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Use of polysaccharide-based aerogels in iced tea clarification process

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ABSTRACT

Polysaccharide-based aerogels, particularly those produced from chitosan and xanthan gum, offer a sustainable and efficient alternative for clarifying ready-to-drink (RTD) tea infusions. In this study, the aerogels were applied to black and green teas to reduce turbidity and tea cream formation during 20 days of storage. Key quality attributes were evaluated, including clarity (transmittance, tea cream), color (L^* , a^* , b^* , ΔE), and catechin content. Water mobility assessed by NMR relaxation and diffusion, molecular interactions with tea components examined by FTIR.

Chitosan aerogels significantly reduced cream formation and helped maintain color stability in black tea. In green tea, however, they caused more noticeable color changes, reflected by higher ΔE values, though catechin losses remained moderate. FTIR results showed surface-level interactions between aerogels and tea components such as proteins and polyphenols. NMR relaxometry also indicated reduced water mobility after treatment, supporting surface adsorption. Xanthan gum aerogels produced similar clarity improvements, although color shifts were slightly more pronounced in green tea. Despite these effects, both aerogels preserved essential quality characteristics.

Overall, food-grade polysaccharide aerogels serve as effective alternatives to conventional adsorbents. Their porous structure allows selective removal of turbidity-related compounds, while their easy handling and biodegradability make them promising options for beverage clarification.

1. Introduction

Ready-to-drink (RTD) iced tea has become an increasingly popular beverage worldwide due to its refreshing taste and the potential health benefits associated with tea polyphenols. Although various production methods exist, including the use of tea extracts, modern consumers increasingly favor more authentic and premium-tasting beverages. As a result, high-quality RTD iced tea products are commonly produced from real brewed teas, typically prepared through large-scale industrial batch brewing processes that yield flavorful and visually clear infusions (Dubey et al., 2020). The process involves the brewing of tea leaves with hot water, followed by filtration and clarification steps to eliminate tea cream and enhance the product's shelf life.

Black and green teas differ in their polyphenolic composition, oxidation level, antioxidant activity, and astringency. While green tea retains higher levels of catechins due to minimal processing, black tea is produced through full fermentation, leading to the formation of theaflavins and thearubigins. These compounds contribute to black tea's

characteristic color, brightness, strength, and taste, and they are also known to be the main components of tea cream (Dubey et al., 2020; Rao et al., 2011).

Due to the presence of proteins, polyphenols, free amino acids, caffeine, starch, polysaccharides, and pectin, tea infusions possess a colloidal unstable structure (Cifte et al., 2025; Rao et al., 2011). When stored below 4 °C, the interactions among these components become stronger, gradually leading to their precipitation and resulting in the formation of tea cream or tea haze, which disrupts the clarity of the infusion (Bindes, Cardoso, et al., 2019; Kim & Talcott, 2012). In addition to affecting the visual appearance of the product, tea haze also accelerates color and flavor degradation (Bindes et al., 2020; Rao et al., 2011).

There are several methods for the removal of tea cream including membrane filtration, ultrafiltration, adsorbents and enzymatic treatment (Argyle & Bird, 2015; Bindes et al., 2020; Chandini et al., 2013; Huang et al., 2007; Jöbstl et al., 2005; Kawakatsu et al., 1995; Rocha et al., 2017a; Su et al., 2009; Subramanian et al., 2014; Todisco et al.,

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2002). Among these, filtration is the most conventional approach, however, it often leads to a reduction in the concentration of bioactive compounds and organoleptic components (Dubey et al., 2020). The use of enzymes such as tannase, on the other hand, may reduce astringency but also negatively affect overall tea quality and is considered impractical due to the difficulty of enzyme recovery (Dubey et al., 2020). Adsorbent-based clarification offers a promising approach for removing tea cream and improving clarity without significantly altering the flavor, color, or polyphenol content of the infusion. Unlike filtration or enzymatic methods, adsorbents can provide selective removal of proteins, polyphenols, and other colloidal compounds responsible for turbidity (Rao et al., 2011).

Aerogels are considered a novel class of adsorbents due to their high surface area and large pore volume, and they have been effectively applied in various processes such as the regeneration of frying oil (Delice et al., 2024), treatment of dye- and oil-contaminated water (Nguyen et al., 2022), clarification of grape juice (Turhan Kara et al., 2024), and patulin removal from apple juice (Liu et al., 2021). Although chitosan and xanthan gum have been effectively used as clarifying agents in tea and juice clarification processes in their powder forms (Chatterjee et al., 2004; Chen et al., 2019; Domingues et al., 2012; Dubey et al., 2020; Erkan-Koç et al., 2015; Fang et al., 2007; Genovese & Lozano, 2001; Lachowicz et al., 2018; Rao et al., 2011; Rocha et al., 2017b; Tastan & Baysal, 2015; Taştan & Baysal, 2017), to the best of our knowledge, their aerogel forms have not been utilized for tea clarification applications. Given the superior adsorption properties of aerogels derived from their high porosity and surface area, it is assumed that chitosan and xanthan gum aerogels may offer an effective and innovative approach for tea clarification. Therefore, the aim of this study is to evaluate the clarification efficiency of chitosan and xanthan gum-based aerogels in black and green tea infusions, and to compare their performance with that of a conventional commercial adsorbent (silica).

2. Materials and method

2.1. Materials

Aerogels prepared from 2 g/100 mL chitosan and 10 g/100 mL xanthan gum were obtained from a previously conducted study in our laboratory (C2 and X10; (Namlı et al., 2025)). Green tea and black tea leaves were kindly provided from Dogadan Tea Company (Ankara, Türkiye). Silica (magnesium silicate) was obtained from Aropi Kimya (Konya, Türkiye). All other chemicals were purchased from Sigma-Aldrich Chemical Co. (St. Louis, MO, USA), and all reagents were of analytical grade.

2.2. Preparation of tea infusions and clarification

Black and green tea samples were brewed at 70 °C using a water-to-tea ratio of 1:30 (w/w) for 20 min. Throughout the brewing process, the infusions were stirred every 2 min to minimize leaf sedimentation and enhance extraction efficiency. Upon completion, the brewed teas were filtered through filter paper and subsequently cooled to room temperature. 0.1% sodium azide was added to tea infusions to prevent spoilage and then samples were centrifuged at 2862×g for 20 min and supernatants were collected as the samples. Control samples were taken from this step, and they were denoted as C-BT for black tea and C-GT for green tea infusion.

For the clarification of tea samples, chitosan and xanthan gum aerogels were used as adsorbents. The physical and chemical properties of these aerogels (C2 for chitosan aerogel and X10 for xanthan gum aerogel) were described in previous studies (Namlı et al., 2025). To evaluate the efficiency of these bio-based aerogels in comparison with a commercial adsorbent, silica (SiO₂) was also tested at the same concentration (Bindes, Reis, et al., 2019; Rao et al., 2011). Adsorbents were individually added to tea infusions at an adsorbent-to-tea ratio of 2%

(w/v) and stirred for 30 min. After the adsorption treatment, the infusions were filtered and centrifuged at 2862×g for 20 min. The resulting samples were stored at 4 °C until further analysis. Control samples for black tea (C-BT) and green tea (C-GT) were prepared without the addition of any adsorbent. However, control infusions were subjected to the same filtration, centrifugation and storage conditions as the adsorbent-treated samples to ensure comparability. Tea samples treated with chitosan and xanthan gum aerogels were labeled as Ch-BT, X-BT (for black tea) and Ch-GT, X-GT (for green tea), respectively. Samples treated with silica were labeled as Si-BT and Si-GT for black and green tea, respectively.

2.3. Determination of total phenolics, total solids and purity index

The total phenolics content (TPC) of tea infusions was determined using the Folin–Ciocalteu method, adapted from de Santana Magalhães et al. (2018). Briefly, 0.5 mL of tea sample was mixed with 2.5 mL of 10 mL/100 mL Folin–Ciocalteu reagent. After 3 min, 2.5 mL of sodium carbonate solution (7.5 g/100 mL) was added. The mixture was then incubated in the dark for 1 h. Following incubation, the absorbance was measured at 760 nm using a UV-Vis spectrophotometer (Optizen POP NanoBio, Mecasys Co., Ltd, Korea). A calibration curve was constructed using gallic acid standards (0.01–0.1 mg/mL), and results were expressed as mg gallic acid equivalents (GAE) per g of dry tea.

Total solid content (TSC) was determined gravimetrically by drying 5 mL of each sample in a drying oven at 105 °C for 24 h, followed by cooling in a desiccator and weighing to constant mass (Bindes et al., 2020). Tea purity was calculated as the ratio of total phenolics content to total solid content as described by Balyan and Sarkar (2018):

$$\text{Purity (\%)} = \frac{\text{TPC}}{\text{TSC}} \cdot 100$$

2.4. Determination of protein content

Since it is known that adsorbents can also bind proteins, the total protein content of tea infusions was determined by using the Bradford method (Bradford, 1976). For the analysis, a standard microplate protocol was applied. Briefly, 150 µL of tea infusion was pipetted into each microplate well, followed by the addition of 150 µL Coomassie Plus reagent. The microplates were shaken for 30 s and then incubated in dark at room temperature for 10 min. The absorbance was measured at 595 nm. Bovine serum albumin was used as the standard for constructing the calibration curve, and the results were expressed as mg protein per mL of tea infusion (mg/mL).

2.5. Quantification of catechins, gallic acid, caffeine, and L-theanine

Epicatechin (EC), epicatechin gallate (ECG), epigallocatechin (EGC), epigallocatechin gallate (EGCG), gallic acid (GA), caffeine and L-theanine were quantified using a high performance liquid chromatography (HPLC) system (Shimadzu Scientific Instruments Co. Ltd, Kyoto, Japan) equipped with a degasser (DGU-20A5), a pump (LC-20AD), an auto-sampler (SIL-20AHT), a column oven (CTO-20A) and a PDA detector (SPD-M20A). Separation was achieved using a Hypersil GOLD Phenyl column (250 x 4.6, 5 µm, Thermo Fisher Scientific Inc., USA). For the analysis of catechins, gallic acid, and caffeine, a linear gradient method was employed as described by Cifte et al. (2025). Prior to injection, all samples were filtered through a 0.45 µm nylon syringe filter. The mobile phase consisted of acetonitrile (A) and 0.1% (v/v) acetic acid in water (B) was supplied at a flow rate of 1 mL/min. The linear gradient elution program was as follows: 0–15 min, 10% to 20% A; 15–25 min, 20% to 40% A; 25–30 min, 40% to 10% A. The injection volume was 10 µL and the column temperature was maintained at 25 °C. Detection was performed at 275 nm. Quantification was based on external standard calibration, using individual linear calibration curves for each compound.

L-theanine was quantified according to the method described by Cifte et al. (2025), adapted from Murugesu et al. (2018). The analysis was conducted using a linear gradient elution program with a mobile phase consisting of acetonitrile (A) and water (B), applied as follows: 0-5 min, 2% A; 5-12 min, 2% to 50% A; 12-30 min, 50% A; 30-40 min, 50% to 2% A; 40-45 min, 2% A. The injection volume, column temperature, and flow rate was 10 μ L, 25 $^{\circ}$ C, and 1 mL/min, respectively. The peaks were identified at 200 nm and quantification was based on the peak area obtained from the standard linear calibration curve of L-theanine.

2.6. Viscosity measurement of tea infusions

The viscosity of tea infusions before and after clarification was measured to determine whether the adsorbents had any effect on flow behavior. Measurements were performed using a Kinexus rheometer (Malvern, UK). A cup-and-bob geometry was used to measure the viscosity at 25 $^{\circ}$ C, under a shear rate range of 5-100 s^{-1} .

2.7. Measurement of tea turbidity

The turbidity of tea samples was determined by measuring the light transmittance at 640 nm by using a UV-Vis spectrophotometer (Optizen POP NanoBio, Mecasys Co., Ltd, Korea) and de-ionized water was used as a blank. Transmittance values were recorded on days 0, 10, and 20 during storage.

2.8. Quantification of tea cream formation

The formation and accumulation of tea cream were monitored throughout the storage period. Tea samples were collected on days 5, 10, 15, and 20. Preweighed tea samples were centrifuged at 2862 \times g for 10 min at 4 $^{\circ}$ C, and the supernatant was discarded. The resulting precipitate, representing the tea cream, was weighed, and its percentage was calculated based on the initial sample weight.

2.9. Color analysis by CIELAB system

Color measurements of tea samples were performed using a colorimeter (SL400, Serlab Co. Ltd., İstanbul, Türkiye) based on the CIELAB color space. Results were expressed as lightness (L^*), chromaticity coordinates (a^* , b^*), and total color difference (ΔE). To evaluate color stability, measurements were also conducted after 20 days of storage. The total color difference (ΔE) was calculated by using the following equation:

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2}$$

where L^* , a^* , and b^* represent the color parameters of the sample after treatment or storage, and L_0^* , a_0^* , and b_0^* correspond to the initial color values of the control samples (C-BT and C-GT) measured on day 0.

2.10. Fourier Transform Infrared (FTIR) spectroscopy analysis

The changes of aerogels at a molecular level after adsorption were investigated by FTIR analysis (IR Affinity-1 Spectrometer, Shimadzu Corporation, Japan) by using Attenuated Total Reflectance unit with zinc selenide crystal in the range of 4000-600 cm^{-1} at a resolution of 4 cm^{-1} with 32 scans.

2.11. Time domain nuclear magnetic resonance (TD-NMR) relaxometry and self diffusion analysis

T_2 relaxation times were measured using a CPMG sequence at 0.5 T (proton Larmor frequency 20.34 MHz) with a Time-Domain Nuclear

Magnetic Resonance (TD-NMR) system (Spin Track, Resonance Systems GmbH, Germany). Measurements of chitosan aerogels were performed with an echo time of 2000 μ s, 1500 echoes, and 32 scans, while measurements of xanthan gum aerogels were performed with an echo time of 1000 μ s, 500 echoes, and 4 scans.

Self-diffusion coefficient measurements were conducted using a bench-top MRI system (Pure Devices GmbH, Germany) operating at a frequency of 24.15 MHz, equipped with a gradient amplifier (maximum gradient strengths: x-axis 1.229 T/m, y-axis 1.230 T/m, z-axis 1.515 T/m) and a 10 mm RF coil. A pulsed gradient spin echo (PGSE) sequence was applied for the analysis, with an estimated diffusion coefficient of $2.3 \times 10^{-9} m^2/s$ and an echo time of 20 ms.

Prior to measurement, aerogels were removed from tea, gently blotted with paper towels to remove excess liquid, and then analyzed. For comparison, measurements were also carried out on aerogels that had been dipped in water.

2.12. Statistical analysis

To investigate the effects of adsorbents, statistical analyses were conducted using analysis of variance (ANOVA) in MINITAB software (version 16, Minitab Inc., Coventry, UK). Tukey's comparison test was applied with a 95% confidence interval. All measurements were performed in triplicate or more, and results were presented as the mean \pm standard deviation. Prior to analysis all ANOVA assumptions were verified. Different letters in the tables indicate statistically significant differences among the samples ($p < 0.05$).

3. Results and discussion

3.1. Total phenolics, total solids and purity and total protein content

The total phenolics content (TPC) provides a general estimation of the overall phenolic composition in tea extracts, which is closely associated with their antioxidant capacity. Green tea is particularly rich in polyphenols, including both low-molecular-weight compounds such as flavan-3-ols (catechins) and more complex structures such as flavonols (Parvez & Wani, 2024). On the other hand, the polyphenolic profile of black tea differs from that of green tea, as some of the basic catechins are oxidized and polymerized during the fermentation process. This transformation leads to the formation of theaflavins and thearubigins, which are responsible for the characteristic color and astringency of black tea (Zhang et al., 2019).

Results showed that total phenolics content of black tea samples did not significantly differ among treatments ($p > 0.05$), ranging from 148 to 181 mg GAE/g (Table 1). Although Ch-BT showed a slightly lower TPC (148 mg GAE/g), possibly due to polyphenol binding affinity of chitosan, this reduction was not statistically significant. Chitosan is known to interact with negatively charged molecules such as polyphenols, and it can coagulate these compounds during clarification (Rao et al., 2011). Therefore, the absence of a significant decrease in TPC after chitosan aerogel treatment may indicate that black tea polyphenols were relatively well preserved.

On the other hand, treatment with chitosan aerogels led to a significant reduction in the TPC of green tea samples (167 mg GAE/g), indicating stronger interactions between chitosan and green tea polyphenols compared to those in black tea. In contrast, Si-GT and X-GT retained relatively high levels of TPC like the control samples, suggesting that silica powder and xanthan gum aerogel treatment caused less polyphenol loss. While TPC offers an overall picture, specific phenolic compounds were also identified and quantified via HPLC analysis, and their individual contributions will be discussed in the following sections.

Clarification led to varying reductions in total solid content (TSC) (Table 1). X-BT had the highest TSC (299 mg/g), among black tea samples, suggesting a minimal removal of soluble solids by xanthan gum aerogel. Ch-BT exhibited the lowest TSC (218 mg/g), indicating a more

Table 1
Total phenolics content, total solids, purity and protein content of tea samples.

	TPC (mg GAE/g dry tea)	TSC (mg/g)	Purity (%)	Protein Content (mg/mL)
C-BT	174 ± 10 ^a	252 ± 22 ^{ab}	63.8 ± 4.9 ^{ab}	1.81 ± 0.02 ^a
Si-BT	180 ± 18 ^a	248 ± 9 ^{ab}	76.0 ± 1.1 ^a	1.73 ± 0.01 ^b
Ch-BT	148 ± 9 ^a	218 ± 17 ^b	69.3 ± 3.1 ^{ab}	0.86 ± 0.08 ^c
X-BT	181 ± 9 ^a	299 ± 21 ^a	60.7 ± 4.8 ^b	1.82 ± 0.02 ^a
C-GT	182 ± 3 ^A	355 ± 15 ^A	51.2 ± 2.1 ^B	1.78 ± 0.03 ^A
Si-GT	187 ± 5 ^A	311 ± 4 ^B	60.3 ± 1.3 ^A	1.76 ± 0.03 ^A
Ch-GT	167 ± 6 ^B	264 ± 25 ^C	60.8 ± 2.6 ^A	1.15 ± 0.03 ^B
X-GT	182 ± 5 ^A	345 ± 4 ^A	52.5 ± 0.8 ^B	1.71 ± 0.04 ^A

Lower case and upper case letters indicate significant differences among black tea and green tea samples, respectively ($p < 0.05$).

aggressive removal of soluble material, which is in line with chitosan's coagulating ability. A similar trend was observed in green tea samples as well, where Ch-GT had the lowest TSC (264 mg/g), while X-GT and C-GT retained the highest levels, again highlighting that xanthan aerogel-based clarification has minimal impact on soluble solids.

The results are also in-line with purity of tea samples (Table 1). In black tea samples, Si-BT achieved the highest purity (76.0%), reflecting an effective clarification, while X-BT showed the lowest (60.7%). The purity increase was more pronounced in silica-treated samples for both tea types (Si-BT and Si-GT), confirming silica's superior ability to remove turbidity-causing compounds. In green tea samples, Si-GT and Ch-GT displayed the highest purity (around 60%), while X-GT and the control (C-GT) remained lower (around 52%), supporting that xanthan gum aerogel may be less effective in removing non-soluble impurities.

Protein content of tea samples was also affected by the clarification treatments, with the most pronounced reduction observed in the chitosan-treated samples (Table 1). Ch-BT and Ch-GT showed significantly lower protein concentrations (0.86 and 1.15 mg/mL, respectively) compared to their controls and other treatments. This reduction can be attributed to chitosan's strong affinity for proteins, which is mainly due to electrostatic interactions between the positively charged amino groups of chitosan and the negatively charged sites on protein molecules (Rao et al., 2011). As a result, proteins are effectively bound and precipitated during the clarification process. In contrast, silica and xanthan gum treatments caused only minor changes in protein content, suggesting weaker or negligible interactions with soluble tea proteins. The ability of chitosan to remove protein compounds is advantageous for improving clarity, but it may also reduce the nutritional or functional protein content of the final product as well (Yahyaoui et al., 2018).

3.2. Catechins, gallic acid, caffeine, and L-theanine content of tea samples

The analysis of tea individual tea components such as catechins, gallic acid, caffeine and L-theanine showed notable differences among the treated and untreated samples (Table 2). Chitosan and xanthan gum aerogel treatment resulted in a significant reduction in gallic acid (GA) levels in both black and green tea samples. Gallic acid is a naturally

occurring phenolic acid found in tea, and it contributes to the antioxidant properties while also playing an important role in the cream formation through hydrogen bonding interactions with other compounds (Chandini et al., 2011; Cifte et al., 2025). It exists both in its free form and as part of gallated catechins, while in black tea it may also originate from the degradation of catechins during fermentation (Zhang et al., 2019). In the recent studies, the gallic acid content in black tea was reported to range from 118 to 377 mg/100 g dry tea, whereas in green tea it ranged from 45 to 180 mg/100g dry tea (Aydemir et al., 2024; Jakabová et al., 2024; Zhao et al., 2019). The values obtained in the presented study after clarification fall within these reported ranges. Therefore, while the removal of tea cream forming compounds is beneficial for visual clarity, it is also essential to preserve antioxidant constituents such as gallic acid to maintain the overall functional quality of tea. The current findings suggest that although some loss of phenolic compounds may occur with aerogel treatments, a balanced approach can be achieved by optimizing the clarifier type and treatment conditions to retain bioactive compounds while improving clarity as well.

Green tea samples naturally exhibited much higher concentrations of catechins than black tea, as expected. Chitosan aerogel caused a significant reduction in EGC and EC levels in Ch-GT compared to control, indicating a strong interaction with monomeric flavan-3-ols. Similarly, EGC content dropped from 1340 to 1070 mg/100 g, although this change was not statistically significant ($p > 0.05$). In black tea samples, differences were less pronounced. Ch-BT showed a slight reduction in EGC and ECG, but EC and EGCG levels were relatively unaffected. This supports the idea that oxidative polymerization during black tea fermentation leads to more stable polyphenol structures, such as theaflavins and thearubigins, which are less effective towards chitosan and xanthan gum aerogel (Zhang et al., 2019). On the other hand, gallated catechins such as EGCG and ECG have a higher tendency to promote tea cream formation due to the presence of additional hydroxyl groups from the gallic acid moiety. These hydroxyl groups increase their hydrogen bonding capacity, thereby facilitating the aggregation of polyphenols, caffeine, and other tea constituents that contribute to tea cream development (Chandini et al., 2011). Therefore, a controlled reduction in these catechins could help reduce tea cream formation and improve

Table 2
Catechins, gallic acid, caffeine and L-theanine content of tea (mg/100g dry tea).

	GA	EGC	EC	EGCG	ECG	Caffeine	L-theanine
C-BT	358.7 ± 15.4 ^a	317.2 ± 2.0 ^a	496.2 ± 6.0 ^a	483.9 ± 43.6 ^a	351.3 ± 18.7 ^a	1922.1 ± 141.9 ^a	485.9 ± 32.9 ^a
Si-BT	358.1 ± 2.4 ^a	313.5 ± 3.3 ^a	407.5 ± 18.6 ^b	480.3 ± 13.5 ^a	344.3 ± 30.0 ^a	1916.5 ± 95.8 ^a	472.0 ± 13.7 ^a
Ch-BT	182.6 ± 5.7 ^b	233.4 ± 4.7 ^b	437.1 ± 29.8 ^{ab}	469.6 ± 5.6 ^a	232.8 ± 2.2 ^b	1798.1 ± 16.5 ^a	422.7 ± 37.3 ^a
X-BT	191.5 ± 13.4 ^b	318.2 ± 11.9 ^a	480.0 ± 25.5 ^a	482.0 ± 20.7 ^a	351.0 ± 5.0 ^a	1876.2 ± 132.9 ^a	476.6 ± 8.4 ^a
C-GT	198.3 ± 4.9 ^A	2706.7 ± 109.7 ^A	909.0 ± 51.9 ^A	3007.7 ± 188.7 ^A	1340.6 ± 109.4 ^A	1576.6 ± 72.0 ^A	658.9 ± 12.3 ^A
Si-GT	196.3 ± 6.6 ^A	2558.4 ± 219.1 ^A	754.3 ± 72.0 ^{AB}	3004.7 ± 48.6 ^A	1328.7 ± 2.0 ^A	1527.8 ± 85.6 ^A	532.2 ± 49.9 ^B
Ch-GT	168.1 ± 1.7 ^C	1929.9 ± 116.0 ^B	674.9 ± 6.5 ^B	2653.2 ± 48.4 ^A	1070.3 ± 111.0 ^A	1439.0 ± 32.5 ^A	488.6 ± 24.7 ^B
X-GT	183.0 ± 3.7 ^B	2629.0 ± 45.6 ^A	753.7 ± 43.7 ^{AB}	2860.5 ± 37.8 ^A	1330.7 ± 59.0 ^A	1530.4 ± 88.0 ^A	536.7 ± 46.8 ^B

Lower case and upper case letters indicate significant differences among black tea and green tea samples, respectively ($p < 0.05$).

clarity, if the nutritional and functional qualities of the tea are maintained. In this regard, while chitosan aerogel showed some ability to reduce catechin levels in green tea, particularly EGC and EC, none of the adsorbents caused a statistically significant decrease in EGCG content. Importantly, both the initial and post-treatment catechin contents fall within the ranges previously reported for black and green tea samples, supporting the validity of the results and indicating that the clarification process did not drastically alter the catechin profile (Aydemir et al., 2024; Jakobová et al., 2024; Zhao et al., 2019).

When the total catechin concentration (GA + EGC + EC + EGCG + ECG) of tea samples after treatment with aerogels and silica was evaluated, the results showed a trend consistent with the total phenolic content (TPC) values. While control, silica and xanthan gum aerogel-treated samples exhibited similar catechin levels in both tea types, chitosan aerogel-treated samples showed a slight decrease in total catechin content, indicating some adsorption of these polyphenolic compounds.

Caffeine, one of the main purine alkaloids found in tea, is known to contribute to the health-promoting effects of tea, especially by supporting its antioxidant and anticancer properties. Caffeine content was largely stable across all adsorbent treatments for both tea types. This suggests that caffeine, being a small, neutral molecule does not strongly interact with the tested adsorbents.

L-theanine, a non-protein amino acid in tea, is responsible for anticancer properties and characteristic umami taste of tea (Shojaei-Zarghani et al., 2021). L-theanine contents were significantly affected by clarification treatments in green tea samples, with all adsorbent-treated groups showing a lower content compared to control ($p < 0.05$). This indicated that clarification may lead to partial removal of free amino acids, possibly due to weak ionic or hydrogen bonding interactions with the adsorbents. However, since there was no statistically significant difference among the chitosan aerogel, xanthan gum aerogel and silica, no definitive conclusion could be done regarding the specific affinity of each material for L-theanine. In contrast, no statistically significant difference was observed among black tea sample ($p > 0.05$), suggesting that the matrix composition or lower initial L-theanine content in black tea may limit its interaction with the adsorbents.

3.3. Viscosity of tea infusions

Viscosity measurements of the clarified tea samples showed relatively small variations depending on the type of adsorbent used (Table 3). In black tea samples, viscosity ranged between 1.659 and 1.810 mPa s, with the highest value observed in X-BT. Although the differences are not drastic, this increase may be attributed to the residual presence or solubilization of xanthan gum components in the clarified tea extract, which are known to increase solution viscosity even at low concentrations due to its strong water-binding capacity and extended molecular conformation. In contrast, chitosan aerogel and silica treatments resulted in slightly lower viscosities compared to the control. This behavior suggests that these adsorbents may remove the soluble solids in tea infusions, or disrupt macromolecular structures such as protein-

Table 3

Viscosity of tea infusions.

	Viscosity (mPa.s)	R ²
C-BT	1.683	0.987
Si-BT	1.659	0.991
Ch-BT	1.670	0.993
X-BT	1.810	0.989
C-GT	1.760	0.991
Si-GT	1.726	0.989
Ch-GT	1.747	0.989
X-GT	2.009	0.991

polyphenol complexes which also affects the apparent viscosity of tea infusions.

In green tea samples, the viscosity values were generally higher than those in black tea, consistent with their greater content of catechins, amino acids, and soluble solids. The highest viscosity again obtained in the X-GT sample, suggesting that xanthan gum aerogels had a similar effect in both tea types. On the other hand, chitosan aerogel and silica did not much alter viscosity compared to control, further supporting their role in selective clarification without significantly impacting the fluid properties of the tea. From an industrial perspective, the limited changes in viscosity observed after clarification are advantageous, as they suggest that aerogel-based clarification processes do not require additional modifications to existing production systems or commonly used operations such as pumping, filling, or heat exchange.

The high R² values observed in all samples indicate a strong linear relationship between the shear stress and the shear rate, suggesting that the tea infusions behaved as a Newtonian fluid, as expected. This result indicates that clarification by tested aerogels does not cause undesirable non-Newtonian flow characteristics of tea infusions.

3.4. Tea turbidity and tea cream

It is known that tea cream develops more prominently during cold storage, as polyphenols, caffeine, and other components aggregate and precipitate at lower temperatures (Kim & Talcott, 2012). Therefore, the combined analysis of transmittance and tea cream formation provided a comprehensive understanding of the visual clarity and physical stability of tea infusions during cold storage of 20 days (Tables 4 and 5). In black tea samples, cream formation increased significantly with storage time ($p < 0.05$). C-BT showed a rapid increase in tea cream amount over time, reaching 6.9% at day 20, accompanied by a substantial drop in transmittance from 44.2% to 14.6%. This strong inverse relationship confirms that cream formation is a major contributor to turbidity and visual deterioration. Among the treatments, Ch-BT exhibited the lowest tea cream accumulation and a moderate final transmittance value, indicating its effectiveness in reducing precipitable compounds without completely eliminating turbidity. Xanthan gum aerogel treatment (X-BT) resulted in comparable tea cream content to Ch-BT by day 20, but with a much lower transmittance value, suggesting that although less visible precipitate was formed, suspended particles or colloidal haze might still persist in the infusion, contributing to turbidity.

In green tea samples, all treatments maintained high transmittance throughout storage, with final values ranging from 64.3% to 78.4%. However, differences in tea cream formation were more pronounced. Control and silica treated green tea samples exceeded 7% tea cream by day 20, whereas chitosan aerogel and xanthan gum aerogel treatment significantly reduced this value to around 5%, highlighting aerogel's selective removal or stabilization of cream-forming components.

Table 4

Transmittance (%) of tea infusions during storage time of 20 days.

	Time (days)		
	0	10	20
C-BT	44.2 ± 0.0 ^d	21.5 ± 0.8 ^b	14.6 ± 0.4 ^b
Si-BT	59.5 ± 0.1 ^b	22.6 ± 1.3 ^b	15.1 ± 0.5 ^b
Ch-BT	64.4 ± 0.3 ^a	75.2 ± 0.1 ^a	63.5 ± 2.9 ^a
X-BT	54.2 ± 0.0 ^c	22.2 ± 1.3 ^b	16.1 ± 1.0 ^b
C-GT	74.3 ± 0.1 ^B	78.5 ± 0.2 ^C	69.6 ± 0.3 ^B
Si-GT	77.5 ± 0.1 ^A	82.3 ± 0.5 ^A	77.1 ± 0.7 ^A
Ch-GT	72.6 ± 0.1 ^C	79.2 ± 0.1 ^{BC}	78.4 ± 0.0 ^A
X-GT	72.1 ± 0.0 ^D	79.9 ± 0.2 ^B	64.3 ± 2.9 ^C

Lower case letters indicate significant differences among black tea samples, and uppercase letters indicate significant difference among green tea samples at the same storage day ($p < 0.05$).

Table 5
Tea cream formation (%) during storage time of 20 days.

	Time (days)			
	5	10	15	20
C-BT	1.0 ± 0.0 ^h	3.2 ± 0.1 ^d	3.9 ± 0.1 ^c	6.9 ± 0.1 ^a
Si-BT	0.7 ± 0.0 ⁱ	2.3 ± 0.1 ^e	2.9 ± 0.1 ^d	4.3 ± 0.2 ^b
Ch-BT	0.6 ± 0.0 ⁱ	1.8 ± 0.1 ^{fg}	2.1 ± 0.1 ^{ef}	3.8 ± 0.2 ^c
X-BT	1.7 ± 0.1 ^g	2.0 ± 0.1 ^{fg}	2.4 ± 0.1 ^e	6.7 ± 0.3 ^a
C-GT	1.7 ± 0.0 ^H	3.4 ± 0.0 ^{EF}	4.7 ± 0.0 ^C	7.6 ± 0.1 ^A
Si-GT	1.7 ± 0.1 ^H	3.9 ± 0.2 ^D	4.5 ± 0.2 ^C	7.8 ± 0.2 ^A
Ch-GT	1.8 ± 0.0 ^H	3.3 ± 0.1 ^{EF}	3.6 ± 0.1 ^{DE}	4.8 ± 0.1 ^{BC}
X-GT	2.4 ± 0.1 ^G	2.8 ± 0.1 ^{FG}	3.1 ± 0.1 ^F	5.2 ± 0.1 ^B

Lower case letters indicate significant differences among black tea samples and uppercase letters indicate significant difference among green tea samples across all storage days, based on two-way ANOVA analysis ($p < 0.05$).

Notably, Ch-GT had both the highest transmittance and the lowest tea cream formation, suggesting that chitosan aerogel may be especially effective in preserving optical quality of green tea without compromising the stability. The findings were further supported by two-way ANOVA, which confirmed that both sample type and storage time had a statistically significant effect on tea cream formation ($p < 0.05$). This result suggests that even if treatment effects are notable, the storage time remains a dominant driver of tea cream accumulation.

The results are also consistent with the literature, where chitosan was reported to interact with anionic colloids such as pectin and proteins, contributing to its clarifying potential (Bindes, Reis, et al., 2019). However, aerogel formulations offer distinct practical advantages over conventional chitosan dispersions, such as easy removal after treatment and the possibility to customize selectivity, allowing removal of larger macromolecules while preserving beneficial components like polyphenols and color pigments.

3.5. Color change of tea infusions

Color is one of the key quality attributes of tea infusions and an important indicator of chemical changes during storage. The color parameters L^* (lightness), a^* (red-green), and b^* (yellow-blue) as well as the total color difference (ΔE) were used to evaluate the visual appearance changes in tea samples after 20 days of storage (Table 6).

In black tea samples, a noticeable increase in lightness (L^*) values was observed for all treatments over 20 days. Among them, although Si-BT showed the highest increase in L^* , the visual turbidity remained high, indicating that clarification was not effective.

This was also reflected in the highest ΔE , suggesting substantial color change. On the other hand, Ch-BT and X-BT demonstrated much smaller ΔE values after 20 days of cold storage (1.9 and 2.1, respectively), indicating better color stability, with minimal visual changes.

Table 6
Color parameters of tea infusions during 20 days of storage.

	L^*		a^*		b^*		ΔE	
	Day 0	Day 20	Day 0	Day 20	Day 0	Day 20	Day 0	Day 20
C-BT	28.1 ± 0.3 ^c	31.3 ± 0.1 ^b	1.8 ± 0.1 ^e	5.7 ± 0.1 ^b	3.2 ± 0.2 ^d	8.8 ± 0.1 ^b	-	7.7 ± 0.1 ^b
Si-BT	27.0 ± 0.1 ^e	32.3 ± 0.1 ^a	1.9 ± 0.1 ^e	6.5 ± 0.2 ^a	1.6 ± 0.1 ^g	9.7 ± 0.1 ^a	1.9 ± 0.1 ^{cd}	9.2 ± 0.2 ^a
Ch-BT	27.5 ± 0.1 ^d	28.0 ± 0.1 ^c	0.8 ± 0.1 ^f	2.8 ± 0.0 ^d	2.7 ± 0.1 ^e	4.8 ± 0.0 ^c	1.3 ± 0.1 ^f	1.9 ± 0.2 ^{de}
X-BT	26.8 ± 0.1 ^e	27.5 ± 0.0 ^d	2.0 ± 0.1 ^e	3.2 ± 0.0 ^c	2.3 ± 0.1 ^f	4.8 ± 0.1 ^c	1.7 ± 0.1 ^e	2.1 ± 0.0 ^c
C-GT	29.4 ± 0.0 ^{AB}	27.8 ± 0.0 ^D	-0.7 ± 0.1 ^E	-0.3 ± 0.0 ^{BC}	3.0 ± 0.1 ^D	4.8 ± 0.0 ^B	-	2.5 ± 0.0 ^B
Si-GT	26.5 ± 0.2 ^F	28.1 ± 0.3 ^C	-0.5 ± 0.0 ^D	-0.3 ± 0.0 ^{BC}	4.4 ± 0.1 ^C	5.7 ± 0.1 ^A	3.2 ± 0.2 ^A	3.0 ± 0.1 ^A
Ch-GT	29.2 ± 0.0 ^B	27.1 ± 0.1 ^E	-0.9 ± 0.0 ^F	0.7 ± 0.0 ^A	2.9 ± 0.1 ^D	4.3 ± 0.1 ^C	0.3 ± 0.0 ^C	3.0 ± 0.0 ^A
X-GT	29.5 ± 0.1 ^A	27.8 ± 0.1 ^D	-0.6 ± 0.0 ^{DE}	-0.2 ± 0.0 ^B	3.1 ± 0.0 ^D	5.0 ± 0.2 ^B	0.2 ± 0.0 ^C	2.7 ± 0.1 ^B

ΔE values were calculated using C-BT (day 0) and C-GT (day 0) as benchmarks. Lower case letters indicate significant differences among black tea samples and uppercase letters indicate significant differences among green tea samples across all storage days, based on two-way ANOVA analysis for each color parameter ($p < 0.05$).

Particularly for Ch-BT, the limited change in L^* , a^* , and b^* values indicate effective stabilization of the color pigments in the black tea matrix during storage.

In green tea samples, initial L^* values were generally higher than in black tea, consistent with their lighter natural hue. Also, the a^* values in green tea shifted toward zero during storage, suggesting a loss of green hue and the b^* values increased significantly in all samples, indicating enhanced yellowness. Also, the Ch-GT sample initially exhibited a greener hue compared to the C-GT sample, suggesting that chitosan aerogel may have interacted more strongly with chlorophyll-related pigments, potentially due to its cationic nature and affinity toward negatively charged compounds (Rao et al., 2011; Yahyaei et al., 2018).

In green tea samples, chitosan aerogel and silica treatments led to greater ΔE values compared to control, suggesting that while effective in clarification, these treatments may have caused more noticeable color shifts during cold storage. These findings suggest that polysaccharide-based aerogels not only contribute to the clarification of tea infusions but can also affect the visual stability of the product depending on tea type. Therefore, their impact on color should be carefully evaluated, particularly for green teas where minor color differences may influence consumer perception and product quality.

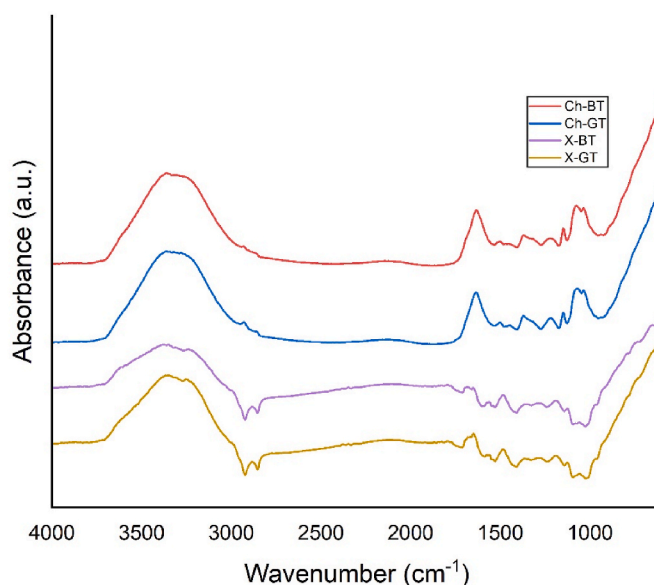


Fig. 1. FTIR spectroscopy of aerogels.

3.6. FTIR spectroscopy of aerogels

Fourier Transform Infrared (FTIR) spectroscopy was employed to investigate the interactions between tea components and polysaccharide-based aerogels. The spectra in Fig. 1 show distinct differences among the pure aerogels and the aerogels recovered after treatment with black and green tea infusions. These changes reflect molecular interactions, particularly with proteins, polyphenols (such as catechins), and other soluble compounds present in the teas.

In both chitosan and xanthan gum aerogels, a broad absorption band was observed around 3300-3400 cm^{-1} , which is typically attributed to the stretching vibrations of hydroxyl (O-H) groups. This band primarily reflects the presence of physically adsorbed water and the intrinsic hydrophilic character of the polysaccharide matrix. After treatment with tea infusions, a slight increase in band intensity and minor shifts were detected, especially in chitosan aerogels. These spectral changes may indicate an alteration in hydrogen bonding patterns due to interactions with tea components. However, the influence of hydration changes must also be taken into account when interpreting this region. In chitosan samples treated with black tea and green tea, a clear increase in the amide I region was noted (1650 cm^{-1}), suggesting adsorption of tea proteins, which are known contributors to turbidity and tea cream formation (Wu & Bird, 2010). Also, several bands between 1650 and 900 cm^{-1} shifted, most likely because of catechin adsorption onto chitosan aerogels (Chanphai & Tajmir-Riahi, 2018).

In the region around 1020-1080 cm^{-1} , typically attributed to C-O-C stretching vibrations of polysaccharides and polyphenol-related structures, minimal absorbance changes were observed following tea treatment (Ozesme Taylan et al., 2025). In chitosan aerogels, a slight decrease in this region was noted, particularly after green tea exposure. This reduction may be attributed to interactions between soluble tea components, such as phenolics, carbohydrates, or organic acids, and the hydroxyl groups on the chitosan backbone (Giorgini et al., 2023). Such interactions, likely involving hydrogen bonding, can restrict the vibrational freedom or alter the dipole behavior of C-O bonds, resulting in diminished signal intensity. However, the slight shifting suggests that the interactions were either limited to surface adsorption or did not induce substantial structural rearrangement within the aerogel matrix.

Additionally, another distinctive change was observed between 1200 and 1400 cm^{-1} , where bands related to C-N stretching and O-H bending became more pronounced after tea treatment, especially in chitosan-based aerogels. This enhancement may reflect the adsorption of nitrogen-containing compounds or polyphenol-protein complexes from tea, contributing to the increased band intensity. These spectral results suggest that tea components interacted mainly on the surface of the aerogels without significantly changing their overall structure, supporting the idea of selective adsorption in reducing turbidity.

Overall, FTIR results confirm that polysaccharide-based aerogels can interact with tea components primarily through hydrogen bonding and other non-covalent interactions. However, the spectral changes were relatively limited, indicating that these interactions occurred mainly at the surface and did not lead to major alterations in the polysaccharide structure. Among the samples, chitosan aerogels showed slightly more pronounced spectral changes, particularly after black tea treatment, suggesting moderate adsorption of tea-derived compounds such as proteins or polyphenol-protein complexes. These findings support the proposed clarification mechanism based on selective surface adsorption and are consistent with the turbidity and color retention results, particularly the efficient reduction of tea cream and color preservation in chitosan aerogel treated tea samples.

3.7. TD-NMR relaxometry and diffusion coefficient analysis of aerogels

NMR relaxometry and diffusometry results provided insight into the hydration and molecular mobility characteristics of chitosan and xanthan gum aerogels following tea treatments. The T_2 relaxation time

Table 7

T_2 relaxation times and diffusion coefficients of aerogels.

	T_{21}	T_{22}	Diffusion Coefficient (m^2/s)* 10^9
Ch-Water	14.5 \pm 0.0 ^b	647.4 \pm 31.4 ^a	2.23 \pm 0.02 ^b
Ch-BT	12.9 \pm 0.4 ^b	315.9 \pm 12.4 ^c	2.31 \pm 0.00 ^a
Ch-GT	23.1 \pm 1.3 ^a	430.0 \pm 17.5 ^b	2.30 \pm 0.01 ^a
X-Water	143.2 \pm 0.0 ^A	465.1 \pm 31.4 ^A	2.27 \pm 0.01 ^B
X-BT	49.1 \pm 1.3 ^B	62.4 \pm 3.5 ^B	2.34 \pm 0.02 ^A
X-GT	43.0 \pm 1.6 ^B	74.8 \pm 2.6 ^B	2.38 \pm 0.03 ^A

Lower case and upper case letters indicate significant differences among chitosan aerogel and xanthan gum aerogel, respectively, for each parameter ($p < 0.05$).

shows how easily protons can move in the water trapped inside the polymer (Ates et al., 2021). T_2 relaxation times (T_{21} and T_{22}) and self-diffusion coefficients significantly changed depending on the sample type. For chitosan aerogels, Ch-Water sample showed a longer T_{22} time (647.4 ms), suggesting higher mobility of loosely bound water (Table 7). Upon black tea treatment (Ch-BT), T_{22} decreased markedly to 315.9 ms, indicating reduced water mobility, likely due to the adsorption of tea compounds leading to partial pore blockage or altered surface hydration. Interestingly, green tea-treated chitosan (Ch-GT) showed an intermediate T_{22} (430.0 ms) and a notably longer T_{21} (23.1 ms), which may reflect a more hydrated or less rigid bound water population in these samples.

Xanthan gum aerogels exhibited a different trend. While X-Water sample had long T_{21} (143.2 ms) and T_{22} (465.1 ms) times, both decreased substantially after treatment with black or green tea, with T_{22} dropping to around 60-75 ms and T_{21} to around 43-49 ms. These reductions suggest a stronger restriction of water and decreased mobility after tea adsorption, likely caused by tighter polymer-tea interactions or blockage of hydration pathways.

T_2 relaxation times provide general information on the water content in hydrogels, but the signal originates from protons not only in water but also in the surrounding macromolecular environment, which can mask the specific mobility of water molecules. In contrast, the self-diffusion coefficient directly reflects the translational mobility of water within the matrix. Diffusion coefficient values are highly sensitive to polymer-water interactions; as these interactions increase, water diffusion slows down, leading to reduced self-diffusion coefficient values. The values also mirrored T_2 relaxation time trends. Both tea-treated chitosan and xanthan aerogels exhibited significantly higher diffusion coefficients compared to their water controls, indicating that despite reduced relaxation times, molecular motion within the pores remained active, possibly due to the presence of smaller mobile tea solutes contributing to the average diffusion signal. These findings suggest that tea treatment leads to measurable changes in the hydration state and mobility within aerogels, with distinct patterns for black and green tea and between different polysaccharide types.

4. Conclusions

Polysaccharide-based aerogels, particularly chitosan and xanthan gum, effectively clarified RTD tea infusions by reducing turbidity and tea cream formation without major losses in catechins or changes in viscosity. Chitosan aerogels performed best in black tea, showing minimal color change and efficient removal of haze-forming compounds. Although green tea showed slightly greater color shifts, key antioxidant compounds were largely preserved. Compared to traditional powder adsorbents, aerogels offer additional benefits such as easier removal and the potential for selective adsorption. These findings support the use of food-grade aerogels as a promising and sustainable alternative for tea clarification. However, despite these promising results, there are still some limitations to this study. The clarification performance of the

aerogels was tested in laboratory scale using a single adsorbent concentration and a limited storage time. The adsorption behavior may change under industrial processing conditions, with different tea formulations and extended storage times. Also, the sensorial properties of the tea infusions after clarification have not been tested yet. Future studies should focus on adsorption selectivity for a different kinds of tea formulations and for consumer related sensory properties to validate the practical applicability of aerogel-based adsorption systems for clarification processes.

CRedit authorship contribution statement

Serap Namli: Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **Özge Güven:** Methodology, Investigation. **Emre Taskin:** Methodology, Conceptualization. **Mecit Halil Öztop:** Writing – review & editing, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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