University of Thi-Qar Journal of agricultural research

Thi-Qar Journal of agricultural research

ISSN Onlin:2708-9347, ISSN Print: 2708-9339 Volume 14, Issue 2 (2025) PP 270-281

https://jam.utq.edu.iq/index.php/main https://doi.org/10.54174/utjagr.v13i1.323

Effect of Conventional Thermal Processing Duration on Hydroxymethyel furfural, Antioxidant Activity, and physicochemical Properties of Pomegranate Molasses

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Abstract

This study investigated the effect of traditional heating duration on the physicochemical characteristics, hydroxymethylfurfural (HMF) formation, antioxidant activity, and mass yield during the production of pomegranate molasses. Pomegranate juice was thermally concentrated under traditional conditions for up to 14 hours, with periodic sampling to monitor changes. Heating significantly affected all measured parameters ($p < 0.05$). HMF content showed a non-linear increase, with initial low values, a sharp rise between 1–3 hours, slight decline during intermediate heating, and a final increase at 12–14 hours. Importantly, HMF levels remained below the maximum allowable limit of 75 mg/kg, ensuring product safety. Polynomial regression confirmed a strong quadratic relationship between heating time and HMF concentration ($R^2 = 0.912$, $p < 0.001$). Antioxidant activity decreased gradually with heating, while total soluble solids and viscosity increased, and pH and moisture content decreased. These results emphasize the need to optimize traditional heating time to maximize yield and quality while maintaining HMF within safe limits.

Keywords: Pomegranate molasses- Hydroxymethyl furfural - Antioxidant activity – Traditional methods.

I. Introduction

Pomegranate (Punica granatum L.) is widely acknowledged for its broad spectrum of bioactive properties, including antioxidant, anti-inflammatory, antiviral, antibacterial, and antifungal effects. Its juice is particularly rich in antioxidants, with concentrations often exceeding those found in other commonly consumed fruit juices and beverages. The primary phytochemical constituents of pomegranate include polyphenols such as flavonoids, hydrolysable and condensed tannins, as well as various organic and phenolic acids. These compounds are largely responsible for its health-promoting potential (Legua et al., 2016). Pomegranate molasses is a concentrated syrup traditionally produced by the thermal evaporation of pomegranate juice. This process, which involves prolonged boiling, results in a viscous product with a dark color and a distinctive sweet-sour, mildly astringent flavor. It is widely utilized in Middle Eastern and Mediterranean gastronomy, where it serves as a flavoring agent in a variety of dishes, including salads, raw meat preparations, dolma, chutneys, curries, and as a natural meat tenderizer. Beyond its culinary applications, pomegranate molasses is valued for its high nutritional content and notable bioactive properties. It exhibits potent antioxidant, antitumoral, and antimicrobial activities, which have been associated with the prevention of several non-communicable diseases, including diabetes mellitus, cardiovascular disorders, and certain types of cancer. The traditional preparation method enhances both the organoleptic characteristics and the concentration of health-promoting phytochemicals through the gradual reduction of juice volume under heat. (El Darra et al., 2017 ; Özmert Ergin, 2020) Pomegranate molasses can be produced through either traditional or commercial methods. In traditional practices, the process involves washing, crushing, granulating, pressing the pomegranate fruits, followed by the evaporation of the juice to achieve the desired consistency. This method typically excludes the use of added sugars or artificial additives. In contrast, commercial production incorporates more advanced processing steps, such as juice pasteurization, enzymatic treatment, clarification, filtration, and controlled evaporation. During these stages, various additives—including glucose or fructose

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syrup, citric acid, antioxidants, colorants, and preservatives—may be introduced to enhance product stability, appearance, and shelf life (Bige et al., 2017;Karabiyikli & Kisla, 2012) additionally, Pomegranate molasses is considered a nutritionally dense product with notable bio functional attributes, particularly its strong antioxidant activity. These characteristics contribute to its potential role in mitigating the risk of various non-communicable diseases, including diabetes mellitus, cardiovascular disorders, and certain forms of cancer. In addition to its antioxidant potential, pomegranate molasses is recognized for its richness in phenolic compounds and essential minerals such as potassium, magnesium, calcium, and zinc, which collectively support its health-promoting and therapeutic potential. (Özmert Ergin, 2020a) Maillard reactions are commonly observed during the thermal processing and storage of food products, resulting in non-enzymatic browning and the formation of compounds such as hydroxymethyl furfural (HMF). In fruit molasses concentrated syrups produced from various fruit juices—these reactions are particularly pronounced due to the high content of reducing sugars and amino compounds. Browning becomes more evident at elevated temperatures and during extended storage, negatively affecting the sensory attributes and nutritional quality of the product. HMF, typically absent in fresh foods, forms as a result of thermal exposure and is widely used as a marker for heat treatment and product quality. Its accumulation is influenced by factors such as pH, acidity, mineral composition, packaging materials, and exposure to thermal or light-induced stress. Although the toxicological profile of HMF is still under investigation, elevated levels have demonstrated cytotoxic effects in animal studies. As a result, various food safety authorities have set maximum allowable limits for HMF in food products: for example, the European Union permits a maximum of 20 mg/kg in fruit juices and concentrates, while in Turkey, the limit for fruit molasses is set at 75 mg/kg. (Kus et al., 2005; R. Oral et al., 2012). Pomegranate is a valuable fruit in the Kurdistan Region of Iraq, particularly in Halabja city, annually, approximately 55,000 to 65,000 tonnes of production are obtained from an estimated 11,000 dunams of cultivated land (Al Dulaimi & Mohamed, 2022), where it is consumed fresh or traditionally processed into molasses during peak production periods. The traditional method involves extracting juice from washed, crushed pomegranates, which is then slowly concentrated by evaporation over a low flame or charcoal heat in open pans until reaching about 60 °Brix. This artisanal process, free from additives, produces a thick syrup widely used in local cuisine to enhance flavors, tenderize meats, and as a customary remedy for managing hypertension. The preservation of these traditional techniques underscores their cultural importance and the demand for naturally processed, nutrient-rich products. The objective of this study was to evaluate the effect of prolonged heating time on the antioxidant properties, bioactive compound content, and physicochemical characteristics of pomegranate molasses. Specifically, it aimed to determine the optimal heating duration that maximizes antioxidant activity and bioactive retention while minimizing nutrient degradation and undesirable browning.

II. Materials and Methods

Preparation of pomegranate molasses and sampling

Two different local producers of traditional pomegranate molasses were selected for the study in Halabja city. Pomegranate fruits were first washed, sorted, and cut to separate the arils, which were then subjected to juice extraction and filtration. The clear juice was concentrated using the traditional open pan heating method and the temperature range between 70-90 C° until it reached 70 °Brix. During the concentration process, samples were systematically collected at one-hour intervals to monitor changes in physicochemical and bioactive properties over time. At each interval, three replicate samples were obtained to ensure data reliability and statistical validity. All samples were immediately transferred to a cool box to prevent further thermal reactions and to preserve their integrity prior to laboratory analysis.

Physiochemical properties

The pH of the pomegranate molasses samples was measured using a pH meter (Eutech pc 700). Titratable acidity was determined by dissolving 5 g of sample in 100 mL of distilled water and titrating with 0.1 N sodium hydroxide to a pH endpoint of 8.3. followed by (KAMIŞ et al., 2022). Total soluble solids were assessed using a hand-held refractometer (ATC, China). Moisture content was determined by oven (Memmert, Germany) drying 5 g of the sample at 105°C until a constant weight was achieved according to (Kamiloglu & Capanoglu, 2014; Shakir & Rashid, 2019).

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Determination of hydroxymethyl furfural

Hydroxymethyl furfural was evaluated by spectrophotometric method. p-toluidine solution was prepared by dissolving 10 g p-toluidine in 50ml isopropanol followed by warming in a water bath, mixed with 10ml glacial acetic acid and stored in the dark for 24h before use. 2g of samples were diluted in 5ml water in 10ml volumetric flask to make a sample solution. 2ml of this solution and 5ml of p-toluidine were put into two different test tubes: in one test tube added 1ml barbituric acid 0.5% (Sample solution) and 1ml distilled water was added to other test tubes (Reference solution). Absorbance values were measured at wavelength 550 nm and the following formula was used for calculation: {Absorbance of Sample solution - Absorbance of Reference solution} ×192 where:192 is a factor for dilution and extinction coefficient.(Bige et al., 2017; Turkben et al., 2016).

Antioxidant Activity

DPPH Radical Scavenging Activity was determined to assess the free radical neutralizing capacity of the samples. DPPH (2,2-diphenyl-1-picrylhydrazyl) in methanol was mixed with the sample extract and incubated in the dark for 30 minutes. The decrease in absorbance was measured at 517 nm measured by (Thermo fisher scientific, USA) spectrophotometer. (Akpinar-Bayizit et al., 2016).

Browning Index

Browning Index (BI) was evaluated by measuring the absorbance at 420 nm, which corresponds to the formation of brown pigments due to thermal degradation and Maillard reactions. The browning index reflects the degree of pigment change during concentration or storage (Dorris et al., 2018)

Statistical analyses

Statistical analyses were conducted using SPSS version 25 (IBM, USA). Trends in HMF formation and antioxidant activity were visualized graphically, and simple linear regression and Least Significant Difference (LSD) test at $p < 0.05$ were performed used to assess the effect of heating time on quality properties and antioxidant activity.

III. Results and discussion

Mass distribution of pomegranate components and final molasses yield

In traditional pomegranate molasses production, especially among local producers, understanding the mass distribution of fruit components is essential for evaluating efficiency and reducing waste. Based on the processing of 150 kg of whole pomegranate fruits, the results showed in Table 1, that arils represented approximately 52% of the total mass, followed by peel (47.5%) and seeds (13.5%). Juice yield was calculated as 38%, and final molasses production accounted for only 8.44% of the total fruit weight. These values were determined through step-by-step weighing and measurement during each processing stage, providing quantitative insight into the transformation of raw fruit into concentrated molasses. Such information is critical for local producers to estimate expected yield, plan resource usage, and assess the economic return of molasses production. Additionally, by-products like peel and seeds, which collectively exceed 60% of the fruit mass, represent valuable sources of dietary fiber, phenolic compounds, and natural antioxidants that are often underutilized. Their potential applications in food, drugs, cosmetics, or animal feed industries may offer added value and reduce production waste (Al-Rawahi, 2014; Waly et al., 2012; Wang et al., 2011). The low yield of molasses (only 8.44%) reflects the high concentration involved in the traditional boiling process and highlights the importance of optimizing heating time, energy use, and juice recovery methods to improve both quality and sustainability (Helvacıoğlu et al., 2018a). Documenting these values also supports future comparisons between traditional and modern production techniques and contributes to the preservation of indigenous food processing knowledge.

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Table 1. Mass distribution of pomegranate components and final molasses yield

Component	Mass	Unit	% Total fruit mass and yield
Pomegranate	150.00	Kg	100.00%
Arils	78.00	Kg	52.00%
Peel	71.25	Kg	47.50%
Juice	57.00	L	38.00%
Seeds (after filtration)	20.25	Kg	13.50%
Molasses	12.67	Kg	8.44%

Effect of heating time on hydroxymethyl furfural formation

Figure 2 illustrates the changes in hydroxymethyl furfural (HMF) during thermal concentration of pomegranate juice into molasses during heating exhibited a non-linear trend, indicating that its formation is not strictly dependent on time alone. To evaluate the quantitative relationship between heating time and HMF concentration, the data were fitted using polynomial regression as the pattern showed a non-linear trend. At the beginning of the heating process (0 hours), the HMF content was relatively high in fresh juice (27.68 mg/kg), possibly due to pre-existing HMF formed during storage or initial concentration steps prior to the controlled heating period. Also, HMF can form even at low temperatures over time, especially if the juice is acidic and rich in sugars like pomegranate, apple, or grapes (Agcam, 2022; Kowalski et al., 2013) furthermore, in a previous study by (Özcan et al., 2015) on traditionally produced grape molasses, detectable levels of HMF ranging from 8 to 10 mg/kg were already present at the must stage, before the application of any heat treatment. This suggests that HMF can form even prior to thermal processing, possibly due to natural fermentation, storage conditions, or the intrinsic composition of the juice Interestingly, a sharp decrease was observed at 1 hour, where HMF dropped to 4.22 mg/kg. This may reflect early evaporation or dilution effects before thermal degradation reactions became dominant. Between 1 and 3 hours, HMF levels rose sharply, peaking at 38.78 mg/kg. This surge is likely associated with the onset of significant sugar degradation, especially the acid-catalyzed dehydration of fructose, which is known to occur under elevated temperatures and low pH conditions. Pomegranate juice naturally contains both hexose sugars and organic acids, making it highly prone to HMF formation during thermal processing. This behavior is consistent with literature showing rapid HMF accumulation in fruit-based systems when subjected to moderate-to-high heat (Agcam, 2022) Following this early peak, a noticeable decrease in HMF concentration was recorded from 4 to 7 hours, with values falling as low as 2.50 mg/kg. This decline may be attributed to two factors. First, HMF itself is thermally unstable and may degrade into secondary compounds such as levulinic acid and humic acid during prolonged heating (Ramli et al., 2024) or this reduction may be attributed to the degradation of HMF into other compounds, particularly melanoidins, which are known as the final products of non-enzymatic browning reactions (Aliyazicioglu et al., 2009). Second, agitation and temperature fluctuations commonly observed in traditional heating setups often fueled by charcoal or firewood could lead to intermittent cooling phases that limit further HMF generation. In the later stages of processing, particularly between 12 and 14 hours, HMF content began to rise again, reaching 36.86 mg/kg at 12 hours and 33.02 mg/kg at 14 hours. This second increase likely corresponds to intensified heat application near the end of the

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concentration process except that, metal ions and phenolic compounds are known to enhance the Maillard reaction. Additionally, HMF and other products formed during thermal processing can contribute to the acceleration of browning reactions during subsequent storage, particularly under elevated temperatures (CHEYNIER et al., 1988). Overall, the data illustrate that HMF formation in traditional pomegranate molasses production does not follow a simple linear relationship with heating time. Instead, it is shaped by a complex interplay of time, temperature, pH, sugar content, and heating variability. The observed fluctuations in HMF content suggest multiple phases: an initial loss or instability, a rapid formation phase, potential degradation, and a final accumulation. such patterns highlight the need for better control of heating intensity during traditional production to prevent excessive HMF levels, which are not only indicators of over-processing but may also pose potential health concerns. Regression analysis revealed a significant quadratic relationship between heating time and HMF concentration ($R^2 = 0.912$, $p < 0.001$), confirming that HMF formation does not follow a simple linear behavior. Among the heating durations evaluated, HMF concentrations at 3, 12, and 14 hours reached 38.784, 36.864, and 33.024 mg/kg, respectively. Although these values represent the highest levels observed during the heating process, they remain well below the maximum allowable limit of 75 mg/kg established by the Turkish Food Codex for pekmez and fruit concentrates (Oral et al., 2012) . Additionally, (Özmert Ergin, 2020b) emphasized that maintaining HMF levels should not exceed 50 mg/kg is essential for preserving product quality and adhering to standard processing guidelines. This indicates that, under national food safety regulations, the product remains within acceptable HMF levels even during extended thermal processing. These findings emphasize the importance of monitoring heating conditions to maintain product safety while preserving traditional production methods. Without such adjustments, the final product may not comply with recommended HMF limits, thereby affecting both its regulatory acceptance and nutritional quality.

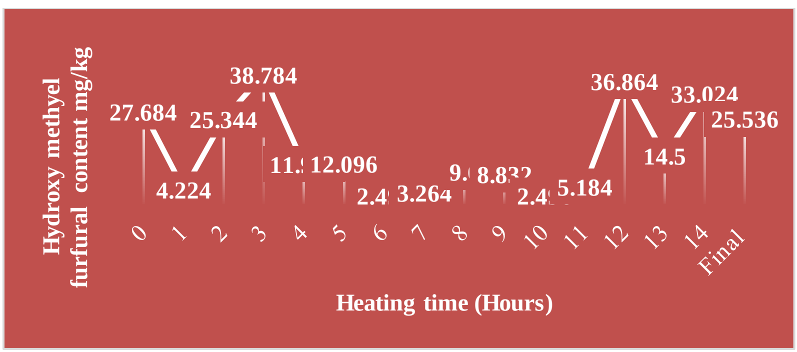

Figure 1. Effect of heating time on HMF hydroxymethyl furfural levels in pomegranate molasses

Effect of heating time on physiochemical properties during production of grape molasses

The influence of heating time on moisture content, total soluble solids (°Brix), titratable acidity, and pH during pomegranate molasses processing was analyzed using linear regression models and ANOVA, and LSD post-hoc comparisons at $p < 0.05$. Values at different time points are indicated in Table 2 by different letters. The results revealed statistically significant changes ($p < 0.05$) in all parameters with increasing heating duration. Moisture content showed a strong negative linear relationship with heating time ($R^2 = 0.979$, $p < 0.001$), decreasing from 96.46% at 0 hours (juice) to 20.35% (molasses) in the final sample this results close to results showed by (İncedayi et al., 2010; Yilmaz & Isik, 2005) . This indicates substantial water evaporation due to prolonged thermal exposure. In contrast, °Brix values significantly increased ($R^2 = 0.988$, $p < 0.001$), reflecting concentration of sugars and solutes during processing, with values rising from 6.4 to 74.3 this result is consistent with the findings of Yilmaz et al. (2007) who reported a 73.90. Similarly, titratable acidity increased markedly ($R^2 = 0.972$, $p < 0.001$), from 4.22% to 15.75%, suggesting the accumulation of acidic compounds as heating progressed this trend is consistent with previous findings, where the titratable acidity of pomegranate juice concentrate was reported to range from 5.8% to 14.27% (Vardin et al., 2008a).

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The pH exhibited a significant decreasing trend ($R^2 = 0.943$, $p < 0.001$), declining from 3.18 to 2.61 were in range with (Vardin et al., 2008b), consistent with the observed increase in acidity. This acidification may be attributed to organic acid formation, types of pomegranates, and concentration effects. According to the quality criteria outlined by (Özmert Ergin, 2020) pomegranate molasses should contain a minimum of 68.0% water-soluble dry matter, reflecting a high concentration of sugars and soluble solids that contribute to the product's consistency, sweetness, and shelf stability. The titratable acidity, expressed as citric acid, must not be lower than 7.5%, which ensures the desired tart flavor and enhances microbial safety. The pH value is expected to be approximately 3.0, maintaining the acidic environment necessary for preservation. Heating time significantly affected all measured properties of pomegranate molasses ($p < 0.05$). Moisture decreased steadily, TSS and titratable acidity increased, and pH gradually declined as heating progressed. LSD letters indicate that early, mid, and final time points were significantly different from each other, confirming that heating duration strongly influences the chemical and physical characteristics of the molasses. All these correlations were found to be statistically significant ($p < 0.05$), indicating that heating time had a strong and consistent effect on the physicochemical properties of pomegranate juice during its transformation into molasses.

Table 2. Effect of heating time on Quality properties in pomegranate molasses

Heating time (Hours)	Moisture %	Total soluble solid (°Brix)	Total Acidity (citric) %	pH
0	96.46^{a}	6.4^{a}	4.220^{a}	3.18^{a}
1	95.981^{a}	6.8^{a}	4.252^{a}	3.15^{a}
2	83.55^{b}	18.3^{b}	4.814^{b}	2.99^{b}
3	81.401^{b}	19.5^{b}	4.658^{b}	2.98^{b}
4	78.135^{b}	23.2^{c}	5.549^{c}	2.95^{b}
5	74.132^{c}	26.5^{c}	6.321^{c}	2.93^{b}
6	70.66^{c}	30.3^{c}	6.956^{d}	2.93^{c}
7	67.336^{d}	33.8^{d}	7.797^{d}	2.91^{c}
8	63.541^{d}	38^{d}	8.596^{e}	2.88^{c}
9	54.752^{e}	44^{e}	9.967^{e}	2.84^{c}
10	50.689^{e}	49.1^{e}	11.105^{f}	2.81^{d}
11	45.354^{f}	54.6^{f}	12.099^{f}	2.78^{d}
12	40.452^{f}	60^{f}	13.505^{g}	2.75^{d}
13	38.969^{g}	61.7^{f}	13.439^{g}	2.73^{d}
14	38.751^{g}	62.5^{g}	14.169^{g}	2.71^{d}
Final	20.353^{h}	74.3^{h}	15.747^{h}	2.61^{e}

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Different letters indicate statistically significant differences ($p < 0.05$) among the heating times for each parameter as determined by one-way ANOVA followed by LSD post-hoc test. Values sharing the same letter are not significantly different. Each parameter (Moisture, TSS, Titratable Acidity, pH) is analyzed independently.

Effect of heating time on Antioxidant activity

In the current study, antioxidant activity initially increased shown in figure 2, peaking between 6 and 7 hours of heating, followed by a gradual decline until hour 15. This pattern aligns with previous findings that suggest moderate heating facilitates the release of phenolic and flavonoid compounds from plant cell structures and promotes the formation of Maillard reaction products (MRPs), which possess antioxidative potential themselves (Akpinar-Bayizit et al., 2016); (Chalfoun-Mounayar et al., 2012). In the initial stages of heating (0–6 hours), DPPH inhibition rose from 83% to a peak of 95%, suggesting a significant increase in free radical scavenging capacity. The initial increase in antioxidant activity during the first 6 hours may be attributed to thermal breakdown of cell walls, releasing bound antioxidant compounds within the juice matrix. This process may also enhance the formation of Maillard reaction products and other thermally induced compounds that contribute to antioxidant capacity (Gülçin, 2012; Singleton et al., 1999). Such an increase is commonly observed in thermally processed fruit products, where moderate heat improves extractability and chemical transformation of antioxidant constituents. Supporting these finding, Helvacıoğlu et al., (2018) reported that traditionally produced grape molasses had significantly higher antioxidant capacity than those made with modern industrial methods. This difference may be duo to longer and more intense heat treatment in traditional processing, which can lead to both degradation and formation of new antioxidant compounds. Özcan et al., (2015) noted that antioxidant activity in red grape molasses increased steadily from the must stage to the final product, black and white grape molasses showed fluctuations throughout processing, possibly duo to instability or transformation of phenolic compounds under heat. Our results algin with these observations, showing a clear increase in antioxidant activity in red grape molasses, while black and white varieties exhibited inconsistent patterns during thermal treatment. Paul and Ghosh, (2012) found that the degradation of ascorbic acid in pomegranate juice was slower and less pronounced during heating within the range of 70 to 90 ℃, a stability attributed to various phytochemicals and their synergistic effects. Despite this, in our study after 6 hours of heating, a gradual decline in antioxidant activity was observed. this may be linked to thermal degradation or transformation of heat-sensitive antioxidant molecules, reducing their bioactivity. However, the linear regression analysis indicated that the downward trend in antioxidant activity was not statistically significant ($p > 0.05$), suggesting that heating time alone is not a strong predictor of antioxidant activity under these conditions. One possible explanation is the traditional pomegranate molasses processing method, which often involves low fire fueled by charcoal or controlled wood heat especially in villages setting. The intensity of heating can vary depending on producer technique or fuel conditions, with occasional increases in heat to accelerate evaporation at later stages. Such fluctuations in heating intensity may influence antioxidant stability in ways not captured by heating time alone, explaining the weak correlation found in the regression analysis. Overall, these findings suggest that antioxidant activity in pomegranate molasses is affected by heating, but not in a strictly linear or time-dependent manner. Instead, the interaction of heating time, intensity, and traditional processing techniques collectively influence antioxidant preservation.

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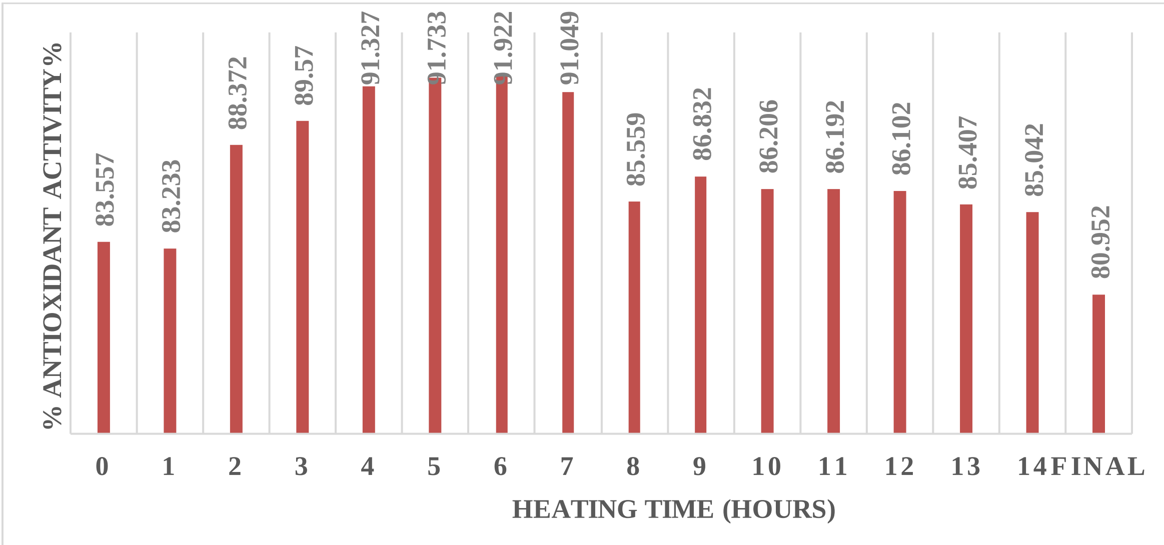

Figure 2: Effect of heating time on Antioxidant activity % in pomegranate molasses

Effect of Heating Time on Browning Index

The browning index (BI), which measures absorbance at 420 nm (A420), is a widely accepted parameter to evaluate the extent of non-enzymatic browning in fruit juices, particularly during thermal processing and storage. In the data presented, the BI steadily increased from 0.12 at hour 0 to 0.65 at the final time point (16 hours), indicating a progressive accumulation of browning pigments. This trend clearly suggests that prolonged heating enhances Maillard reaction activity and associated browning development in the juice matrix. Non-enzymatic browning, primarily due to the Maillard reaction and ascorbic acid degradation, is thermally driven and can be exacerbated by extended heat exposure, as supported by multiple studies. The Maillard reaction is a critical pathway in browning development during juice concentration. According to Guo & Nong-Xue (2010), browning in apple juice concentrates follows first-order kinetic behavior, with the reaction rate accelerating under higher temperatures, resulting in increased absorbance at 420 nm. In current dataset, HMF levels rise substantially in later stages (e.g., 25.3 mg/kg at the final point), correlating with the increase in BI. Furthermore, shows that °Brix levels increased from 6.4 to 74.6 during heating, which further supports Maillard reaction acceleration, as reducing sugars are key reactants. Studies such as that by (Alonso-Riaño et al., 2024) demonstrated the importance of sugar-amino interactions in browning kinetics and stressed that temperature and sugar concentration play key roles in BI development

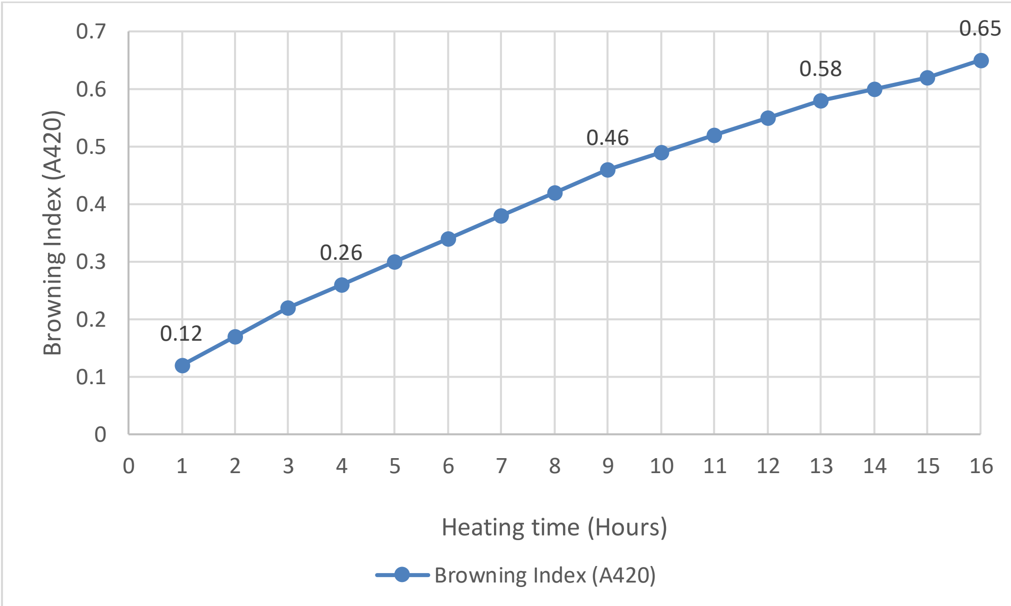

Figure 3: Effect of heating time on Browning index in pomegranate molasses

IV. Conclusions

The findings of this study clearly demonstrate that heating time plays a crucial role in determining the quality and nutritional profile of pomegranate molasses. final HMF levels remained within acceptable limits. Antioxidant activity levels increased significantly during the first 6–7 hours of heating, reaching peak values (DPPH: 95%) and an increase in browning index (from 0.12 to 0.65), indicating thermal degradation and Maillard reaction progression. These changes were accompanied by increased sugar concentration and acidity, affecting both taste and nutritional value. Overall, this study highlights that careful control of heating time, particularly limiting it to 6–7 hours can maximize antioxidant potential and bioactive retention while minimizing nutrient loss and quality deterioration in traditional pomegranate molasses production. These findings provide practical guidance for small-scale and industrial producers to optimize processing condition for higher quality molasses with improved functional value, improved consistency and grater market competitiveness.

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