ÇUKUROVA UNIVERSITY
INSTITUTE OF NATURAL AND APPLIED SCIENCES

MSc THESIS

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FORMATION OF FURFURALS AND DEGRADATION OF BIOACTIVE COMPOUNDS DURING MICROWAVE ROASTING OF ARABICA COFFEE BEANS

DEPARTMENT OF FOOD ENGINEERING

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ABSTRACT

MSc THESIS

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In this study, the effects of microwave roasting process conditions (time and temperature) on *Coffea Arabica* beans physicochemical properties such as phenolic profiles, caffeine, hydroxymethylfurfural (HMF), Furfural (F), sugar degradation, browning index (BI) and antioxidant capacity were investigated. Convection roasting is the common method for roasting coffee, but this method negatively affects coffee. Therefore, in the present study, the microwave roasting method was applied. Its effect on (colour, dry matter content, total phenolic content, phenolic compounds, sugar degradation, HMF, F formation and sensory properties) L* value is a good indicator for determining the roasting degree of coffee beans. It decreased remarkably by increasing roasting temperature and time. HMF and furfural rose to a certain level but degraded remarkably by increasing time and temperature. A higher decomposing rate was determined for coffee beans roasted at higher temperature. Caffeine content increased by increasing roasting time and temperature. Total phenolic content and antioxidant activities of coffee beans had decreasing tendency according to roasting conditions. The kinetic study findings indicated that higher degradation rate constant values were calculated for the samples roasted at a higher temperature.

Key Words: Arabica Coffee, Microwave Roasting, Hydroxymethylfurfural and Furfural, Sugar degradation.

ÖZ

YÜKSEK LİSANS TEZİ

KAHVE ÇEKİRDEKLERİNİN MİKRODALGA İLE KAVRULMASI SIRASINDA FURFURALARIN OLUŞUMU VE BİYOAKTİF BİLEŞİKLERİN PARÇALANMASI

Yasmeen Badawi Yaseen JAMJOUM

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Bu çalışmada, caffee Arabica çekirdeklerinde mikrodalga kavurma proses koşullarının (zaman ve sıcaklık) fenolik profiller, kafein, hidroksimetilfurfural (HMF), şeker bozunması, esmerleşme indeksi (BI) ve antioksidan kapasite gibi fizikokimyasal özelliklere etkileri araştırılmıştır. Konveksiyon kavurma, kahveyi kavurmak için yaygın bir yöntemdir ancak bu yöntem kahveyi olumsuz etkiler. Bu nedenle, bu çalışmada mikrodalga kavurma yöntemi uygulanmış ve (renk, kuru madde içeriği, toplam fenolik içerik, fenolik bileşikler, şeker bozunması, HMF, F oluşumu ve duyusal özellikler) üzerindeki etkisi L* değerinin belirlenmesinde iyi bir göstergedir. Kahve çekirdeklerinin kavurma derecesi ve kavurma sıcaklığının ve süresinin artmasıyla önemli ölçüde azalmıştır. HMF ve furfural belirli bir seviyeye yükselmiş, ancak zaman ve sıcaklığın artmasıyla önemli ölçüde parçalanmışlardır. Daha yüksek sıcaklıkta kavrulmuş kahve çekirdeklerinde daha yüksek bozunma hızı tespit edilmiştir. Kavurma süresinin ve sıcaklığının artmasıyla kafein içeriği artmıştır. Kahve çekirdeklerinin toplam fenolik içeriği ve antioksidan aktiviteleri kavurma koşullarına göre azalma eğilimi göstermiştir. Kinetik çalışma bulguları, daha yüksek sıcaklıkta kavrulmuş örn içinkler daha yüksek bozunma hızı sabit değerlerinin hesaplandığını göstermiştir.

Anahtar Kelime: Arabica kahve, Mikrodalga kavurma, Hidroksimetilfurfural ve Furfural, Şeker parçalanması

EXTENDED ABSTRACT

Coffee is one of the most-consumed beverages worldwide after water due to its flavour, which attracts the consumers, and its distinctive aroma. Although the genus coffee has over a hundred species and subspecies, the focus is only on the two main types of coffee, Arabica coffee (Arabica coffea) and Robusta coffee (Robusta coffea). The present study was on Arabica coffee beans. The higher value of Arabica coffee is attributed to its various sensory and aromatic properties. On the other hand, the high extractability of canephora coffee is considered a significant factor contributing to the market's growth. The taste of coffee is affected by many factors; age, culture, geography, storage, and preparation methods all significantly impact the diversity of coffee consumers, but the main factor is roasting.

The roasting process is a thermal process causing several physical and chemical changes in coffee beans. Roasted coffee produces melanoidins, a type of brown pigment; these substances, along with trigonelline and caffeine, are thought to be strong antioxidants that have many benefits for human health. The roasting stage of natural coffee processing is considered to be crucial for generating changes in bioactive components like polyphenols. The degree of roasting significantly impacts the antioxidant and sensory qualities. The roasting process depends on two factors (time and temperature). It is known that the common coffee roasting method is the convection roasting method by using an oven at different temperatures (180-250 ^{o}C) for varying lengths of time; for example, the coffee was roasted at (160 ^{o}C, 180 ^{o}C, and 220 ^{o}C) for 10, 20, 30, and 40 mins, hold at each temperature to produce the three primary levels of roasting being light, medium and dark. But this method has many harmful effects on the coffee beans, which are the long roasting time to produce the product, non-uniform roasting of coffee beans and burns the surface of coffee beans, which negatively affect the taste, smell, and colour of coffee.

Therefore, in the present study, the microwave roasting method was applied. Using a microwave for roasting has numerous advantages due to the possibility of increasing control over the roasting process in terms of start-up and shutdown times, process speed, and energy savings compared with the traditional roasting process. Furthermore, its effect on Arabica coffee beans' physical and chemical properties (colour, dry matter content, TPC, sugar degradation, HMF formation and general impression of coffee taste) were studied. Microwave roasting was applied by controlling two variables, namely time and temperature.

Three temperature degrees were chosen according to the previous studies, which are 180, 200, and 220 °C degrees for 120, 40, and 20 minutes, respectively. Therefore, a temperature of 200 °C was chosen as the optimum degree for roasting Arabica coffee beans using a microwave, which can produce light, medium, and dark roasted coffee beans.

Microwaves affect coffee beans positively by increasing the content of TPC and antioxidant activity of the coffee bean extract by increasing microwave power and roasting length of time; however, extreme microwave power levels and roasting durations had a negative impact because of roasting for a longer time or at higher microwave power levels that caused their degradation. In addition, it was observed that hydroxymethylfurfural (HMF) increased to a certain extent, but increasing time and temperature decomposed significantly.

It was found that microwave roasting decreases roasting time, improves coffee flavour compared to convection roasting, and increases coffee beans' nutritional value. In addition, microwave roasting produced a better quality of coffee beans with the desired flavour.

It is recommended to use the microwave roasting method for coffee beans to produce high-quality products with high nutritional value. Its recommended to expand on the studies of HMF decomposition and follow the reactions passing through to define any compounds that are converted at high temperature and to study the physical properties of coffee beans and the best position inside microwave ovens in large quantities so that this method can be realistically applied.

GENİŞLETİLMİŞ ÖZET

Kahve, tüketicileri kendine çeken ve kendine özgü aroması nedeniyle sudan sonra Dünya'da en çok tüketilen içeceklerden biridir. Kahvenin yüzden fazla türü ve alt türü vardır, sadece iki ana kahve türü, Arabica kahvesi (Arabica coffea) ve Robusta kahvesi (Robusta coffea)dır. Bu çalışmada Arabica kahve çekirdekleri kullanılmıştır. Arabica kahvesi kuvvetli duyusal ve aromatik özelliklerinden dolayı daha çok tüketilmektedir. Öte yandan, canephora kahvesinin yüksek özütlenebilirliği, pazarın büyümesine katkıda bulunan önemli bir faktör olarak kabul edilmektedir. Kahvenin tadı birçok faktörden etkilenir; yaş, kültürel önlemler, coğrafya, saklama ve hazırlama yöntemleri, kahve tüketicilerinin çeşitliliği üzerinde büyük bir etkiye sahiptir, ancak bunlardan en önemlisi kavurma işlemidir.

Kavurma işlemi, kahve çekirdeklerinde bir takım fiziksel ve kimyasal değişikliklere neden olan termal bir işlemdir. Kavrulmuş kahvede, bir tür kahverengi pigment olan melanoidinler oluşur, bu maddelerin trigonellin ve kafein ile birlikte insan için birçok faydası olan güçlü antioksidanlar olduğu düşünülmektedir. Doğal kahvenin işlenmesinin kavurma aşamasının, polifenoller gibi biyoaktif bileşenlerde değişiklikler meydana getirmek için çok önemli olduğu düşünülmektedir. Kavurma derecesi, antioksidan ve duyusal nitelikler üzerinde büyük bir etkiye sahiptir. Kavurma işlemi iki faktöre bağlıdır (zaman ve sıcaklık). Kahve kavurmanın yaygın yönteminin, farklı sıcaklıklarda (180-250 ^{o}C) farklı sürelerde fırın kullanılarak konveksiyonel kavurma yöntemi olduğu bilinmektedir. Örneğin kahve (160 ^{o}C, 180 ^{o}C ve 220 ^{o}C), açık, orta ve koyu olmak üzere üç temel kavurma seviyesini üretmek için her sıcaklıkta 20, 30 ve 40 dakika tutulmaktadır. Ancak bu yöntemin, ürünün üretilmesi için uzun kavrulma süresi, kahve çekirdeklerinin düzensiz kavrulması ve kahve çekirdeklerinin yüzeyini yakması gibi kahve çekirdekleri üzerinde birçok zararlı etkisi vardır, bu da kahvenin tadını, kokusunu ve rengini olumsuz etkilemektedir.

Bu nedenle, bu çalışmada mikrodalga kavurma yöntemi uygulanmış olup, geleneksel kavurma işlemine kavurma sıcaklıkları ve süreleri, işlem hızı ve enerji tasarrufu açısından kavurma işlemi üzerindeki kontrolü artırma olasılığı nedeniyle kavurma için mikrodalga kullanılması pek çok avantaja sahiptir. Arabica kahve çekirdeklerinin fiziksel ve kimyasal özelliklerine (renk, kuru madde içeriği, topşam fenolik madde (TPC), şeker bozulması, HMF oluşumu ve kahve tadı genel izlenimi) ve prosesin etkisi incelenmiştir. Mikrodalga kavurma, zaman ve sıcaklık olmak üzere iki değişken kontrol edilerek uygulanmıştır.

Önceki çalışmalara göre sırasıyla 120, 40, 20 dakika için 180, 200, 220°C olmak üzere üç sıcaklık derecesi seçilmiştir. Arabica kahve çekirdeklerinin hafif, orta ve koyu kavrulmuş kahve çekirdekleri üretebilen mikrodalga kullanılarak kavrulması için optimum derece olarak 200°C seçilmiştir.

Mikrodalga, mikrodalga gücünü ve kavurma süresini artırarak kahve çekirdeği ekstraktının TPC içeriğini ve antioksidan aktivitesini artırarak kahve çekirdeklerini olumlu yönde etkiler; ancak aşırı mikrodalga güç seviyeleri ve kavurma süreleri, daha uzun süre kavurma nedeniyle olumsuz etki yapmış veya daha yüksek mikrodalga güç seviyelerinde kavurma bozulmalarına neden olmuştur. Hidroksimetilfurfuralın (HMF) belirli bir oranda arttığı ancak süre ve sıcaklığın artmasıyla önemli ölçüde parçalandığı gözlemlenmiştir.

Mikrodalga kavurmanın kavurma süresini kısalttığı, konveksiyonel kavurmaya göre kahvenin lezzetini geliştirdiği ve kahve çekirdeklerinin besin değerini arttırdığı tespit edilmiştir.

Besin değeri yüksek kaliteli kahve çekirdekleri üretmek için mikrodalga kavurma yönteminin kullanılması tavsiye edilmekte ve HMF parçalanma çalışmalarının genişletilmesi ve tanımlanan herhangi bir bileşiğe geçen reaksiyonların takip edilmesi önerilir. Bu yöntemin gerçekçi bir şekilde yüksek kapasiteli üretimlerde uygulanabilmesi için yüksek sıcaklıkta kavrulan kahve çekirdeklerinin fiziksel özelliklerinin ve mikrodalga fırınlardaki en uygun konumularının araştırılması önerilmektedir.

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LIST OF ABBREVIATIONS

C. arabica	: *Coffea Arabica*
5-HMF	: 5-Hydroxymethylfurfural
BI	: Browning index
FDA	: Food and Drug Administration
WHO	: World Health Organisation
FAO	: Food and Agricultural Organisation
ERH	: Equilibrium relative humidity
AOAC	: Association of Official Agricultural Chemists
EPA	: Environmental Protection Agency
HPLC	: High-performance liquid chromatography
TPC	: Total phenolic compounds
TFC	: Total flavonoid content
DPPH	: 2,2-diphenyl-1-picrylhydrazyl
FRAP	: Ferric Reducing Antioxidant Power
5-CQA	: Chlorogenic acid
CA	: Caffeic acid
3-CQA	: Neochlorogenic acid
3.4 di CQA	: 3,4-Dicaffeoylquinic acid
QDA	: The quantitative descriptive analysis
aW	: Water activity
Dm	: Dry matter
$T_{1/2}$	: half shelf time
R^2	: Coefficient of determination
k value	: Activation energy
*E*a	: Reaction rate constant
n.d.	: Indicates not detected.
Fe	: Iron
Na_2CO_3	: Sodium carbonate

1. INTRODUCTION

This research focuses on the world's most consumed beverage, coffee. Coffee consumption is considered one of the world's most popular beverages and has a strong cultural and economic impact (Stiefel et al., 2022). Age, culture, and geography significantly impact coffee consumers' diversity (Santoso et al., 2021). In 2020, around 169 million/60 kg bags of coffee were consumed globally (Stiefel et al., 2022). Although the genus coffee has over a hundred species and subspecies, only the two main types of coffee, Arabica coffee (*Arabica coffea*), accounting for about 64% of total global coffee production worldwide and Robusta coffee (*Robusta coffea*) 36%, which play a significant role in the global market (Campuzano-Duque et al., 2021, Maman et al., 2021). The higher value of Arabica coffee is attributed to its various sensory and aromatic properties. On the other hand, canephora coffee's great extractability is considered to be an important factor that contributes to the market's growth.

Besides being a popular beverage, coffee's health benefits are also intensely interesting (Stiefel et al., 2022). Coffee's health benefits are related to bioactive chemicals from four primary structural groups, including phenolics, alkaloids, and Millard reaction products such as melanoidins and terpenoids (Herawati et al., 2019). Caffeine, chlorogenic acid isomers, caffeic acid, ferulic acid, and trigonelline are some of the most important components known for their antioxidant and radical scavenging properties (Stiefel et al., 2022). Arabica coffee beans (*Coffea arabica*) are the most widespread in most coffee supply chains. Several meta-analyses suggest that regular coffee consumption may effectively reduce the incidence of oxidative stress-related chronic diseases (Shahinfar et al., 2021). Although coffee is known to have adverse effects on various health conditions such as diabetes and heart disease, newer studies have shown that regular consumption of coffee can also help improve various aspects of human

health. Some of these include the development of metabolic disorders, such as Alzheimer's and Parkinson's disease, and liver function (Stiefel et al., 2022).

Excessive consumption of coffee could be harmful to the human body's health due to exposure to some of the toxic components formed in coffee (Farias-Pereira, Park, and Park, 2019). As coffee is being roasted, the Maillard reaction generates several toxic products (Park and Lee, 2021). The most important product of the Maillard reaction in coffee is the 5-Hydroxymethylfurfural (5-HMF) compound (Murkovic and Pichler, 2006). In addition, the thermal decomposition of sugars and carbohydrates produces (5-HMF), which is also produced as a reaction product in the Maillard reaction (Park and Lee, 2021). Recent research indicated that 5-Hydroxymethylfurfural might have carcinogenic potential due to metabolic activation by sulfotransferases; the compound has gained interest (Murkovic and Pichler, 2006). Coffee bean type, roasting temperature, and roasting time impact the levels of 5-HMF in the beverage (Kim et al., 2021).

Food processing is important for maximising food's nutritional value and sensory quality. Roasting is the most important processing method for enhancing colour, flavour, and aroma. By several reactions, such as the Maillard reaction, Strecker degradation, the breakdown of sugars, and the degradation of amino acids, the roasting method can produce aromatic compounds and a new flavour, such as a bitter flavour from burned coffee beans (Santoso et al., 2021). Time and temperature are the two important variables in roasting, producing three roasting degrees by colour standards (light roast, medium roast, and dark roast) (Bolka and Emire, 2020). To produce high-quality coffee, roasters must have the knowledge and abilities to develop physical characteristics, chemical compositions, and biological activity. As a result of different methods, which produce various coffee qualities, the ideal roasting procedure is highly complicated. Therefore, accurate determination of the roasting and cooling processes is required to prevent excessive coffee ripening (Santoso et al., 2021). In addition, coffee has antioxidant effects connected to polyphenols and chlorogenic acid. The main health benefits of coffee,

such as its antioxidant activity and the presence of polyphenolic compounds like ferulic, caffeic, and *n*-coumaric acids, are related to chlorogenic acid, which is found in coffee.

The roasting process begins with heating the coffee beans, evaporating the moisture, and then roasting. In this process, more than two hundred chemical reactions gradually change coffee colour to black (Kelly, 2015). Roasted coffee produces melanoidins, a brown pigment; these substances, along with trigonelline and caffeine, are considered to be potent antioxidants that have many benefits for human health. The roasting stage of natural coffee processing is believed to be crucial for generating changes in bioactive components like polyphenols. The roasting degree greatly impacts the antioxidant and sensory qualities (Bobková et al., 2020); the time required to stop roasting must be known to obtain the desired colour and flavour (Kelly, 2015).

The traditional roasting process was carried out using a drying oven as a single layer of green coffee beans at different temperatures of 180-250 ^{o}C (Mehaya and Mohammad, 2020) for varying lengths of time (Yüksel et al, 2020); for example, the coffee will be roasted at (160 ^{o}C, 180 ^{o}C, and 220 ^{o}C) for 10, 20, 30, and 40 mins hold at each temperature to produce the three primary levels of roasting being light, medium and dark (Somporn et al, 2011), then roasting coffee beans can be classify according to the weight loss of the beans as light (weight loss of 11%), a medium roast (weight loss of 14%) and dark roast (weight loss of 20%) (De Luca, 2016).

Almost always, the conventional method of roasting coffee is applied, which depends on high temperatures for long periods of time, which results in an uneven and non-uniform roasting. Sometimes this method can cause over-roasting which produces an undesirable flavour, and surface burns to the coffee beans. Therefore, the use of the microwave for roasting has many advantages due to the possibility of increasing control over the roasting process in terms of start-up and shutdown times, process speed and energy savings compared with the traditional

roasting process (Bolek and Ozdemir, 2017; Bhattacharya and Basak, 2017). Therefore, microwave roasting has been used to preserve the quality attributes compared to the traditional roasting method (Bolek and Ozdemir, 2017; Zhou, 2016).

Microwave roasting produced a better quality of coffee beans and preserved most of the volatile aromatic compounds. Nebesny et al. (2007) reduced the roasting time and the ultimate temperature of roasted coffee beans by using microwave roasting (Bolek and Ozdemir, 2017). In terms of fatty acid composition, total phenol and antioxidant activity of coffee beans, microwave roasting can be recommended (Saeed Alkaltham et al., 2020).

Several studies have been conducted to study the conventional roasting of coffee beans, and a few studies are looking at the subject of microwave coffee roasting. This study aims to produce the most desirable coffee product with the highest nutritional value by determining the change of physicochemical properties of microwave roasting process conditions by studying their effects on the phenolic profiles, caffeine, hydroxymethylfurfural (HMF), sugar degradation, browning index (BI), and antioxidant capacity of roasted Arabica coffee beans.

2. LITERATURE OVERVIEW

2.1. Green Coffee beans

Coffee is one of the most consumed products worldwide, with over 400 billion cups of coffee consumed worldwide (Gokavi et al, 2021). It is the 2nd most common beverage in the world after water and the second greatest commodity after oil (Butt and Sultan, 2011). Excessive coffee drinking has been thought to have adverse effects on human health due to the stimulating properties of caffeine. But there is much evidence that coffee contains various bioactive components and antioxidants that may positively affect human health (Franca and Oliveira, 2016). Coffee has so many essential components for human health such as phenolic compounds and antioxidant activity (Murthy and Madhava, 2012). Consuming moderate amounts of coffee may reduce the incidence of chronic conditions related to oxidative stress (Shahinfar et al., 2021).

Due to its outstanding antioxidant capacity, coffee's protective effects on human health are mostly attributed to its high concentration of bioactive components, particularly various phenolic compounds and the products formed by the Maillard reaction. Depending on individual dietary preferences, a daily intake of approximately 200 mg of phenolic acids could be achieved (Wu et al., 2022). As a result, the quantity of coffee consumed daily affects the bioavailability of bioactive compounds in coffee beans. Phenolic compounds pass through many stages during the digestion process, as they first pass through the stomach and small intestine, some phenolic compounds are digested and absorbed with low efficiency after drinking coffee. The rest of the granules of undigested phenolic compounds reach the colon, where the remaining nutrients that weren't absorbed could be further digested and used, mainly during fermentation by gut microorganisms. The concentration of phenolic and antioxidant compounds obtained through drinking coffee is affected by many factors. The most important of which is the roasting process, which depends on specific temperatures and time.

Therefore, the degree of roasting and digestion combination could determine the bio-accessibility and bioactivity of phenolic compounds after coffee consumption (Wu et al., 2022).

2.2. Arabica and Robusta coffee beans

The two most common species of coffee are Coffea canephora Pierre ex A. Froehner (Robusta) and *Coffea arabica* (Arabica) (Gokavi et al, 2021). Arabica coffee (*Arabica coffea*) accounts for about 64% of the total global coffee production worldwide and Robusta coffee (*Robusta coffea*) 36% (Maman et al, 2021). Total global coffee production is 10.18 million tons and consumption is 9.7 million tons, making it one of the sectors that provide many jobs, especially for small farmers and workers in rural areas (Gokavi et al, 2021). Comparison between the chemical composition of green Arabica and Robusta coffee beans (g/100 g) after harvest was shown in Table 2.1. (Ghosh and Venkatachalapathy, 2014).

Table.2.1. Chemical composition of green Arabica and Robusta coffee beans (g/100 g) after harvest.

Components		**Average content (g/100g)**	
		Arabica	Robusta
Macronutrients	Soluble carbohydrates	9–12.5	6–11.5
	Protein	9.8	9.5
	Lipids	16.5	10
	Water	8-12	8-12
	Fiber	46-53	35-44
Micronutrients Acids and Phenols	Minerals	4.2	4.4
	Quinin acids	0.4	0.4
	Chlorogenic acid	6.5	10
	Nonvolatile aplitic acids	2-2.9	1.3-2.2
Other bioactive compounds	Bioactive amines	0.011	0.002
	Caffeine	0.8-1.4	1.7-4.0
	Kahweol	0.7-1.1	n.d
	Trigonelline	0.6-1.2	0.3-0.9

* n.d. indicates not detected.

Over 60% of the world's coffee supply is made from Arabica coffee beans, which customers widely appreciate for their superior smooth, mild, and rich flavour. Compared to Robusta beans, Arabica coffee beans are often sold for at least twice as much (Mihailova et al., 2022).

Coffee has a pleasant taste, aroma, antibacterial activity, and antioxidant activity of coffee, which is an important compound in cancer and coronary disease prevention (Nebesny and Budryn, 2003). The origin cultivation area of *C. arabica* is Ethiopia. More than 80 developing countries export it to all industrialised countries. The botanical variety, topographical and weather conditions, and the care taken during growing, harvesting, processing, storage, export preparation, and transport form a combination to determine the quality of coffee. Recently, natural bioactive components in coffee have become important for studies according to functional and healthy effects and an important business worldwide. For example, Starbucks is the largest chain of cafes worldwide, receiving more than 100 million dollars annually through its business receipts (Shang et al, 2017).

Different factors can influence the final quality of coffee. For example, coffee is affected by 40% at pre-harvest, 40% at post-harvest practices, and 20% at secondary/export processing. Therefore, it is important to study the physical properties of roasted coffee beans since most physical changes happen during the roasting process to determine the final quality of coffee (Oromia, 2016).

2.3. Compounds in Roasted Coffee Beans

During the roasting process, many chemical reactions occur, which produce several physical changes and form substances responsible for the sensory qualities of coffee beverages.

The difference is apparent between the composition of different coffee bean species in terms of analytical methods, origins, or roasting degrees, chlorogenic acids, the degradation of polysaccharides, trigonelline, and oligosaccharides (especially sucrose), which are commonly noticed in the green coffee beans and decreasing dramatically, these components produce the characteristic aroma, flavour, taste, and colour of the roasted coffee bean.

During roasting, many compounds form, such as carbohydrates, protein fragments, caffeine, low-molecular-weight acids, lipids, trigonelline, unknown molecules called melanoidins, and more than 900 volatile compounds.

A comparison between the compositions of green and roasted coffee beans is shown in Table 2.2 (Wei and Tanokura, 2015).

Table.2.2. Chemical Compounds of Green Coffee Beans and Roasted Coffee Beans

	Arabica		**Robusta**	
Components	Green Coffee Bean	Roasted Coffee Bean	Green Coffee Bean	Roasted Coffee Bean
Polysaccharides	50.0-55.0	24.0-39.0	37.0-47.0	-
Oligosaccharides	6.0-8.0	0-3.5	5.0-7.0	0-3.5
Lipids	12.0-18.0	14.5-20.0	9.0-13.0	11.0-16.0
Free amino acids	2.0	0	2.0	0
Proteins	11.0-13.0	13.0-15.0	11.0-13.0	13.0-15.0
Chlorogenic acids	5.5-8.0	1.2-2.3	7.0-10.0	3.9-4.6
Caffeine	0.9-1.2	0-1.0	1.6-2.4	0-2.0
Trigonelline	1.0-1.2	0.5-1.0	0.6-0.8	0.3-0.6
Fatty acids	1.5-2.0	1.0-1.5	1.5-2.0	1.0-1.5
Minerals	3.0-4.2	3.5-4.5	4.0-4.5	4.6-5.6
Melanoidins	-	16.0-17.0	-	16.0-17.0

2.4. Roasting Process

The roasting process is important with most of the physical and chemical changes happening in coffee beans, which are researched to understand (Illy and Viani, 2005). Although coffee roasting is a thermal process of green coffee beans (Kelly, 2015), this process depends on time and temperature (Mehaya and Mohammad, 2020), which produces coffee's distinctive colour and flavour. The roasting process begins with heating the coffee beans, evaporating the moisture, and then roasting. In this process, more than two hundred chemical reactions cause the changes in coffee colour gradually to black. In this process, the time required to stop roasting must be known to obtain the desired colour and flavour (Kelly, 2015).

Mehaya and Mohammad (2020) used a drying oven to roast green coffee beans traditionally by using a single layer at various temperatures (180-250 ^{o}C) for varying lengths of time (Yüksel et al., 2020). For example, the coffee roasted at (160 ^{o}C, 180 ^{o}C and 220 ^{o}C) for 10, 20, 30, and 40 min held at each temperature to produce the three primary levels of roasting being light, medium and dark (Somporn et al, 2011) after roasting coffee beans can be classified according to the weight loss of the beans as light (weight loss of 11%), medium (weight loss of 14%) and dark roast (weight loss of 20%) (De Luca, 2016).

2.5. The roasting degrees

The roasting degree is an important factor in producing high-quality coffee. The roasting time of the coffee determines the colour of the coffee beans. The longer the time, the darker the colour of the coffee beans. The degree of roasting can be measured by judging the colour of the bean in many ways, such as through the human eye, the weight loss of water after roasting, or by the colourimeter. The next table (Table 2.3) shows that the higher the degree of roasting, the lower the L^* value (for lightness in the CIELAB colour space) (Wei and Tanokura, 2015).

Table.2.3. Roasting Degree of Coffee Bean

Roasting Degree	Roasting Name	*L** value[a]	Weight Loss (% of Green Coffee Bean)
Light	Light roast	30.2	10.0-14.0
	Cinnamon roast	27.3	
	Medium roast	24.2	
Medium	High roast	21.5	14.0-17.0
	City roast	18.5	
	Full city roast	16.5	17.0-21.0
Dark	French roast	15.5	
	Italian roast	14.2	>21.0

[a]The *L** value ranges from 0 (black) to 100 (white)

2.6. Microwave Heating

Recently, the spread of microwave heating around the world has become so widespread that it has become one of the most common methods used in homes to heat food, as microwave heating is known for the safety of its operation and the reduction of food loss of heat-labile nutrition (e.g., phenols, vitamins C and B, antioxidants and carotenoids) (Puligundla et al, 2013).

The microwave has been used since 1940 in food industries as a heat source, as it was used to heat, reheat, dry, free drying, cook, melt, pasteurise, and sterilise food (Oliveira and Franca, 2002). Microwaves are a part of electromagnetic waves, which have a frequency range between 300 MHz and 300 GHz corresponding to wavelengths from 1 mm to 1 m (Puligundla et al, 2013).

Microwave frequencies of 915 MHz and 2.45 GHz can be utilised for industrial, scientific, and medical applications. Compared to conventional heating,

microwave heating consumes less energy, less processing time (Puligundla et al, 2013), and space savings (Oliveira and Franca, 2002). In addition, it reduces the amount of temperature difference between the surface and the food interior. In conventional treatments, heat is transferred from the surface of food by convection and conduction, which causes a temperature gradient between outside and inside food which takes a long time. However, in microwave heating, heat enters the food's interior in a short time when the microwave penetrates through it (Puligundla et al, 2013).

Microwave functioning is based on water, primarily because water is present in a substantial percentage of food products. The microwave interacts with polar water molecules and charged ions in food via strong dipole rotation of water molecules.

The microwave works to constantly rotate the particles of polar water molecules in the food and couple with an electromagnetic field. As a result of this rotational movement of the water heat is generated (Puligundla et al., 2013), also heat can be generated in the microwave by changing the electrical and magnetic fields in the product (Oliveira and Franca, 2002), through the ionic migration which depends on the interaction between positive and negative ions of dissolved salts in food with the electric (Puligundla et al., 2013).

Microwave heating is used for most semi-cooked foods, such as baked food products and pre-cooked meat patties, which need baking or oven roasting before consumption (Puligundla et al., 2013).

According to previous studies, it was concluded that the use of the microwave heating method, in addition to reducing the time and energy consumed for the cooking or roasting process, plays an important role in pasteurisation or sterilisation in eliminating pathogenic microorganisms and minimising the loss of important nutrients (Puligundla et al, 2013). But many previous studies also showed that microwave heating could result in an uneven temperature distribution in food material. As it is one of the greatest risks that can cause significant damage

to the final product, which harms the sensory quality and the possibility of microbes remaining in cold spots therefore, a rotating glass disk, and a motor were used that were placed in microwave ovens to allow the electromagnetic waves to be distributed almost evenly over the dimensions of the food products (Yang et al, 2021).

2.7. Microwave roasting

The traditional method of roasting coffee is usually used, which depends on high temperatures for long times, which results in an uneven and non-uniform roasting. Sometimes this method can cause over-roasting, producing an undesirable flavour, and surface burns to the coffee beans. Therefore, the use of microwaves for roasting has numerous advantages due to the possibility of increasing control over the roasting process in terms of start-up and shut down times, process speed and energy savings compared with the traditional roasting processes (Bolek and Ozdemir, 2017; Bhattacharya and Basak, 2017). Therefore, microwave roasting has been used to preserve the quality attributes compared to the traditional roasting method (Bolek and Ozdemir, 2017; Zhou, 2016).

During the roasting process, many changes happen in green coffee beans' physical and chemical properties. These changes affect the phenolic acid, carbohydrate, moisture, and protein. Consequently, roasting can cause caramelization, Maillard reactions, oxidation, pyrolysis, and the formation of colour and aroma, which produces the desired product by consumers (Liu, 2016; Mehaya and Mohammad, 2020).

2.8. Sugar degradation during roasting

The conversion of sugars during the roasting process contributes to the formation of the flavour compounds of the coffee. The two main polysaccharides in coffee beans are arabinogalactan type II and galactomannans. The conversion

reactions of polysaccharides occurring during coffee bean roasting are shown in Figure 2.1 that modified from Oosterveld (2003).

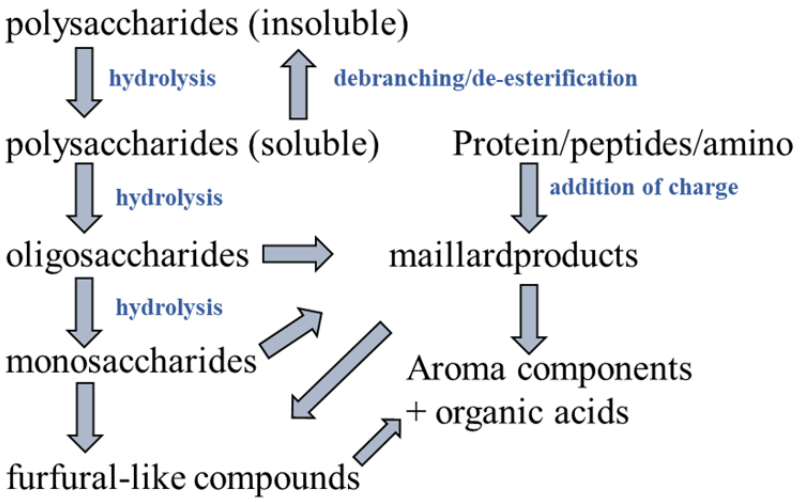

Figure 2.1. Processes of polysaccharide conversion that occur during coffee bean roasting process

The roasting process opens the outer wall of the coffee beans and then begins the degradation reactions of polysaccharides (Oosterveld, 2003).

Sucrose is the most abundant carbohydrate in green coffee beans, which is degraded quickly in the first step of the roasting process and produces aliphatic acids (acetic, lactic, glycolic, and formic), as sucrose is considered a leading source of aliphatic acids during the roasting process. Furthermore, sucrose plays an important role as an aroma precursor during roasting. Acid formation in roasted coffee beans mainly results from the thermal treatment of carbohydrates. The main polysaccharide responsible for these reactions is sucrose, the polysaccharide fraction, particularly arabinogalactan, contributes (Wei and Tanokura, 2015).

In addition to the formation of aliphatic acids, as a result of carbohydrates degradation, there is an important compound produced by the breakdown of carbohydrates during the roasting process, which is 5-Hydroxymethylfurfural (HMF). The formation of HMF depends on many factors like water activity, time, temperature, and sugar used. Therefore, it has been studied as an indicator for monitoring food storage conditions, an indicator of heat damage, and a chemical index for ensuring adequate heat processing.

HMF is produced by the division of sucrose to monomeric carbohydrates and then to glyceraldehyde and methylglyoxal during the coffee roasting process. As a summary of what was mentioned previously, sucrose is converted into aromatic compounds and aliphatic acids (acetic acid, lactic acid, glycolic acid, and formic acid) during coffee roasting. Water-soluble polysaccharides are produced after roasting, which helps to save the volatile substances and improve the viscosity of the coffee brew, produces the texture of coffee, and which is called "body" (creamy sensation) (Wei and Tanokura, 2015).

2.9. The formation of furfural and 5-hydroxymethyl-2-furfural during roasting

Maillard reaction is one of the most important reactions occurring because of heat treatment for food products. In addition to adding flavour, aroma, and smell to food products, the Maillard Reaction also generates several heat-treatment contaminants, such as furfural and 5-HMF, which may be dangerous to human health (Basaran, B. et al., 2022). The formation and breakdown of furfural and HMF from xylose and glucose are shown in Figure 2.2. that modified from Almeida et al. (2009).

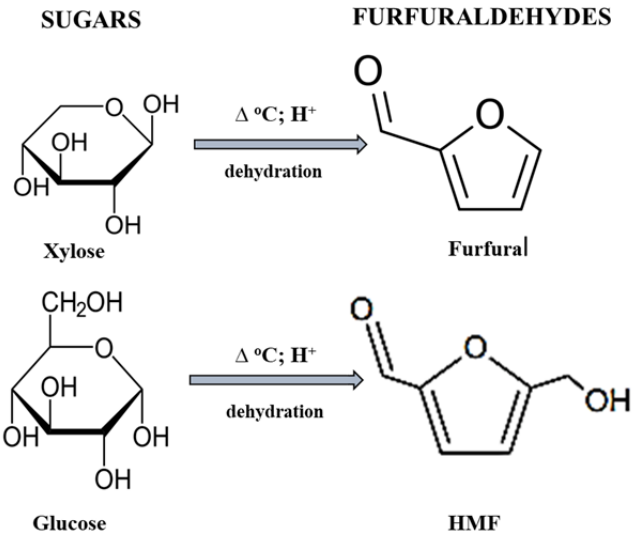

Figure 2.2. HMF and furfural formation and breakdown from xylose and glucose, respectively

Around 1500 organic components have been found in roasted coffee. One of them is furfural, an undesirable toxic compound generated by Maillard reactions while roasting coffee. According to recent research, consuming furfural may increase the risk of developing skin cancer and liver cancer. Furthermore, according to the World Health Organisation (WHO), furfural may have cytotoxic effects. Furthermore, an individual with an average body weight of 70 kg should take 0.2 mg of furfural orally every day, according to the Environmental Protection Agency (EPA). Additionally, because furfural affects the organoleptic properties of foods, the amount of the substance is strictly controlled in food products (Piñeiro-García et al., 2020).

Every food containing free carbohydrates has HMF, the concentration of it depends on the temperature and/or duration of production or storage (Murkovic, 2007). HMF occurs as a result of overheating or storage. Many pathways can form it as acid-catalysed degradation of reducing sugars or via the Maillard reaction, for example, dehydration of carbohydrates e. g. fructose to HMF through a sequence,

fructofuranose ring intact; this compound is formed with and without acid catalysis. A necessary fructofuranosyl cation is produced directly by the hydrolysis of sucrose, which reacts to produce HMF. The Millard reaction, which creates Amadori molecules in the beginning and HMF in the later stages of the reaction, is another mechanism (Murkovic, 2006). Another pathway is the cleavage of a-dicarbonyl-compounds and recombination of methylglyoxal with glyceraldehyde (Figure 2.3) that modified from Murkovic (2007).

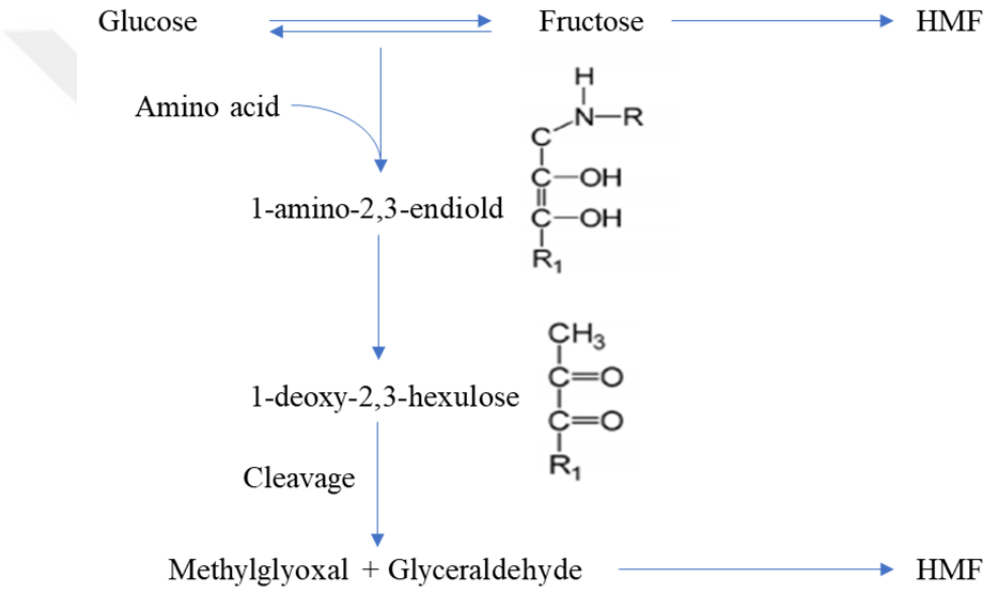

Figure 2.3. Formation process of HMF from hexoses

The importance of HMF began to appear through the carcinogenic potential of the metabolic activation by sulfotransferases (Murkovic, 2006). The previous studies showed that HMF is not a highly dangerous compound (Murkovic, 2007) despite its relatively low toxicity. However, recent results on the metabolic activation of HMF show a genotoxic potential of this compound (Murkovic, 2006). The risks and toxicity of this compound have not yet been clarified (Murkovic, 2006; Murkovic, 2007). High concentration will cause cytotoxicity and cause

irritation to the eyes, the upper respiratory tract, the skin, and the mucous membrane. But, if the uptake of HMF is between 0.5 – 1 mg/kg body weight, it will not cause any health risk. The most food that has a high amount of HMF is dried fruits with a high sugar content, the lowest HMF amount observed in figs (1 mg/kg) and the highest observed in plums (2200 mg/kg), and the lowest concentrations found in meat and meat products (below 0.9 mg/kg). In addition, according to a human study with seven healthy volunteers, 0.75% of the ingested HMF was excreted within six hours of the urine excretion (Murkovic, 2006).

The roasting process produces coffee's taste, aroma, and flavour due to chemical reactions in which many substances are formed. One of the constituent substances is HMF. In coffee, the concentration of HMF depends on sucrose as the dominant carbohydrate of green coffee and alanine being the dominant amino acid (Murkovic, 2007). The content of HMF in all types of coffee ranges from 300 to 2900 mg/kg, 5 to 420 mg/L (Murkovic, 2006). The commercial roasted coffee concentration ranges between 0.3-1.9 mg/g. 5-hydroxymethylfurfural (HMF) formation happens during the roasting of coffee. The maximum concentration of HMF occurs at 240 ^{o}C after 3 minutes with fast decreases during 10 minutes with a higher degree of roasting. The highest concentration of HMF in coffee (909 μg/g) was obtained after 3 min, and commercially roasted coffee contains less HMF than expected. (Murkovic, 2007). HMF has been analysed by RP chromatography with methanol-water mixtures as eluent with UV detection at 280 nm (Murkovic, 2006).

The amounts of furfural and 5-HMF formation in coffee are affected by the type of coffee bean, type of sugar, water activity, pH, roasting temperature, and time (Toker et al., 2013). However, until now, there is no study showing if coffee is beneficial or harmful for human health because of the isolated substances it includes, except the effects of xanthine alkaloids (caffeine, theobromine) (Murkovic, 2007).

2.10. Caffeine

Despite the advantages of caffeine consumption, there are many disadvantages, such as headaches, anxiety, restlessness, and nausea. These symptoms have appeared due to excessive caffeine intake; it will be mild and temporary when caffeine intake stops suddenly. In addition, some studies have shown an increased risk of cardiovascular disease and hypertension. But if caffeine consumption is less than 400 mg/day, it will not cause any health effects.

According to some studies that indicate the positive effect of caffeine consumption on weight loss, reducing the risk of developing Parkinson's disease, improving glucose tolerance, thus reducing the incidence of type II diabetes, and also reducing the chance of developing cancer (Mitchell et al., 2014), relaxation of bronchial muscle, gastric acid secretion, stimulation of the central nervous system (Belayet al, 2008). 85% of the U.S. population consumes at least one caffeinated beverage daily, equaling 165 ± 1 mg for all ages combined. Most of this caffeine comes from beverages. Beverages are the most products containing caffeine like coffee, tea, carbonated soft drinks, and energy drinks. The primary source of caffeine is carbonated soft drinks compared to coffee in adults.

According to the FDA, in 2012, caffeine intake without health effects shouldn't be more than 400 mg/day for healthy adults, with special recommendations for pregnant women (<300 mg/day) and children. However, there are also some exceptions to subgroups like military personnel, whose caffeine intake can be 1000 mg/day and doses of 600 mg/day as a consideration of physical demands and sleep deprivation, even though it is hard to take this amount of caffeine from only beverages (Mitchell et al., 2014).

Coffee has caffeine compared with other beverages, e.g., tea, energy drinks, soft drinks, and solid food products such as chocolate, but this type of food like chocolate, sweets, and cocoa contains a small amount of caffeine. More than 400 million cups of coffee daily were consumed globally, which is the main resource of caffeine in the adult diet.

Caffeine is a natural alkaloid in more than 60 plants, including coffee beans. It is a bioactive compound that has obvious effects in stimulating the central nervous system, and it has a good impact on long-term memory. It also has a physiological function of reducing lactic acid accumulation after exercise improving endurance performance during exercise. The concentration of caffeine in the plants depends on many factors such as species (Arabica beans contain about 1.0–1.2%, whereas the caffeine content in Robusta beans is about 1.6–2.5%) (De-Marco et al, 2018), roasting degree (light, medium or dark), the type of product, agronomic and processing, variations in raw materials, agricultural practices, duration and conditions of storage. The variation of caffeine content according to the preparation process of coffee is given in Table 2.4 (Gonzalez de Mejia and Ramirez-Mares, 2014). This discussed the low possibility of drinking two cups of coffee with the same chemical composition even though both have coffee from the same store.

Table.2.4. Variation of caffeine content

Product	**Serving size (fl. Oz)**	**Caffeine in one serving (mg)**
Coffee		
Regular drip or percolated	8	95-330
Brewed or percolated, decaffeinated	8	3-12
Instant, prepared from powder	8	30-70
Espresso	1	50-150

Coffee's pleasant taste and aroma depend on more than 1000 compounds besides caffeine. There are many active compounds in coffee, e.g. methylxanthines (caffeine, theophylline, theobromine), flavonoids (anthocyanins, catechins), diterpene alcohols (kahweol, cafestol), tocopherols, and melanoidins, which

produce a long chain of chemical transformations occur from the coffee bean until the coffee cup to create the desired final product (Gonzalez de Mejia and Ramirez-Mares, 2014). If caffeine consumption is (e.g., (400 mg/day) it will not cause any health effects (Mitchell et al, 2014).

The amount of caffeine in coffee determines the quality of coffee, as by increasing the amount of caffeine in the coffee, you want to improve its quality. Several physical and chemical methods have been developed to calculate the caffeine content, and these analyses are considered a tool for assessing the quality of coffee. UV/vis spectrophotometer is the easiest, cheap, and available in most laboratories to determine the caffeine in coffee and other beverages (Belayet al, 2008).

2.11. Phenolic compounds and antioxidant activity in coffee

Polyphenols are the most abundant antioxidants in the diet. Their daily intake is the highest component consumed compared with all other phytochemicals and known dietary antioxidants, which could be more than one g/d. For example, its consumption is 10 times higher than vitamin C and 100 times higher than vitamin E and carotenoids, polyphenols have a big effect on human health (Scalbert et al., 2005) (Priftis et al., 2015), as when consuming plant foods, the absorbed polyphenols stimulate many vital activities that positively impact human health (Priftis et al., 2015), and helps in prevention of neurodegenerative diseases, degenerative diseases, particularly cardiovascular diseases, cancers, osteoporosis, and diabetes mellitus.

The studies of polyphenols, flavonoids, and the antioxidant activity of polyphenols and their effects on disease prevention started after 1995. The experts before that focused on studies on the antioxidants produced from vitamins, minerals, and carotenoids. The reason for delayed studies about polyphenols is the variety and complexity of their chemical structures (Scalbert et al., 2005).

A coffee bean contains many natural biological substances such as polyphenols, melanoidins, and caffeine, which play an important role in decreasing the risk of cardiovascular diseases, reducing brain injury, antioxidative, anti-mutagenic, anti-carcinogenic activity, and improving the coffee's bitterness (Shang et al., 2017).

Coffee is one of the most consumed plant products in the world, which provides the consumer with polyphenols, and its great consumption is due to its pleasure and pleasant aroma. In the past, it was consumed because of its taste, quality, and health effects centred on the benefits of caffeine, the most studied ingredient. However, with the progress of science and the development of studies on coffee, more beneficial components for human health have been discovered in coffee, such as antioxidants, which have a great health value as it contains primarily polyphenols, and the most polyphenols present are chlorogenic acid (CGA). The intense antioxidant activity in coffee is due to the presence of polyphenols.

The different content and composition of polyphenols and the different molecules produced during roasting are the reasons for the differences in antioxidant activity between different types of coffee. Antioxidant activity depends on the roasting time, at which point there can be an increase or decrease in antioxidant activity. When coffee is roasted as a result of many reactions, such as Maillard interactions, the polyphenol profile of the beans is changed, specially affecting CGA, which produces new compounds from the products of this hydrolysis, which maybe will change the overall antioxidant capacity of the coffee beans, leading to their hydrolysis, and accordingly, the antioxidant activity in the coffee beans is changed (Priftis et al., 2015).

Alkaltham et al. (2020) improved that according to coffee beans' antioxidant capacity, total phenol content, and fatty acid composition, microwave roasting can be recommended. The coffee samples' bioactive properties, phenolic components, and fatty acids showed significant differences. Linolenic, linoleic,

arachidonic, and oleic acids increased after roasting. Antioxidant activity, moisture, and total phenol values of the green and roasted coffee samples all significantly decreased. Coffee beans roasted in a microwave contained less than 3,4-dihydroxybenzoic acid, gallic acid, 1,2-dihydroxybenzene, resveratrol, p-coumaric acid, naringenin and kaempferol than coffee roasted in an oven. In general, it was discovered that coffee roasted in both a microwave and an oven contained higher amounts of phenolic compounds than the control group. Except for linolenic acid, the amounts of fatty acids in coffee beans roasted in microwave and oven systems differed significantly from the control.

Salamatullah et al. (2022) showed that TFC (Total Flavonoid Content), TPC, DPPH scavenging capacity, mineral content, and colour indicate moderate microwave roasting conditions favourable for coffee beans. Furthermore, it was noticed that the TPC, TFC, and antioxidant activity of the coffee bean extract was enhanced by increasing microwave power and roasting length of time; however, extreme microwave power levels and roasting durations had a negative impact because of roasting for a longer time and at higher microwave power levels caused their degradation.

3. MATERIALS AND METHODS

3.1. Material

In this study, 10 kg of green Arabica coffee beans were purchased from the Turkish market in Adana, as shown in Figure 3.1. Samples of green coffee beans were roasted by microwave in the laboratory. A professional microwave oven (Berghof MWS-2, Germany) was used during the roasting process. The system can maintain time, temperature, and power.

Water activity, dry matter, colour values, antioxidant activity, TPC, phenolic compounds, caffeine, sugar, furfural, HMF, and the browning index were determined in ground coffee samples.

Figure 3.1. Green Arabica coffee beans

3.2. Method

3.2.1. Preparing sample

Arabica coffee beans were divided into ten portions (~1kg) inside reinforced plastic bags. After preparing the samples inside the bags, the bags were vacuum sealed using a vacuum sealer (Propack HVC-410F/2A, Turkey), as shown in Figure 3.2 to be preserved during the experiment without being exposed to physical or chemical changes due to environmental factors.

Figure 3.2. Green coffee beans sealed under vacuum conditions bags

3.2.2. Microwave roasting

Coffee beans were roasted using a speedwave microwave (1000W, Berghof MWS-2, Germany), as shown in Figure 3.3, which was determined with a patented infrared thermometer to measure the temperatures of the samples. In the roasting process three factors were controlled; power, temperature, and time. The

preliminary studies indicated that up to 75% of the microwave power could be used without damaging coffee beans. Therefore, the microwave power was arranged between 0-75% (0 – 750 W) to maintain the roasting temperature. According to preliminary studies and literature overview, three roasting temperatures were decided to work on which are 220 ^{o}C for 20 minutes, and the samples were collected every 2 minutes, 200 ^{o}C for 40 minutes and the samples were collected every 4 minutes, and finally 180 ^{o}C for 120 minutes, and samples were collected every 12 minutes. Samples were roasted inside beakers to make the microwaves reach all the beans. After that samples were ground by a grinding machine (Bosch, Germany), and the ground coffee beans were kept at 2 ^{o}C degrees till further analysis. All the analyses were conducted in 10 days to eliminate the storage effect.

Figure 3.3. Microwave Digestion System speedwave (Berghof MWS-2, Germany)

3.2.3. Analysis of colour measurement

Minolta (CR-400, Japan) was used for colour (CIE L*, a*, b*) analysis. 50g of coffee was transferred into the glass optical cell and analysed. The CIELAB colour system was used to evaluate the results. L* stands for brightness (0: black; 100: white), a* for red/green value ((+): red; (-): green), and b* for yellow/blue value ((+): yellow; (-): blue) in this system. In addition, the Hue*, C^* and ΔE^* colour values were calculated by using the following formulas (Ağçam and Akyıldız, 2014):

$$Hue^* = \arctan\left(\frac{b^*}{a^*}\right)$$

$$C^* = \sqrt{(a^*) + (b^*)}$$

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

3.2.4. Analysis of dry matter content

Each sample's weight was recorded at the beginning and end of the drying process, and weight loss was calculated according to the AOAC method 934.06. The moisture level of the dried samples was measured by using an oven (MMM, Venticell, Germany) at 105 ^{o}C until the constant weight (AOAC, 1990).

3.2.5. Analysis of water activity

Water activities of coffee powder were carried out at 25 ± 0.2 ^{o}C using the specially designed instrument (Novasina, Sweeden). The instrument is sensitive to the change in equilibrium relative humidity (ERH) around the sample, not directly to moisture content.

3.2.6. Analysis of sugar components

Sugar components of coffee samples were analysed by HPLC-PDA/RID system (Sturm et al, 2003). For this, 1g of coffee sample was transferred to a 15 mL centrifuge tube and after adding 9 mL of ultrapure water, mixing was done in

vortex for 1 minute. The sample extracts were centrifuged at 6000 rpm, 4 ^{o}C for 10 minutes (Hettich, Germany). Finally, the resulting clear fractions were injected into the HPLC-PDA/RID device (Shimadzu, Japan) after passing through a 0.45 µm nylon membrane filter. The chromatography conditions to be applied during the analysis are given below.

Chromatography conditions;

- Column: Core gel Ion 300 (Concise, USA)
- Column temperature: 30 ^{o}C
- Mobile phase: 5mM H2SO4, isocratic flow
- Mobile phase flow: 0.4 mL/min
- Injection volume: 20 µL
- Elution time: 55 min
- Detector: PDA/RID (PDA wavelength: 210 and 244 nm)

The identification of sugar peaks was carried out using certified standard materials. The retention time and spectra of the peaks (Appendix 3) was defined by comparing them with the standard. For the concentration calculation of the peaks, 5 different concentrations of standard substances were injected into the system, and the obtained calibration curves were used for this purpose (Sturm et al, 2003).

3.2.7. Analysis of HMF and furfural (F)

In order to improve extraction of the HMF and furfural, 2 g ground coffee was mixed with 20 mL pure water and kept for one hour for equilibrium at room temperature. After incubation, the samples were mixed, and centrifuged at 6000 rpm, 4 ^{o}C for 10 min (Hettich, Germany). First, the supernatant was used for the extraction of furfural compounds. And then, 5 mL of the supernatant solution was

exposed to the liquid phase extraction method suggested by Gokmen and Acar (1996).

The collected extract enriched with furfurals by following the procedures mentioned above was injected into the HPLC system (Shimadzu, Japan) that consists of LC (20AT), PDA detector (SPD-20A), auto-sampler (SIL-20A), degasser (DGU-20A5R), and column oven (CTO-10AS). Twenty µL extract was injected into C18-column (ACE, 4.6×250mm, 5 µm) at 30 ^{o}C. Ultra-pure water/methanol/acetic acid (79/20/1, v/v/v) solution was used as the mobile phase with the isocratic flow at 0.5 mL/min. PDA detector was set at 275 nm and 285 nm (Appendix 2) during analysis for furfural and HMF, respectively. The furfural peaks were identified by comparing the UV spectrums and retention times of HMF and furfural standards. Five different concentrations of the standards were injected, and after that the obtained calibration curve was used to quantify the concentration of HMF and furfural in the samples.

3.2.8. Analysis of browning index (BI)

In Teflon tubes, 0.2 g of each variety of coffee was combined with 5 mL ethyl alcohol (95%) and centrifuged (4000 rpm, 10 min, 4 ^{o}C). After that the supernatant was passed through a 0.45 µm Teflon membrane filter. The absorbance of the supernatant was measured at 420 nm in a spectrophotometer (Perkin Elmer Lambda 25-UV/VIS, USA) (Ağçam and Akyıldız, 2014; Meydav et al., 1977).

3.2.9. Preparation of dilution/stock solution of phenolic and caffeine

In order to obtain an extract rich in bioactive compounds, 1 g coffee was mixed with 25 ml methanol solution (80%), and then the mix was vortexed and incubated for 10 min in the dark. After that, the samples were sonicated in an ultrasonic water bath (Bandelin Sonerex, Germany) for 10 min at room temperature. The sample extracts were centrifuged at 6000 rpm, 4 °C for 10 minutes (Hettich, Germany). Thus, the main stock extracts were obtained for the

analysis of total phenolic contents (TPC), antioxidant activity (FRAP and DPPH), caffeine and individual phenolic compounds.

3.2.9.1. Analysis of total phenolic content (TPC)

1 g of coffee was mixed with 25 ml methanol of 80% in a 50ml centrifuge tube, then mixed by vortex and incubated for 10 min in the dark. After that the tubes were put in an ultrasound water bath for 10 min. The sample extracts were centrifuged at 6000 rpm, 4 ^{o}C for 10 minutes (Hettich, Germany). 1000 µL from the clear fraction with 3000 µL ultra-pure water was mixed. 100 µL from Na_2CO_3 and Folin was added. The absorbance of the solution was recorded at 510 nm. The result was represented as mg catechin equivalents per gram dried extract (mg CE/g DW).

3.2.9.2. Analysis of phenolic compounds and caffeine

The primary stock extract was passed through a 0.45 µm Teflon membrane filter, and then the filtered extract was injected into the HPLC-PDA system (Ağçam et al, 2014).

A Shimadzu LC-20AT (Japan) system was used for HPLC analyses, which included a quaternary pump, a column temperature control oven (CTO-10AS), an autosampler unit (SIL-20A), a degasser module (DGU-20A5), and a photodiode array detector (SPD-M20A). The C18 XTerra (Waters, 4.6 250 mm) column was injected with 20 µL of supernatant. The flow rate was 1 mL/min, and the column was kept at 30 ^{o}C. The wavelengths of the photodiode array detector were set to 280 and 320 nm. For mobile phases, A and B; 5% formic acid (A) and Phase A: acetonitrile solutions (CAN), 20:80 (B) was utilised. The optimal gradient elution, according to preliminary studies, was:0 min: 100% A; 10 min: 95% A + 5% B; 25 min: 90% A + 10%; 55 min: 80%A + 20%B; 70min: 55%A + 45%B; 90min: 100%B; 95 min: 100% A.

The phenolic compounds were identified by comparing the certified standards' UV–visible spectra and retention times. The phenolic compounds' quantification was conducted using a standard external method at 280 and 320 nm (Appendix 1). Commercial standards of the concentrations commonly present in coffee were used to create calibration curves (Ağçam and Akyıldız, 2014).

3.2.10. Analysis of antioxidant activity

3.2.10.1. DPPH Scavenging

The DPPH (2,2-diphenyl-1-picryl-hydrazyl-hydrate) free radical-scavenging activity determination of coffee was performed using Klimczak et al. (2007) procedure with slight modifications. The main stock extract of samples prepared earlier was used. The absorbance of the samples was measured using a spectrophotometer at 515 nm after a 60-minute incubation period in the dark. The antioxidant activity was expressed as mg Trolox equivalents per 100 g dry matter (mg TE/100g DM) (Ağçam and Akyıldız, 2014).

3.2.10.2. Ferric reducing antioxidant power (FRAP)

This method is based on the ability of the sample to reduce Fe^{+3} to Fe^{+2} ions. Briefly 100 µL of sample extract and 2.9 mL of working FRAP reagent were added. After incubation for 1 hour at room temperature, the absorbance was measured at 593. The FRAP activity was expressed as the mg Trolox equivalents per 100 g dry matter (mg TE/100g DM).

3.2.11. Sensory analysis

In the present study, to obtain coffee beans with different roasting degrees under microwave conditions, a temperature of 200 ^{o}C was chosen. For the light, medium and dark coffee beans, green coffee was roasted with a microwave at 200 ^{o}C for 2, 16 and 40 min, respectively. The roasting degree of coffee beans was classified according to the L* value. Following the grinding process, these coffee

samples were used for sensory evaluation. On the other hand, the traditionally roasted and ground coffee was purchased from the local market in Adana, Turkey, in a light, medium and dark roasting degree.

Sample preparation: 7 g of ground coffee was gently mixed for 5 minutes with 100 mL of boiling water, and this extract was cooled to 55 ^{o}C before any further analysis. This coffee beverage was served to the panel of the sensory evaluation committee, which consisted of 16 person (10 women and 6 men). Sensations of taste and aroma were rated on a scale of 1 (extremely dislike) to 10 (Extremely like). Each sensory determinant was given a significance index. The acceptability of the brew was calculated as the sum of the sensory attribute scores multiplied by the significance indices of those scores.

3.2.12. Kinetic Study

In the present study, the bioactive and sugar compounds degradation at constant roasting temperature was expressed by the following differential equation:

$$\frac{d[C]}{dt} = -k[C]^n \quad (1)$$

where C is the concentration of bioactive and sugar compounds (mg or g/100g dm), k is the reaction rate constant, t is time (min) and n is reaction order. When Eq.1 is solved for zero and first reaction order, the following equations can be obtained, respectively:

$$[C] = [C]_0 - kt \quad (2)$$

$$[C] = [C]_0 e^{-kt} \quad (3)$$

where, $[C]_o$ is the initial concentration of the compound, and k is the reaction rate constant ("mg or g/100gdm.min" or "1/min"). The roasting time requirement for the bioactive and sugar compounds to lose half of their initial concentrations can also be calculated ($t_{1/2}$, min). To achieve that, Eq. 4 and Eq. 5 can be used for zero and first reaction order, respectively:

$$t_{\frac{1}{2}} = \frac{[C]_0}{2k} \quad (4)$$

$$t_{1/2} = \frac{0.693}{k} \quad (5)$$

On the other hand, the caramelization reaction and Maillard reaction pathways were used to express mathematically the formation of HMF and furfural (F) during roasting at a constant temperature. While caramelization is one of the reaction chains responsible for formation of HMF and furfural by basic sugar degradation pathways. Maillard reaction pathways are responsible to form them from the reaction chains between amino acids and reducing sugars. In the medium containing reducing sugars and amino acids like coffee beans, both caramelization and Maillard reaction chains are responsible from formation of HMF and Furfural (Figure 3.4). Therefore, both of them must be taken into consideration for expressing kinetic models.

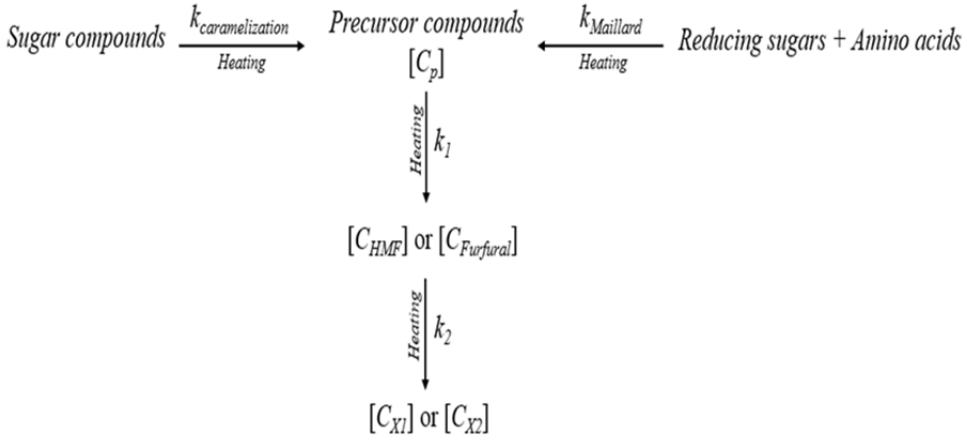

Figure 3.4. The main precursor compounds in coffee beans for the formation of HMF and furfural

The experimental results showed that HMF and furfural are not stable during the roasting process due to low water activity and high temperature conditions. This means that the formed HMF and furfural decompose to the compounds, which are called unknown compounds ($[C_{X1}]$ for HMF and $[C_{X2}]$ for furfural). Therefore, sequential reaction approaches were applied to express the HMF and furfural accumulation during roasting of coffee beans.

$$\frac{d[C_{HMF,F}]}{dt} = k_1[C_p] - k_2[C_{HMF,F}] \quad (6)$$

After integration of Eq.6, the following mathematical expression (Eq. 7) can be derived for change in HMF or furfural concentration during roasting at a constant temperature:

$$[C_{HMF,F}] = [C_{HMF,F}]_0 e^{-k_2 t} + \left(\frac{k_1}{k_2 - k_1}\right)[C_p]_0 \left(e^{-k_1 t} - e^{-k_2 t}\right) \quad (7)$$

Where, $[C_{HMF,\,F}]$ and $[C_{HMF,\,F}]_o$ are HMF or furfural concentration at any roasting time and initial concentration (µmol/100g dm) respectively, $[C_p]_o$ is the initial concentration of total precursor compounds induced by caramelization and Maillard reaction pathways (µmol/100g dm), t is the roasting time (min), k_1 is the reaction rate constant for formation of the HMF or furfural (1/min), and k_2 is the reaction rate constant for formation of unknown compounds (1/min, HMF decomposition compound is X1 or furfural decomposition compound is X2).

The activation energy for the degradation of bioactive and sugar compounds was determined using the Arrhenius' equation that defines the relationship between reaction rate constant and temperature.

$$k = k_o e^{-\frac{Ea}{RT}} \tag{8}$$

where Ea is the activation energy (J/mol), R is the universal gas constant (J/mol.K), T is the temperature (K) and k_o is the pre-exponential factor. $Ln(k)$ values were plotted against $1/T$ values, and the obtained slope value was used in the calculation of activation energy for the degradation of the compounds.

3.2.13. Statical analysis

The evaluation of the collected results, plotting of the figures, and calculation of the kinetic outputs was concluded using Excel 2010 (Microsoft Corp., USA) and MATLAB (MathWorks, USA) software. The SPSS 22 was used to determine significant differences between the treatments using analysis of variance (ANOVA) and Duncan's multiple comparison test (SPSS Inc., USA). All analyses were done at least three times, and the results are expressed as the mean value.

4. RESULTS AND DISCUSSION

4.1. Quality attributes of green Arabica coffee beans

Chlorogenic acids (CGA), caffeine (CAF), sucrose, and trigonelline content were observed in Arabica coffee as biochemical compounds of coffee.These biochemical components of coffee are the basic components of aroma. As a result, correlations between the quality of a coffee cup and various chemical characteristics could be used as additional tools for assessing the quality of coffee (Sualeh et al., 2020).

Green coffee beans have a large number of high antioxidant capacities and phenolics. Tyrosol and chlorogenic acid were found to be more abundant in green coffee than roasted coffee, while ferulic acid, m-hydroxybenzoic acid, and p-hydroxyphenylacetic acid were shown to be more abundant in roasted coffee. (Muñoz et al., 2020).

Sualeh et al. (2020) reported that the range of trigonelline, chlorogenic acids, and caffeine on a dry matter basis was, respectively, 0.80 to 1.08 g/100g, 2.80 to 5.42 g/100g, and 0.85 to 1.73 g/100g of green coffee.

In present study color values, caffeine, phenolic compounds, antioxidant activity, TPC and main sugars for Arabica green coffee beans were reported in table 4.1.

Color paremeters values reported as, L value is 53.48, a value is 1.67, b value is 20.46, C value id 20.53, Hue value is 85.34, and browning index is 0.065.

The phenolic compounds in the samples reported as, green coffee bean has 811.9 mg/100g dm of 5-CQA, 102.8 mg/100g of CA, 95.0 mg/100g of 3-CQA, 55.2 mg/100g of 3,4-di CQA, 85.0 mg/100g of 4,5-diCQA, 93.3 mg/100g of 3,5-di CQA, 6.5 mg/100g dm of 4-CQA, 0.3 mg/100g dm of 5-FQA, 26.2 mg/100g dm of 4-FQA, 692.4 mg/100g dm of caffeine, and 3-FQA it was not didicted. Antioxidant capacity of green coffee beans measured by FRAP was 2439 mg/100g dm, and

DPPH was 6268 mg/100g dm. TPC in green coffee beans was 1692±114 mg/100g dm.

The main sugars values in green coffee beans were reported as, sucrose was 5.30 g/100g dm, fructose was 0.47 g/100g dm, and glucose was 0.49 g/100g dm. In the last the content of HMF in green coffee beans was 0.46 µmol/100g dm and Furfural was 0.01 µmol/100g dm, as shown in table 4.1.

Table 4.1. Physical/Chemical atributes of untreated Arabica coffee beans

Physical/Chemical Atributes		Green coffee beans
Color measurements	L*	53.48±1.46
	a*	1.67±0.28
	b*	20.46±0.39
	C*	20.53±0.40
	Hue*	85.34±0.74
	BI	0.065±0.013
	DM(%)	89.6±0.07
	aw	0.601±0.018
Caffeine and phenolic compounds (mg/100g dm)	5-CQA	811.9±19.0
	CA	102.8±2.4
	3-CQA	95.0±2.2
	3,4-di CQA	55.2±1.3
	4,5-diCQA	85.0±2.0
	3,5-di CQA	93.3±2.2
	3-FQA	N.D.
	4-CQA	6.5±0.2
	5-FQA	0.3±0.0
	4-FQA	26.2±0.6
	Caffeine	692.4±16.3
t activity and TPC (mg/100g dm)	FRAP	2439±182
	DPPH	6268±30
	TPC	1692±114
Main sugars (g/100g dm)	Sucrose	5.30±0.46
	Fructose	0.47±0.04
	Glucose	0.49±0.04
µmol/ 100g dm	HMF	0.46±0.07
	Furfural	0.01±0.00

4.2. Time and Temperature History Data for Microwave Roasting

Roasting time increased by decreasing temperature as shown in Figure 1.4. Arabica coffee beans roasted at three different temperatures (180, 200 and 220 °C). Each degree needed specific time to reach the required temperature degree to start the roasting process, namely come up time. 180 °C needed 2.0 minutes, 200 °C needed 2.7 minutes and 220 °C needed 3.3 minutes as mentioned in Table 4.2.

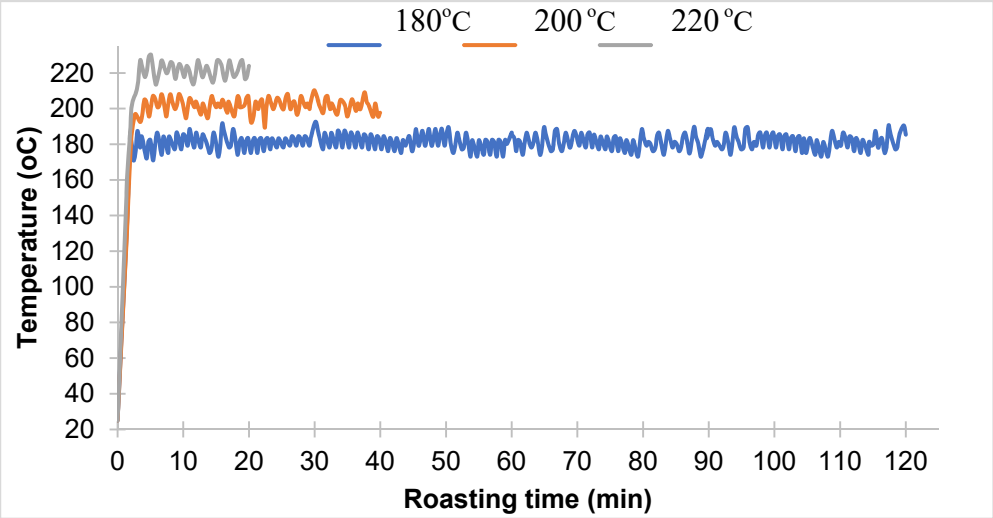

Figure 4.1. Microwave roasting temperature and time

Table 4.2. Come up time for target temperature under microwave roasting

Temperature (°C)	**Come up Time (CUT, min)**	**Ta (°C)**	**SD**
180	2.0	180.7	4.3
200	2.7	201.7	4.1
	**N.D: Not detected*		
220	3.3	221.4	4.3

SD: Standard Deviation

4.3. Colour analysis

Roasted coffee beans were divided into three degrees based on the L*, a*, and b* CIE parameters: light, medium, and dark roasted coffee beans. The L* value changes dramatically during the roasting process among colour parameters (Duarte et al., 2005). Therefore, it can be used as an indicator to divide the roasting degree of coffee beans. Da Porto et al. (1991) and Duarte et al. (2005) reported that the L* value decreased with increased roasting time and temperature. Each stage of the roasting process can be recognized in a clearly defined colour zone when the chromatic parameters a* and b* are checked because they represent the colour directions: Red, green, yellow, and blue are the colours that are represented by the symbols +a*, -a*, +b*, and -b*, respectively (Duarte et al., 2005) (Figure 4.2).

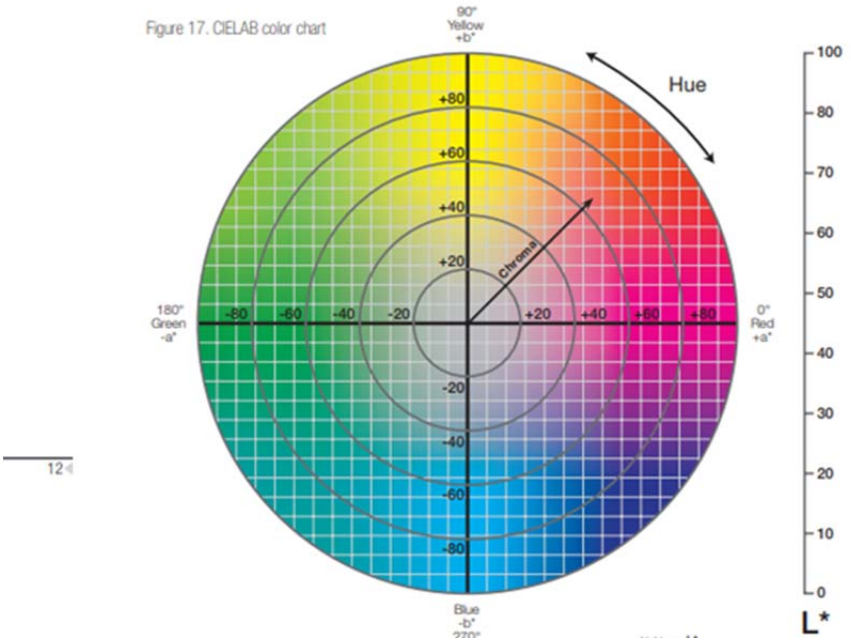

Figure 4.2. CELAB colour value

The following figures explain the colour values of different coffee samples (Figure 4.3-4.5). For example, L* values of ground arabica coffee beans at 180 ^{o}C for 120 minutes ranged from 52.6 to 19.3, a* values ranged from 8.8 to 10.4, b* values ranged from 30.7 to 15.4, C* values ranged from 31.9 to 18.6, Hue* values ranged from 74.1 to 56.0 and ΔE* values ranged from 12.5 to 35.6 as explained in Figure 4.3, at 200 ^{o}C for 40 minutes L* values ranged from 26.1 to 15.1, a* values ranged from 10.0 to 6.5, b* values ranged from 19.1 to 8.6, C* values ranged from 21.5 to 10.7, Hue* values ranged from 62.4 to 52.8 and ΔE* values ranged from 28.6 to 40.5 as explained in Figure 4.4, at 220 ^{o}C for 20 minutes L* values ranged from 17.5 to 12.5, a* values ranged from 4.8 to 4.3, b* values ranged from 8.8 to 5.0, C* values ranged from 10.0 to 6.6, Hue* values ranged from 61.1 to 49.6 and ΔE* values ranged from 38.0 to 43.9 as explained in Figure 4.5.

In the present study, L* was significantly ($p < 0.05$) higher than all the coffee samples at 180 ^{o}C for 120 minutes. L* value decreased with increased roasting time and temperature, as expected. This is in agreement with results reported by Da Porto et al. (1991), Nicoli et al. (1997), and Duarte et al. (2005).

4.4. Browning index (BI)

The roasting degree determined by the browning index, which plays an important role in the overall acceptability of the coffee beverage**.** Browning is formed during the roasting process due to the changes in the physical characteristics of coffee samples, which generate colour-forming compounds during the roasting process.

BI values of different samples of coffee were explained in the following figures. BI values of ground arabica coffee beans at 180 ^{o}C for 120 minutes ranged from 0.17 to 0.91 as explained in Figure 4.3, at 200 ^{o}C for 40 minutes ranged from 1.07 to 1.62 as explained in Figure 4.4, at 220 ^{o}C for 20 minutes ranged from 1.20 to 1.80 as explained in Figure 4.5. In the present study BI value was significantly ($p < 0.05$) higher than all the coffee samples at 220^{o}C for 20 minutes.

This study's results are in accordance with the findings of Nicoli et al. (1997), Doğan et al. (2019), and Salamatullah et al. (2021), that reported a significant increase ($p < 0.05$) in browning index values of Arabica coffee beans by increasing roasting time and temperature as shown in Table 4.3.

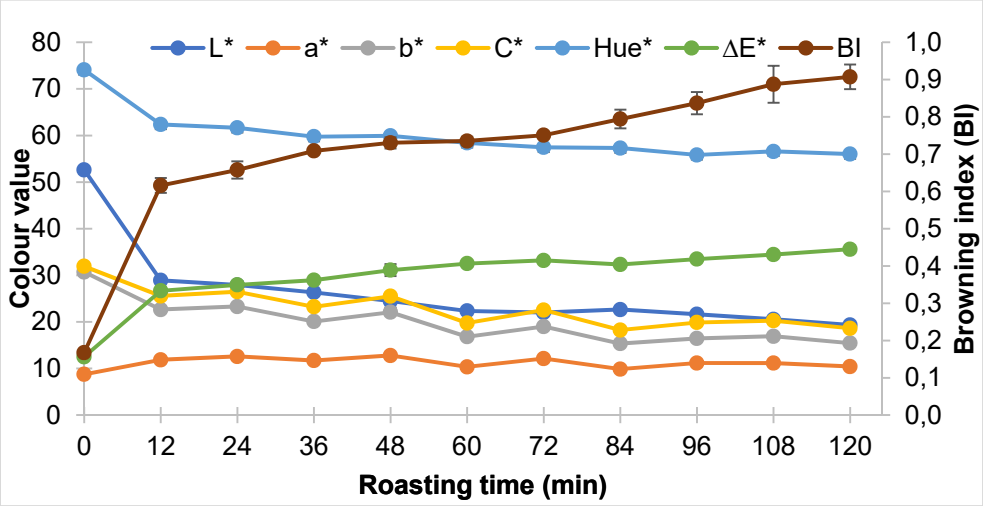

Figure 4.3. Browning index (BI) and colour values for roasted coffee beans at 180 °C, for 120 minutes

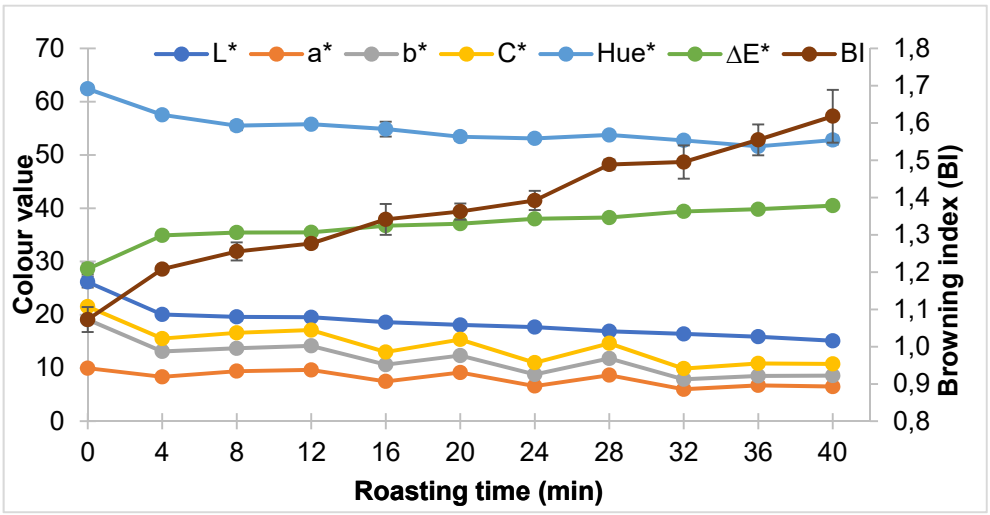

Figure 4.4. Browning index (BI) and colour values for roasted coffee beans at 200°C, for 40 minutes

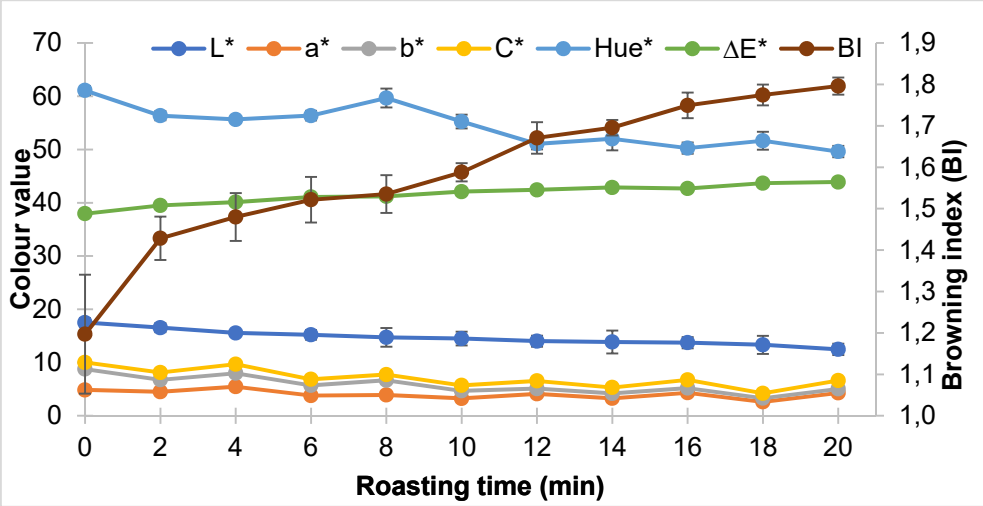

Figure 4.5 Browning index (BI) and colour values for roasted coffee beans at 220 °C, for 20 minutes

Table 4.3. Effect of microwave roasting at different times and temperatures on Colour values and browning index BI

T	*t* (min)	L*	a*	b*	C*	Hue*	ΔE*	BI
180 °C	CUT	52.6±0.7^{a}	8.8±0.2^{h}	30.7±0.2^{a}	31.9±0.3^{a}	74.1±0.2^{a}	12.5±0.3^{i}	0.17±0.00^{g}
	12	28.9±0.4^{b}	11.9±0.1^{d}	22.6±0.3^{c}	25.6±0.3^{c}	62.4±0.3^{b}	26.7±0.4^{h}	0.62±0.02^{f}
	24	27.9±0.8^{c}	12.6±0.1^{b}	23.3±0.1b	26.5±0.2^{b}	61.6±0.2c	28.0±0.8^{g}	0.66±0.02^{e}
	36	26.3±0.5^{d}	11.7±0.1^{d}	20.1±0.3^{e}	23.2±0.3^{d}	59.8±0.3^{d}	28.9±0.5^{f}	0.71±0.00^{d}
	48	24.5±1.4^{e}	12.8±0.2^{a}	22.1±0.4^{c}	25.5±0.4^{c}	59.9±0.4^{d}	31.1±1.3^{e}	0.73±0.002^{d}
	60	22.3±0.7^{f}	10.3±0.1^{f}	16.8±0.4^{g}	19.8±0.3^{g}	58.4±0.5^{e}	32.5±0.7^{d}	0.74±0.01^{d}
	72	22.0±0.6^{f}	12.1±0.2c	19.0±0.3f	22.5±0.3^{e}	57.4±0.2f	33.2±0.6^{c}	0.75±0.00^{d}
	84	22.6±0.4^{f}	9.9±0.3^{g}	15.4±0.4^{i}	18.2±0.4^{i}	57.3±0.3f	32.3±0.3d	0.79±0.03^{c}
	96	21.6±0.3^{g}	11.2±0.4^{e}	16.4±0.4^{h}	19.9±0.5^{g}	55.8±0.4^{h}	33.5±0.3c	0.84±0.03^{b}
	108	20.6±0.3^{h}	11.2±0.2^{e}	16.9±0.4^{g}	20.3±0.4^{f}	56.6±0.5^{g}	34.4±0.2b	0.89±0.05^{a}
	120	19.3±0.8^{i}	10.4±0.2^{f}	15.4±0.4^{i}	18.6±0.4^{h}	56.0±0.5^{h}	35.6±0.8^{a}	0.91±0.03^{a}
200 °C	CUT	26.1±1.0^{a}	10.0±0.1^{a}	19.1±0.2^{a}	21.5±0.2^{a}	62.4±0.4^{a}	28.6±1.0^{g}	1.07±0.03^{g}
	4	20.0±0.3^{b}	8.3±0.2^{f}	13.1±0.3^{d}	15.5±0.3^{d}	57.5±0.4^{b}	34.9±0.3^{f}	1.21±0.01^{f}
	8	19.6±0.7^{bc}	9.4±0.3^{e}	13.7±0.6^{c}	16.6±0.7^{c}	55.5±0.5^{c}	35.4±0.7^{e}	1.26±0.02^{ef}
	12	19.5±0.4^{c}	9.6±0.2^{b}	14.1±0.4^{b}	17.1±0.5^{b}	55.8±0.4^{c}	35.4±0.4^{e}	1.28±0.01^{e}
	16	18.6±0.3^{d}	7.5±0.2^{g}	10.6±0.3^{g}	13.0±0.4f	54.9±0.4^{d}	36.7±0.3^{d}	1.34±0.04^{d}
	20	18.1±0.2^{e}	9.1±0.1^{d}	12.3±0.2^{e}	15.4±0.2^{d}	53.4±0.4^{ef}	37.1±0.2^{d}	1.36±0.02^{d}
	24	17.7±0.3^{e}	6.6±0.2^{ih}	8.8±0.2^{h}	11.0±0.2^{g}	53.1±0.4^{fg}	38.0±0.3^{c}	1.39±0.03^{d}
	28	16.9±0.4^{f}	8.7±0.1^{e}	11.8±0.1^{f}	14.6±0.1^{e}	53.7±0.5^{e}	38.2±0.4^{c}	1.49±0.01^{c}
	32	16.4±0.3^{g}	6.0±0.2^{j}	7.9±0.3^{i}	9.9±0.3^{h}	52.7±0.8^{g}	39.4±0.3^{b}	1.50±0.04^{bc}
	36	15.9±0.3^{h}	6.7±0.3^{h}	8.5±0.4^{h}	10.8±0.5^{g}	51.6±0.5^{h}	39.8±0.3^{b}	1.55±0.04^{b}
	40	15.1±0.7^{i}	6.5±0.2^{i}	8.6±0.2^{h}	10.7±0.2^{g}	52.8±0.9^{g}	40.5±0.6^{a}	1.62±0.07^{a}
220 °C	CUT	17.5±0.3^{a}	4.8±0.2^{b}	8.8±0.3^{a}	10.0±0.3^{a}	61.1±0.8^{a}	38.0±0.4^{h}	1.20±0.14^{g}
	2	16.5±0.03^{b}	4.5±0.2^{c}	6.8±0.3^{c}	8.1±0.4^{b}	56.3±0.8^{c}	39.5±0.3^{g}	1.43±0.05^{f}
	4	15.5±0.2^{c}	5.5±0.1^{a}	8.0±0.2^{b}	9.7±0.2^{c}	55.6±0.6^{c}	40.1±0.2^{f}	1.48±0.06e^{f}
	6	15.2±0.1^{d}	3.8±0.2^{f}	5.7±0.3^{d}	6.8±0.3^{e}	56.4±0.9^{c}	41.1±0.1^{e}	1.52±0.05^{def}
	8	14.7±0.2^{e}	3.9±0.3^{f}	6.7±0.3^{c}	7.7±0.4^{d}	59.7±1.8^{b}	41.2±0.2^{e}	1.54±0.05^{de}
	10	14.5±0.3^{e}	3.3±0.2^{g}	4.7±0.2f	5.7±0.3^{g}	55.2±1.3^{c}	42.1±0.3^{d}	1.59±0.02^{cd}
	12	14.0±0.3^{f}	4.1±0.1^{e}	5.1±0.2^{e}	6.5±0.2^{f}	51.0±1.0^{de}	42.4±0.4^{c}	1.67±0.04^{bc}
	14	13.9±0.2^{fg}	3.3±0.2^{g}	4.2±0.2^{g}	5.3±0.2^{h}	52.0±2.1^{d}	42.9±0.2^{b}	1.70±0.02^{ab}
	16	13.7±0.2^{g}	4.3±0.3^{cd}	5.2±0.2^{e}	6.7±0.3^{fe}	50.3±1.0^{ef}	42.7±0.2^{b}	1.75±0.03^{ab}
	18	13.3±0.2^{h}	2.6±0.2^{h}	3.3±0.1^{h}	4.2±0.2^{i}	51.6±1.7^{d}	43.7±0.1^{a}	1.77±0.03^{ab}
	20	12.5±0.5^{i}	4.3±0.2^{de}	5.0±0.1^{e}	6.6±0.2^{fe}	49.6±1.1^{f}	43.9±0.5^{a}	1.80±0.02^{a}

Superscript letters on the same column indicate the significant difference between the roasting time for regarding temperature ($p < 0.05$). CUT: Come up time for target roasting temperature.

4.5. Water activity (a_w)

Water activity (a_w) significantly affects coffee's physical and chemical properties. There are differences between raw coffee beans and roasted coffee beans in textural properties due to water activity. Roasted coffee beans had less strength and toughness than green coffee beans, and this resulting textural property was strongly affected by a_w. Water activity for green coffee beans was 0.523 for roasted coffee beans it was in a range of 0.253- 0.207 (Pittia, et al., 2007).

The water activity (a_w) value of different samples of coffee is explained in the following Figures. The water activity of ground Arabica coffee beans at 180 ^oC for 120 minutes ranged from 0.347 to 0.128 as explained in Figure 4.6, at 200 ^oC for 40 minutes ranged from 0.282 to 0.135 as explained in Figure 4.7, at 220 ^oC for 20 minutes ranged from 0.159 to 0.122 as explained in Figure 4.8.

Results show that during the roasting process, a_w values significantly ($p < 0.05$) decreased by increasing the temperature, producing less strength and toughness in coffee beans, as shown in Table 4.4.

4.6. Dry matter content

The dry matter content of coffee beans is affected by several factors, main factors are the storage period and roasting process (Król, K. et al., 2020). In the present study, the effect of the roasting process on the amount of dry matter content was studied.

Dry matter content of different samples of coffee is illustrated in the following figures. Dry matter of ground Arabica coffee beans at 180 ^oC for 120 minutes ranged from 94.9% to 99.2% as shown in Figure 4.6, at 200 ^oC for 40

minutes ranged from 98.1% to 99.5% as shown in Figure 4.7, at 220 ^{o}C for 20 minutes ranged from 98.9% to 99.9% as shown in Figure 4.8.

The roasting temperature was directly proportional to dry matter content values, and a significant increase ($p < 0.05$) of dry matter content was determined as the temperature increased. These results are highly in agreement with obtained results for Brazil beans reported by Dybkowska et al. (2017) and (*Coffea arabica*) coffee beans reported by Król et al. (2020), which showed a significant effect of roasting degrees on dry matter of coffee beans, mainly the highest dry matter content was in dark roasted coffee (98.74 g $100g^{-1}$) while the smallest in light roasted (96.24 g $100g^{-1}$).

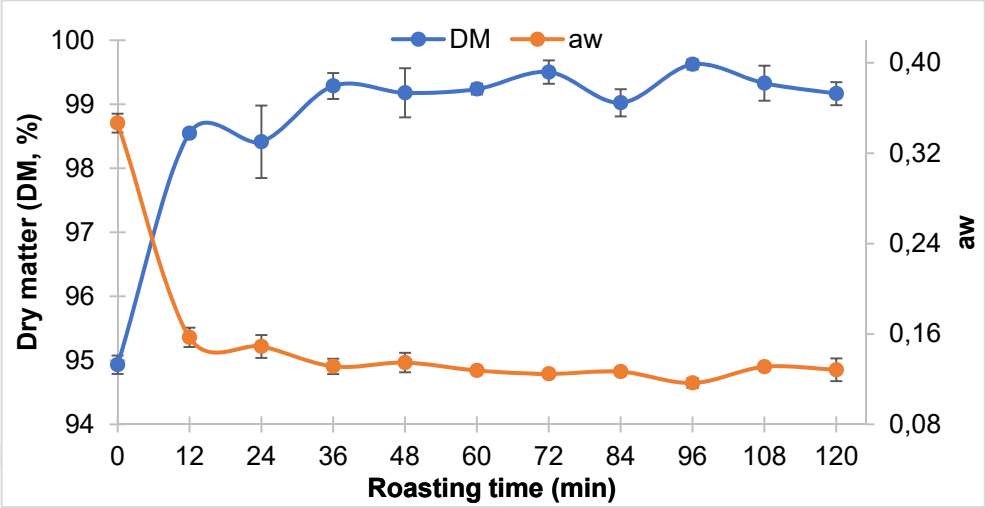

Figure 4.6. Dry matter content and water activity values for roasted coffee beans at 180 °C, for 120 minutes

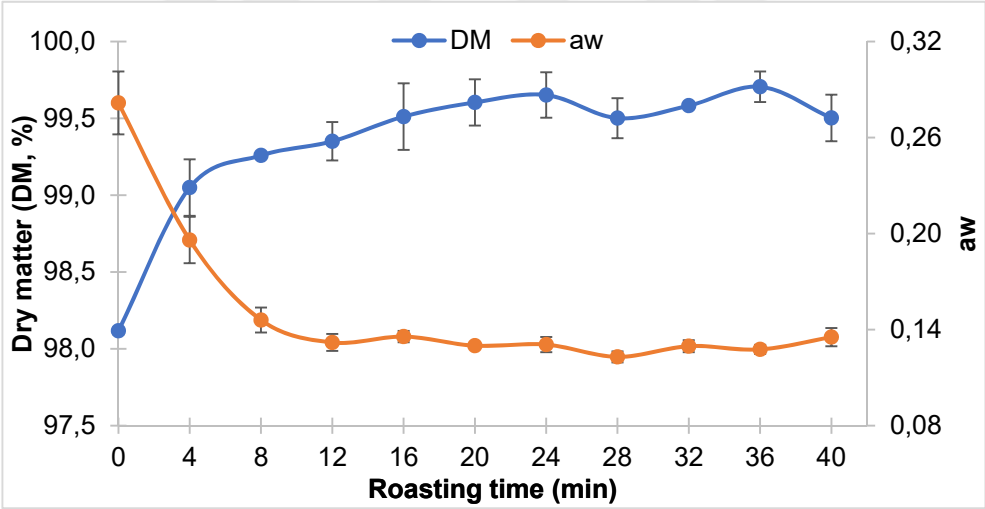

Figure 4.7. Dry matter content and water activity values for roasted coffee beans at 200 °C, for 40 minutes

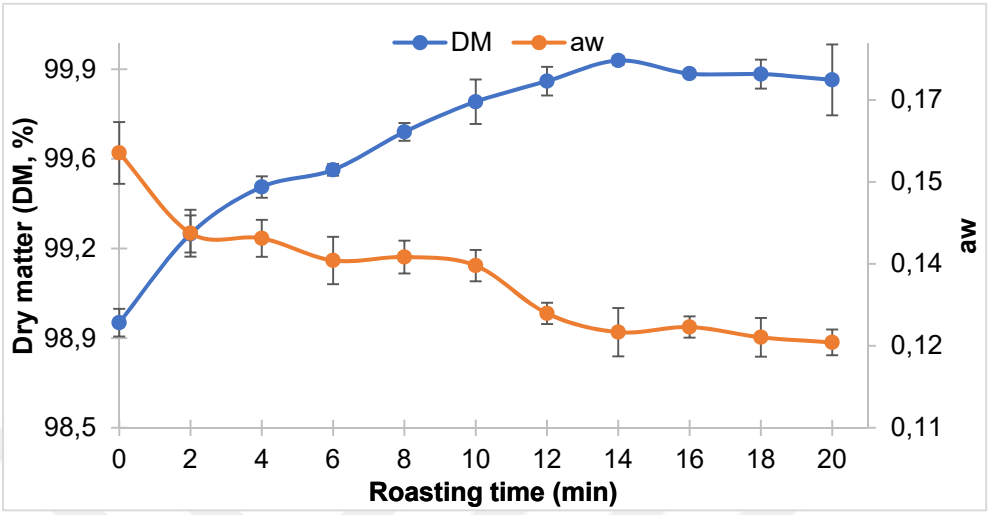

Figure 4.8. Dry matter content and water activity values for roasted coffee beans at 220 °C, for 20 minutes

Table 4.4. Effect of microwave roasting at different times and temperatures on dry meter content and water activity

Temperature	Time(min)	DM (%)	aw
180 °C	CUT	94.9±0.14^{d}	0.347±0.008^{a}
	12	98.5±0.05^{c}	0.157±0.009^{b}
	24	98.4±0.57^{bc}	0.149±0.010^{b}
	36	99.3±0.20^{ab}	0.131±0.007^{c}
	48	99.2±0.38^{a}	0.135±0.009^{c}
	60	99.2±0.09^{a}	0.128±0.002^{c}
	72	99.5±0.18^{a}	0.125±0.003^{cd}
	84	99.0±0.21^{a}	0.127±0.003^{cd}
	96	99.6±0.08^{a}	0.117±0.005^{cd}
	108	99.3±0.27^{a}	0.131±0.002^{cd}
	120	99.2±0.18^{a}	0.128±0.010^{d}
200 °C	CUT	98.1±0.02^{e}	0.282±0.020^{a}
	4	99.0±0.18^{d}	0.196±0.015^{b}
	8	99.3±0.02^{cd}	0.146±0.008^{c}
	12	99.4±0.12^{bcd}	0.132±0.005^{cd}
	16	99.5±0.22^{abc}	0.136±0.004^{cd}
	20	99.6±0.15^{abc}	0.130±0.002^{cd}
	24	99.7±0.15^{abc}	0.131±0.005^{cd}
	28	99.5±0.13^{ab}	0.123±0.003^{cd}
	32	99.6±0.00^{ab}	0.130±0.004^{d}
	36	99.7±0.10^{ab}	0.128±0.003^{d}
	40	99.5±0.15^{a}	0.135±0.006^{d}
220 °C	CUT	98.9±0.05^{d}	0.159±0.006^{a}
	2	99.3±0.07^{c}	0.143±0.005^{b}
	4	99.4±0.04^{b}	0.142±0.004^{b}
	6	99.5±0.02^{b}	0.138±0.005^{b}
	8	99.7±0.03^{d}	0.138±0.003^{b}
	10	99.8±0.09^{bc}	0.137±0.003^{b}
	12	99.9±0.06^{a}	0.127±0.002^{c}
	14	99.9±0.02^{a}	0.124±0.005^{c}
	16	99.9±0.00^{a}	0.125±0.002^{c}
	18	99.9±0.06^{a}	0.123±0.004^{c}
	20	99.9±0.14^{a}	0.122±0.003^{c}

Superscript letters on the same column indicate the significant difference between the roasting time for regarding temperature ($p < 0.05$). CUT: Come up time for target roasting temperature.

4.7. Caffeine and phenolic compounds

According to the average quantitative results from this investigation (Table 4.5 and Table 4.6), chlorogenic acid (5-CQA, 1227.9–53.1 mg/100g dm) and caffeic acid (CA 335.1–29.1 mg/100g dm) were significantly (p <0.05) the higher phenolic compound values present in all the coffee samples. On the other hand, the samples of coffee beans showed that 5-feruloylquinic acid (5-FQA) had the lowest concentration of phenolic compounds (16.6–3.7 mg/100g dm).

The following tables give the phenolic compound values of different coffee samples. 5-CQA content of ground Arabica coffee beans at 180, 200, and 220 ^{o}C ranged from 1227.9 to 186.2 mg/100g dm, 757.1 to 146.3 mg/100g dm and 566.3 to 53.1 mg/100g dm, for 120, 40 and 20 minutes respectively. As explained in Table 4.5. 5-CQA values of light roasted coffee at 180 ^{o}C were significantly (p <0.01) higher than all the coffee samples.

CA values of ground arabica coffee beans at 180, 200 and 220 ^{o}C ranged from 335.1 to 76.6 mg/100g dm, 261.0 to 69.6 mg/100g dm and 198.5 to 29.1 mg/100g dm, for 120, 40 and 20 minutes respectively, as explained in Table 4.5. CA values of lightly roasted coffee at 180 ^{o}C were significantly (p <0.01) higher than all the coffee samples.

Neochlorogenic acid (3-CQA) values of ground arabica coffee beans at 180, 200 and 220 ^{o}C ranged from 320.9 to 64.5 mg/100g dm, 263.6 to 61.7 mg/100g dm and 183.8 to 35.3 mg/100g dm, for 120, 40 and 20 minutes respectively. As explained in Table 4.5. 3-CQA values of lightly roasted coffee at 180 ^{o}C were significantly (p < 0.01) higher than all the coffee samples.

3,4-dicaffeoylquinic acid (3.4-diCQA) values of ground arabica coffee beans at 180, 200 and 220 ^{o}C ranged from 97.8 to 13.1 mg/100g dm, 52.2 to 9.0 mg/100g dm and 40.3 to 2.7 mg/100g dm, for 120, 40 and 20 minutes respectively. As explained in Table 4.5. (3.4-diCQA values of lightly roasted coffee at 180 ^{o}C were significantly (p <0.05) higher than all the coffee samples.

4,5-dicaffeoylquinic acid (4.5-diCQA) values of ground arabica coffee beans at 180, 200 and 220 °C ranged from 156.3 to 22.5 mg/100g dm, 84.5 to 15.2 mg/100g dm and 61.4 to 4.5 mg/100g dm, for 120, 40 and 20 minutes respectively. As explained in Table 4.5. 4.5 di-CQA values of lightly roasted coffee at 180 °C were significantly ($p < 0.05$) higher than all the coffee samples.

3,5-dicaffeoylquinic acid (3.5-diCQA) values of ground arabica coffee beans at 180, 200 and 220 °C ranged from 90.4 to 10.5 mg/100g dm, 45.0 to 7.1 mg/100g dm and 33.8 to 2.1 mg/100g dm, for 120, 40 and 20 minutes respectively. As explained in Table 4.5. 3.5-diCQA values of lightly roasted coffee at 180 °C were significantly ($p <0.05$) higher than all the coffee samples.

4-caffeoylquinic acid (4-CQA) values of ground arabica coffee beans at 180, 200 and 220 °C ranged from 31.2 to 15.7 mg/100g dm, 24.9 to 14.2 mg/100g dm and 19.5 to 7.1 mg/100g dm, for 120, 40 and 20 minutes respectively. As explained in Table 4.6. 4-CQA values of lightly roasted coffee at 180 °C were significantly ($p <0.01$) higher than all the coffee samples.

5-feruloylquinic acid (5-FQA) values of ground arabica coffee beans at 180, 200 and 220 °C ranged from 16.6 to 6.4 mg/100g dm, 16.0 to 6.6 mg/100g dm and 12.7 to 3.7 mg/100g dm, for 120, 40 and 20 minutes respectively. As explained in Table 4.6. 5-FQA values of lightly roasted coffee at 180 °C were significantly ($p <0.01$) higher than all the coffee samples.

4-feruloylquinic acid (4-FQA) values of ground arabica coffee beans at 180, 200 and 220 °C ranged from 38.6 to 8.2 mg/100g dm, 24.9 to 7.2 mg/100g dm and 18.9 to 2.8 mg/100g dm, for 120, 40 and 20 minutes respectively. As explained in Table 4.6 4-FQA values of lightly roasted coffee at 180 °C were significantly ($p <0.01$) higher than all the coffee samples.

These results showed that the coffee beans roasted at 180 °C ($p <0.05$) had significantly higher individual total phenolic compounds values. The decrease of total phenolic compounds by increasing roasting time and the temperature was observed. A significant stability of total phenolic compound content was observed

in the sample roasted at 200 °C, and a significant decrease (p <0.05) of total phenolic compound content was reported in the samples roasted at 220 °C.

Increasing roasting time and temperature causes breakage of the carbon-carbon bonds of 5-CQA and the levels of 5-CQA declined significantly. Isomerization and partial hydrolysis of diCQA to monoester derivatives may also take place in decreasing these phenolic compounds. This might be the result of other, heat-sensitive molecules being lost. Total 5-CQA was lost as the roasting time increased. Other chemical reactions may take place besides isomerization and degradation. Phenolic compound values depend on the roasting time and temperature, as a result of this study, the best roasting temperature to produce the most phenolic acids values in coffee is a light and medium roast degree (180, 200 °C), whereas darker roasts produce less phenolic compounds values (Farah et al., 2005).

Similar results were reported by Duarte et al. (2005) and Król et al., (2020), which showed that phenolic compounds decreased with increased roasting time and temperature. In addition, light roasting coffee could be the best coffee beverage to protect cells against oxidative damage according to phenolic compounds values.

Lee et al. (2016) show that the samples with the highest phenolic compound values were determined at the lowest temperature (180 °C).

The main acid in both Arabica and Robusta green beans, where it made up 66% and 56% of all chlorogenic acids, respectively, was 5-CQA. Furthermore, roasting green coffee beans at 205 °C was reported to significantly reduce the total CGA content. These investigations indicated that changing the roasting time and temperature curve is the most effective way to dramatically reduce the amount of CGA. The amount of CGA in the final coffee products appears to be strongly impacted by changes in roasting times and temperatures. This might explain the significant variations in pH and CGA levels across the commercial coffees tested.

However, rigorous treatment of food and beverages causes the loss of factors that contribute to palatability, such as flavor and color (Fujioka and Shibamoto, 2008).

Similar observations were made by Somporn et al. (2011), which reported that chlorogenic acid was the most abundant phenolic acid of the ten phenolic acids identified in coffee beans. Light-roasted coffee has the highest phenolic content and radical-scavenging activities of all types of roasted coffee. The importance of phenolic acid in roasted coffee comes from its antioxidant properties. As a result, coffee must be prepared so that it has a low enough amount of phenolic acid to prevent acid reflux while having a sufficient amount for antioxidant activities.

All the phenolic acids accumulate differently depending on the roasting degree. They all decreased with the degree of roasting. Medium roasting produces the lowest amounts, and unroasted coffee beans produce the highest phenolic amounts. The heating process has a significant impact on phenolic compounds with different rates of reactivity.

Depending on the degree of roasting, total phenolic acids and individual phenolic acid amounts changed. Individual phenolic acids decreased as temperatures rose, with the exception of some phenolic acids, such as chlorogenic acid, which were reduced by increasing the temperature. While higher roasting temperatures increased the concentrations of protocatechuic acid, vanillic acid, and ferulic acid, suggesting that heat treatment had an impact both positively and negatively on phenolic compounds.

Previous research reported that thermal processing decreased the phenolic content of vegetables. Although breakdown of cell walls also releases oxidative and hydrolytic enzymes that can damage the antioxidants in fruits and vegetables, thermal processing may release more bound phenolic acids. Therefore, the heat processing of polyphenol compounds may lead to the release of antioxidant compounds with various chemical and biological properties (Somporn et al., 2011).

The flavour of coffee is reported to be significantly influenced by CGA. They provide astringency and bitterness to the beverage, as well as contribute to its

final acidity. Release of caffeic acid occurs due to the Maillard and Strecker reactions. Roasting causes the creation of lactones and other phenol derivatives responsible for flavour and aroma, as well as an increase in bitterness (Somporn et al., 2011).

The roasting process causes a part of the CGA to be isomerized. Part of it is going to become quinolactones as a result of dehydration and the formation of an intramolecular bond, and the other part is going to be hydrolyzed and decomposed into low-molecular-weight compounds. The formation of polymeric substances like melanoidins is also promoted by CGA. Depending on the type of processing, blend, roasting degree, and analytical conditions, high roasting conditions may result in losses of up to 95% of CGA (Farah and Donangelo, 2006).

When compared to the majority of food sources of CGA, the CGA content of light-to medium-roasted coffees still stands out. At this roasting stage, diCQA may be partially hydrolyzed into monoesters and caffeic acid, which may then be hydrolyzed once more, decarboxylated, and degraded to a variety of simple phenols. Volatile phenol concentrations rise as the process continues (Farah and Donangelo, 2006).

However, roasting did result in changes in the relative contents, most notably a 33% drop in 5-O-caffeoylquinic acid that was accompanied by increases in 3- and 4-O-caffeoylquinic acids. However, the medium-roasted beverage's predominant chlorogenic acid remained 5-O-caffeoylquinic acid. Finally, it should be noted that significant amounts of chlorogenic acids survive to be extracted into domestic brews and instant coffee despite their progressive destruction during roasting. For many consumers, the beverage is a major dietary source of not only chlorogenic acids but also other antioxidants (Stalmach et al., 2006).

Thermal breakage of carbon–carbon covalent bonds occur during isomerisation in the initial roasting stages, and epimerisation, lactonisation, and degradation occur during the later roasting stages, which results in a decrease in CQAs levels. A previous study performed with other cultivars of C. arabica and C.

canephora, showed decreasing CQAs levels under stronger roasting conditions (Tfouni et al., 2012).

Caffeine values of different samples of coffee are explained in Tables 4.6. For example, caffeine values of ground arabica coffee beans determined at 180, 200, and 220 ^{o}C ranged from 5437.4 to 6485.4 mg/100g dm, 6514.8 to 8363.3 mg/100g dm, and 7056.7 to 7907.2 mg/100g dm, for 120, 40 and 20 minutes respectively.

In this study, caffeine values increased by increasing roasting time and temperature. Caffeine values (at 220 ^{o}C, 20 minutes) were significantly ($p < 0.05$) higher than all the coffee samples.

An increase in caffeine content was observed by Tfouni et al. (2012), when roasting temperatures were between 140 and 160 or 200 °C. Caffeine levels could possibly decrease as a result of sublimation. The fact that the weight of the green beans can decrease by 20% or more during roasting (10% water, 10% dry matter), and that findings are given on a dry matter basis, may be the reason for the increase in caffeine levels during roasting.

Tsai and Jioe (2021) reported similar results, in which, after roasting, caffeine levels in coffee significantly increased and reached around 6.12 mg/g under the dark roast condition. The caffeine level of Indonesian coffee was lowest in light roasting (2.62 mg/g), and it significantly increased in medium roasting (4.1 mg/g) and dark roasting (3.5 mg/g).

Table 4.5. Effect of microwave roasting at different microwave times and temperatures on phenolic acids (mg/100g dm)

T	T(min)	5-CQA	CA	3-CQA	3,4-di CQA	4,5-diCQA	3,5-di CQA
180 °C	**CUT**	1227.9±28.7^{a}	335.1±7.9^{a}	320.9±7.9^{a}	97.8±2.3^{a}	156.3±3.^{a}	90.4±2.1^{a}
	12	730.8±19.9^{b}	254.3±7.4^{b}	221.2±10.2^{b}	57.6±1.4^{b}	97.9±2.3^{b}	56.3±1.5^{b}
	24	559.1±13.2^{c}	202.4±4.8^{c}	198.1±5.8^{c}	41.6±1.0^{c}	76.9±1.8^{c}	39.1±0.9^{c}
	36	459.8±11.1^{d}	150.6±3.7^{d}	134.0±4.2^{d}	33.3±0.8^{d}	54.0±1.4^{d}	27.8±0.7^{d}
	48	361.6±8.6^{e}	137.1±3.3^{e}	124.7±4.3e	25.7±0.6e	45.5±1.3^{e}	22.3±0.5^{e}
	60	323.5±7.6^{f}	122.4±3.0^{f}	107.5±2.5^{f}	22.8±0.7^{f}	37.1±0.9^{f}	20.2±0.5^{f}
	72	268.8±6.5^{g}	108.5±2.8^{g}	93.0±2.2^{g}	18.2±0.4g	32.8±1.0^{g}	14.3±0.3^{g}
	84	258.1±6.1^{g}	100.5±2.5^{h}	87.4±2.2^{gh}	17.2±0.4^{g}	29.5±0.7^{h}	14.1±0.3^{g}
	96	236.3±5.7^{h}	96.4±2.5^{h}	80.9±1.9^{h}	15.4±0.7^{h}	26.7±0.7^{i}	12.8±0.3^{gh}
	108	199.9±4.8^{i}	79.0±1.8^{i}	64.6±1.6^{i}	14.3±0.3^{hi}	22.7±0.7^{j}	11.6±0.3^{hi}
	120	186.2±4.5^{i}	76.6±2.2^{i}	64.5±1.5^{i}	13.1±0.6^{i}	22.5±0.5^{j}	10.5±0.3^{i}
200 °C	**CUT**	757.1±21.4^{a}	261.0±6.4^{a}	263.6±15.2^{a}	52.2±1.3^{a}	84.5±2.4^{a}	45.0±1.1^{a}
	4	502.6±16.4^{b}	198.4±7.9^{b}	187.3±12.1^{b}	35.7±0.9^{b}	58.3±1.4^{b}	27.4±0.8^{b}
	8	359.8±14.9^{c}	167.3±7.6^{c}	143.0±7.3^{c}	23.9±0.6^{c}	41.6±1.2^{c}	20.5±0.7^{c}
	12	341.2±12.7^{c}	147.9±4.1^{d}	137.8±7.1^{c}	21.1±0.6^{d}	33.9±1.1^{d}	17.9±0.7^{d}
	16	285.3±13.2^{d}	121.9±6.5^{e}	115.2±6.4^{d}	17.9±0.5^{e}	28.6±0.9^{e}	15.3±0.5^{e}
	20	231.2±5.6^{e}	103.0±3.1^{f}	103.3±4.2^{de}	14.9±0.2^{f}	24.7±0.7^{f}	12.0±0.6^{f}
	24	219.1±5.2^{e}	100.4±4.3^{f}	95.6±1.2^{e}	14.5±0.4^{f}	23.4±0.7^{fg}	10.8±0.5^{g}
	28	211.7±5.1^{e}	94.3±4.6^{fg}	90.7±2.7^{ef}	14.1±0.4^{f}	22.1±0.6^{g}	10.5±0.4^{g}
	32	188.9±4.5^{f}	89.1±4.7^{g}	81.8±6.1^{f}	11.4±0.2^{g}	17.5±0.4^{h}	8.7±0.4^{h}
	36	167.5±4.0^{g}	73.6±1.9^{h}	65.2±2.1^{g}	10.8±0.2^{g}	16.8±0.4^{hi}	8.6±0.4^{h}
	40	146.3±3.4^{h}	69.6±2.4^{h}	61.7±2.0^{g}	9.0±0.5^{h}	15.2±0.4^{i}	7.1±0.4^{i}
220 °C	**CUT**	566.3±14.1^{a}	198.5±4.7^{a}	183.8±4.3^{a}	40.3±0.9^{a}	61.4±1.5^{a}	33.8±1.0^{a}
	2	329.4±8.2^{b}	136.5±4.2^{b}	139.9±13.4^{b}	24.7±0.6^{b}	36.4±0.9^{b}	18.7±0.5^{b}
	4	283.2±6.6^{c}	115.1±2.7^{c}	107.0±3.2^{c}	21.0±0.8^{c}	29.9±1.0^{c}	15.7±0.5^{c}
	6	245.5±6.7^{d}	101.0±2.9^{d}	95.2±4.2^{d}	16.9±1.0^{d}	24.9±1.4^{d}	12.9±0.7^{d}
	8	234.9±5.5^{d}	93.8±2.2^{e}	83.7±2.4^{e}	16.1±0.7^{d}	21.8±0.8^{e}	12.70.3^{d}
	10	208.0±4.9^{e}	82.2±1.9^{f}	79.1±2.9^{e}	13.9±0.3^{e}	16.3±0.4^{f}	10.9±0.3^{e}
	12	108.3±5.1^{f}	55.5±4.8^{g}	51.8±5.8^{f}	5.5±0.3^{f}	8.4±0.3^{g}	4.2±0.2^{f}
	14	93.3±8.2^{g}	45.2±5.8^{h}	43.8±5.4^{fg}	4.6±0.1^{fg}	6.8±0.2^{h}	3.5±0.3^{fg}
	16	73.9±1.7^{h}	37.5±1.6^{i}	35.6±2.0^{gh}	4.1±0.1^{gh}	6.5±0.2^{h}	3.3±0.2^{g}
	18	65.3±4.2^{h}	34.3±3.1^{ij}	29.4±1.5^{gh}	3.3±0.1^{hi}	4.9±0.1^{i}	2.4±0.3^{h}
	20	53.1±1.4^{i}	29.1±1.9^{j}	35.3±2.9^{h}	2.7±0.1^{i}	4.5±0.2^{i}	2.1±0.1^{h}

Superscript letters on the same column indicate the significant difference between the roasting time for regarding temperature ($p < 0.05$). CUT: Come up time for target roasting temperature.

Table 4.6. Effect of microwave roasting at different microwave times and temperatures on phenolic acids and caffeine (mg/100g dm).

T	t (min)	4-CQA	5-FQA	4-FQA	Caffeine
180 °C	**CUT**	31.2±0.7^{a}	16.6±0.4^{a}	38.6±0.9^{a}	5437.4±146.4^{f}
	12	25.8±0.7^{b}	14.5±0.8^{b}	26.7±0.6^{b}	5632.6±134.8^{ef}
	24	25.1±0.6^{b}	12.5±1.1^{c}	23.2±0.5^{c}	5809.1±136.7^{de}
	36	23.1±1.2^{c}	11.6±1.3^{cd}	17.6±0.4^{d}	5914.5±120.3^{cd}
	48	21.5±0.6^{d}	10.9±1.0^{d}	14.8±0.3^{e}	5972.9±158.5^{bcd}
	60	20.5±0.5^{de}	9.1±0.2^{e}	12.4±0.3^{f}	6026.1±144.5^{bcd}
	72	20.2±0.5^{e}	9.0±0.5^{e}	10.4±0.9^{g}	6081.6±143.1^{bc}
	84	18.1±0.5^{f}	8.2±0.7^{ef}	10.6±0.3^{g}	6195.4±163.1^{b}
	96	17.0±1.1^{f}	8.1±0.4^{ef}	10.0±0.2^{g}	6218.6±153.5^{b}
	108	17.4±0.3^{f}	7.0±0.4^{fg}	8.3±0.2^{h}	6232.9±139.8^{b}
	120	15.7±0.5^{g}	6.4±0.4^{g}	8.2±0.2^{h}	6485.4±152.1^{a}
200 °C	**CUT**	24.9±1.3^{a}	16.0±0.4^{a}	24.9±0.8^{a}	6514.8±167.0^{e}
	4	23.8±0.5^{b}	13.2±0.7^{b}	17.5±0.4^{b}	6641.9±155.5^{e}
	8	23.4±0.6^{bc}	11.8±0.3^{c}	15.4±0.4^{c}	7267.8±170.9^{d}
	12	22.3±1.2^{c}	11.3±0.3^{c}	13.4±0.3^{d}	7388.0±152.0^{d}
	16	20.0±0.5^{d}	9.9±0.1^{d}	11.3±0.3^{e}	7471.7±186.6^{cd}
	20	19.8±0.5^{d}	10.1±0.7^{de}	10.4±0.3^{f}	7557.2±220.4^{bcd}
	24	19.4±0.5^{d}	9.1±0.4^{ef}	9.6±0.2^{g}	7741.8±162.8^{bc}
	28	16.6±0.5^{e}	8.4±1.1^{fg}	9.7±0.3^{g}	7757.7±190.9^{bc}
	32	16.3±0.5^{ef}	7.8±0.5^{g}	8.6±0.2^{h}	7795.6±194.5^{bc}
	36	15.2±0.4^{fg}	6.9±0.4^{h}	7.3±0.2^{i}	7851.5±184.1^{b}
	40	14.2±0.4^{g}	6.6±0.4^{h}	7.2±0.2^{i}	8363.3±197.3^{a}
220 °C	**CUT**	19.5±0.5^{a}	12.7±0.5^{a}	18.9±0.4^{a}	7056.7±169.7^{d}
	2	18.1±0.5^{b}	10.0±0.4^{b}	12.6±0.3^{b}	7109.6±171.9^{d}
	4	17.3±0.5^{c}	8.8±0.3^{c}	10.6±0.3^{c}	7085.7±180.1^{d}
	6	16.2±0.5^{d}	8.0±0.2^{d}	9.4±0.3^{d}	7280.9±170.7^{cd}
	8	14.5±0.3^{e}	7.6±0.3^{d}	8.9±0.2^{e}	7313.4±178.6^{cd}
	10	13.0±0.3^{f}	6.7±0.3^{e}	8.2±0.2^{f}	7278.5±172.8^{cd}
	12	13.0±0.7^{f}	5.9±0.5^{f}	5.1±0.2^{g}	7450.3±176.9^{c}
	14	10.2±0.6^{g}	4.9±0.5^{g}	4.1±0.1^{h}	7521.9±179.0^{bc}
	16	10.1±0.7^{g}	4.6±0.5^{g}	3.7±0.1^{i}	7601.4±190.7^{abc}
	18	8.2±0.2^{h}	3.5±0.4^{h}	2.8±0.1^{j}	7790.1±191.6^{ab}
	20	7.1±0.4^{i}	3.7±0.2^{h}	3.0±0.1^{j}	7907.2±197.3^{a}

Superscript letters on the same column indicate the significant difference between the roasting time for regarding temperature ($p < 0.05$). CUT: Come up time for target roasting temperature.

4.8. Total phenolic contents

The microwave roasting process significantly affects coffee beans' total phenolic content (TPC) (Salamatullah et al., 2022). Polyphenols in roasted coffee beans appeared to be lower than in green coffee beans (Nebesny and Budryn, 2003, Salamatullah et al., 2021), due to the degradation of polyphenols during the roasting process. More than 60% of the chlorogenic acid in green coffee was degraded during roasting. Microwaving caused a smaller decomposition in the ratio of polyphenols in coffee beans compared to the convection roasting method (Nebesny and Budryn, 2003). Friedman and Dao (1990) reported that chlorogenic acid content in ergot degraded strongly by convection heating compared to the microwave method.

TPC of different samples of coffee is explained in the following figures. TPC of ground arabica coffee beans at 180 ^{o}C for 120 minutes ranged from 2951.7 to 2013.7 mg/100g dm as explained in Figure 4.9, at 200 ^{o}C for 40 minutes ranged from 2874.9 to 2411.9 mg/100g dm as explained in Figure 4.10, at 220 ^{o}C for 20 minutes ranged from 2548.8 to 2023.5 mg/100g dm as explained in Figure 4.11.

These results, as shown in table 4.6, reported that coffee beans roasted at 200 ^{o}C significantly (p <0.05) had higher TPC values. The decreasing of TPC by increasing roasting time and the temperature was observed. 200 ^{o}C roasting degree showed significant stability of TPC content, which ranged from 2874.9 to 2411.9 mg/100g dm and the significant (p <0.05) decrease of TPC content reported in the samples was roasted at 220^{o}C.

Similar results were reported by Al-Juhaimi et al. (2018), which showed that medium power of microwave treatments increased the TPC and antioxidant activity of apricot kernels while decreasing it at higher powers. The phenolic compounds content may have decreased due to the polymerization, auto-oxidation, and degradation of the compounds during roasting (Wongsa et al., 2019). The increase of microwave power affects TPC positively, while high microwave power reduces TPC value in roasted coffee beans (Salamatullah et al., 2022).

According to Salamatullah et al. (2021) reported that (Arabica and Robusta) green coffee beans had higher possessed enhanced antioxidant activity and TPC compounds compared to roasted coffee beans, in contradiction to Diviˇset al. (2019) and Król et al. (2020) studies, which reported significantly (p <0.05) increasing of total phenolic compounds during roasting process comparing to green coffee beans, and coffee beans roasted at the light and medium roasting parameters were found to have the highest TPC content (Salamatullah et al., 2021).

These results are also in accordance with the findings of Ghafoor et al. (2019) reported that TPC decreased in poppy seeds and oil after being microwave-roasted at 720 W for five minutes, showing that roasting has similar effects on other plant materials.

4.9. Antioxidant activity

4.9.1. Ferric reducing antioxidant power (FRAP)

In the FRAP method, the polyphenol extract's ability to reduce ferric ions Fe^{3+} to Fe^{2+} reflects antioxidant capacity, which can be quantitatively assessed for varied samples. For 100 g, FRAP activity of Trolox Eq. was recorded. Higher FRAP concentrations indicated higher antioxidant potentials (Manasa et al., 2021).

In the present study, antioxidant capacities were determined by using the FRAP method. The antioxidant capacity of different samples of coffee is explained in the following Figures. FRAP of ground arabica coffee beans at 180 ^{o}C for 120 minutes ranged from 2687.6 to 1386.7 mg TE /100g dm as explained in Figure 4.9, and at 200 ^{o}C for 40 minutes ranged from 3550.3 to 2081.6 mg TE /100g dm as explained in Figure 4.10, at 220 ^{o}C for 20 minutes ranged from 3875.1 to 2094.5 mg TE/100g dm as explained in Figure 4.11 and Table 4.7.

These results showed a high content of antioxidant activity in 200 and 220 ^{o}C and increased antioxidants by increasing roasting temperature. The most stable temperature for antioxidant activity was at 200 ^{o}C, ranging from 3550.3 to 2081.6

mg TE /100g dm, and the lowest FRAP value reported in the samples was roasted at 180 °C.

4.9.2. 2,2-diphenyl-1-picrylhydrazyl (DPPH)

The DPPH radical scavenging technique was used to evaluate the antioxidant activity of untreated and microwave-roasted coffee beans (Salamatullah et al., 2022). This method is used because of the relatively fast reaction compared to other methods used for the same purpose. In addition, a change from purple to yellow in the colour of the solution occurs as a result of the reduction of the DPPH radical, which is an acceptor of hydrogen atoms from antioxidants (Nebesny and Budryn, 2003).

DPPH scavenging value of ground arabica coffee beans at 180 °C for 120 minutes ranged from 4316.1 to 4094.7 mgTE/100g dm, as explained in Figure 4.9, at 200 °C for 40 minutes ranged from 5493.6 to 5331.4 mgTE/100g dm, as explained in Figure 4.10, and at 220 °C for 20 minutes ranged from 5482.6 to 5318.6 mgTE/100g dm, as explained in Figure 4.11. Results show that by increasing the temperature, the DPPH value increases during the roasting process, which explains the degradation of antioxidant value by increasing the time and temperature.

The result of the present study, as shown in table 4.7, supports the conclusions of Nebesny and Budryn's (2003) research that detected the highest antioxidative capacity in green coffee beans. Compared to the convection roasting process, it has been determined that using microwaves for coffee roasting is less damaging to the antioxidative capacity of coffee beans, and microwaved samples were more active as antioxidants. The microwave roasting process of green coffee beans decreases the decline in DPPH scavenging capacity related to the greater total polyphenol content. Strong DPPH scavenging activity is indicated in roasted coffee beans using conventional or microwave methods. However, the initial

antioxidative capacity decreased due to the roasting processes. The concentration of polyphenols is most strongly connected with antioxidative effectiveness.

Similar results were reported by Nicoli et al. (1997), which showed that green coffee beans' higher antioxidative action is caused by their higher concentration of polyphenols. During the roasting process, polyphenols have been through chemical interactions or degradation, and some of their decomposition products, including phenylindane, have significant antioxidative activity. Also, Salamatullah et al. (2021) and Dogan et al. (2019) reported that untreated samples of coffee beans showed a significantly higher antioxidant activity than the microwave roasted coffee beans.

Perrone et al. (2012) and Opitz et al. (2017) are consistent with the present study that found that *Coffea arabica*'s antioxidant capacity decreased as roasting temperatures increased.

On the other hand, Hayat et al. (2010) and Ludwig et al. (2013) reported an increase in the DPPH scavenging of the roasted coffee. In addition, microwave roasting boosted the DPPH scavenging of other plant materials, including pomace and citrus peels, apricot kernels, and fennel seeds. It is believed that high-temperature processing causes an increase in the TPC of the plant materials, which increases their antioxidant activity. Furthermore, the Maillard reaction might occur due to the roasting process at high temperatures, producing several compounds that could enhance the product's antioxidant activities.

Microwaving plant materials can break down their cell walls and make it easier for phytochemicals to release from the plant matrix. This increases the content of bioactive components that are widely available, which increases the antioxidant activities of plant food materials (Salamatullah et al., 2022). Salamatullah et al. (2022) reported that microwave roasting protected the antioxidant capacity better than oven roasting. However, the same study found that pre-dried microwave-roasted coffee beans (750 W for 7.42 min) had higher TPC

and antioxidant activity than pre-dried convective-roasted beans (230 °C for 3.78 min).

Therefore, the compounds that appear or decompose during the roasting process of coffee beans explain the increase or loss of the antioxidant activities of roasted coffee beans. Although, as a result of the microwave roasting process, the TPC and antioxidant activity of the microwave-roasted coffee beans were significantly lower than untreated beans, even for a short time. Traditional heating at a high temperature of 120 °C causes the loss of 15–36% of the bioactive compounds (Salamatullah et al. 2021; Salamatullah et al. 2022)

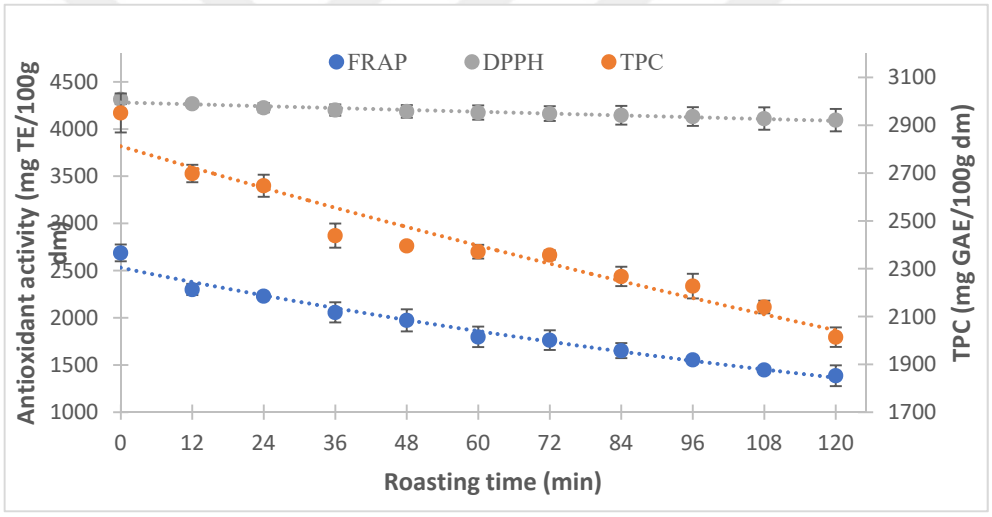

Figure 4.9. TPC and Antioxidant activity (DPPH and FRAP) value for roasted coffee beans at 180 °C, for 120 minutes

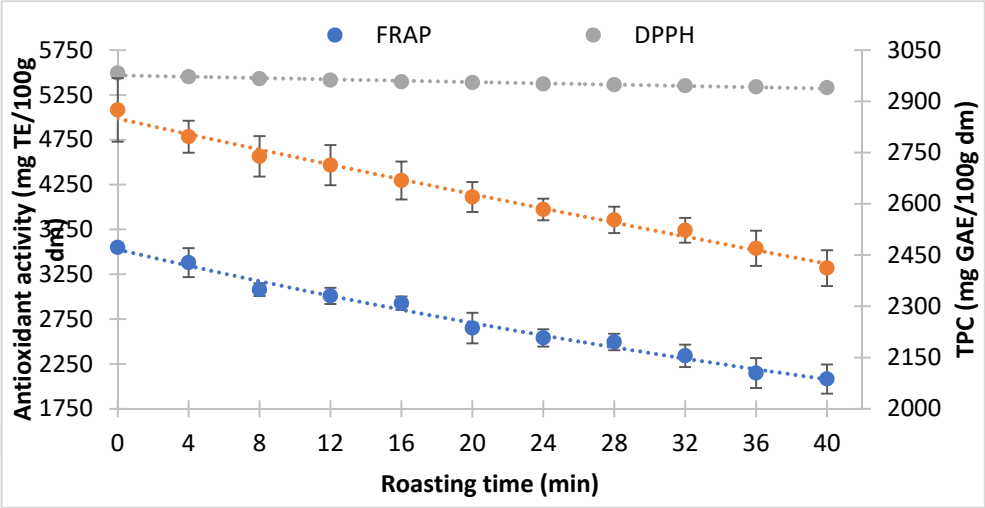

Figure 4.10. TPC and Antioxidant activity (DPPH and FRAP) value for roasted coffee beans at 200 °C, for 40 minutes

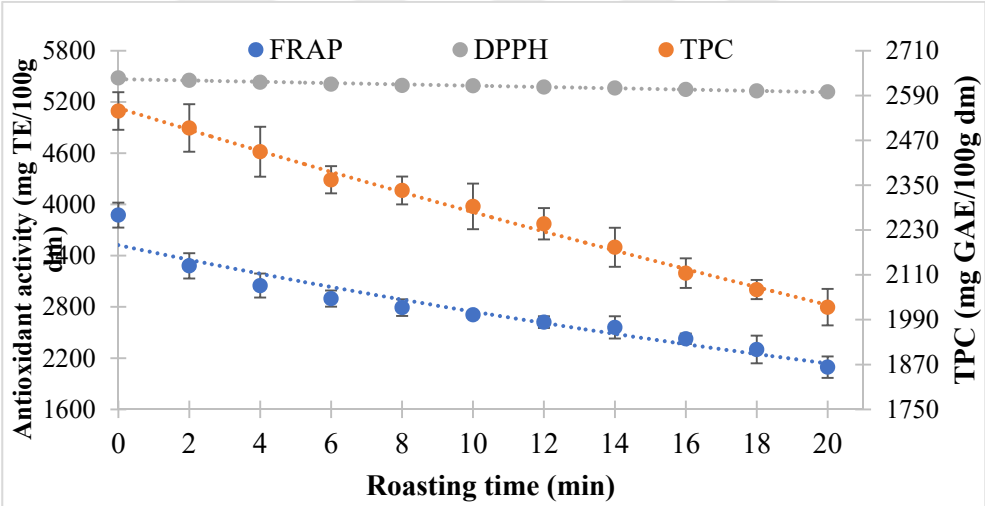

Figure 4.11. TPC and Antioxidant activity (DPPH and FRAP) value for roasted coffee beans at 220 °C, for 20 minutes

Table 4.7. Effect of microwave roasting at different times and temperatures on bioactive compounds.

Temperature	Time	FRAP (mgTE/100g dm)	DPPH (mgTE/100g dm)	TPC (mg/100g dm)
180 °C	**CUT**	2687.6±88.3^{a}	4316.1±51.8^{a}	2951.7±81.6^{a}
	12	2300.0±59.4^{b}	4267.5±30.2^{ab}	2698.2±36.8^{b}
	24	2229.1±19.5^{b}	4226.3±49.5^{abc}	2647.0±46.4^{b}
	36	2057.4±107.4^{c}	4201.4±59.4^{abc}	2438.4±50.3^{c}
	48	1973.1±117.2^{c}	4186.8±67.1^{abc}	2395.5±10.2^{cd}
	60	1797.7±108.6^{d}	4175.0±75.3^{abc}	2371.4±28.8^{d}
	72	1763.7±104.6^{d}	4163.3±77.9^{abc}	2357.4±19.1^{d}
	84	1651.9±79.3^{de}	4145.5±99.2^{bc}	2267.9±40.3^{e}
	96	1554.1±31.6^{ef}	4132.8±98.7^{bc}	2227.4±51.4^{e}
	108	1448.4±35.3^{fg}	4111.2±118.2^{bc}	2140.1±26.5^{f}
	120	1386.7±110.1^{g}	4094.7±118.4^{c}	2013.7±41.0^{g}
200 °C	**CUT**	3550.3±43.7^{a}	5493.6±44.5^{a}	2874.9±92.9^{a}
	4	3380.1±162.4^{a}	5453.3±25.7^{b}	2796.5±47.0^{b}
	8	3079.1±71.7^{b}	5431.4±14.5^{bc}	2739.0±59.3^{bc}
	12	3009.3±89.9^{b}	5415.5±18.2^{cd}	2713.3±58.7^{c}
	16	2926.8±75.0^{b}	5397.5±17.5^{cde}	2668.2±55.5^{cd}
	20	2650.1±170.2^{c}	5387.5±13.7^{def}	2620.0±43.6^{de}
	24	2540.7±96.2^{cd}	5371.9±9.8^{efg}	2583.4±31.6^{ef}
	28	2494.6±91.3^{cd}	5362.6±8.8^{efgh}	2553.0±38.5^{ef}
	32	2340.5±125.5^{de}	5352.5±4.1^{fgh}	2522.6±36.4^{fg}
	36	2148.8±167.1^{ef}	5340.8±8.8^{gh}	2469.8±51.4^{gh}
	40	2081.6±161.7^{f}	5331.4±12.3^{h}	2411.9±52.5^{h}
220 °C	**CUT**	3875.1±146.5^{a}	5482.6±31.5^{a}	2548.8±50.3^{a}
	2	3280.8±148.5^{b}	5453.9±10.4^{ab}	2503.2±63.7^{ab}
	4	3049.8±141.1^{c}	5432.0±14.0^{bc}	2439.8±66.8^{b}
	6	2897.4±95.5^{cd}	5409.2±22.4^{cd}	2364.6±86.5^{c}
	8	2791.7±97.8^{de}	5393.1±13.4^{cde}	2336.0±37.4^{c}
	10	2709.0±17.6^{def}	5389.5±13.7^{de}	2293.3±61.0^{cd}
	12	2621.6±69.0^{efg}	5375.6±29.2^{def}	2246.7±41.9^{de}
	14	2559.4±130.2^{fg}	5364.6±25.0^{efg}	2183.9±52.4^{ef}
	16	2426.5±61.0^{gh}	5347.0±14.1^{fgh}	2114.7±39.8^{fg}
	18	2300.5±162.1^{h}	5329.7±28.1^{gh}	2070.7±25.7^{gh}
	20	2094.5±126.1^{i}	5318.6±30.1^{h}	2023.5±48.8^{h}

Superscript letters on the same column indicate the significant difference between the roasting time for regarding temperature ($p < 0.05$). CUT: Come up time for target roasting temperature.

4.9.3. Kinetic outputs for degradation of bioactive compounds during roasting of coffee beans

Coefficient of determination (R^2) value of zero and first reaction orders and kinetic outputs of main bioactive compounds (FRAP, DPPH, and TPC) degradation in microwave roasted coffee samples at 180, 200, and 220 ^{o}C for processing time between 20 and 120 minutes is shown in Table 4.8. The average results of zero and first-order R^2 values indicated that first-order kinetic fits better to explain bioactive compound degradation in coffee samples than zero-order kinetic.

k values of bioactive compounds for thermally processed coffee samples are given in Table 4.8. *k* value was calculated for FRAP as 5.15, 13.23, 24.97 1/min for heat treatment at 180, 200 and 220 ^{o}C, respectively. *k* value was calculated for DPPH as 0.39, 0.69 and 1.41 1/min for heat treatment at 180, 200 and 220 ^{o}C, respectively. *k* value was calculated for TPC as 2.67, 4.04 and 11.54 1/min for heat treatment at 180, 200 and 220 ^{o}C, respectively.

$t_{1/2}$ values of bioactive compounds for thermally processed coffee samples are given in Table 4.8. $t_{1/2}$ value was calculated for FRAP as 134.7, 52.4 and 27.8 min for heat treatment at 180, 200 and 220 ^{o}C, respectively. $t_{1/2}$ value was calculated for DPPH as 1786.5, 1011.9 and 490.2 min for heat treatment at 180, 200 and 220 ^{o}C, respectively. $t_{1/2}$ value was calculated for TPC as 260.1, 171.7 and 60.0 min for heat treatment at 180, 200 and 220 ^{o}C respectively.

Ea values of bioactive compounds for thermally processed coffee samples are given in Table 4.8. *Ea* value was calculated for FRAP, DPPH and TPC as 73.5, 59.9 and 67.6 kJ/mol, respectively.

According to the kinetic study findings, the roasting temperature was directly proportional to the *k* value. Therefore, the higher roasting temperature was caused due to a higher *k* value. However, it is inversely proportional to the value of

$t_{1/2}$, which shows a decreasing tendency for the half-life time of compounds of coffee beans by increasing the temperature.

Table 4.8. Kinetic outputs for degradation of bioactive compounds during roasting of coffee beans

Compound	Temperature (°C)	Coefficient of determination (R^2)		Kinetic outputs*		
		Reaction order		k x 10^3	$t_{1/2}$	Ea
		0	1	(1/min)	(min)	(kJ/mol)
FRAP	180	0.956	0.984	5.15	134.7	73.5
	200	0.983	0.989	13.23	52.4	
	220	0.899	0.941	24.97	27.8	
DPPH	180	0.945	0.948	0.39	1786.5	59.9
	200	0.954	0.956	0.69	1011.9	
	220	0.976	0.977	1.41	490.2	
TPC	180	0.923	0.941	2.67	260.1	67.6
	200	0.989	0.991	4.04	171.7	
	220	0.996	0.996	11.54	60.0	

* The kinetic outputs were calculated according to the first-order kinetic model

4.10. Sugar degradation

Coffee carbohydrates decreased with increased roasting temperature and time (Redgwell and Fischer, 2006). Total changes in cell wall polysaccharide content of Arabica coffee beans occurring during roasting. After a long roasting, up to 40 % of the polysaccharides were degraded (Oosterveld et al., 2003).

The most abundant simple carbohydrate in green coffee beans, sucrose, interacts as an aroma precursor during roasting and produces a variety of compounds, including carboxylic acids, furans, and aldehydes that have an impact on coffee flavour. However, it has been discovered that sucrose degrades rapidly during the first stage of roasting (Wei and Tanokura, 2015).

The major source of the aliphatic acids (formic, acetic, glycolic, and lactic) produced during coffee roasting is sucrose. Glucose and fructose rearrange to give acid precursors due to the well-known thermal dehydration of these sugars (Wei and Tanokura, 2015).

The Carbohydrate contents of different samples of coffee beans (Sucrose, Glucose, and fructose) are explained in the following Figures. For example, sucrose content of ground arabica coffee beans at 180 ^{o}C for 120 minutes ranged from 5.28 to 0.58 g/100g dm as explained in Figure 4.12, at 200 ^{o}C for 40 minutes ranged from 2.49 to 0.66 g/100g dm as explained in Figure 4.13, at 220 ^{o}C for 20 minutes ranged from 2.26 to 0.44 g/100g dm as illustrated in Figure 4.14.

The fructose content of ground arabica coffee beans at 180 ^{o}C for 120 minutes ranged from 0.291 to 0.072 g/100g dm as explained in Figure 4.12, at 200 ^{o}C for 40 minutes ranged from 0.281 to 0.112 g/100g dm as explained in Figure 4.13, at 220 ^{o}C for 20 minutes ranged from 0.221 to 0.111 g/100g dm as explained in Figure 4.14.

Glucose-Fructose content of ground arabica coffee beans at 180 °C for 120 minutes ranged from 0.327 to 0.091 g/100g dm as explained in Figure 4.12, at 200 °C for 40 minutes ranged from 0.270 to 0.138 g/100g dm as explained in Figure 4.13, at 220 °C for 20 minutes ranged from 0.264 to 0.162 g/100g dm as explained in Figure 4.14.

The result of the present study supports the conclusions of Wei and Tanokura's (2015) research that the highest carbohydrate content in roasted coffee beans is sucrose. All carbohydrate content (sucrose, fructose and glucose) decomposes by increasing roasting temperature due to thermal reactions. These results shown in Table 4.9, reported that the high content of sucrose in 180 °C decreases by increasing roasting time and temperature.

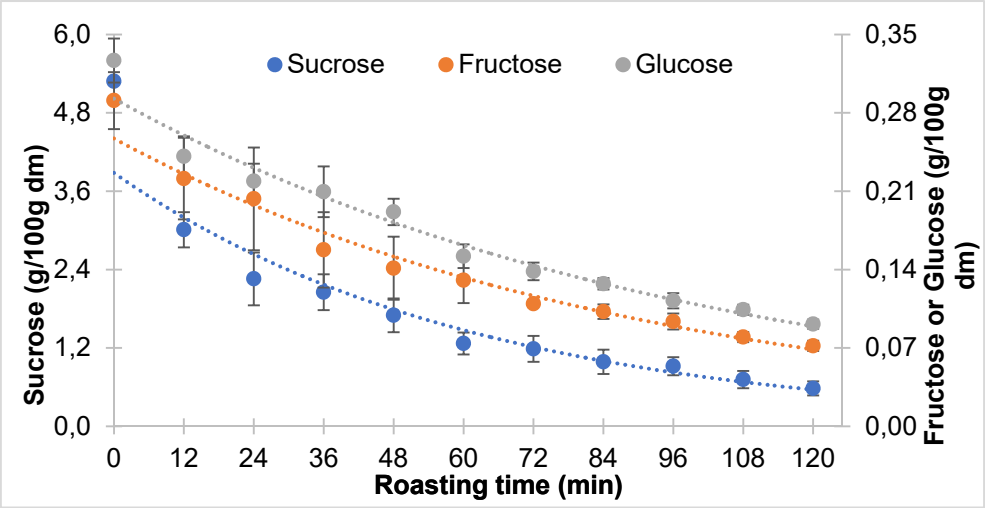

Figure 4.12. Sucrose, Fructose and Glucose contents for roasted coffee beans at 180 °C, for 120 minutes

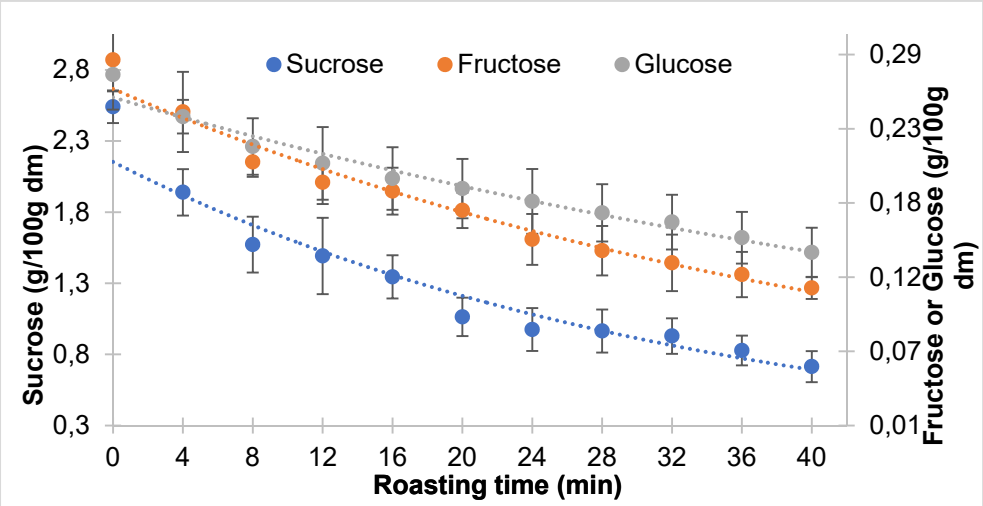

Figure 4.13. Sucrose, Fructose and Glucose contents for roasted coffee beans at 200 °C, for 40 minutes

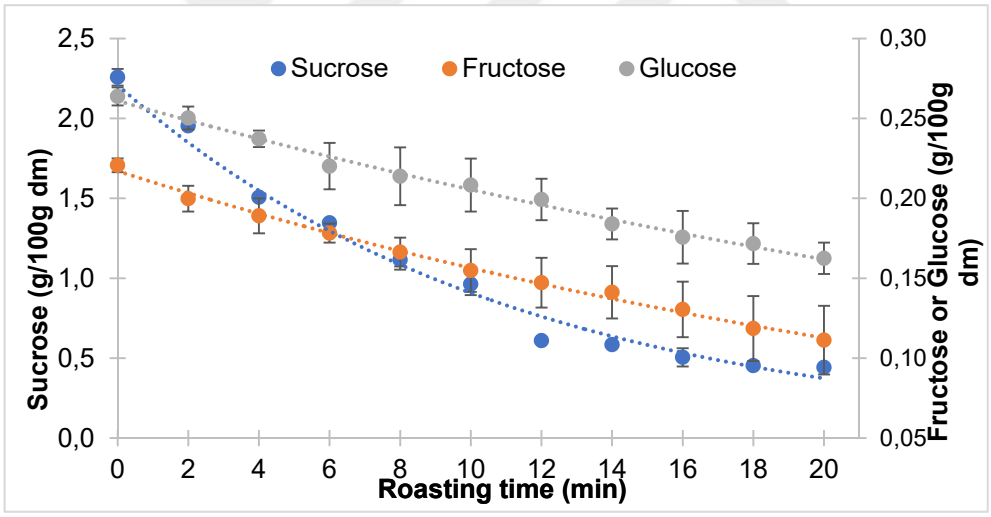

Figure 4.14 Sucrose, Fructose and Glucose contents for roasted coffee beans at 220 °C, for 20 minutes

Table 4.9. Effect of microwave roasting at different times and temperatures on sugar contents (sucrose, fructose and glucose).

Temperature	T (min)	Sucrose (g/100g dm)	Fructose (g/100g dm)	Glucose (g/100g dm)
180 °C	CUT	5.28±0.03^{a}	0.291±0.025^{a}	0.327±0.020^{a}
	12	3.01±0.27^{b}	0.221±0.036^{b}	0.241±0.018^{b}
	24	2.26±0.40^{c}	0.203±0.046^{b}	0.219±0.016^{bc}
	36	2.05±0.27^{cd}	0.158±0.034^{c}	0.209±0.023^{cd}
	48	1.70±0.26^{d}	0.141±0.028^{cd}	0.191±0.012^{d}
	60	1.27±0.17^{e}	0.130±0.020^{cde}	0.152±0.011^{e}
	72	1.19±0.20^{e}	0.109±0.002^{def}	0.138±0.008^{ef}
	84	0.99±0.19^{ef}	0.102±0.007^{def}	0.127±0.005^{fg}
	96	0.92±0.14^{efg}	0.094±0.007^{ef}	0.112±0.007^{gh}
	108	0.72±0.13^{fg}	0.080±0.004^{f}	0.104±0.004^{gh}
	120	0.58±0.11^{g}	0.072±0.005^{f}	0.091±0.004^{h}
200 °C	CUT	2.49±0.11^{a}	0.281±0.037^{a}	0.270±0.012^{a}
	4	1.89±0.16^{b}	0.242±0.030^{b}	0.239±0.013^{ab}
	8	1.52±0.20^{c}	0.205±0.011^{c}	0.217±0.021^{bc}
	12	1.44±0.27^{c}	0.190±0.016^{c}	0.204±0.027^{bcd}
	16	1.29±0.15c	0.184±0.017^{cd}	0.193±0.023^{cde}
	20	1.01±0.14^{d}	0.170±0.013^{cde}	0.186±0.022^{cdef}
	24	0.92±0.15^{ed}	0.148±0.019^{def}	0.176±0.024^{defg}
	28	0.91±0.15^{ed}	0.140±0.018^{ef}	0.168±0.021^{defg}
	32	0.88±0.12^{ed}	0.131±0.021^{f}	0.161±0.020^{efg}
	36	0.78±0.10^{ed}	0.122±0.017^{f}	0.149±0.019^{gf}
	40	0.66±0.11^{e}	0.112±0.008^{f}	0.138±0.018^{g}
220 °C	CUT	2.26±0.05^{a}	0.221±0.004^{a}	0.264±0.006^{a}
	2	1.95±0.02^{b}	0.200±0.008^{ab}	0.250±0.007^{ab}
	4	1.51±0.02^{c}	0.189±0.011^{bc}	0.237±0.005^{bc}
	6	1.35±0.01^{d}	0.178±0.006^{bcd}	0.220±0.015^{cd}
	8	1.12±0.06^{e}	0.166±0.009^{cde}	0.214±0.018^{d}
	10	0.96±0.07^{f}	0.155±0.013^{def}	0.208±0.017^{d}
	12	0.61±0.01^{g}	0.147±0.016^{ef}	0.199±0.013^{de}
	14	0.58±0.00^{g}	0.141±0.016^{efg}	0.184±0.010^{ef}
	16	0.51±0.06^{h}	0.131±0.017^{fgh}	0.176±0.016^{f}
	18	0.45±0.01^{h}	0.119±0.020^{gh}	0.172±0.013^{f}

Superscript letters on the same column indicate the significant difference between the roasting time for regarding temperature ($p < 0.05$). CUT: Come up time for target roasting temperature.

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4.10.1. Kinetic for degradation of sugar compounds

Coefficient of determination (R^2) value of zero and first reaction orders and kinetic outputs of main sugar compounds (sucrose, fructose and glucose) degradation in microwave roasted coffee samples at 180, 200 and 220 ^{o}C for processing time between 20 and 120 minutes is shown in Table 4.10. The average results of zero and first order R^2 values indicated that first order kinetic fits better to explain sugar compound degradation in coffee samples than zero order kinetic.

k values of sugar compounds for thermally processed coffee samples are given in Table 4.10. k value was calculated for sucrose as 16.14, 29.68 and 88.83 1/min for heat treatment at 180, 200 and 220 ^{o}C, respectively. *k* value was calculated for fructose as 11.0, 21.69 and 32.77 1/min for heat treatment at 180, 200 and 220 ^{o}C, respectively. k value was calculated for glucose as 9.90, 15.03 and 23.92 1/min for heat treatment at 180, 200 and 220 ^{o}C, respectively.

$t_{1/2}$ values of sugar compounds for thermally processed coffee samples are given in Table 4.10. $t_{1/2}$ value was calculated for sucrose as 42.9, 23.4 and 7.8 min for heat treatment at 180, 200 and 220 ^{o}C, respectively. $t_{1/2}$ value was calculated for fructose as 63.0, 32.0 and 21.2 min for heat treatment at 180, 200 and 220 ^{o}C, respectively. $t_{1/2}$ value was calculated for glucose as 70.0, 46.1 and 29.0 min for heat treatment at 180, 200 and 220 ^{o}C, respectively.

Ea values of sugar compounds for thermally processed coffee samples are given in Table 4.10. *Ea* value was calculated for sucrose, fructose and glucose as 78.8 50.8 and 40.9 kJ/mol, respectively.

Table 4.10. Kinetic outputs for degradation of sugar compounds during roasting of coffee beans

Compound	Temperature (°C)	Coefficient of determination (R^2)		Kinetic outputs*		
		Reaction order		k x 10^3	$t_{1/2}$	Ea
		0	1	(1/min)	(min)	(kJ/mol)
Sucrose	**180**	0.755	0.961	16.14	42.9	
	200	0.858	0.951	29.68	23.4	78.8
	220	0.916	0.973	88.83	7.8	
Fructose	**180**	0.895	0.98	11.00	63.0	
	200	0.933	0.981	21.69	32.0	50.8
	220	0.987	0.995	32.77	21.2	
Glucose	**180**	0.915	0.981	9.90	70.0	
	200	0.947	0.979	15.03	46.1	40.9
	220	0.984	0.992	23.92	29.0	

* The kinetic outputs were calculated according first order kinetic model.

4.11. Hydroxymethylfurfural (HMF) and furfural (F) formation

The amino-carbonyl reaction is the main pathway in the formation of furfural and HMF responsible for the browning of coffee beans. HMF content in samples of roasted coffee was found to be 17.97 to 115.34 µmol/100g dm. It was observed that HMF values increased significantly (p <0.05) at 180 ^{o}C until 48 minutes (72.16 µmol/100g dm), and after that decreased significantly (p < 0.05) as shown in Table 4.11.

HMF and furfural contents of different samples of coffee are explained in the following Figures. HMF of ground arabica coffee beans at 180 ^{o}C for 120 minutes ranged from 50.53 to 38.00 µmol/100g dm as explained in Figure 4.15, at 200 ^{o}C for 40 minutes ranged from 115.34 to 34.53 µmol/100g dm as explained in Figure 4.16, at 220 ^{o}C for 20 minutes ranged from 91.02 to 17.97 µmol/100g dm as explained in Figure 4.17. Furfural of ground arabica coffee beans at 180 ^{o}C for 120 minutes ranged from 5.52 to 7.12 µmol/100g dm as explained in Figure 4.15, at 200 ^{o}C for 40 minutes ranged from 35.05 to 11.23 µmol/100g dm as explained in Figure 4.16, at 220 ^{o}C for 20 minutes ranged from 15.76 to 5.12 µmol/100g dm as explained in Figure 4.17.

According to this study's results HMF and furfural formation kept continuous until determining degree and after that, it started to decompose significantly (p < 0.05) as shown in Table 4.11. Significantly (p < 0.05) highest HMF and furfural contents between all coffee samples was seen at 200 ^{o}C, 0 minute 115.34 µmol/100g dm and 35.05 µmol/100g dm, respectively, and significantly (p < 0.05) lowest HMF and furfural content values was seen at 220 ^{o}C, 20 minutes 17.97 µmol/100g dm and 5.12 µmol/100g dm, respectively.

The roasting process showed that the levels of furan and methyl furan are positively correlated with the roasting degree. However, the 5-HMF level quickly rose during the first stage of roasting and then dropped dramatically until roasting was completed. In an effort to reduce the content of the thermal contaminants, it was recommended to choose a medium roasting degree as the optimum roasting

condition when the 5-HMF concentration reaches its maximum value (Zhu et al., 2022).

Sucrose plays a main role in HMF formation in coffee during the roasting process. Glucose and a reactive fructofuranosyl cation are produced during the breakdown of sucrose. Rather than acrylamide synthesis, glucose plays a larger role in the formation of intermediates, specifically glyoxal and 3-deoxyglucosone (3-DG). On the other hand, the production of 5-hydroxymethylfurfural, which was discovered to be the most significant intermediate precursor of acrylamide generated in coffee after roasting, was mostly enhanced by the fructofuranosyl cation (Hamzalıoğlu and Gökmen, 2020).

Hamzalıoğlu and Gökmen (2020) reported that the content of acrylamide rose dramatically to an apparent maximum, and then decreased exponentially as a result of the rapid decrease of sucrose concentration. After 15 minutes of roasting at 240 °C, almost all the sucrose had been decomposed, whereas this process took 30 minutes at 220 °C and 45 minutes at 200 °C. In addition, asparagine concentration dramatically decreases during the roasting process, which is responsible for the formation of acrylamide, the second predominant amino acid in green coffee . Similarly, other free amino acids are rapidly depleted during the roasting process. The roasting process induces α-dicarbonyl compound formation in coffee beans. α-dicarbonyl compounds are formed with similar kinetics to acrylamide. It was observed that, during 200 °C roasting, significantly more α-dicarbonyl compounds were formed. The highest concentration of α-dicarbonyl compounds to be formed in coffee during roasting was 3-DG, which is a well-known intermediate in HMF formation through sugar degradation. The 3-DG concentration peaked after 10 minutes of roasting at 200 °C. Through sugar fragmentation, α-dicarbonyl compounds could be formed in the early stages of roasting; however, their concentration decreased in the final stages of roasting due to both their degradation and their involvement in the Maillard reaction's advanced stages, which leads to the formation of melanoidin. The concentration of HMF

increased quickly during roasting. According to the results, at all roasting temperatures, the concentration of HMF reaches its maximum when the concentration of 3-DG starts to decrease.

The formation of α-dicarbonyl compounds, including HMF as critical intermediates, was significantly influenced by the breakdown of sucrose into glucose and a highly reactive fructofuranosyl cation. It was observed that 3-DG formation from glucose, not Amadori/Heyns products, and HMF formation from fructofuranosyl cation, not 3-DG, were the processes that were quantitatively significant. Instead of using glucose and fructofuranosyl cation, HMF was used to produce acrylamide. Asparagine is thermodynamically more favourable for being converted into acrylamide rather than glucose as a result of the lower melting point of HMF. Considering that many other roasted nuts and seeds have compositional characteristics similar to coffee (low moisture, neutral pH, high free amino acids and proteins, limited reducing sugars, and relatively high sucrose), the details of the complex chemical mechanism described for roasted coffee could also be applicable to them.

Sucrose was found to be more effective in the production of HMF. HMF is produced from glucose through 3-DG synthesis, but sucrose produces it more effectively through fructofuranosyl cation. However, the production of HMF during roasting was significantly assisted by the dehydration of the fructofuranosyl cation. Furthermore, different studies have demonstrated that the efficiency of fructose in forming HMF is kinetically more important (Hamzalıoğlu and Gökmen, 2020).

Similar results were determined by Ağçam (2022), which reported that, in fruit juice the ability of fructose to induce HMF formation through the Maillard and caramelization reaction pathways is significantly higher. As a result of the main precursor of HMF, fructofuranosyl cation, being created when sucrose is hydrolyzed in an acidic medium at a high temperature.

The pathways of HMF formation in an acidic medium at a high temperature containing sugar and amino acids from glucose depend on the critical precursor compound, which is 3-deoxy glucosone, while from fructose it is fructofuranosyl cation. The fructofuranosyl cation is directly formed when fructose directly dehydrates from its cyclic form at high temperatures. While glucose can produce HMF by cyclizing 3-deoxyglucosone, it cannot produce HMF by dehydrating its cyclic form. Therefore, the rate of HMF generation from 3-deoxyglucosone disintegration is dramatically lower than that of the fructofuranosyl cation produced from the fructose moiety. The results showed that HMF formation continued till 90 °C, but samples higher than 90 °C significantly started to decompose. According to one explanation, HMF rapidly decomposes at temperatures above 90 °C in a medium made of fruit juice and amino acids. In other words, if the processing temperature is higher than 90 °C in the presence of an amino acid, the HMF concentration produced by the caramelization and Maillard reactions will change into another compound. According to these results, it can be concluded that HMF is an unstable compound under high temperatures and acidic conditions, especially when amino acids are present. As a result, it interacts with other substances in the medium and decomposes at a rapid rate (Ağçam, 2022).

Similar results were reported by Nebesny and Budryn (2003), which found that these compounds were intensively formed during the first stages of this process, and thereafter decreased. In the present study, the humidity of raw coffee beans affected HMF and furfural contents in roasted beans. Its higher concentrations were detected in the coffee samples dried prior to roasting. The huge influence of water content was observed on the synthesis of Maillard reaction products during microwaving, which agrees with this study’s results.

Table 4.11. Effect of microwave roasting at different times and temperatures on HMF and furfural (µmol/100g dm).

Temperature	Time(min)	HMF	Furfural
180 °C	CUT	50.53±3.07^{de}	5.52±1.91^{h}
	12	75.44±3.10^{b}	11.85±1.29^{bc}
	24	85.51±4.56^{a}	15.92±1.58^{a}
	36	73.69±0.28^{b}	14.78±2.13^{a}
	48	72.16±1.65^{b}	12.65±1.79^{b}
	60	55.63±0.07^{c}	11.22±0.86^{bcd}
	72	51.19±3.14^{d}	10.14±0.60^{cde}
	84	47.00±2.04^{ed}	9.62±0.93^{def}
	96	43.93±1.20^{f}	9.01±0.58^{efg}
	108	41.50±0.82^{gh}	7.96±0.25^{fg}
	120	38.00±4.35^{h}	7.12±0.65^{gh}
200 °C	CUT	115.34±3.62^{a}	35.05±2.30^{a}
	4	79.85±3.11^{b}	28.92±2.12^{b}
	8	74.72±1.95^{c}	23.32±1.52^{c}
	12	68.92±1.96^{d}	21.29±2.12^{d}
	16	55.37±0.75^{e}	17.21±0.74^{e}
	20	54.21±0.24^{e}	15.27±0.63^{ef}
	24	46.74±0.72^{f}	14.39±0.86^{fg}
	28	45.27±0.51^{f}	13.42±0.97^{fgh}
	32	38.67±1.18^{g}	12.67±0.42^{ghi}
	36	35.03±0.71^{h}	12.12±0.37^{hi}
	40	34.53±0.70^{h}	11.23±0.74^{i}
220 °C	CUT	91.02±0.95^{a}	15.76±0.71^{a}
	2	80.46±0.63^{b}	14.06±0.50^{b}
	4	71.80±2.14^{c}	13.42±0.48^{b}
	6	68.64±1.50^{d}	12.41±0.27^{c}
	8	52.35±0.75^{e}	9.62±0.34^{d}
	10	50.28±0.60^{f}	9.04±0.34^{d}
	12	25.53±0.21^{g}	8.15±0.78^{e}
	14	24.56±0.77^{g}	7.06±0.14^{f}
	16	22.67±1.01^{h}	6.65±0.21^{fg}
	18	19.88±1.40^{i}	6.02±0.71^{g}
	20	17.97±1.58^{j}	5.12±0.84^{h}

Superscript letters on the same column indicate the significant difference between the roasting time for regarding temperature ($p < 0.05$). CUT: Come up time for target roasting temperature.

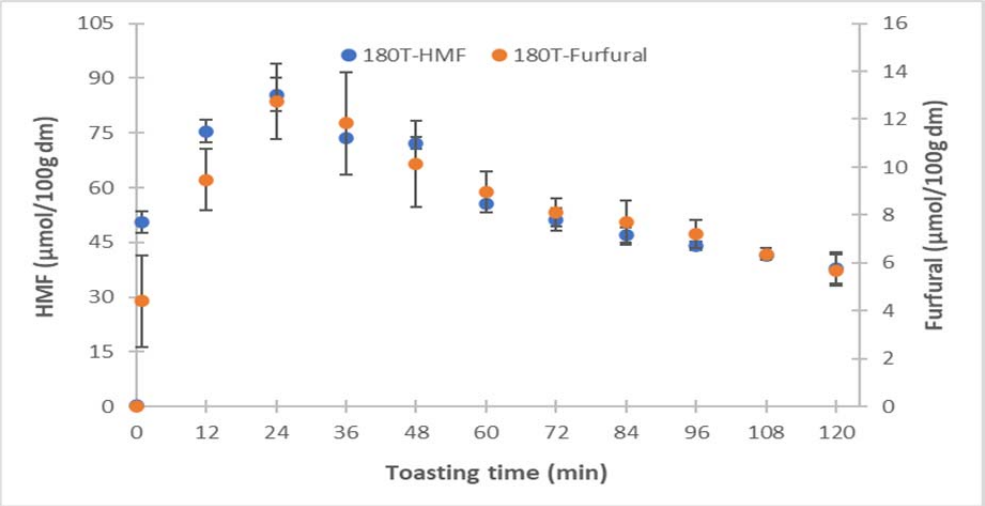

Figure 4.15. HMF and furfural values for roasted coffee beans at 180 °C for 120 minutes

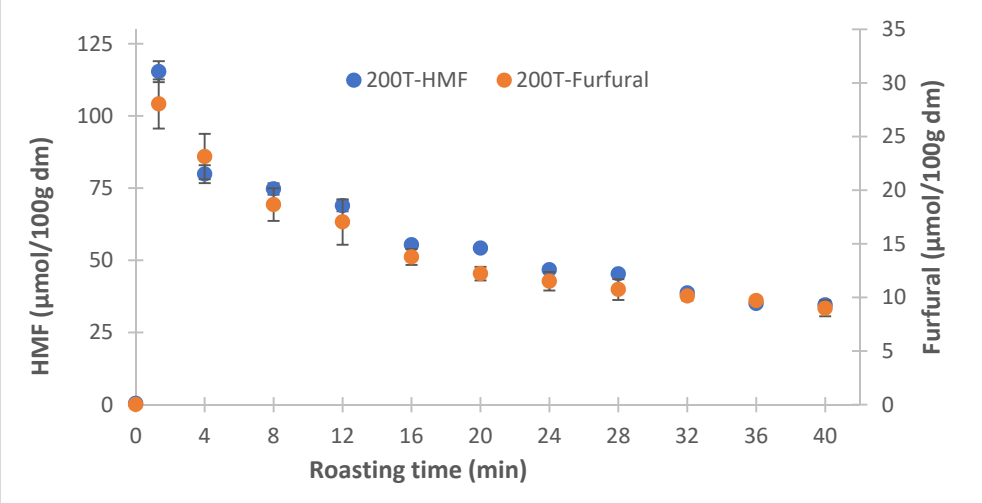

Figure 4.16. HMF and furfural values for roasted coffee beans at 200 °C for 40 minutes

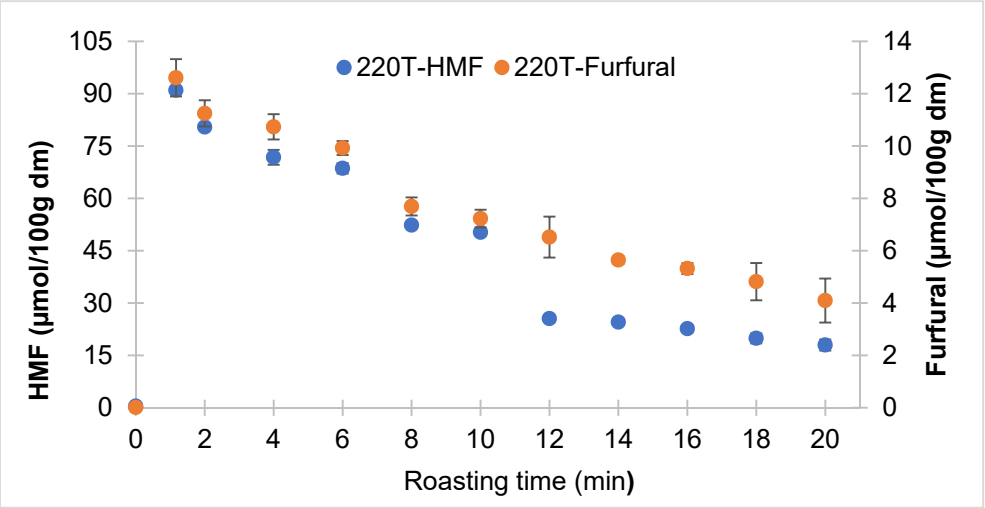

Figure 4.17. HMF and furfural values for roasted coffee beans at 220 oC for 20 minutes

4.11.1. Hydroxymethylfurfural (HMF) and furfural (F) formation kinetics during microwave roasting process

Coefficient of determination (R^2) value of zero and first reaction orders and kinetic outputs of hydroxymethylfurfural (HMF) and furfural (F) formation in microwave roasted coffee samples at 180, 200 and 220 °C for processing time between 20 and 120 minutes are shown in Table 4.12. The average results of zero and first order R^2 values indicated that first order kinetic fits better to explain sugar compound degradation in coffee samples than zero order kinetic.

k values of furfurals for thermally processed coffee samples are given in Table 4.12. k value was calculated for HMF as 0.81, 19.07 and 28.92 1/min for heat treatment at 180, 200 and 220 °C, respectively. *k* value was calculated for furfural as 0.38, 14.89 and 233.30 1/min for heat treatment at 180, 200 and 220 °C, respectively. k value was calculated for Unknown-1 as 0.0075, 0.0321 and 0.0889 1/min for heat treatment at 180, 200 and 220 °C, respectively. *k* value was calculated for Unknown-2 as 0.0062, 0.0331 and 0.0586 1/min for heat treatment at 180, 200 and 220 °C, respectively.

Ea value was calculated for HMF, furfural, Unknown-1 and Unknown-2 as 167.7, 300.1, 114.8 and 104.8 kJ/mol, respectively.

Roasting temperature is directly proportional to the value of *k*, in other words, the higher roasting temperature results in a higher *k* value for the formation and degradation of furfurals.

Table 4.12. Kinetic outputs for formation and degradation of furfurals during roasting of coffee beans

Compound	Temperature (°C)	Coefficient of determination (R^2)	Kinetic outputs	
			k **(1/min)**	***Ea*** **(kJ/mol)**
HMF	**180**	0.957	0.81	
	200	0.943	19.07	167.7
	220	0.968	28.92	
Furfural	**180**	0.914	0.38	
	200	0.959	14.89	300.1
	220	0.991	233.30	
Unknown-1 [C_{X1}]*	**180**	0.957	0.0075	
	200	0.943	0.0321	114.8
	220	0.968	0.0889	
Unknown-2 [C_{X2}]**	**180**	0.914	0.0062	
	200	0.959	0.0331	104.8
	220	0.991	0.0586	

* Unknown-1 is the unknown decomposition compound of HMF.
** Unknown-2 is the unknown decomposition compound of furfural.

4.12. Sensory Evaluation

Sensory attributes of microwave and traditional roasting coffee beans were determined according to the roasting degree (light, medium and dark), which included four determinants of taste (sweet, sour, astringent, bitter), aroma, and texture (body).

There is no significant difference between the sensory attributes of microwave roasted coffee and traditional roasted coffee as presented in Table 4.13 and Figure 4.18. The highest score of aroma was detected in T-Medium, acidity was detected in T-Light, bitterness was detected in MW-Dark, taste was detected in T-Medium, the body was detected in MW-Dark and T-Dark, and general impression was detected in MW-Light.

According to the results of this study there is a significant difference between traditional roasted coffee samples and microwave roasted coffee samples, because of that microwave roasting method can be an effective method for the future roasting processes.

Table 4.13. Effect of microwave roasting at different microwaved roasted coffee beans at three degrees (light, medium and dark) on sensorial attributes

Sample	Aroma	Acidity	Bitterness	Taste	Body	General impressions
MW-Light	5.7±2.0^{a}	4.7±1.6^{a}	5.0±1.9^{ab}	5.5±1.7^{ab}	4.7±1.9^{a}	5.6±2.1^{a}
MW-Medium	5.4±2.1^{a}	4.0±2.0^{a}	6.1±1.7^{a}	4.3±1.7^{b}	5.1±1.7^{a}	4.8±2.1^{a}
MW-Dark	5.7±2.1^{a}	4.6±1.7^{a}	6.2±1.7^{a}	5.1±1.8^{ab}	5.6±1.5^{a}	4.5±2.2^{a}
T-Light	5.0±1.7^{a}	4.9±1.9^{a}	4.1±2.4^{b}	5.1±2.1^{ab}	4.2±1.9^{a}	5.0±1.6^{a}
T-Medium	5.9±1.6^{a}	4.8±2.1^{a}	4.8±1.9^{ab}	5.8±1.5^{a}	5.0±1.7^{a}	5.5±1.9^{a}
T-Dark	5.8±2.1^{a}	4.2±1.9^{a}	5.7±1.9^{a}	4.5±2.0^{ab}	5.6±1.8^{a}	5.3±2.1^{a}

*MW is microwave roasting of coffee beans.
** T is traditional roasting of coffee beans.

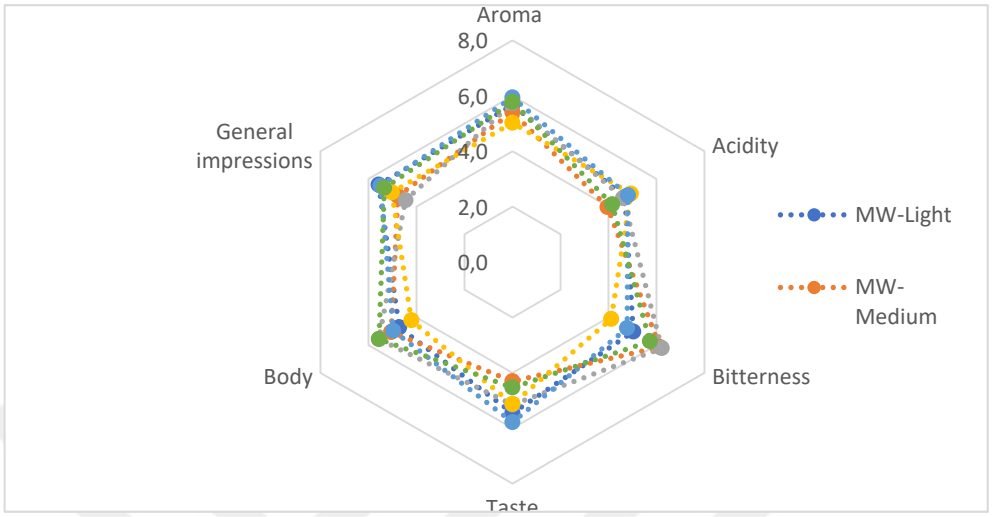

Figure 4.18. Sensory analysis for microwave and traditional roasted coffee samples

5. CONCLUSION

In the present study, Arabica coffee beans were successfully roasted in a microwave oven for three temperatures (180, 200, and 220 ^{o}C) and various times (0-120 min). When all the collected results were evaluated together, the following highlighted results are summarised below:

- L* value is a good indicator in determining the roasting degree of coffee beans, and it decreased remarkably by increasing roasting temperature and time. Similarly, the browning index value decreased during the roasting process values significantly by increasing the temperature.
- The roasted coffee beans obtained higher dry matter contents and lower a_w values.
- Chlorogenic acid (5-CQA) and caffeic acid (CA) were the most abundant phenolic compounds in all the coffee samples, and 5-feruloylquinic acid (5-FQA) had the lowest concentration.
- Phenolic compounds decreased with increased roasting time and temperature, and moderate roasting conditions (Light or medium roasting) could be the best way to obtain a final product rich in phenolic compounds.
- Caffeine content increased by increasing roasting time and temperature.
- Total phenolic content and antioxidant activities of coffee beans had decreasing tendency according to roasting conditions. The kinetic study findings indicated that higher degradation rate constant values were calculated for the samples roasted at the higher temperature.
- The most abundant basic sugar component was determined as sucrose in coffee beans, followed by fructose and glucose sugars. Due to caramelization and Maillard reactions, the main sugar content in coffee beans (sucrose, fructose, and glucose) decomposed by increased roasting temperature. According to the decomposition reaction rate values of sugar

compounds, the roasting temperature and time dramatically affected the decomposition of basic sugars.

- It was observed that HMF and furfural increased to a certain level, but they degraded remarkably by increasing time and temperature. A higher decomposing rate was determined for coffee beans roasted at a higher temperature. The results highlighted that the formation of furfurals was induced at the beginning of the roasting process. Then they decreased with a certain tendency due to low water activity and high temperature. Moreover, furfurals are not stable under these conditions and decompose remarkably to the further compounds.
- *k* value was calculated for HMF as 0.81, 19.07 and 28.92 1/min, for furfural as 0.38, 14.89 and 233.30 1/min for heat treatment at 180, 200 and 220 ^{o}C, respectively. *Ea* value was calculated for HMF, furfural, Unknown-1 and Unknown-2 as 167.7, 300.1, 114.8 and 104.8 kJ/mol, respectively.
- According to sensory evaluation (general impressions), the highest score was obtained for light coffee beans roasted under microwave conditions, followed by the medium traditional roasted coffee beans.

The microwave roasting method can be used to roast coffee beans as a result of producing high-quality products with high nutritional value. In addition, microwave roasting can be used to quickly obtain coffee beans with various roasting degrees. Also, the results highlighted that HMF and furfural are not stable under roasting conditions. Therefore, it can be recommended to reveal decomposition compounds (cited as unknown compounds in the present study) of HMF and furfural via caramelization and Maillard reaction pathways. On the other hand, it is necessary to focus on the physical properties of coffee beans and the best position inside microwave ovens in large quantities, making this method more acceptable for food processing. Finally, it is important to study the shelf-life

stability of coffee beans roasted under microwave conditions and to compare them with traditionally roasted coffee beans.

Lastly, the microwave roasting process positively affects the coffee beans by increasing the content of bioactive compounds in the coffee beans. However, extreme microwave-based roasting conditions (>200 ^{o}C) and roasting times negatively impacted the quality attributes of coffee beans. Therefore, microwave roasting at 200 ^{o}C can be suggested as roasting conditions for collecting coffee beans with different roasting degrees (such as light, medium and dark), reducing roasting time, meeting consumer expectations, and saving total processing energy.

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CURRICULUM VITAE

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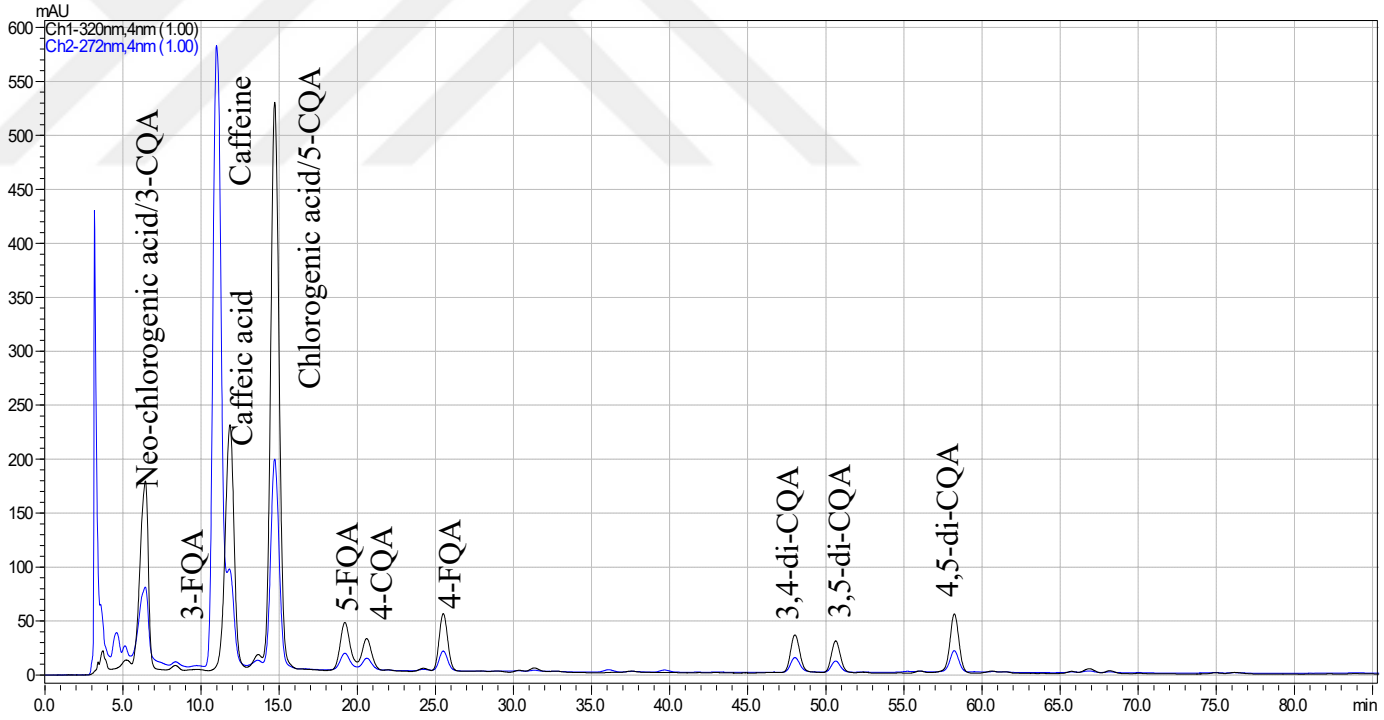
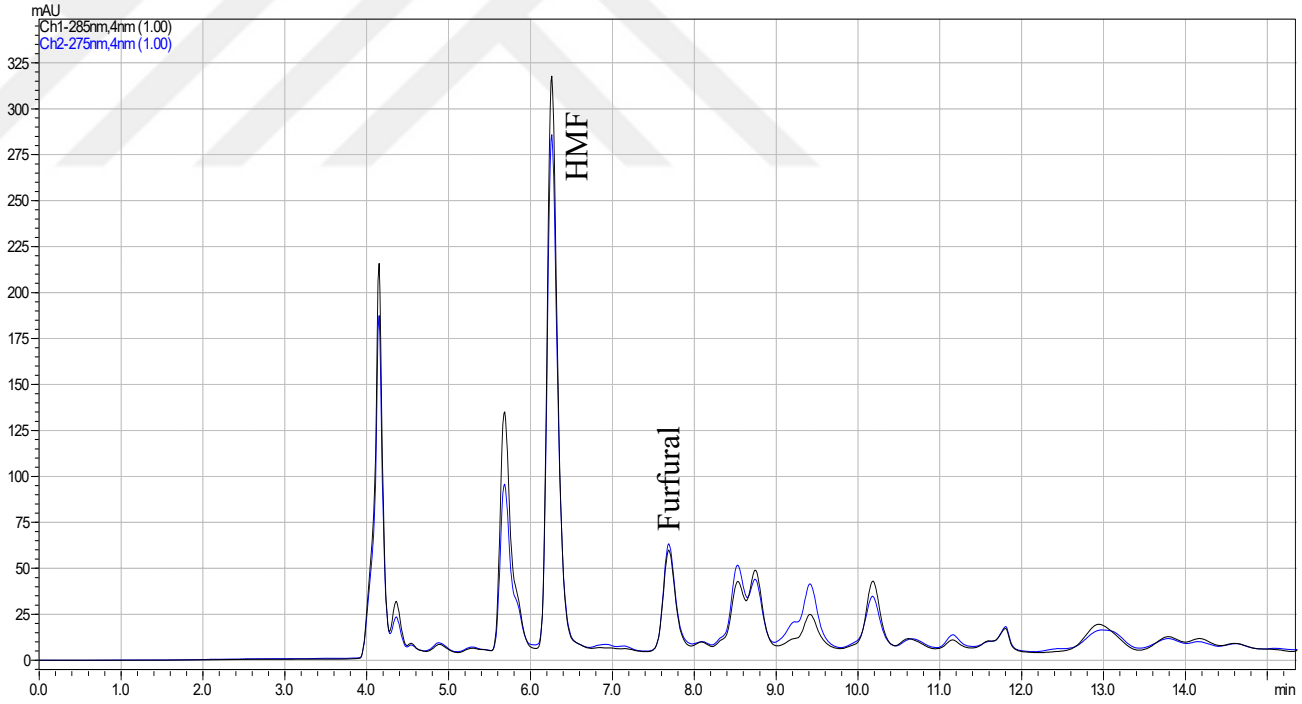
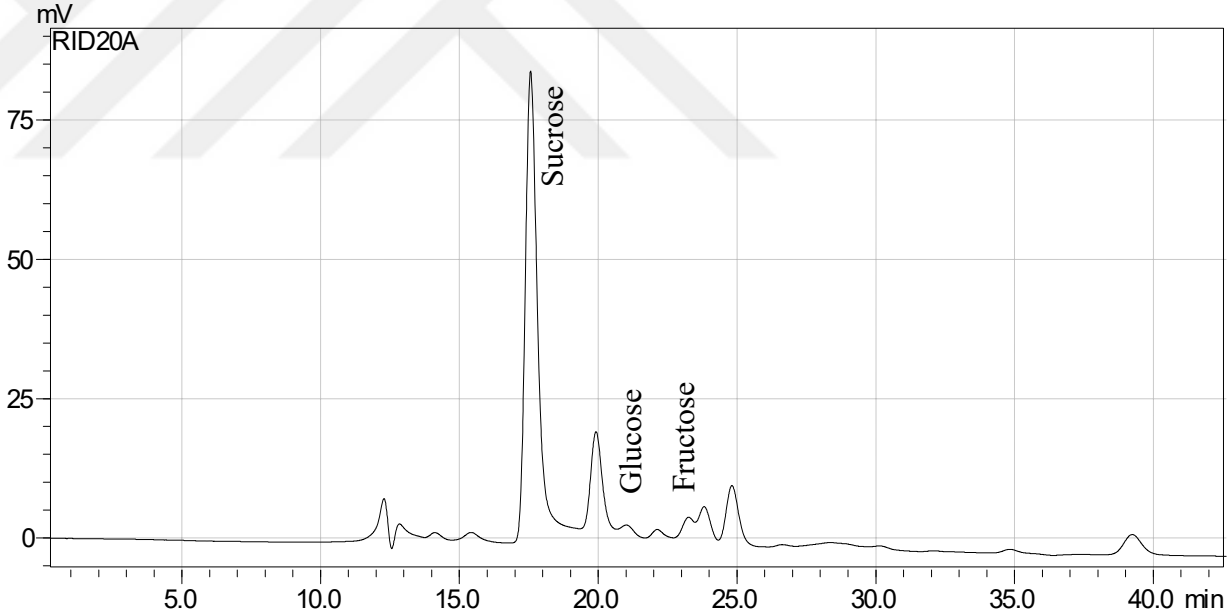
APPENDICES



Appendix 1. HPLC chromatograms of caffeine and phenolic compounds at 272 and 320 nm



Appendix 2. HPLC chromatograms of HMF and Furfural at 275 and 285 nm



Appendix 3. HPLC chromatogram of sugar compounds