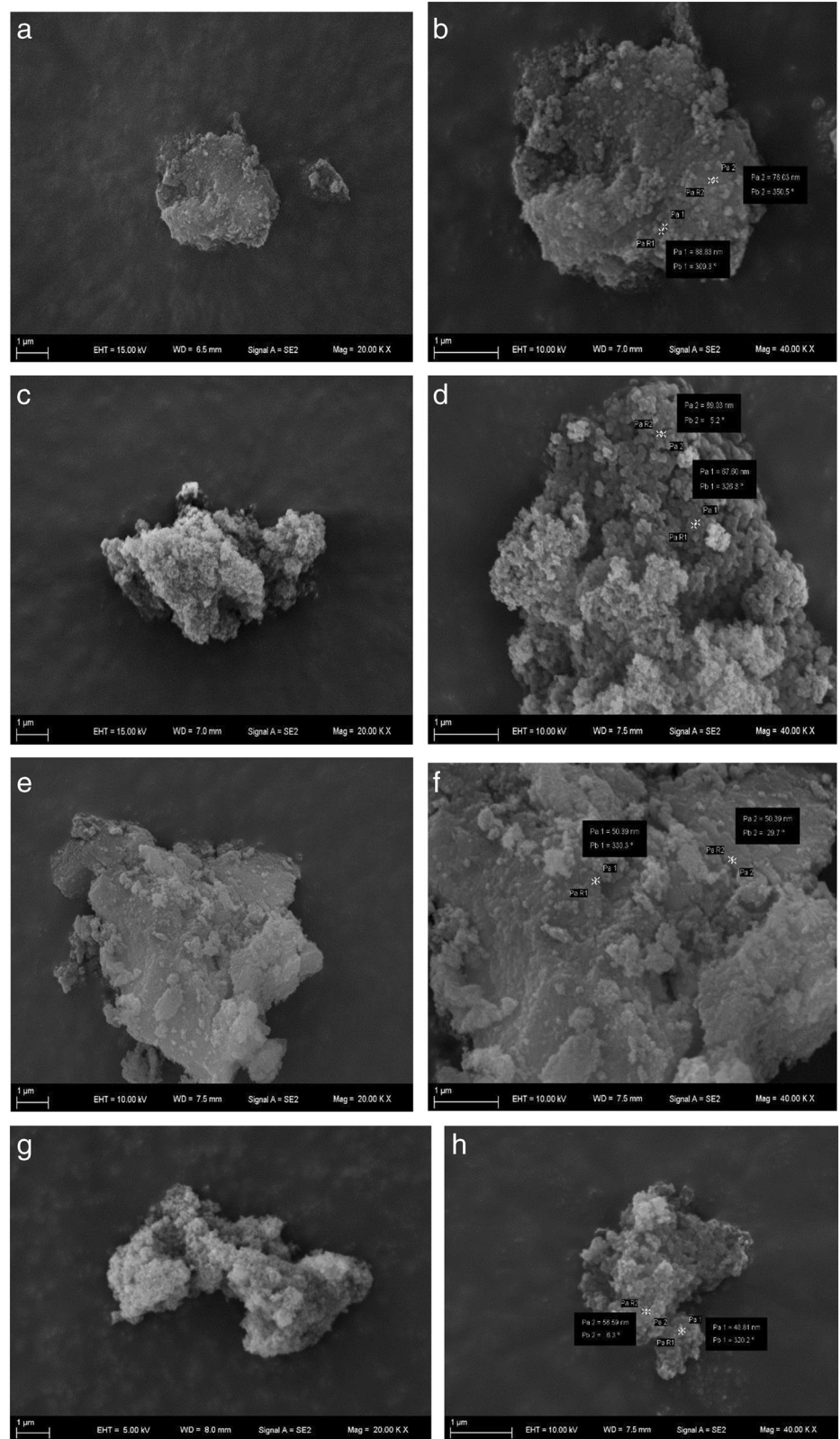


Table 2 Effect of TEOS addition on the textural and physical properties of silica aerogels neutralized with nitric acid

TEOS	Dryer type	S_{BET} ($\text{m}^2 \text{g}^{-1}$)	BJH Pore size (nm)	V_{meso} ($\text{cm}^3 \text{g}^{-1}$)	Density (g cm^{-3})	Porosity (%)
Without	Oven	140.7	12.05	0.418	0.38	82
With	Oven	234.0	7.33	0.333	0.19	91
Without	Air	247.8	5.38	0.524	0.71	68
with	Air	287.7	11.19	0.923	0.29	87

Table 3 Effect of acid type on the textural and physical properties of air-dried silica aerogels

Acid type	S_{BET} ($\text{m}^2 \text{g}^{-1}$)	BJH Pore size (nm)	V_{meso} ($\text{cm}^3 \text{g}^{-1}$)	Density (g cm^{-3})	Porosity (%)
Acetic ($\text{C}_2\text{H}_4\text{O}_2$)	294.4	10.70	1.022	0.37	83
Hydrochloric (HCl)	268.0	11.79	0.998	0.38	83
Nitric (HNO_3)	287.7	11.19	0.923	0.29	87
Oxalic ($\text{C}_2\text{H}_2\text{O}_4$)	322.5	10.85	1.048	0.21	90
Sulfuric (H_2SO_4)	294.9	10.33	1.044	0.39	82

Aerogels which were prepared using TEOS as the additive resulted in higher porosity and smaller tapping density, while without TEOS the structure had a lesser porosity and higher tapping density. From the Table 2, it is clear that the oven dried aerogel prepared with TEOS addition had the lowest density (0.19 g cm^{-3}). This is due to the fact that the gel strengthens with the addition of TEOS into the sol, and also it is hindering cracking of gel. However, in the absence of TEOS, the shrinkage caused by capillary tension occurs on the gel network which leads to cracking of gel, hence reducing the pore size and specific surface area [19]. In the same way, Tadjardi et al. [19] synthesized silica aerogel from rice husk ash in the presence of sulfuric acid and dried under atmospheric pressure for 10 h at 40°C . It was observed that with TEOS addition the BET surface area and pore size was raised from 220 to $315 \text{ m}^2 \text{g}^{-1}$ and 8.3 to 9.8 nm, respectively. Results obtained in this study are in agreement with the study mentioned above.

Figure 3 illustrates the FT-IR spectra of silica aerogels prepared with TEOS and without TEOS. The intensity of asymmetric bending of the Si–O–Si band at 1062 cm^{-1} is stronger in silica aerogel samples prepared with TEOS addition (Fig. 3) due to the silica content of TEOS. In a similar study, Nayak et al. [22] specified usage of TEOS in aging step of silica gel and by this treatment more silica was introduced into the silica network.

3.3 Effect of acid type on textural and physical features of silica aerogels

The type of acid affects the textural and physical properties of the resulting silica aerogels. In order to evaluate the effect of the acid on the textural and physical properties, silica aerogel samples were prepared using various acids.

The textural and physical properties of silica aerogels prepared using the different acids are compared in Table 3. The largest BET specific surface area was obtained from the sample prepared with oxalic acid, and the lowest value was obtained from the sample prepared with hydrochloric acid. Additionally, it can be observed that the pore size of the samples prepared with all acid types were relatively similar. This observation is presumably attributed to the occurrence of sodium salts with differences in the size during formation of weak gel [23]. The water washing step was more efficient in the elimination of sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$), sodium sulfate (Na_2SO_4) and sodium acetate (NaOAc) from weak gel however not in the elimination of sodium nitrate (NaNO_3) and sodium chloride (NaCl). Sodium chloride may have agglomerated in the gel network due to its smaller size, and hence may block the pores [24]. Moreover, the effect of sodium salts may not be only due to the size but also related to hydrophobic and some other interactions between the solvent and silica particles [25, 26].

The aerogel prepared with oxalic acid had a relatively low tapping density (0.21 g cm^{-3}) than the other samples; therefore the calculated % porosity for the aerogel prepared with oxalic acid was very high (90%). However, aerogels prepared with acetic and hydrochloric acid showed relatively high density (0.37 and 0.38 g cm^{-3} , respectively). The presence of sulfuric acid tended to obtain denser aerogels (0.39 g cm^{-3}).

The energy dispersive X-ray spectroscopy (EDS) results of aerogel samples produced using different acids are presented in Table 4. The EDS results indicated that, silica aerogel produced with sulfuric acid has the lowest sodium content while aerogel produced with hydrochloric acid has the highest sodium content. On the other hand, the silica content of aerogels prepared with hydrochloric, acetic and

sulfuric acids were higher which were also denser. The aerogel prepared with oxalic acid has the lowest silica which also has the lowest density. The results were in accordance with the BET and density results.

3.4 Nitrogen physisorption studies

N₂ adsorption and desorption isotherms of the silica aerogels were investigated to identify the effect of process variables on the structure of the pores in the three-dimensional network of aerogels. Figure 6 shows the N₂ adsorption–desorption isotherms of aerogel samples. Aerogels exhibited the features of Type IV isotherms which are generally coincided in many mesoporous materials [27, 28]. There are two kind hysteresis loops; H1 hysteresis is observed cylindrical pores open at both ends, H2 hysteresis

loops is associated with “ink-bottle” pores. Most of the aerogel samples exhibited H1 hysteresis loops; however, aerogel prepared without TEOS addition and also vacuum dried aerogel had H2 hysteresis loop. In line with these data, it can be concluded that the pore shape of aerogel was affected by the presence of TEOS and type of drying method.

4 Conclusion

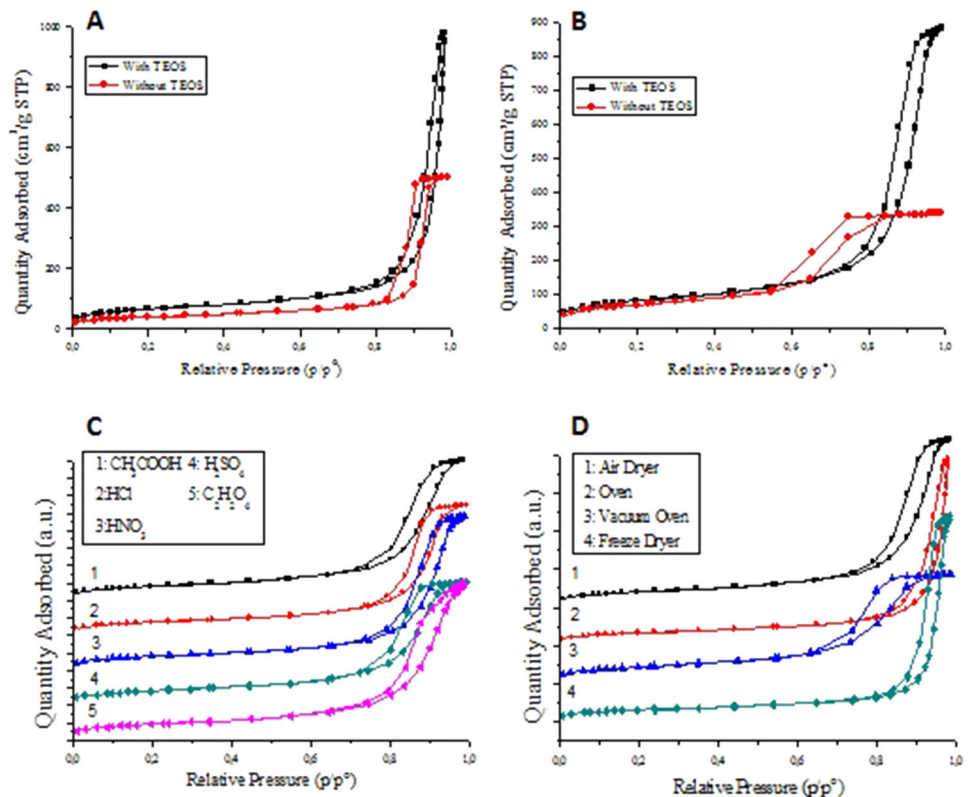
There is an increasing requirement to obtain advanced materials with suitable properties to apply in a specific application. Silica aerogels are among the advanced materials which are attracting widespread interest due to their unique properties. However, various production conditions have an influence on the final properties of silica aerogels. Therefore, the determination of optimum production conditions is crucial. In this study, the effect of dryer type, additive and acid type on the physicochemical properties of silica aerogels were examined. The following major findings can be summarized based on the experimental results:

1. Although usually replaced by more complex dryer types, air dryer found to be precisely sufficient in many cases, particularly when aerogels are needed with enhanced textural properties. The aerogel dried

Table 4 EDS results of silica aerogel samples produced using different acids

Element	Atomic %				
	Acetic	Hydrochloric	Nitric	Oxalic	Sulfuric
O	60.01	59.7	68.73	70.75	57.98
Si	38.72	38.87	30.31	27.96	41.38
Na	1.21	1.43	0.96	1.29	0.64

Fig. 6 N₂ adsorption/desorption isotherms of the (a). Oven dried aerogels prepared with TEOS addition and without TEOS addition (b). Air dried aerogels with TEOS addition and without TEOS addition (c). Aerogels prepared using various acids (d). Aerogels prepared using different drying methods



using air dryer had a high BET specific surface area ($287.7 \text{ m}^2 \text{ g}^{-1}$), pore size (11.19 nm) and mesopore volume ($0.923 \text{ cm}^3 \text{ g}^{-1}$) as compared to the freeze and oven dried samples.

- The silica aerogels were comprised of nano-structure. The maximum pore size of aerogel was only 12.05 nm.
- The results showed that the BET specific surface areas of the silica aerogels increased with the addition of TEOS. The addition of TEOS is favorable for reducing the tapping density of silica aerogels.
- Silica aerogels obtained using different acid types. Oxalic acid was found to be the best acid amongst the other acids for silica aerogel production. Because the silica aerogel prepared with oxalic acid had comparable BET specific surface area and low tapping density to those prepared with other acid types. The results of EDS analysis also showed that the silica content was the lowest in the aerogel prepared with oxalic acid which was consistent with surface area and density results.
- The pore structure of obtained aerogels was affected from dryer type and TEOS addition. Silica aerogels which are produced in vacuum oven dryer and without TEOS additive have shown ink bottle pore shape while the other samples had cylindrical pores open at both ends.

Further efforts should be realized on the synthesis parameters and the drying techniques in order to widen the range of accessible silica aerogels with advanced textures.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no competing interests.

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