

STUDIES ON THE THERMAL PROPERTIES, LYCOPENE AND BETA-CAROTENE CONTENTS OF PUMPKIN FRUIT FLOUR

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Abstract

The pumpkin plant (*Cucurbita pepo*) is a vital crop that has potential to significantly contribute to food sustainability, particularly in Nigeria and other tropical countries. Its rich carotenoids and essential nutrients often decrease during processing, underscoring the need for further exploration in this field. This study then examined the effects of pretreatment techniques (blanching and brining), and drying methods (oven and sun-drying), on the preservation of lycopene and beta carotene in pumpkin fruit flour, and to determine its thermal properties that would enhance its usage during food formulation. All investigations were carried out using standard procedures. The results showed that the blanched samples exhibited a lower phase transition temperature range (370-375°C) than the control (unblanched) samples (330-395°C). Brined samples had the highest phase transition temperature ranges (395-410°C). For sun-drying (and oven drying), the densities, thermal conductivities, heat transfer coefficients, specific heat capacities and thermal diffusivities of the control (unblanched), blanched and brined samples are 1.0186 (1.10143)g/ml, 0.5905(0.5918)g/ml, 0.4764 (0.70646)g/ml; 0.0461(0.0527)W/mK, 0.0307(0.0307)W/mK, 0.3687(0.1844)W/mK; 3.8409(4.3896)W/m².K, 2.5606(2.5606)W/m². K, 30.7272 (15.3636)W/m². K; 0.0061(0.0061)J/kg.°C, 0.0054(0.0067)J/kg.°C, 0.0739(0.0232)J/kg.°C; and 7.3745 (8.5389)m²/s, 9.7033(7.7626)m²/s, 10.4796(11.2558)m²/s respectively. The lycopene and beta-carotenoids content after sun-drying (and oven drying) for control (unblanched), blanched and brined samples are: 10.32(9.72)mg/100g, 9.73(9.95)mg/100g, 10.64(10.18)mg/100g and 34.13(41.68)mg/100g, 37.25(37.47)mg/100g, 32.78(39.58)mg/100g respectively. Controlled oven drying retained more lycopene and beta carotene than sun drying, and blanching had no significant effect on beta carotene levels.

Keywords: DTA/TGA; Thermal Properties; Pumpkin Fruit; Phytochemicals; Food Sustainability

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Introduction

Pumpkins, the vividly colored yellow to orange fruits belonging to the *Cucurbita* genus within the *Cucurbitaceae* family, exhibit a diverse array of shapes, ranging from round to oblong, and are covered with a smooth yet resilient rind. Their complex composition significantly affects their responses to processing conditions, particularly temperature variations (Jamali *et al.*, 2018). A comprehensive understanding of the thermal properties of these materials is essential for optimizing their application in diverse contexts, including food processing.

Food processing is inherently comprised of various unit operations, many of which are dependent on temperature. This dependence results in unavoidable alterations to the composition of food materials due to heat treatment. Such compositional changes can significantly impact the final product, highlighting the critical importance of mastering a range of heat treatment methods to mitigate undesired effects. Techniques such as blanching, steaming, heating, cooling, pasteurization, sterilization, and drying vary from mild to intense, each exerting distinct influences on the properties of food (Oriola *et al.*, 2022). A comprehensive understanding and application of these methods will enable professionals to enhance food quality and achieve optimal processing results.

To thoroughly investigate and evaluate heat treatments, it is imperative to employ two primary approaches: absolute and relative thermal evaluation methods. Absolute methods, such as differential thermal analysis (DTA) and thermogravimetric analysis (TGA), are conducted in isolation, providing essential insights into material behavior devoid of external influences. Conversely, relative thermal evaluation methods incorporate various parameters, including density, specific heat, thermal conductivity, and thermal diffusivity (Oriola *et al.*, 2022). A comprehensive understanding of these methodologies is essential for professionals in the field of food processing, as it enables the optimization of processes and the enhancement of product quality.

Differential thermal analysis (DTA) serves as a powerful thermodynamic tool for assessing the thermal behavior of materials during both heating and cooling cycles. By comparing a sample to a reference under precisely controlled temperature and time parameters, DTA enables the identification of physical transformations and the assessment of chemical changes within materials (Devi *et al.*, 2021). In a similar vein, thermogravimetric analysis (TGA) measures

weight changes associated with the heating, holding, or cooling of a sample, offering deeper insights into material properties (Srinivas *et al.*, 2018). Elkhalfa *et al.* (2020) further explain that TGA can elucidate a biomaterial's response to various temperature-dependent processes, thereby enhancing the understanding of material stability and integrity. The integration of simultaneous thermogravimetric and differential thermal analysis (TG/DTA) is particularly beneficial for characterizing biomaterials, as it provides essential information concerning thermal stability, phase transition temperatures, drying loss, and the amounts of bound and free water present within the material (Mekasha, 2020).

This research aims to enhance the industrial applications of pumpkin fruits as nano and functional materials within the rapidly advancing field of technological processing. Specific objectives include assessing the effects of pretreatment on the thermal properties of this biomaterial and evaluating its bioactive components, including lycopene and carotene.

Materials and Methods

Matured pumpkin fruits used for this work were purchased in-situ from a farm in Osogbo, Osun State. The fruits were processed following the flowchart presented in Figure 1.

Thermo-Gravimetric Analysis (TGA) and Differential Thermal Analysis (DTA)

DTA-TGA thermal Analyzer was employed, the aluminum pan was filled with approximately 10–15 mg of the prepared sample and the sample was heated from the ambient temperature up to 600°C with a heating rate of 10°C/min. The analysis was done under a nitrogen atmosphere with a flow rate of (50 cm³/min), and, it continuously measured the sample's mass as the temperature changes with time.

Thermal properties

An Armfield equipment model HT₁₂ with heat transfer service unit (Armfield equipment model HT_{10XC}) consisting of single heater wire and thermocouple was used to measure the thermal conductivity and the specific heat capacity. The Thermal Conductivity (TC) values of the samples were high when the temperature rises with the subsequent time line increase, the TC calculations are given below;

$$\text{For TC, } \lambda = \frac{Q \cdot X}{A (T_2 - T_1)} \quad (1)$$

Where; λ – Thermal conductivity of sample (W/mK)

Q - Generated heat per unit length of sample/time (W/m²)

X - Thickness of sample (m)

M - Mass (kg)

ρ - Density (kg/m³)

A - Area (m²)

T₀ and T₅ (K) - Initial temperature at time, t₀ and Final temperature at time, t₅ (s)

Given the sample's diameter and length are 0.0018 m and 0.0043 m respectively.

The heat transfer coefficient, specific heat capacity and diffusivity were calculated using the equations below;

$$\text{Heat transfer coefficient, } u = Q \cdot \frac{x}{[A \cdot \Delta T]} \quad (2)$$

$$\text{Specific heat capacity, } c_p = \frac{Q}{(m \cdot \Delta T)} \quad (3)$$

$$\text{Diffusivity, } \alpha = \frac{u}{(\rho \cdot c_p)} \quad (4)$$

Determination of beta (B) carotene and lycopene contents

The method described by Soroka *et al.* (2012) was followed. 500 mg of each prepared sample (from Figure 1) was measured into a vial, mixed with 100 ml of pure hexane in forming a suspension and then corked. The mixture was thoroughly shaken for 30 min for extraction of the colored substances, after which, filter paper (Whatman Grade 41) was used to filter the supernatant collected in the corked container. The residue was re-extracted with 50 ml of hexane and the re-extraction continued till the sample was discolored. The extract was allowed to settle in the dark for 30 min, then the optical density of the clear hexane extract was measured at a maximum absorption wavelength of 453 nm and 417 nm for beta carotene and lycopene respectively. The beta carotene and lycopene concentrations were calculated using equations 5 and 6 respectively;

$$\text{Concentration of lycopene } \left(\frac{\text{mg}}{100 \text{ g}} \right) = \frac{D_{470} \cdot V}{m \cdot l \cdot E^{\wedge}} \quad (5)$$

Britton *et al.* (2012)

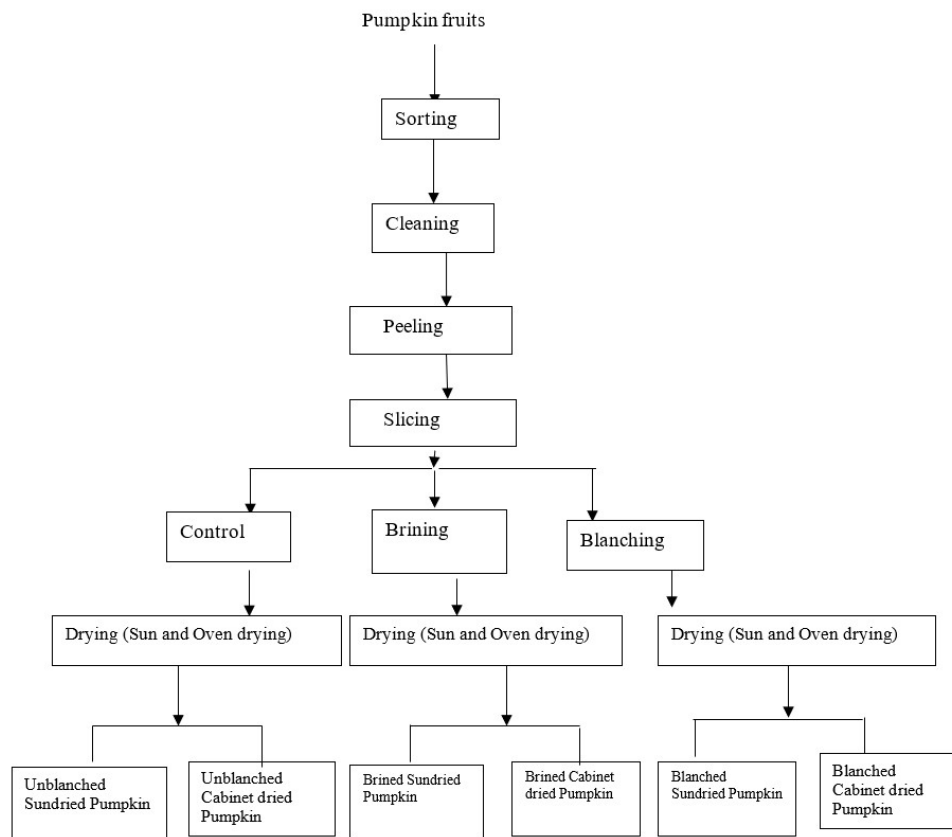


Figure 1. Flowchart for Pumpkin fruit flour processing

Where;

D470 is the optical density of the investigated solution at the wavelength of 470 nm.

V is the hexane volume used for the extraction in ml

m is the sample's weight in g.

l is the thickness of the cuvette using for the optical density measurements in cm.

E[^] is the extinction coefficient of the 1% solution of pure lycopene, which is 3450.

$$\text{Concentration of beta carotene } \left(\frac{\text{mg}}{100 \text{ g}} \right) = \frac{D450 * V}{m * l * E^{\wedge}} \quad (6)$$

Britton *et al.* (2012).

Where;

D450 is the optical density of the investigated solution at the wavelength of 453 nm.

V is the hexane volume used for the extraction in ml.

m is the sample's weight in g.

l is the thickness of the cuvette using for the optical density measurements in cm.

E[^] is the extinction coefficient of the 1% solution of pure lycopene, which is 2592.

Results and Discussion

The thermal characteristics of pumpkin fruit pellets, measured using Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA), are presented in Figure 2 and summarized in

Table 1. All six samples exhibited different regions for exothermic and endothermic reactions, phase transitions, and water distribution, highlighting the uniqueness of each sample based on the treatment applied. The TGA and DTA curves identified three significant phases: dehydration (drying), decomposition, and carbonization. During the constant drying phase, free or unbound water is removed from food samples, while bound water is typically eliminated at elevated temperatures, often leading to the denaturation of the food materials, particularly in the decomposition phase.

In Sample A, free water loss was noted as the sample was heated up to 210°C, during which approximately 3% moisture by weight was lost. This reaction was endothermic, as the heat absorbed by the sample was used to evaporate the free water. The decomposition phase occurred between 230°C and 480°C, resulting in an 82% moisture loss by weight. Comparatively, Sample B also lost the same amount of moisture by weight during its drying phase (≤5%) at a heating temperature of about 200°C. This finding supports the conclusion that blanching increases the drying rate of fruits and vegetables (Oshima *et al.*, 2021), ultimately leading to a higher-quality product. However, Sample B exhibited a 140°C temperature range (from 260°C to 400°C) for its 10% to 50% moisture loss by weight, while Sample A displayed a 150°C temperature range for the same criteria (Figure 2). This indicates that blanching may enhance the preservation of bound water in the sample.

Table 1. Peak weight and Temperature Coordinates from TGA/DTA curves

Sample	Endothermic	Exothermic	Phase Transition (T _{max})
A	(150°C, 93%)	(230°C, 98%)	I: (330°C, 90%)
	(330°C, 4.8%)	(480°C, 93%)	II: (460°C, 13%)
	(520°C, 91%)		III: (605°C, 05%)
B	(130°C, 95%)	(215°C, 97%)	I: (250°C, 96%)
	(370°C, 24%)	(620°C, 98%)	II: (450°C, 26.5%)
	(695°C, 96%)	(750°C, 97%)	III: (585°C, 24.5%)
C	(120°C, 95%)	(200°C, 98%)	I: (290°C, 94.5%)
	(395°C, 17.5%)	(560°C, 98%)	II: (520°C, 22.5%)
	(121°C, 84.5%)	(705°C, 84.5%)	I: (220°C, 96%)
D	(375°C, 04%)	(570°C, 83%)	II: (560°C, 13.5%)
			III: (615°C, 10%)
	(100.1°C, 93.6%)	(260°C, 95%)	I: (165°C, 97%)
E	(395°C, 15%)	(845°C, 95.5%)	II: (465.5°C, 18%)
	(725°C, 93%)		III: (770°C, 15%)
	(140°C, 82%)	(245°C, 83.3%)	I: (275°C, 95.5%)
F			II: (385.5°C, 60%)
			III: (470°C, 14%)
	(410°C, 04%)		IV: (690°C, 06%)

Key:

A: Unblanched sun-dried pumpkin fruit flour

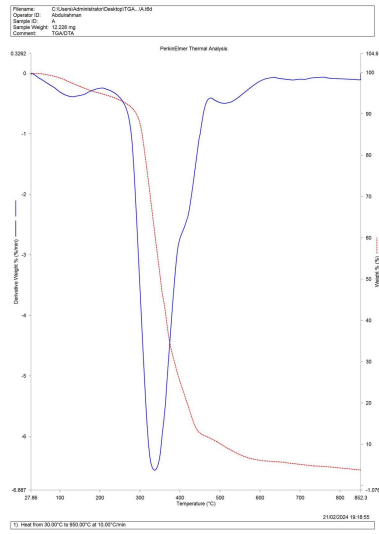
B: Blanched sun-dried pumpkin fruit flour

C: Unblanched oven-dried pumpkin fruit flour

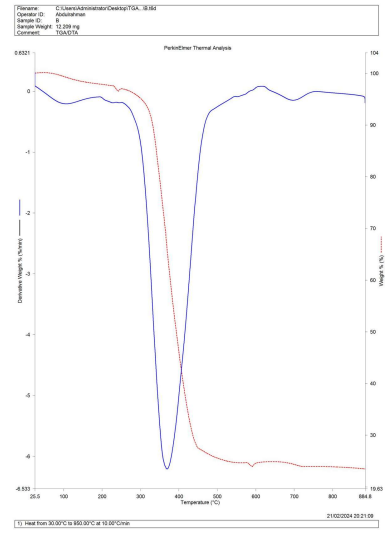
D: Blanched oven-dried pumpkin fruit flour

E: Brined sun-dried pumpkin fruit flour

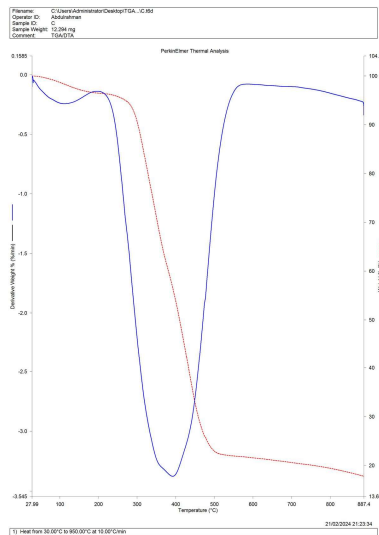
F: Brined oven-dried pumpkin fruit flour



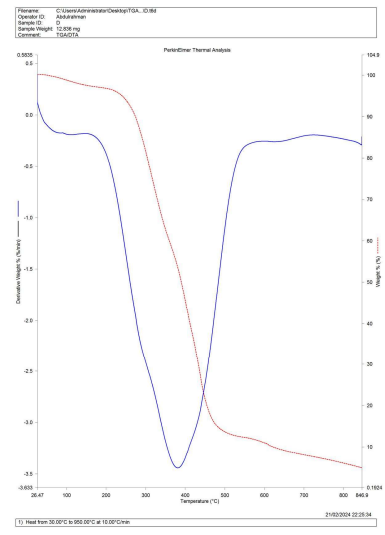
(a)



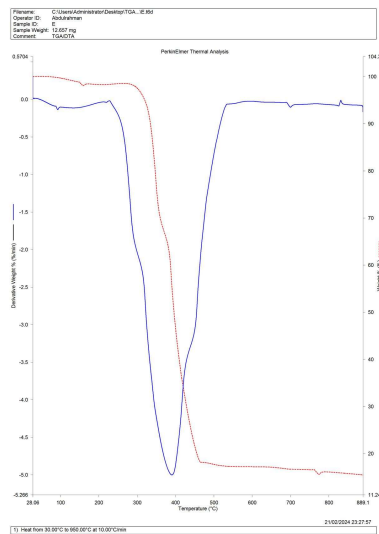
(b)



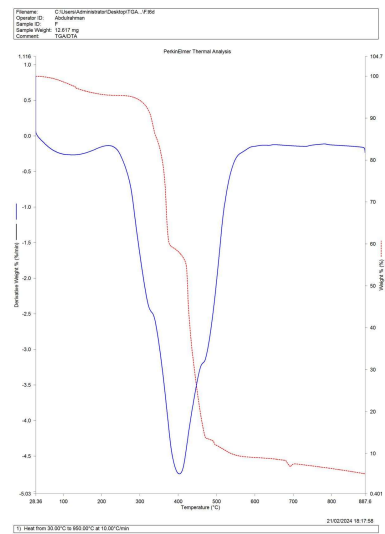
(c)



(d)



(e)



(f)

Figure 2. TGA and DTA graphs of Pumpkin Samples A-F (From L-R)

Similarly, Sample D had a lower maximum drying temperature (170°C) in its dehydration phase compared to Sample C (200°C), reinforcing the positive impact of blanching on food drying. Although Sample D demonstrated a lower decomposition temperature range (180°C to 570°C) than Sample C (205°C to 590°C), its temperature range for the 10% to 50% moisture loss by weight was significantly higher (120°C and 100°C, respectively). This reinforces the advantage of blanching in preserving food samples at higher temperatures and suggests that oven-drying may yield more heat-stable food products than sun-drying.

The maximum temperatures for Samples E and F in the drying phase (approximately 230°C for both samples) were notably higher than those of the previous samples. This could be attributed to the influence of impurities (such as salt) on the boiling point of free water. However, the osmotic treatment applied to Samples E and F resulted in the highest derivative weight loss at 100°C and 130°C, respectively, during their dehydration phase. This indicates that osmotic treatment significantly enhances the drying rate of fruit samples compared to blanching (Özkan-Karabacak *et al.*, 2022). Furthermore, Sample F exhibited a larger temperature range (100°C) for its 10% to 50% moisture loss by weight in comparison to Sample E (50°C), indicating greater heat stability.

In terms of thermal stability at 90% moisture retention (Figure 2), Sample E had the highest peak drying temperature (350°C), while Sample D had the lowest (280°C). Although both Samples E and F demonstrated higher weight retention temperatures (350°C and 330°C, respectively), the addition of brine may significantly affect other physical and engineering properties of these samples. Notably, the meander curve of the brined samples (E and F) at the end of decomposition suggests the presence of impurities from the sodium chloride used in the brining process (Rickard *et al.* 2010).

Moreover, consistent moisture loss occurred at lower temperatures in the oven-dried samples C, D, and F compared to their corresponding sun-dried samples (A, B, and E, respectively), thanks to the ability of an oven dryer to maintain a constant drying temperature, which is not achievable with sun-drying.

Thermal Properties of pumpkin flour. The proximate properties of the pumpkin flour and the thermal properties of the differently processed pumpkin fruit slices from which they were derived, are illustrated in Table 2 and Figure 3 respectively. In Figure 3a, the density properties of all processed samples are presented. Both blanching and brining treatments resulted in a decrease in the density values of all pumpkin slices when compared with the control (unblanched) samples, which aligns with Mythili *et al.* (2021). The drying method, whether sun-drying or oven-drying, did not significantly affect the densities of the unblanched and blanched samples; however, it did impact the brined samples. Specifically, the density values of brined sun-dried samples were lower than those of the brined oven-dried samples (Table 2). This difference may be attributed to the lower moisture content and protein in brined, sundried flour (Hasnadi, 2021). The low density of the brined sun-dried samples indicates the extent of shrinkage the material underwent during drying.

Brining was performed using sodium chloride and water. Sodium chloride is known to regulate extracellular fluids and facilitate the transfer of molecules across cell membranes (Alenyorege *et al.*, 2015). Density is a crucial physical property of food materials as it directly influences the unit price of the product, plays a significant role in handling, determines packaging requirements, and affects transportation. Moisture content also impacts the density of food materials; lower moisture content typically results in lower density, which is advantageous for handling, packaging, and transportation.

Table 2. Proximate properties of the pumpkin fruit flour

Samples	MC	Fat	Fibre	Protein	Ash	CHO	Energy (Kcal/g)
A	7.323 ± 0.11 ^{ab}	1.050 ± 0.02 ^b	1.763 ± 0.03 ^c	4.513 ± 0.04 ^c	6.263 ± 0.05 ^c	79.087 ± 0.10 ^b	343.85
B	7.153 ± 0.02 ^{cd}	0.934 ± 0.03 ^c	1.723 ± 0.02 ^c	4.133 ± 0.03 ^d	7.220 ± 0.04 ^c	78.833 ± 0.02 ^c	340.27
C	6.110 ± 0.08 ^e	1.143 ± 0.03 ^a	2.150 ± 0.03 ^a	4.160 ± 0.04 ^d	6.413 ± 0.03 ^d	80.023 ± 0.10 ^a	347.02
D	7.093 ± 0.04 ^d	0.830 ± 0.02 ^d	1.777 ± 0.04 ^c	5.370 ± 0.05 ^b	7.263 ± 0.02 ^c	77.667 ± 0.11 ^d	339.62
E	7.232 ± 0.05 ^{bc}	0.943 ± 0.04 ^c	1.653 ± 0.03 ^d	5.367 ± 0.05 ^b	8.370 ± 0.04 ^b	76.433 ± 0.08 ^e	335.69
F	7.357 ± 0.06 ^a	0.853 ± 0.04 ^d	1.847 ± 0.04 ^b	5.573 ± 0.12 ^a	8.703 ± 0.06 ^a	75.667 ± 0.14 ^f	332.64

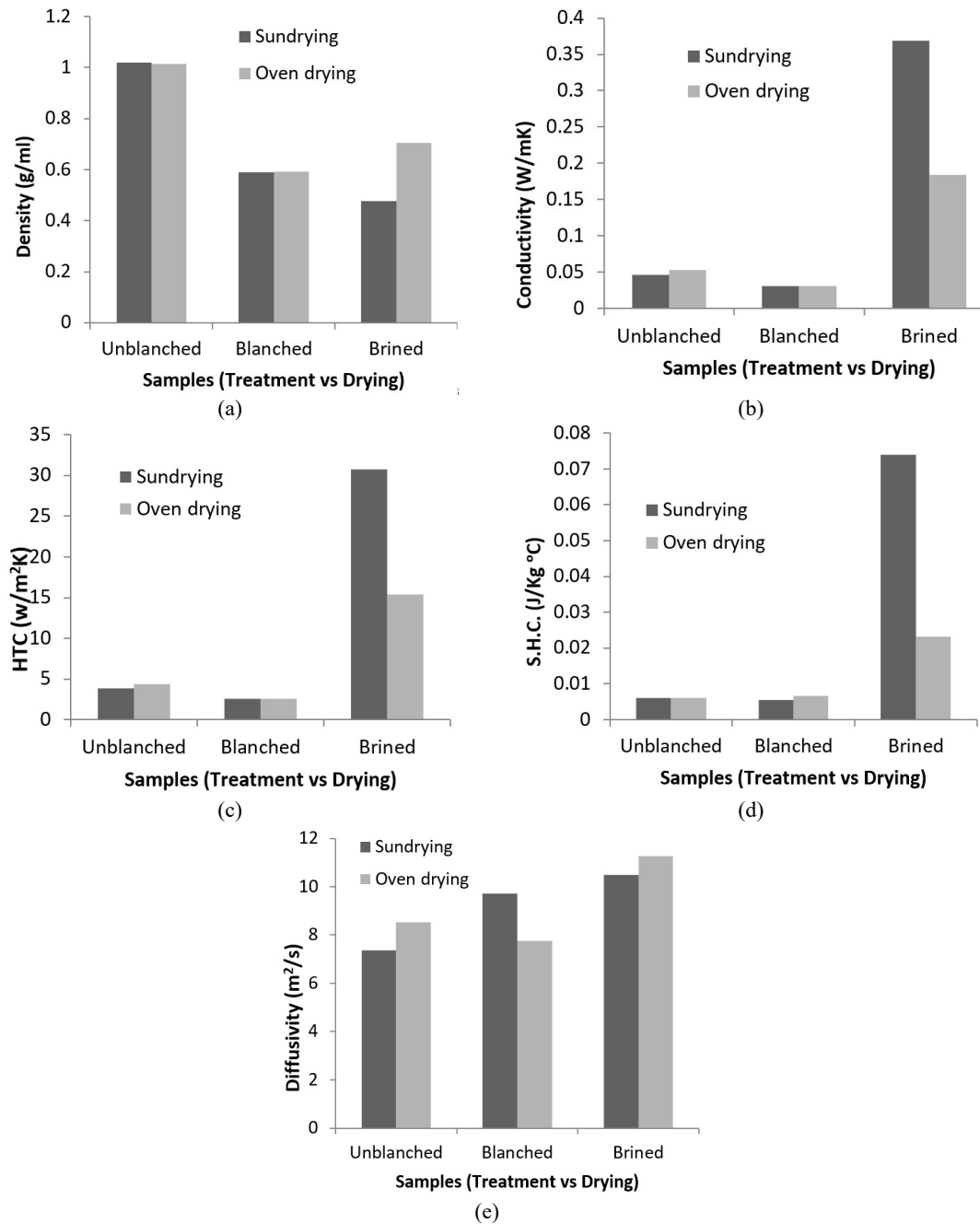


Figure 3. Thermal properties of the pumpkin samples

According to Carson (2017), thermal conductivity is a key physical property for modeling thermal processes such as chilling, freezing, and thawing. Predicting cooling or heating times relies on accurate calculations of thermal conductivity. The thermal conductivity values for the pumpkin samples ranged from 0.0307 W/mK to 0.3687 W/mK, which are higher than 0.026 W/mK,

the thermal conductivity value of air, which is the least conductive component in food materials (Graczykowski *et al.*, 2017). A high thermal conductivity value is desirable during processing, as it indicates lower energy consumption resulting from a reduced rate of heat transfer through the material (Mukama *et al.*, 2020).

The thermal conductivity (TC) values obtained for all pumpkin samples were below 0.5 W/mK, classifying them as poor thermal conductors. While blanching was found to lower the conductivity values, brining significantly increased them. According to Carson *et al.*, (2016), the reduction in thermal conductivity due to blanching is attributed to thermal degradation of plant tissues and removal of air within the plant cells. In contrast, brining reduces moisture content but allows ionic materials to migrate into the product, thereby increasing its conductivity (Wiroonsri and Wattanachant, 2025). This is obvious in the increased ash content of brined samples (E and F) in Table 2.

A similar trend was observed for Heat Transfer Coefficient (HTC). The method of drying did not significantly affect the HTC of the samples, as almost equal values were recorded for the control and blanched samples. However, there is significant increase in the values of HTC of the brined samples with the highest values obtained for brined, sun-dried samples. Unblanched oven-dried samples exhibited higher TC and HTC values than unblanched sun-dried samples, whereas brined sun-dried samples demonstrated higher TC and HTC values compared to brined oven-dried samples.

HTC relates to the basic thermal conductivity of food materials and primarily depends on various material characteristics (such as solid shape, dimensions, surface temperature, and roughness, as well as fluid velocity and turbulence intensity), the heat transfer equipment used, and the thermophysical properties of the material system (including density, specific heat capacity, and thermal conductivity) (Michailidis *et al.*, 2009). HTC is defined as the heat transfer per unit area per Kelvin and is crucial for regulating the temperature of materials during handling. It is an important property for designing food processing equipment, and knowledge of HTC is essential for the effective packaging and storage of biomaterials (Krokida *et al.*, 2002).

Specific heat capacity (SHC) is another important thermophysical property that measures a material's energy storage ability; it indicates how well a material absorbs and eventually releases heat energy. Pumpkin fruits with an SHC higher than that of water may be recommended for patients experiencing internal heat challenges (Atat *et al.*, 2024). Additionally, these fruits could be utilized in food processing where equipment friction generates heat to maintain product temperature.

Figure 3d shows that blanching has no significant effect on SHC of the samples when compared with that of brining. The SHC of the unblanched samples remained consistent despite the

different drying media. Blanching was found to slightly increase the SHC of the oven-dried samples but decreased that of the sun-dried samples. In general, brining resulted in an increased SHC for the samples, likely due to the infusion of sodium chloride into the material, which heightened the ionic concentration of the pumpkin. Low SHC is necessary in food processing for optimal performance. Low specific heat capacity (SHC) is crucial in food processing to reduce energy consumption, while high SHC increases production costs and the unit price of food materials.

The Thermal Diffusivity (TD) of the samples indicated that the different pretreatments increased the materials' TD. In Figure 3e, unblanched samples exhibited a lower TD compared to the brined and blanched samples, likely due to cellular changes that occurred during these pretreatments. Additionally, the drying method used significantly influenced the TD property of the pumpkin. Higher TD values were recorded for the unblanched and brined oven-dried samples, while the blanched oven-dried sample showed lower TD. TD measures the rate at which heat is conducted and transmitted through a material, reflecting the temperature change during thermal processes (Mukama *et al.*, 2020).

Lycopene and Beta-Carotene Contents. Figure 4 presents the effects of various pretreatments (blanching and brining) combined with different drying methods, specifically sun drying and oven drying, on the lycopene content of pumpkin fruit slices. The analysis reveals blanching has a negative effect on lycopene retention of the samples when compared with control (unblanched) samples during sun-drying but a positive effect during oven-drying. Also, there is a consistent higher retention of lycopene in brined samples. These findings imply that brining combined with sun drying results in better retention of lycopene content, as does blanching paired with oven drying. Generally, brining retains more lycopene than blanching, likely due to the equilibrium achieved during brining or the higher SHC of brined samples, which contributes to the lower loss of lycopene. In contrast, the unrestricted blanching treatment leads to an uncontrollable loss of lycopene (TÜRÜT and ARKUN, 2017). Therefore, for optimal lycopene retention after processing, the brining treatment, alongside the sun drying method, is the preferable approach.

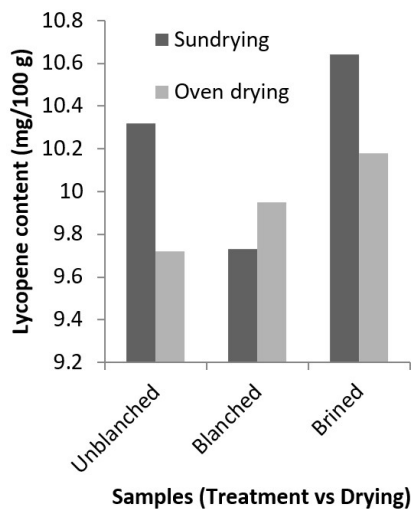


Figure 4. The lycopene content of processes pumpkin fruit

Lycopene is a heat-sensitive natural phytochemical compound known as a potential alternative to synthetic antioxidants and a natural preservative in food processing. According to Li and Yu (2023), lycopene can be modified into various forms, such as emulsions, microcapsules, composite coatings, and active film packaging, for use in food applications. The health benefits of lycopene have also been documented; Leh and Lee (2022) reported that diets rich in lycopene are crucial for chronic disease management. Similarly, Varela *et al.* (2022) highlighted the effects of lycopene on several diseases associated with oxidative stress.

Figure 5 illustrates the beta carotenoid content of pumpkin fruits subjected to various processing methods. It shows that neither blanching nor brining has a significant effect on the retention of beta-carotenoids in the samples. However, samples that were dried in the oven exhibited a higher retention of beta carotene compared to those exposed to sunlight; this is in agreement with Addis *et al.*, 2009. This decline can likely be attributed to the harsh and fluctuating nature of sunlight during the drying process, while oven drying offers a more controlled environment. According to Suryana *et al.* (2023), beta carotene-containing fruits can serve as functional materials to enhance the color of food products during processing, or the beta carotene can be extracted from the fruit for use as a color improver.

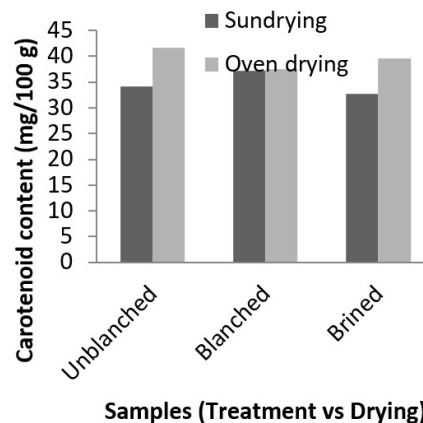


Figure 5. The carotenoid content of processed pumpkin fruit

Conclusion

Blanched pumpkin fruits exhibited a higher drying rate compared to untreated samples. However, this process necessitates greater energy consumption to achieve effective drying, resulting in a significant loss of lycopene. In contrast, brined samples proved to be the most advantageous, requiring less energy, demonstrating better heat stability, and retaining more nutrients than the other samples. It is important to note that brine treatment may substantially affect the sensory characteristics of the final product. Overall, oven-drying method produced samples with greater thermal stability than sun-drying method, while the latter method tended to yield products with enhanced thermal properties and improved nutrient retention.

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