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THERMAL EFFECTS ON 5-HYDROXYMETHYLFURFURAL FORMATION AND QUALITY OF TOMATO PASTE

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ABSTRACT

Tomato paste is popular in cooking for its strong flavour and colour, but high cooking temperatures can lead to the formation of 5-hydroxymethylfurfural (HMF), raising toxicity concerns. This study examined thermal treatments at 150, 170, and 190°C for up to 30 minutes on tomato paste from two commercial brands. Results showed a negative correlation between sugar degradation and HMF formation, with HMF levels increasing with temperature and time, peaking at 62.30 mg per 100 g of dry matter. Kinetic analysis indicated that HMF formation followed zero-order kinetics, with rate constants between 0.78 and 1.74 mg kg^{-1} min^{-1}, and activation energies of 32.01 and 32.50 kJ mol^{-1}. The findings suggest that lower cooking temperatures for longer periods may reduce HMF formation while maintaining quality. Future studies will likely contribute to evaluating the effects of alternative processing methods and components added to the ketchup formulation on HMF kinetics.

Key words: Activation energy, Maillard reaction, sugar degradation, thermal processing, tomato paste, 5-hydroxymethylfurfural

ISIL İŞLEMİN DOMATES SALÇASINDA 5-HİDROKSİMETİLFURFURAL OLUŞUMU İLE KALİTE ÖZELLİKLERİNE ETKİSİ

ÖZ

Domates salçası, yoğun tadı ve karakteristik rengi nedeniyle yemek pişirmede yaygın olarak kullanılmaktadır. Ancak yüksek pişirme sıcaklıkları, 5-hidroksimetilfurfural (HMF) oluşumuna yol açarak potansiyel toksisite açısından endişe yaratabilmektedir. Bu çalışmada, iki farklı ticari markaya ait domates salçaları 150, 170 ve 190°C'de 30 dakikaya kadar ısıl işleme tabii tutulmuştur. Elde edilen sonuçlar, şeker degradasyonu ile HMF oluşumu arasında negatif bir korelasyon bulunduğunu göstermiştir. HMF düzeyleri, sıcaklık ve süre artışına paralel olarak yükselmiş ve 100 g kuru madde başına 62.30 mg seviyesine ulaşmıştır. Kinetik analizler, HMF oluşumunun sıfır dereceden kinetik modele uyduğunu ortaya koymuş; reaksiyon hız sabitlerinin 0.78-1.74 mg kg^{-1} dk^{-1} aralığında ve aktivasyon enerjilerinin ise 32.01 ve 32.50 kJ mol^{-1} olduğunu göstermiştir. Bu bulgular, daha düşük sıcaklıklarda daha uzun süreli pişirme uygulamalarının, ürün kalitesi korunurken HMF oluşumunu sınırlayabileceğini düşündürmektedir. Gelecekte yapılacak çalışmaların, alternatif işleme yöntemlerinin ve salça formülasyonuna ilave edilen bileşenlerin HMF kinetiği üzerindeki etkisinin değerlendirilmesine katkı sağlayacağı düşünülmektedir.

Anahtar kelimeler: Aktivasyon enerjisi, Maillard reaksiyonu, şeker degradasyonu, ısıl işlem, domates salçası, 5-hidroksimetilfurfural

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INTRODUCTION

Tomato (*Lycopersicon esculentum* Mill.) is a popular vegetable, used fresh or in products such as canned sauces, purees, and pastes (Gao et al. 2021). It is well-liked in global cuisine for its intense flavour and colour and has a rich nutritional profile (El-Mansy et al. 2021). It also contains essential nutrients, including sugars, organic acids, and antioxidants such as phenols (Alkanan et al. 2021; Vitucci et al. 2021).

Heat treatment is a widely used method in food processing and cooking that improves flavour, colour, texture, and shelf life (Stadler, 2012). This process leads to the formation of desirable compounds like melanoidins, but high temperatures can also produce mutagenic HMF, a byproduct of Maillard reactions between reducing sugars and amino acids or sugar dehydration under acidic conditions, as shown in Figure 1 (Akıllıoğlu et al. 2015).

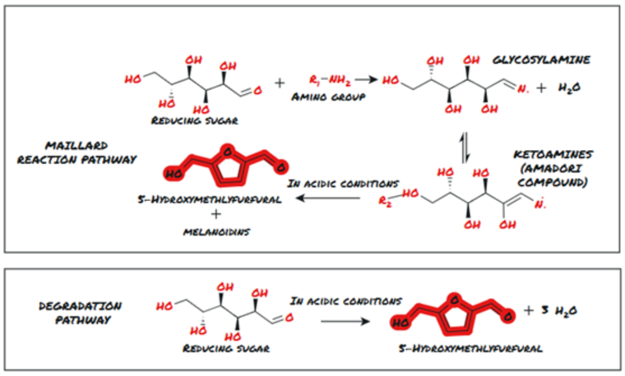

Figure 1. Mechanisms of 5-HMF formation in tomato paste

The accumulation of HMF may compromise food safety and pose potential risks to human health, as it has been associated with chronic metabolic disorders, including obesity, diabetes, and cardiovascular diseases (Noh et al. 2024). Due to its potential toxic effects, HMF is widely recognized as an indicator of thermal processing intensity in various food products. Regulatory limits have been established for certain foods; for example, the maximum allowable level in honey is 40 mg/kg (Codex Alimentarius, 2001), highlighting its relevance to food safety (Choudhary et al. 2020). Although no specific maximum limit has been defined for tomato paste within the European Union, monitoring HMF levels in thermally processed products such as tomato paste remains essential to ensure product quality and protect consumer health.

Previous studies have emphasized the importance of 5-hydroxymethylfurfural (HMF) in tomato products as an indicator of thermal processing intensity. Reported HMF concentrations range from 0.94 to 39.40 µg/g on a dry matter basis, highlighting the strong influence of heat treatment (Akıllıoğlu et al., 2015). In addition, Kus et al. (2005) reported that HMF levels in commercially produced tomato pastes ranged from 3.6 to 18 mg/kg. HMF formation is closely related to both sugar content and processing conditions. In particular, tomato paste, which is rich in sugars such as glucose and fructose, shows a greater tendency for HMF formation. High sugar content and acidic pH promote HMF formation via Maillard reactions, while low moisture conditions further accelerate this process through sugar dehydration (Akıllıoğlu et al., 2015). In this context, the present study investigates the effects of heat treatment on HMF formation in tomato paste and its relationship with key quality parameters, intending to evaluate potential food safety risks and optimize processing conditions for both domestic and industrial applications.

MATERIALS AND METHODS

Materials and Chemicals

Tomato pastes from two popular tin-packaged brands (TP-A and TP-B) were sourced from local markets in Bursa, Türkiye, to assess the impact of thermal processing on 5-hydroxymethylfurfural formation and quality. Acetonitrile (HPLC grade) and sulfuric acid (≥96%) were sourced from Merck (Germany), while standards for glucose, fructose, sucrose, and 5-hydroxymethylfurfural (≥99%) were from Sigma-Aldrich (USA).

Method

Heat Treatment Process

An experimental setup was designed to study changes in tomato paste during thermal processing at three temperatures (150°C, 170°C, and 190°C) for seven durations (0, 5, 10, 15, 20, 25, and 30 minutes). For each combination, 20 g of tomato paste was placed in separate glass jars and processed in a Memmert UNE 500 oven. After each duration, samples were removed, cooled to room temperature, and stored at -18°C until analysis.

Water Activity, Brix, pH and Colour Measurement

Water activity (aw) was measured using a Novasina AG Labmaster AW (Switzerland). pH was determined with a Metrohm meter after homogenizing. Colour parameters were analysed using a HunterLab UltraScan VIS (USA). Water-soluble dry matter content (°Brix) was measured with a Kyoto KEM/RA-600 refractometer (Japan), with all measurements taken at 25°C.

HMF Analysis

The HMF content in tomato paste samples was determined using the method by Tan et al. (2025). One gram of the sample was diluted with 5 mL of distilled water, followed by the addition of 300 µL each of Carrez I and Carrez II solutions. The mixture was sonicated for 1 min, centrifuged at 2700 g for 10 min, and the supernatant was filtered through a 0.22 µm filter. It was then transferred to glass vials for HPLC analysis (Agilent, USA) on a C18 column at 25°C, using a mobile phase of 5% acetonitrile. The injection volume was 20 µL with a flow rate of 1 mL/min. Detection occurred at 284 nm, and HMF concentrations were quantified based on a calibration curve ($R^2>0.99$)

Sugar Analysis

Glucose, fructose, and sucrose were analysed by high-performance liquid chromatography (HPLC) following a modified method from Moe et al. (2022). Samples were dissolved in distilled water and filtered through a 0.22 µm syringe filter. Analysis was performed on an Agilent 1260 Infinity HPLC with a refractive index detector, using an Agilent Hi-Plex H column (300 mm × 7.7 mm). An isocratic mobile phase of 0.05 mM sulfuric acid (H_2SO_4) at a flow rate of 0.6 mL/min was used, with a column temperature of 60°C and detector temperature at 35°C. The injection volume was 20 µL, and sugars were identified by retention times compared to standards, with quantification based on calibration curves ($R^2 = 0.99$).

HMF formation kinetics

Determination of Reaction Degree and Rate Constant (k)

Reaction rate constants (k) were calculated using the zero-order kinetic model according to the equation below:

$$A_t = A_0 - kt \qquad (1)$$

where A_t denotes the initial HMF concentration (mg kg^{-1}), A_t denotes the HMF concentration (mg kg^{-1}) at time t(min), and k is the zero-order reaction rate constant (mg kg^{-1} min^{-1}).

Calculation of activation energy and frequency factor

The Arrhenius equation (Equation 2) was used to determine the activation energy (E_a) of the reaction responsible for changes in HMF concentration:

$$k = k_0\, e^{-E_a/RT} \qquad (2)$$

where k is the reaction rate constant at a given temperature (mg kg^{-1} min^{-1}), kJ mol^{-1} is the frequency factor representing the collision probability of reactants (mg kg^{-1} min^{-1}), T is the absolute temperature (K), E_a is the activation energy (kJ mol^{-1}), and R is the universal gas constant (8.314 J mol^{-1} K^{-1}).

Calculation of the Q10 Value

The Q_{10} value describes the increase in reaction rate resulting from a 10°C rise in temperature. It was calculated using Equation (3), where k_1 and k_2 are the reaction rate constants at temperatures T_1 and T_2(K), respectively:

$$Q_{10} = \left(\frac{k_2}{k_1}\right)^{\frac{10}{T_2 - T_1}} \qquad (3)$$

Statistical analysis

Statistical analyses were conducted with IBM SPSS 30.0 software, utilizing Pearson correlation and assessing significance with p-values at 0.01 and 0.05. One-way ANOVA was applied, with Duncan's Multiple Range Test for post-hoc comparisons ($P<0.05$). All experiments were performed in triplicate.

RESULTS AND DISCUSSION

Quality properties of tomato pastes before treatment

The initial values of tomato pastes (Brix, pH, water activity, ash, dry matter, titratable acidity, and a*/b*) are given in Table 1. No statistically significant differences were observed between the samples except for a*/b*. The Brix, pH, water activity, ash, dry matter, titratable acidity, and a*/b* values for A paste were 27.83±0.29%, 4.22±0.04, 0.89±0.00, 0.03±0.00, 27.75±0.23%, 1.45±0.14, and 1.75±0.13; for B paste, they were 27.67±0.58%, 4.32±0.07, 0.89±0.00, 0.03±0.00, 26.87±0.00%, 1.40±0.07, and 1.58±0.15, respectively. The salt contents in A and B samples were 0.62±0.01% and 0.57±0.01%, respectively, which are well below the maximum limit of 2% (w/w) set by the Food and Drug Administration (FDA) (Sobowale et al. 2012). The obtained initial values are consistent with the tomato paste data reported in the literature. For example, Akıllıoğlu et al. (2015) reported Brix values ranging from 14.5-37.3%, and pH values from 4.09-4.34. Similarly, Sobowale et al. (2012) found pH, total solids, salt content, and TSS (Brix) values in different tomato paste samples to be 4.87-5.30, 54.90-68.90%, 0.68-2.50%, and 42.60-76.40%, respectively. Furthermore, in another study, the titration acidity values of different tomato pastes were found to be in the range of 0.41-0.54 g citric acid/100 g (Marcondes et al. 2021). The literature indicates that pastes with a*/b* values below 1.80 have poor colour quality. The fact that paste B falls below this threshold value indicates that its colour quality is inadequate (Akıllıoğlu et al. 2015).

Table 1. Quality properties of tomato pastes before heat treatment

	TP-A	**TP-B**
Brix (%)	27.83±0.29^{a}	27.67±0.58^{a}
pH	4.22±0.04^{a}	4.32±0.07^{a}
A_w	0.89±0.00^{a}	0.89±0.00^{a}
Salt (%)	0.62±0.01^{a}	0.57±0.01^{a}
Ash (%)	0.03±0.00^{a}	0.03±0.00^{a}
Dry Matter (%)	27.75±0.23^{a}	26.87±0.00^{a}
Titratable Acidity (g citric acid/ 100g)	1.45±0.14^{a}	1.40±0.07^{a}
a/b**	1.75±0.13^{a}	1.58±0.15^{b}

*Different letters (a-b) within the same column indicate significant differences (*P*<0.05).

Quality properties of tomato pastes after treatment

As a result of the thermal treatments applied to tomato paste samples (TP-A and TP-B), changes in pH, water activity (aw), colour values, sugar content, and HMF levels were investigated depending on the combinations of different temperatures (150, 170, and 190°C) and durations (0-30 min). The relationships between HMF and the other quality parameters were evaluated by correlation analysis, and the results are presented in Table 2.

Table 2. Correlation coefficients between HMF and quality parameters in tomato paste

	TP-A			**TP-B**		
T (°C)	150	170	190	150	170	190
Glucose	-0.921**	-0.914**	-0.913**	-0.916**	-0.790*	-0.885**
Fructose	-0.852*	-0.835*	-0.795*	-0.901**	-0.843*	-0.942**
Sucrose	-0.506	-0.754*	-0.838*	-0.706	-0.838*	-0.914**
pH	0.840*	0.558	0.712	-0.423	-0.541	0.224
L*	0.779*	0.686	-0.302	0.889**	0.058	0.388
a*	-0.959**	-0.981**	-0.868*	-0.557	-0.718	-0.808*
b*	-0.981**	-0.981**	-0.745	-0.748	-0.651	-0.787*
C*	-0.968**	-0.982**	-0.840*	-0.634	-0.697	0.800*

*Correlation is significant at the 0.05 level (*P*<0.05); ** Correlation is significant at the 0.01 level (*P*<0.01).

No significant change was observed in water activity and pH values with increasing heat treatment time and temperature. When colour values are examined, no significant difference is observed in terms of L value; however, in the TP-A sample, it was determined that at the end of 190°C, a* value decreased from 19.47 to 12.00, and the b* value decreased from 10.81 to 5.79. Similarly, in the TP-B sample, the a* value decreased from 12.58 to 6.48, and the b* value decreased from 8.19 to 2.96 at the end of 190°C. This decrease is thought to be due to a reduction in colour intensity resulting from Maillard reactions, caramelization, and particularly the thermal degradation of natural pigments such as lycopene at high temperatures.

When HMF changes were examined, the initial HMF content of TP-A was 3.76 mg/kg, increasing to 30.24 mg/kg at 150°C, 34.41 mg/kg at 170°C, and 58.75 mg/kg at 190°C after 30 min (Figure 2). Similarly, in TP-B, the initial HMF level was 4.52 mg/kg and reached 31.25 mg/kg, 40.31 mg/kg, and 62.30 mg/kg at 150, 170, and 190°C, respectively.

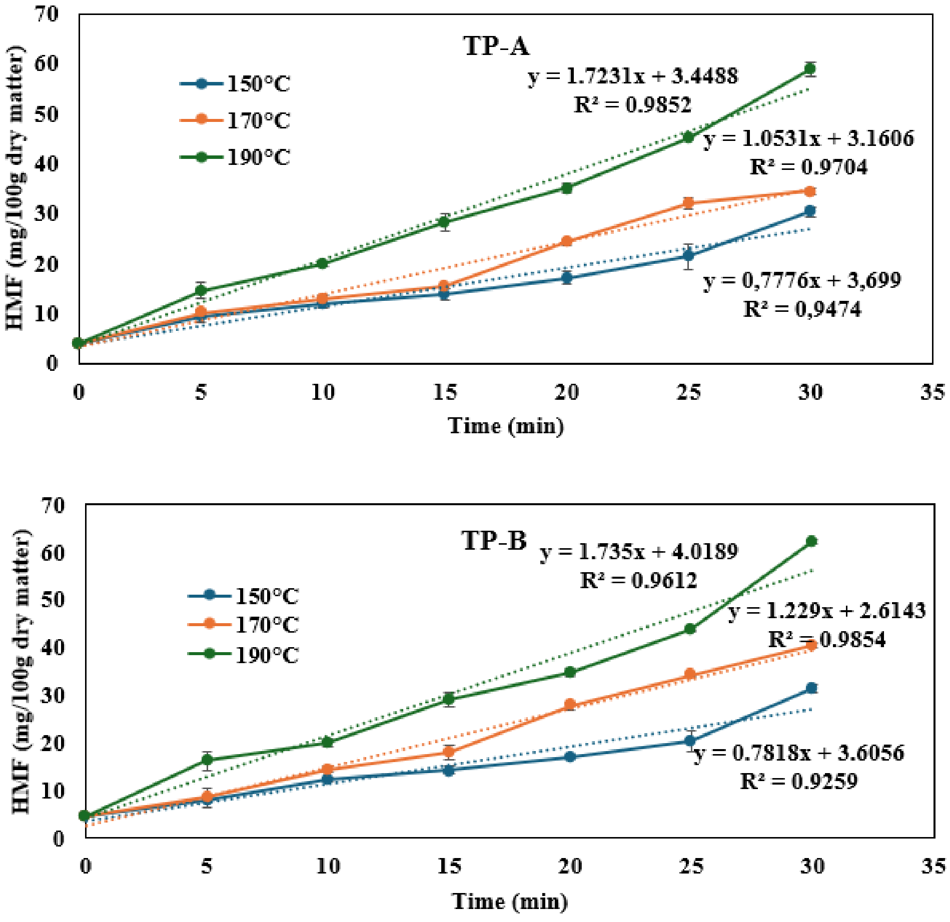

Figure 2. Zero-order formation of HMF in tomato pastes (TP-A and TP-B) at 150°C, 170°C, and 190°C

As shown in Figure 3, sugar contents decreased throughout the processing period at all temperature levels, and this decrease became more pronounced with increasing temperature. Table 2 revealed a statistically significant negative correlation between HMF and reducing sugar content, indicating that as HMF concentration increased over time, reducing sugar levels decreased, demonstrating an inverse relationship between these parameters. Although sucrose is not a reducing sugar and does not directly convert to HMF, its hydrolysis into glucose and fructose is sped up under acidic and high-temperature conditions. These monosaccharides actively participate in HMF formation. Therefore, the observed reduction in total sugar content can be attributed both to the direct conversion of reducing sugars into HMF and to the indirect contribution of sucrose, which is first hydrolyzed into reducing sugars and subsequently converted into HMF.

Correlation analyses also revealed that an increase in HMF levels was accompanied by a decrease in both the a* (redness) and b* (yellowness) colour parameters. Additionally, a negative correlation was found between colour tone (h°) and HMF. These findings indicate that, parallel to the increase in HMF formation during the thermal processing, there are deteriorations in the colour intensity and tone values of the product.

The amount of HMF in tomato paste is affected by heat treatment conditions, depending on temperature and time. Yeom et al. (2016) showed that HMF concentrations increased significantly in tomato paste stored at high temperatures, indicating a direct correlation between storage conditions and HMF accumulation. Shridhar et al. (2022) found that the HMF content in tomato paste was 4.5 ppm after 30 minutes of heat treatment at 55°C, 12.3 ppm after 30 minutes at 85°C, and 16.8 ppm after 30 minutes at 95°C. Similarly, Marcondes et al.

(2021) reported that 5-HMF content in tomato-based products ranged from 1.30 to 312 mg/kg, showing a significant positive correlation with sugar (fructose and glucose) levels and acidity. Ribeiro et al. (2012) evaluated the HMF levels of fresh extracted flower honey exposed to heat at different temperatures ranging from 30 to 100°C at time intervals of 30, 45, 60, 180, and 270 minutes. They found that honey with an initial HMF content of 2.2 mg/kg exceeded the maximum limit value (60 mg/kg) according to the Brazilian Food Codex when exposed to 90°C for 180 minutes.

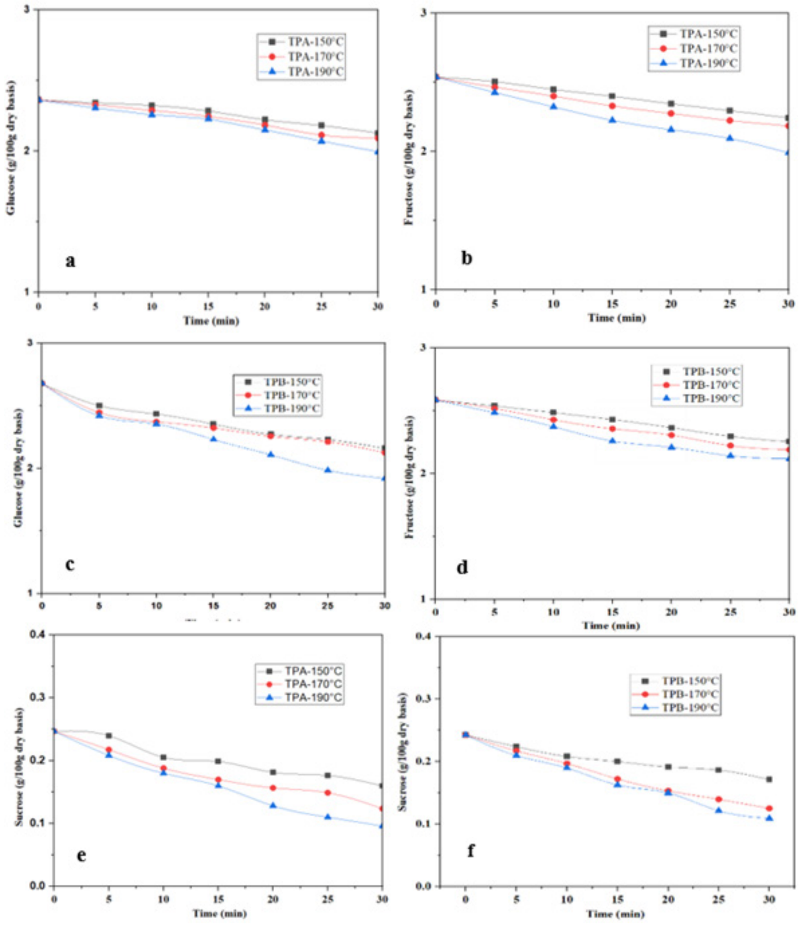

Figure 3. Changes in sugar contents of tomato pastes during thermal treatment at 150°C, 170°C, and 190°C: (a) glucose in TP-A, (b) fructose in TP-A, (c) glucose in TP-B, (d) fructose in TP-B, (e) sucrose in TP-A, and (f) sucrose in TP-B

Kinetic Evaluation of HMF

The HMF formation graph is presented in Figure 2, while the kinetic parameters are given in Table 3. The changes in HMF content for TP-A and TP-B during heat treatment were analysed, with results indicating both samples followed a zeroth-order kinetic model (R^2>0.9). HMF formation rates peaked at 190°C, with degradation rate constants increasing with temperature-TP-A rising from 2.18 to 4.82 $mg.kg^{-1}.min^{-1}$ and TP-B from 2.19 to 4.80 $mg.kg^{-1}.min^{-1}$. Activation energies were similar (32.01 kJ/mol for TP-A; 32.50 kJ/mol for TP-B), indicating comparable thermal stability. The reaction frequency factor was higher for TP-B, but TP-A showed a greater increase in reaction rate at elevated temperatures (170-190°C). Previous studies on HMF formation in various food products have highlighted the impact of temperature and time.

Table 3. Kinetic parameters of TP-A and TP-B samples at different temperatures

Sample	Rate Order	Temperature (°C)	k ($mg.kg^{-1}.min^{-1}$)	R^2	Ea (kj/mol)	F ($mg.kg^{-1}.min^{-1}$)	Q_{10} (150-170°C)	Q_{10} (170-190°C)
TP-A	0	150	0.78	0.95	32.01	6634	1.16	1.27
		170	1.05	0.97				
		190	1.72	0.99				
TP-B	0	150	0.78	0.93	32.50	8103	1.25	1.18
		170	1.23	0.99				
		190	1.74	0.96				

Activation energy (E_a) is the minimum energy needed for a reaction to begin. A low E_a allows for quicker reactions at lower temperatures, while a high E_a slows reactions, requiring higher temperatures. For 5-HMF formation, E_a influences reaction rate and temperature sensitivity. Hidalgo and Pompei (2000) reported an E_a of 139.9 kJ/mol for HMF reactions in tomatoes, whereas Burdurlu et al. (2006) reported values of 181.6-334.8 kJ/mol for citrus juice concentrates. Tan et al. (2025) found activation energies for HMF kinetics in carob molasses to range from 34.51 to 58.02 kJ/mol. Wibowo et al. (2015) reported an activation energy of 136 kJ/mol for HMF accumulation in pasteurized orange juice.

The frequency factor (k_0) indicates how quickly reactions can occur; a higher factor suggests faster HMF formation under certain conditions. Karadeniz et al. (2024) reported a frequency factor of 145.39 for 5-HMF formation in date paste at 25-45°C. Q_{10} shows the effect of a 10°C increase in temperature on reaction rate. Reddy et al. (1999) found Q_{10} values of 2.38 and 2.17 with activation energies of 121.68 and 108.70 kJ/mol for UHT milk, respectively. Other studies have shown that HMF content increases with storage temperature and is significantly higher under oxygenated conditions. Our study found that TP-A and TP-B samples exhibited similar kinetic properties and thermal sensitivity, with any differences likely due to compositional variations. These findings are crucial for understanding the impacts of thermal processing on product stability.

CONCLUSION

Tomato paste is a key ingredient in many cuisines; however, high-temperature processing can lead to the formation of 5-hydroxymethylfurfural (HMF), a compound with mutagenic potential. In this study, HMF formation increased significantly with both temperature and processing time, reaching a maximum of 62.30 mg/100 g dry matter. This increase was accompanied by reductions in sugar content and noticeable changes in colour parameters, with an inverse relationship observed between sugar degradation and HMF formation. Kinetic analysis showed that HMF formation followed a zero-order model, with reaction rate constants ranging from 0.78 to 1.74 mg kg^{-1} min^{-1} and activation energies of 32.01-32.50 kJ mol^{-1}, indicating similar thermal behaviour between samples. These findings demonstrate that HMF formation is strongly dependent on processing conditions and that product quality and safety can be effectively controlled by optimizing temperature-time combinations. Furthermore, this approach offers significant potential for industrial applications by enabling the development of safer and higher-quality food products through controlled thermal processing.

AUTHOR CONTRIBUTIONS

Büşra Ata İrdam: Investigation, Writing - Original Draft; Esranur Tan: Methodology, Writing - Original Draft, Writing - Review & Editing; Rasim Alper Oral: Conceptualization, Methodology, Supervision.

CONFLICTS OF INTEREST

The authors declare no conflict of interest.

DATA AVAILABILITY

Data sharing does not apply to this article.

ARTIFICIAL INTELLIGENCE STATEMENT

Artificial intelligence tools were used only for language editing.

AUTHOR CONTRIBUTIONS

The authors declare that they have contributed equally to the article. All the authors declare that they have seen/read and approved the final version of the article ready for publication.

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