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**Optimization of degassing conditions for reliable BET surface area and BJH pore analyses
of starch aerogels**

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Abstract

Accurate determination of the textural properties of aerogels by nitrogen adsorption–desorption analysis depends strongly on appropriate sample degassing conditions. Inadequate degassing can leave residual moisture and solvent molecules within the pore structure, whereas excessive degassing may alter the porous network, leading to inaccurate characterization. This study investigated the effects of degassing temperature (40–200 °C) and duration (1–24 h) on the nitrogen adsorption behavior, surface area, pore size, and pore volume of starch aerogels prepared using supercritical carbon dioxide (SC-CO₂) drying. The adsorption isotherms exhibited mesoporous characteristics, with nitrogen uptake strongly influenced by the degassing conditions. Increasing the degassing temperature to 75 °C improved nitrogen adsorption by effectively removing residual adsorbates, resulting in the highest BET surface area of 212 m²/g. Above 75 °C, the BET surface area progressively decreased from 212 to 139 m²/g at 200 °C due to a reduction in pore volume, suggesting reduced pore accessibility due to thermal effects on the aerogel structure. Degassing duration had insignificant influence on the measured textural properties, although the adsorption isotherms indicated that 1 h provided the highest nitrogen uptake and most consistent adsorption behavior. Pore size remained relatively constant (16–19 nm) across all conditions. Overall, the results identified 75 °C for 1 h as the optimum degassing condition for starch aerogels and highlight the importance of optimizing degassing protocols to ensure accurate and reproducible nitrogen adsorption measurements of biopolymer aerogels.

Keywords: Starch aerogel, Nitrogen adsorption–desorption, BET surface area, Pore volume

1. Introduction

Aerogels are classified as lightweight, highly porous materials with exceptional surface areas, making them essential for a wide range of applications [1]. Biopolymer-derived aerogels, including protein [2, 3], cellulose [4] and starch aerogels [3, 5], have attracted considerable attention as sustainable porous materials because of their low density, biodegradability, renewability, and microstructure. Owing to their highly porous three-dimensional network, aerogels exhibit large internal surface areas and interconnected pore structures, creating opportunities for applications in bioactive compound encapsulation, thermal insulation, adsorption, catalysis, biomedical scaffolds, drug delivery, and packaging technologies [1, 6]. The physicochemical properties and performance of these aerogels are strongly influenced by their textural characteristics, particularly specific surface area, pore size distribution, and pore volume and are susceptible to structural changes during sample preparation [5]. Therefore, accurate characterization of these parameters is essential for understanding structure-property relationships and optimizing material performance.

The optimization of degassing parameters such as temperature, duration, and vacuum conditions is important to obtain accurate and reproducible surface area and pore characteristics while preserving the native porous structure of the aerogel [7]. Degassing removes physically adsorbed moisture, residual solvents, and volatile impurities from the porous structure, exposing the accessible surface area and pore network to adsorbate gas [7, 8]. Inadequate degassing may leave contaminants within the pores, resulting in underestimated surface area and pore volume values [9]. On the other hand, excessive degassing temperatures or prolonged treatment times can damage fragile porous structures such as biopolymer-based aerogels, leading to pore collapse, shrinkage, or structural modification [7]. Several studies have emphasized the importance of

careful interpretation of nitrogen adsorption data and the challenges associated with analyses. Sing [10] highlighted that experimental conditions such as equilibration time, pressure range, and sample preparation significantly affect the accuracy of surface area and pore size calculations. Similarly, Bardestani, et al. [7] noted that degassing conditions must be optimized according to the thermal and mechanical stability of the material to avoid misleading results in pore structure analysis. Recent studies on adsorption isotherms have further demonstrated that improper experimental procedures can lead to inconsistent interpretations of pore characteristics and hysteresis behavior [9, 11].

Surface area and pore analyses involve multiple theoretical models [7, 8]. The Langmuir model (1916) was the primary theoretical model for characterizing surfaces, though it is limited by the assumption of ideal localized monolayer adsorption [10, 12]. Other established techniques for evaluating textural properties include Mercury Intrusion Porosimetry (MIP), which is effective for macropores but limited in the mesopore range due to the high pressures required, and Small Angle X-ray Scattering (SAXS), which can probe a wider range of pores, including closed ones, but often requires complex corrections for density contrasts [13, 14]. Among the theoretical models used to interpret adsorption data, the Brunauer-Emmett-Teller (BET) theory remains the standard procedure for surface area determination [10, 11]. For the assessment of mesoporosity, the Barrett-Joyner-Halenda (BJH) method, which applies the Kelvin equation to account for capillary condensation, is the most popular approach for deriving pore size distributions. BET method successfully extends the Langmuir model to account for multilayer adsorption, providing a consistent and reproducible way to determine specific surface area [7, 10]. When combined with nitrogen at 77 K, BET physisorption is preferred because nitrogen is inert, inexpensive, and

typically yields a well-defined monolayer capacity for characterizing the complex pore networks [7, 9].

Further, nitrogen adsorption-desorption isotherms provide valuable information regarding pore classification and adsorption mechanisms. An adsorption isotherm describes the relationship between the amount of gas adsorbed and the relative pressure (P/P_0) at a constant temperature [9, 10]. According to IUPAC classification (Figure 1), Type I isotherms are associated with microporous materials, Type II and III with non-porous or weakly interacting surfaces, and Types IV and V with mesoporous materials exhibiting capillary condensation and hysteresis loops. Type VI isotherms represent stepwise multilayer adsorption on uniform surfaces [7, 12]. Hysteresis loops, which arise from differences between adsorption and desorption processes, provide additional insight into pore structure (Figure 1(b)). They are commonly classified as H1–H4. H1 loops indicate uniform cylindrical pores, H2 loops correspond to complex pore networks, H3 loops are associated with slit-shaped pores formed by particle aggregates, and H4 loops are characteristic of narrow slit pores in microporous materials [7, 10]. Together, adsorption isotherms and hysteresis loops are essential for understanding the pore characteristics and surface properties of porous solids. However, accurate pore characterization depends strongly on the quality of the experimental isotherm, which is directly influenced by degassing conditions [9].

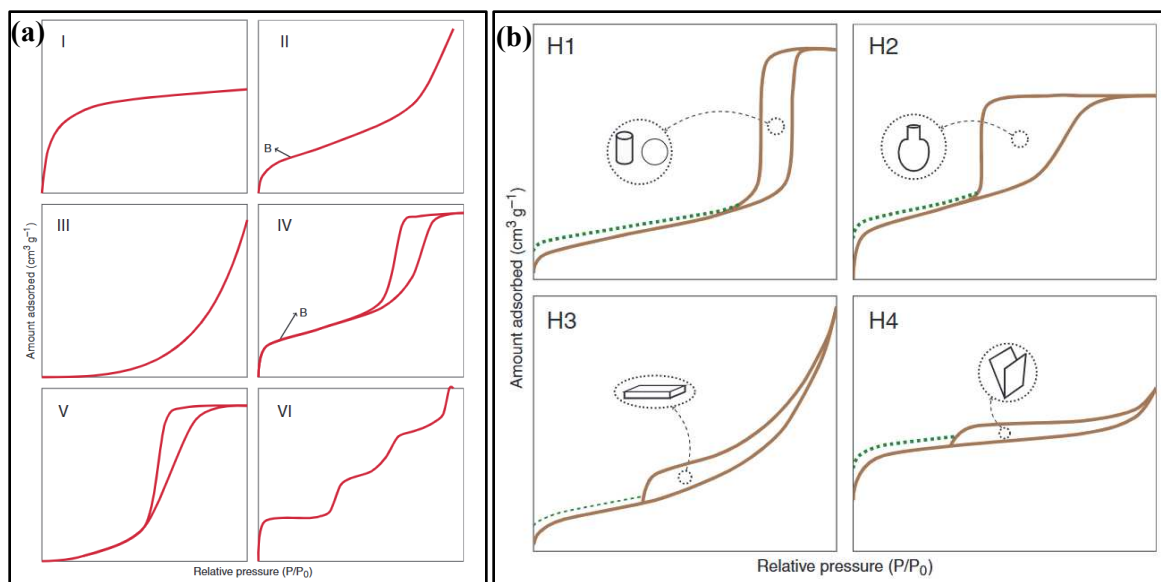


Fig. 1. Types of (a) adsorption isotherms and (b) hysteresis loops. Adapted from Bardestani, et al. [7].

Therefore, this study aims to optimize the degassing conditions for nitrogen adsorption–desorption analysis of starch aerogels to obtain accurate and reproducible BET surface area, BJH pore size, and pore volume measurements. Different degassing temperatures and durations were evaluated to determine their effects on the textural properties and structural stability of the aerogels. The findings of this work contribute to establishing a reliable protocol for the accurate characterization of these high-performance porous biomaterials.

2. Methods

2.1. Materials

Corn starch with high amylose content (Hylon VII; ~70% amylose content) used for aerogel formation was supplied by Ingredion (IL, USA). Ethanol (100%) was purchased from Fisher Scientific (PA, USA). Ultra-high purity nitrogen and helium cylinders (99.999%), liquid nitrogen (99.5%), and carbon dioxide (>99.99%) were supplied by Airgas, Inc. (AR, USA).

2.2. Aerogel formation

Aerogels were formed using three main steps: hydrogel formation, solvent exchange, and SC-CO₂ drying [3]. First, aqueous starch suspensions (12.5%, w/w) were prepared and gelatinized in a Thermomix (Vorwerk, CA, USA) at 95 °C under high shear (4260 rpm) for 25 min [15]. The resulting gels were stored at 4 °C for 48 h to complete retrogradation and form hydrogels. The hydrogels were then cut into small pieces (1-2 cm in length) and subjected to a five-step solvent exchange process by immersion in ethanol-water solutions containing 30%, 50%, 70%, and 100% (v/v) ethanol for 1 h each, followed by immersion in 100% ethanol for 24 h to produce alcogels. The alcogels were subsequently dried using SC-CO₂ (SFT-120, Supercritical Fluid Technologies Inc., DE, USA) at 10 MPa, 40 °C, and a CO₂ flow rate of 1.0 L min⁻¹ (measured at 23 °C and 0.1 MPa). Then, alcogels were loaded into a vessel fitted with stainless-steel frits at the bottom to ensure ethanol coverage during drying. The vessel and micrometering valve temperatures were maintained at 40 and 80 °C, respectively, with the valve heated to prevent freezing due to the Joule-Thomson effect. After reaching the set temperatures, the system was pressurized to 10 MPa and SC-CO₂ drying was carried out for 4 h. Following drying, the vessel was depressurized at the same flow rate, and the resulting starch aerogels were collected and stored in airtight containers at room temperature (23 °C) until further analysis.

2.3. Sample preparation for degassing

The dried aerogel was cut into small-sized pieces (0.06-0.3 g) and placed in the analysis tube without compression to preserve its pore structure. Degassing was performed under vacuum at different temperatures (40-200 °C) for several hours (1-24 h), to remove adsorbed gases, moisture, and other contaminants from the surface and pores. At first, the control (as is, which was at room

temperature (23 °C)) was compared with 40°C to observe any changes in the nitrogen isotherm, then degassing was performed at 50, 75, 100, 150, and 200°C. Finally, based on high surface area and nitrogen adsorption at 75°C, 60-80°C degassing temperatures were investigated. During degassing, the valve should be opened gradually, preferably up to half-open, to prevent the sample from being uplifted by the vacuum and slowly opened to full. The pressure should decrease below 100 mTorr; otherwise, the system should be checked for leaks. After degassing, the sample was accurately weighed, as its mass is important for calculating surface area and pore volume. To prevent moisture uptake, the degassed sample should be protected from atmospheric exposure and promptly transferred to the surface area analyzer for measurement.

2.4. Analysis

The aerogels were analyzed using a Tristar II Plus surface area analyzer (Micromeritics Instrument Corporation, GA, USA). Nitrogen sorption experiments were carried out at -196 °C. The specific surface area was determined by multipoint BET adsorption at a relative pressure (p/p_0 ; equilibrium pressure of nitrogen at the sample surface/saturation pressure of nitrogen) between 0.05 and 0.3. The BJH pore size and volume were determined at relative pressures (p/p_0) greater than 0.35 [15].

2.5. Tristar II Plus mechanism

The instrument operates on the principle of gas physisorption, where nitrogen gas was adsorbed onto and desorbed from the surface of a solid sample under controlled relative pressures (P/P_0). During measurement, the sample tube was immersed in liquid nitrogen (-196 °C), and known quantities of nitrogen gas were introduced incrementally. The amount of nitrogen adsorbed at each relative pressure was determined volumetrically by monitoring pressure changes within the

system. The resulting adsorption–desorption isotherm was used to calculate specific surface area using the BET method, while pore volume and pore size distribution were obtained from the desorption branch using the BJH model. TriStar II Plus provides detailed information on the surface area, pore volume, and pore structure of aerogels.

Figure 2 illustrates the internal instrumental configuration of the TriStar II Plus surface area analyzer. The system operates using a volumetric gas adsorption principle, where precise pressure measurements were employed to determine the amount of nitrogen adsorbed by a sample. The instrument contains several pressure transducers with different measurement ranges to ensure high accuracy throughout the analysis. The 10 Torr transducer (1) was used for highly sensitive measurements at low pressures, which are particularly important for BET surface area determination and micropore analysis. The 1000 Torr manifold transducer (2) monitors pressure changes over a wider range and was used during gas dosing and equilibration. Individual port transducers for ports 1, 2, and 3 (3, 5, and 6) continuously monitor the pressure within each sample station, allowing simultaneous analysis of multiple samples. The elevator (4) raises and lowers the sample tube into the liquid nitrogen Dewar during analysis, maintaining the sample at -196 °C for nitrogen adsorption measurements. The P_o transducer (7) measures the saturation vapor pressure (P_o) of nitrogen, which was required for calculating the relative pressure (P/P_o) used to generate adsorption isotherms. Gas flow and pressure regulation throughout the system are controlled by the servo valve (8), which precisely meters nitrogen into the manifold and sample ports. Together, these components enable accurate measurement of nitrogen adsorption and desorption behavior, from which surface area, pore volume, and pore size distribution are calculated.

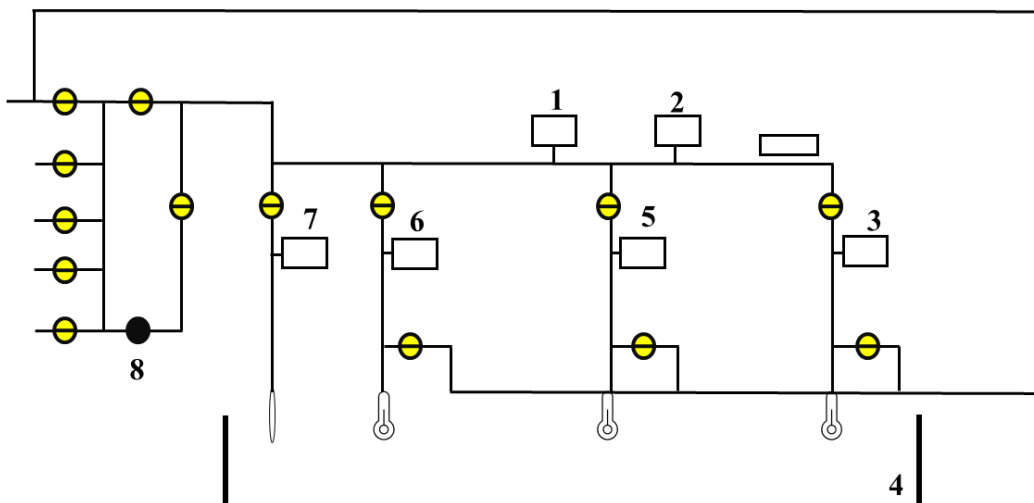


Fig. 2. The instrumental setup of Tristar II Plus. The figure labeling includes (1) 10 Torr transducer, (2) 1000 Torr manifold transducer, (3) port 3 transducer, (4) elevator, (5) port 2 transducer, (6) port 1 transducer, (7) Po transducer, and (8) servo valve.

2.6.BET and BJH assumptions

The BET surface area was determined according to the following equation [7, 16].

$$\text{BET Surface area} = \frac{A \times A_v}{M_v \times (S + Y)}$$

A: gas adsorbed molecular cross-sectional area per molecule; A_v : Avogadro's constant; M_v : molar volume of gas; S: slope of line obtained from BET plot; Y: Y-intercept obtained from BET plot

Assumptions:

- Multiple layers of gas were adsorbing on the surface of the material.
- Neglects any effect of localized phenomenon and treats the surface as uniform.
- Interactions between gas molecules and weakening of adsorption forces were ignored.
- The heat of adsorption for the first layer was higher than for successive layers.
- The heat of adsorption for the second layer and beyond equals the heat of condensation.

207

208 The BJH pore size and pore volume were determined based on Kelvin's equation [7, 16].

209 Kelvin's equation:

210
$$\ln\left(\frac{P}{P_0}\right) = -\frac{2\gamma V_m}{r_k RT}$$

211 P/P_0 : relative pressure; γ : surface tension of liquid nitrogen; V_m : molar volume of liquid nitrogen;

212 r_k : Kelvin radius; R : Universal gas constant; T : temperature

213 BJH equation for pore radius:

214
$$(r_p): r_p = r_k + t$$

215 t : thickness of adsorbed film on pore wall

216 Assumptions for BJH were based on Kelvin's equation and capillary condensation:

- 217 • Pores were cylindrical.
- 218 • Gas was ideal.
- 219 • The condensed liquid was incompressible.
- 220 • The molar volume of the liquid phase was negligible compared to the molar volume of the
- 221 gas.
- 222 • All pores were assumed to be filled at 1.0 relative pressure.

223

224 **2.7.Statistical analysis**

225 The statistical analysis was performed in JMP Pro 17.0 (SAS Institute, NC, USA) using ANOVA

226 and Tukey's HSD test with a 5% significance level. The results were expressed as the

227 mean $\pm$ standard deviation with duplicates.

228

229 **3. Results and discussion**

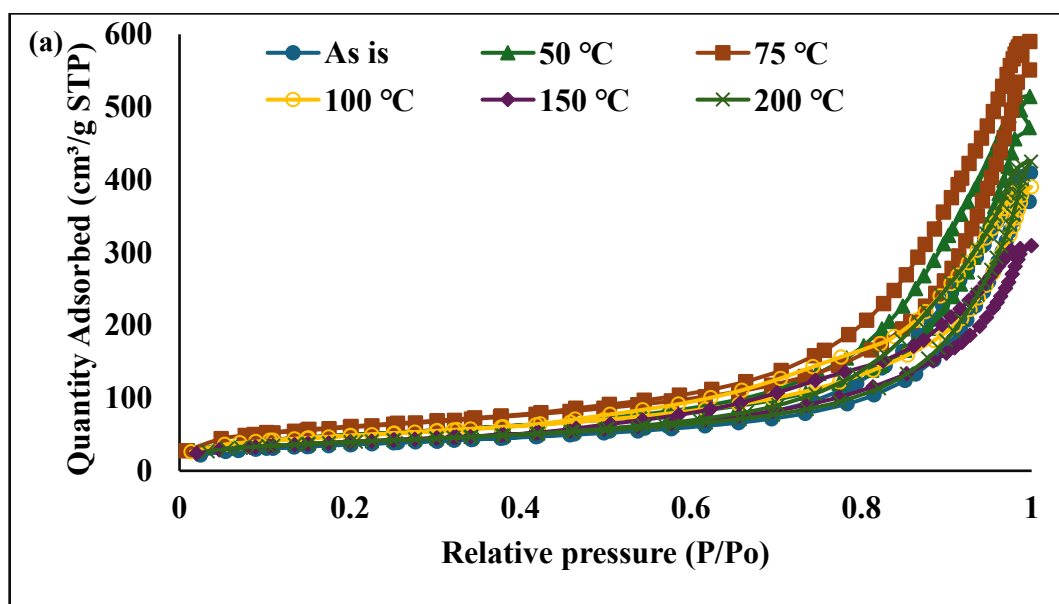
In aerogels and many mesoporous materials, nitrogen adsorption-desorption isotherms typically display Type IV behavior, characterized by a steep uptake at moderate relative pressures (capillary condensation) and a hysteresis loop [17].

3.1.Impact of degassing conditions on nitrogen isotherms

Nitrogen adsorption-desorption isotherms provide valuable information about surface area, pore volume, and pore size distribution. These measurements were carried out at -196 °C, where nitrogen gas interacts with the surface of a solid sample at the relative pressures (P/P_0) of <0.3 [18]. Before analysis, it's crucial to remove any physically adsorbed contaminants such as water, solvents, or other volatile residues; otherwise, it will affect the shape and interpretation of nitrogen adsorption-desorption isotherms for porous materials such as aerogels [9, 19]. The accuracy and reliability of the isotherm can be influenced by the degassing process, which depends on the combination of temperature, time and vacuum [19].

Figure 3 presents the nitrogen adsorption isotherms of aerogel samples degassed at different temperatures (50-200 °C) and compared with the untreated (as-is) sample. All samples exhibited similar adsorption behavior characteristic of mesoporous materials, with a gradual increase in nitrogen uptake at low relative pressures followed by a sharp increase at high relative pressures ($P/P_0 > 0.8$), corresponding to capillary condensation within the pore network. However, the total amount of nitrogen adsorbed varied substantially depending on the degassing temperature, indicating differences in pore accessibility and the effectiveness of contaminant removal. The sample degassed at 75 °C exhibited the highest nitrogen adsorption capacity. This result suggests that 75 °C was sufficient to remove residual moisture and solvent molecules trapped within the aerogel pores while preserving the porous structure. In contrast, the as-is sample showed the lowest

nitrogen uptake over the entire pressure range, confirming that residual adsorbates remained within the pore network and blocked access to adsorption sites. Degassing at 40 °C resulted in only a slight improvement compared with the untreated sample, indicating incomplete removal of physically adsorbed species at this temperature. Further increases in degassing temperatures from 80 to 200 °C did not improve the adsorption capacity. Instead, the nitrogen uptake decreased due to pore filling and capillary condensation. This behavior suggests that temperatures above the optimum condition may induce slight structural rearrangement, shrinkage, or densification of the aerogel network, reducing the accessible pore volume available for nitrogen adsorption [8-10].



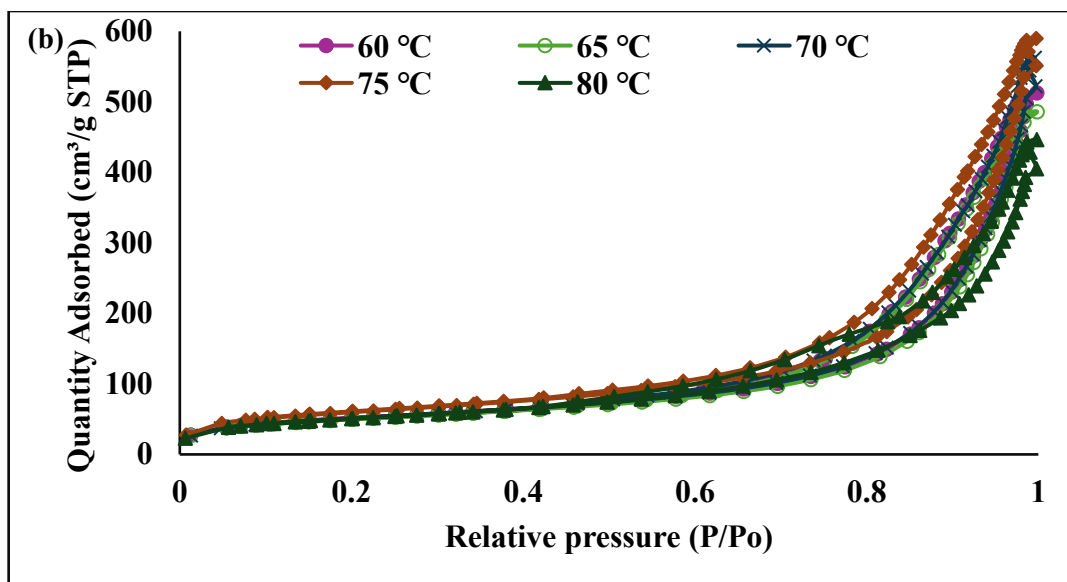


Fig. 3. The impact of different degassing temperatures, (a) 50-200 °C and (b) 60-80 °C, on nitrogen adsorption-desorption isotherm at 1 h degassing.

Further, Figure 4 shows the nitrogen adsorption-desorption isotherms of starch aerogel degassed at 75 °C for different durations ranging from 1 to 24 h. All samples exhibited similar isotherm profiles characteristic of mesoporous materials, indicating that the pore structure remained largely unchanged with degassing time. However, noticeable differences in nitrogen uptake were observed, particularly at high relative pressures ($P/P_0 > 0.8$), where capillary condensation occurs, and the total accessible pore volume can be assessed [8]. Samples degassed for beyond 1 h (4–24 h) showed very similar adsorption behavior and only slightly lower nitrogen uptake, suggesting that 1 h is enough degassing to remove residuals. This suggests that prolonged exposure to vacuum and elevated temperature may induce slight structural rearrangement or shrinkage within the aerogel network, reducing accessible pore volume similar to results observed by Singh, et al. [19] and Lapham and Lapham [9]. Singh, et al. [19] observed a reduction in gas adsorption in grain with a size of <1 mm. Although these changes appear relatively small, they

become evident at high relative pressures where differences in pore filling are most pronounced. The results indicate that degassing duration significantly influences the measured adsorption properties of the aerogel. An optimum degassing time of approximately 1 h at 75 °C was identified, providing sufficient removal of residual contaminants while minimizing potential alterations to the porous structure.

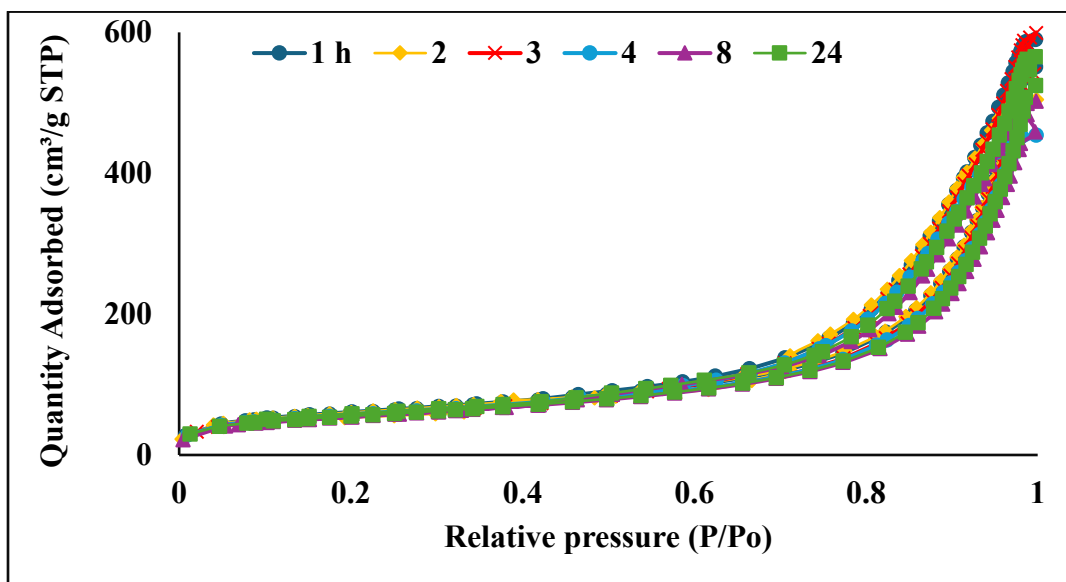


Fig. 4. The impact of different degassing durations on nitrogen adsorption-desorption isotherm at 75 °C.

3.2. Impact of degassing temperature on surface area, pore size and pore volume

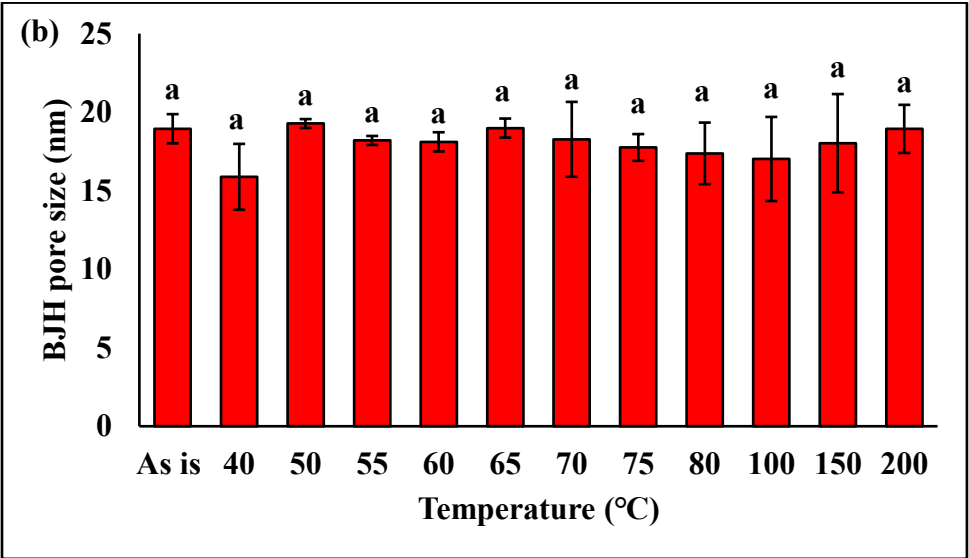
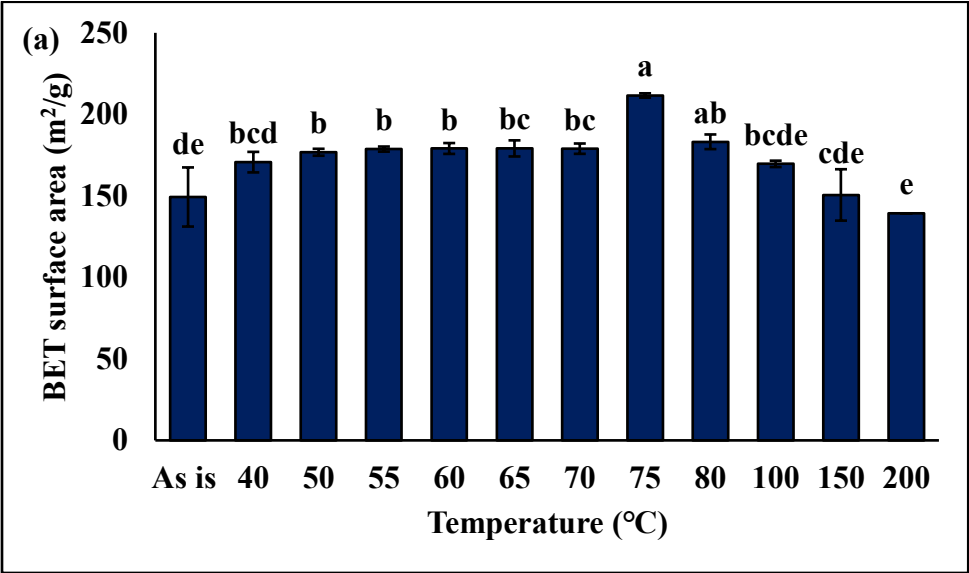
Figure 5 reveals the effect of degassing temperature on the textural properties of the aerogel. Degassing temperature is a critical factor in preparing samples for nitrogen adsorption analysis, as it directly influences the removal of adsorbed water or solvents from the surface and pores of materials [19]. The BET surface area increased from approximately 150 m²/g for the untreated sample to 171 m²/g at 40 °C and gradually increased with increasing degassing temperature,

reaching a maximum value of 212 m²/g at 75 °C (Figure 5a) (p<0.05). This trend is consistent with the nitrogen adsorption isotherms, where higher nitrogen uptake was observed as the degassing temperature increased from 40 to 75 °C. At lower temperatures, residual water and ethanol molecules likely remained trapped within the micropores and mesopores, partially blocking pore entrances and adsorption sites. As the degassing temperature increased from 40 to 75 °C, these contaminants were progressively removed, exposing more internal surface area and improving nitrogen accessibility.

Further, the average pore size difference was insignificant across different temperature ranges, varying between approximately 16 and 19 nm (Figure 5b) (p>0.05). Minor fluctuations were observed, indicating that degassing temperature primarily affected pore accessibility rather than significantly altering pore dimensions. Additionally, the total pore volume showed a similar trend to the surface area (Figure 5c). Pore volume increased from 0.64 cm³/g at 40 °C to a maximum of approximately 0.80 cm³/g at 70 °C (p>0.05). The increase in pore volume between 40 and 75 °C further supports the effective removal of residual adsorbates that initially occupied portions of the pore network. As pores became accessible, greater nitrogen uptake occurred at high relative pressures, resulting in higher pore volumes.

However, further increases in degassing temperature beyond the optimum range resulted in a decline in both surface area to ~178 m²/g and pore volume to 0.61 cm³/g at 200 °C. These results suggest that excessive degassing temperatures may induce structural rearrangement, pore contraction, or partial collapse of the aerogel network, reducing the accessible porosity available for nitrogen adsorption. Although the average pore size remained relatively unchanged, the reduction in surface area and pore volume indicates a loss of accessible internal surface rather than complete destruction of the mesoporous structure. Similar observations were revealed by Singh,

et al. [19] in shale, where higher temperatures (200 and 300 °C) reported lower surface areas and Lapham and Lapham [9] observed reductions in surface area and pore volume of magnesium stearate beyond 70 °C.



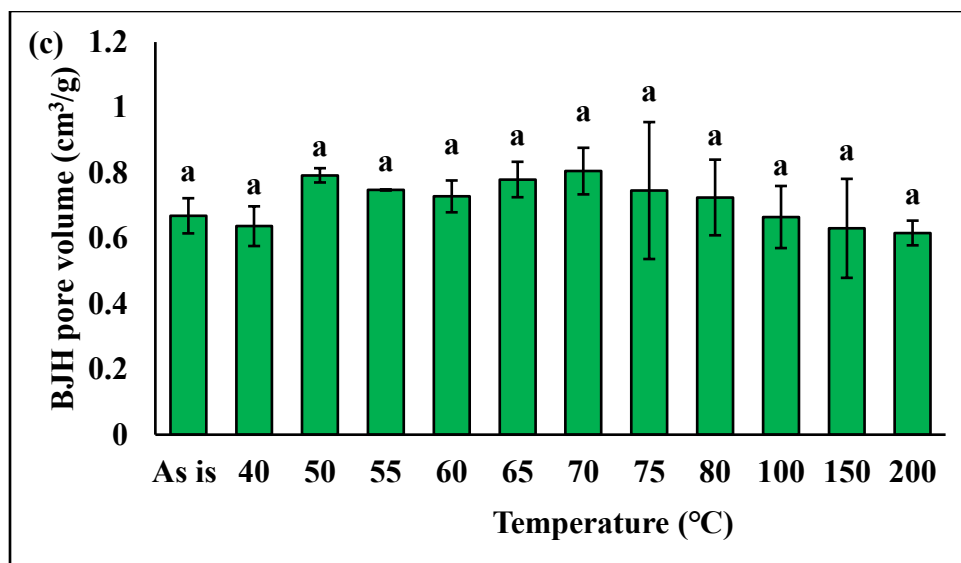


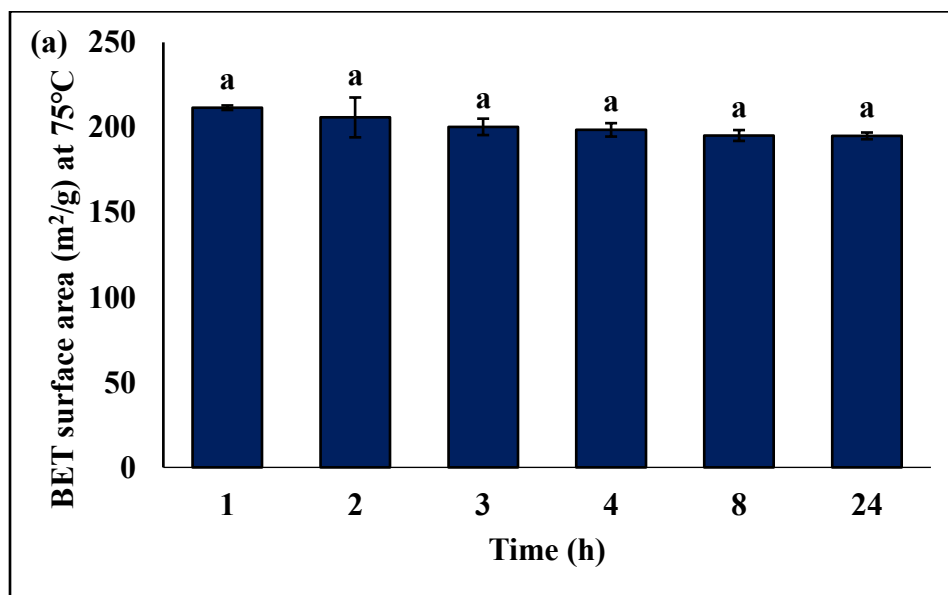
Fig. 5. The impact of degassing temperature on BET surface area (a), BJH pore size (b) and BJH pore volume (c) during 1 h degassing. “As is” was at room temperature (23 °C) without additional heating.

3.3. Impact of degassing duration on surface area, pore size and pore volume

Figure 6 reveals the influence of degassing duration on the textural properties of the aerogel at the optimized temperature of 75 °C. Degassing time plays a critical role in ensuring complete removal of residual moisture, solvents, and adsorbed gases from the pore network prior to nitrogen adsorption analysis. Insufficient degassing may leave contaminants within the pores, restricting nitrogen access and leading to inaccurate determination of surface area and pore volume. In contrast, long degassing durations may provide no additional benefit and may potentially alter the porous structure through prolonged exposure to heat and vacuum [9]. The BET surface area showed a gradual decrease with increasing degassing time (Figure 6a). The highest surface area was obtained after 1 h of degassing ($\sim 212 \text{ m}^2/\text{g}$), and at longer durations resulted in a lower surface area ($\sim 194 \text{ m}^2/\text{g}$) after 8–24 h. Although the differences were relatively small and were

insignificantly different ($p>0.05$), the trend suggests that most adsorbed contaminants were removed within the first hour of degassing. Prolonged degassing did not further increase surface area, indicating that pore cleaning had already reached completion. Instead, the slight reduction observed at longer durations may be associated with shrinkage of the aerogel network under extended vacuum and heating conditions.

Further, the average pore size remained relatively constant throughout the investigated time range, varying between ~ 16 - 18 nm (Figure 6b). The small variations observed suggest that degassing time had minimal influence on the pore size, and pore volume (Figure 6c) ($p>0.05$). Similar impacts of duration were observed by Lapham and Lapham [9] and Singh, et al. [19]. The sufficient duration of 1-4 h is required to remove residual contaminants; further degassing produces only minor changes in surface area, pore size, and pore volume. Consequently, a degassing time of 1 h at 75°C was selected as the optimum condition because it provided the highest nitrogen adsorption capacity together with stable textural properties and reproducible measurements.



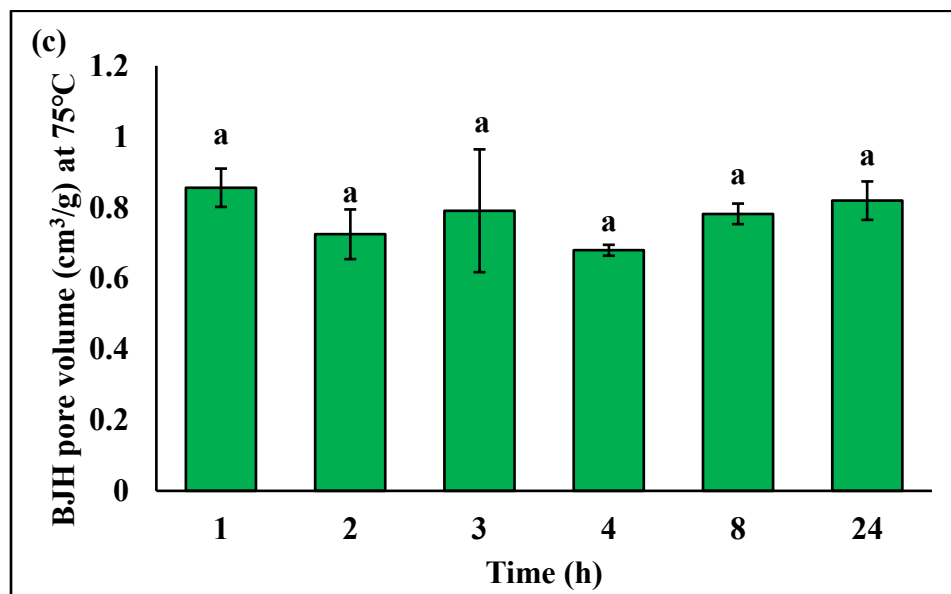
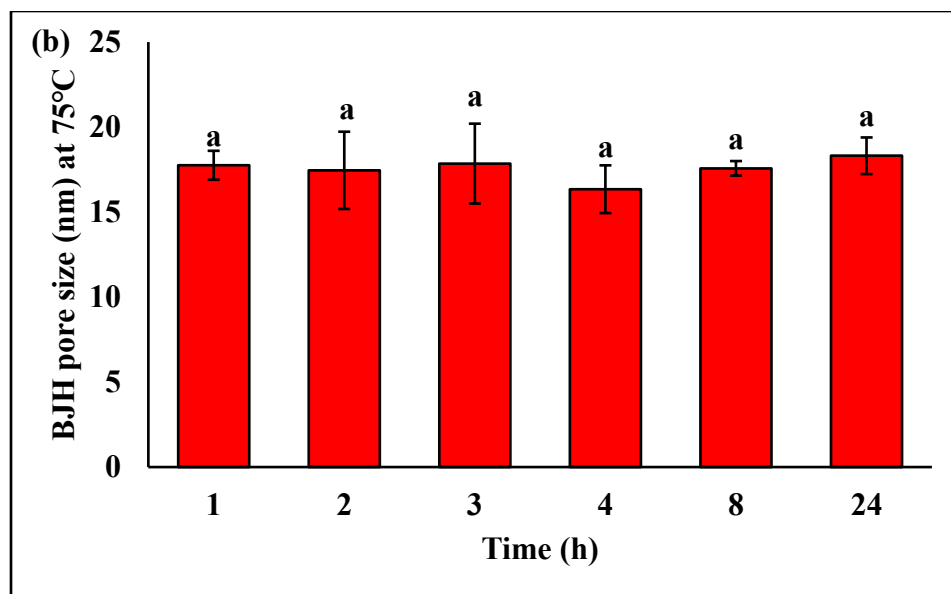


Fig. 6. The impact of degassing duration on surface area (a), pore size (b) and pore volume (c) at 75 °C.

4. Conclusions

This study evaluated the effects of degassing temperature and duration on the nitrogen adsorption characteristics and textural properties of starch-based aerogels. The results demonstrated that

degassing conditions significantly influence the measured surface area, pore volume, and adsorption behavior by affecting the removal of residual moisture and solvent molecules from the porous network prior to nitrogen physisorption analysis. Inadequate degassing resulted in lower nitrogen uptake and underestimated textural properties due to pore blockage by residual adsorbates, whereas excessive degassing conditions reduced adsorption capacity, likely due to structural rearrangement or partial loss of pore accessibility. Among the temperatures investigated, 75 °C revealed the optimum degassing temperature, producing the highest nitrogen adsorption capacity, and based on the combined analysis of adsorption isotherms, surface area, pore size, and pore volume, a degassing condition of 75 °C for 1 h revealed the highest BET surface area (212 m²/g and was the most suitable for accurate characterization of starch aerogels. Overall, the study highlights the importance of optimizing degassing parameters prior to nitrogen adsorption analysis to ensure reliable determination of aerogel textural properties while preserving the integrity of the porous structure.

Author contributions

Sumanjot Kaur: formal analysis, methodology, investigation, visualization, writing-original draft; **Ali Ubeyitogullari:** conceptualization, methodology, investigation, resources, project administration, supervision, funding acquisition, writing-review & editing.

Conflict of interest

The authors declare no conflict of interest.

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Data availability statement

No datasets were generated or analyzed during the current study.

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