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GEBZE TECHNICAL UNIVERSITY

GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

INVESTIGATION of PROCESSING PARAMETERS for INSTANT PISTACIA TEREBINTHUS COFFEE

SİBEL BÖLEK

A THESIS SUBMITTED FOR THE DEGREE OF

DOCTOR OF PHILOSOPHY

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PROF. DR. MURAT ÖZDEMİR

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T.C.
GEBZE TEKNİK ÜNİVERSİTESİ
FEN BİLİMLERİ ENSTİTÜSÜ

TÜKETİME HAZIR PISTACIA TEREBINTHUS KAHVESİ İÇİN İŞLEM PARAMETRELERİNİN ARAŞTIRILMASI

SİBEL BÖLEK

DOKTORA TEZİ
KİMYA MÜHENDİSLİĞİ ANABİLİM DALI

DANIŞMANI
PROF. DR. MURAT ÖZDEMİR

GEBZE
2018

GEBZE TEKNİK ÜNİVERSİTESİ

DOKTORA JÜRİ ONAY FORMU

GTÜ Fen Bilimleri Enstitüsü Yönetim Kurulu'nun 25/10/2017 tarih ve 2017/55 sayılı kararıyla oluşturulan jüri tarafından 16/03/2018 tarihinde tez savunma sınavı yapılan Sibel Bölek'in tez çalışması Kimya Mühendisliği Anabilim Dalında DOKTORA tezi olarak kabul edilmiştir.

JÜRİ

ÜYE (TEZ DANIŞMANI)	: Prof. Dr. Murat Özdemir
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ONAY

Gebze Teknik Üniversitesi Fen Bilimleri Enstitüsü Yönetim Kurulu'nun

...../...../..... tarih ve ...../...../..... sayılı kararı.

SUMMARY

P. terebinthus beans were roasted by a fluidized bed roaster at 180 °C, 200 °C and 220 °C for 5, 20 and 35 min and microwave oven at 360 W, 540 W and 720 W for 5, 11 and 17 min at each roasting level. The effects of the roasting conditions on color, moisture content, density, breaking force and sensory properties of the *P. terebinthus* beans were investigated using a three-level two factor (3^2) full factorial design. The effect of roasting temperature/power and roasting time on quality attributes of *P. terebinthus* beans was analyzed using response surface methodology. Quality characteristics of the roasted *P. terebinthus* beans were affected by the roasting temperature/power and roasting time. Optimum roasting conditions for fluidized bed and microwave roasting of *P. terebinthus* beans were determined. Chemical, structural and thermal analysis of roasted beans were performed. The beans roasted at optimum region were used in the production of instant *P. terebinthus* coffee and investigation of processing parameters for the instant *P. terebinthus* coffee. Oil content of the roasted *P. terebinthus* beans was reduced to obtain instant *P. terebinthus* coffee. Color, particle size distribution, total soluble solids content, total oil content, moisture content, pH and antioxidant activity of the instant *P. terebinthus* coffee were determined. Acceptability tests for the instant *P. terebinthus* coffee beverage were done. The instant *P. terebinthus* coffee beverages obtained from the fluidized bed roasted beans and microwave roasted beans were found to be not different in terms of appearance, flavor, aftertaste and overall impression.

Keywords: *P. terebinthus*, instant coffee, roasting, optimization, sensory properties.

ÖZET

Menengiç taneleri akışkan yatak kurutucuda 180 °C, 200 °C ve 220 °C sıcaklıklarda her bir sıcaklıkta 5, 20 ve 35 dakika ve mikrodalga fırında 360 W, 540 W ve 720 W güç seviyelerinde her bir güç seviyesinde 5, 11 ve 17 dakika kavruldu. Kavurma koşullarının menengiç tanelerinin renk, nem içeriği, yoğunluk, kırılma kuvveti ve duyusal özellikleri üzerine etkisi iki faktörlü üç seviyeli (3^2) tam faktöriyel tasarım kullanılarak araştırıldı. Kavurma sıcaklığı/mikrodalga gücü ve kavurma süresinin menengiç tanelerinin kalite özellikleri üzerindeki etkisi yanıt yüzey yöntemi kullanılarak analiz edildi. Kavrulmuş menengiç tanelerinin kalite özellikleri kavurma sıcaklığı/mikrodalga gücü ve kavurma süresinden etkilendi. Akışkan yatak kavurma ve mikrodalga kavurma için optimum kavurma koşulları belirlendi. Kavrulmuş tanelerin kimyasal, yapısal ve ısısal analizleri yapıldı. Optimum bölgede kavrulmuş taneler tüketime hazır menengiç kahvesinin üretiminde ve işlem parametrelerinin araştırılmasında kullanıldı. Tüketime hazır menengiç kahvesi elde etmek için menengiç kahvesinin yağ içeriği azaltıldı. Tüketime hazır menengiç kahvesinin rengi, partikül boyut dağılımı, toplam çözünür katı madde içeriği, toplam yağ içeriği, nem içeriği, pH ve antioksidan aktivitesi belirlendi. Tüketime hazır menengiç kahvesi içeceği için kabul edilebilirlik testleri yapıldı. Akışkan yatak kavurucuda ve mikrodalga fırında kavrulmuş tanelerden elde edilen tüketime hazır menengiç kahvesi içeceklerinin görünüş, lezzet, ağızda kalan tat ve genel izlenim açısından farklı olmadığı belirlendi.

Anahtar Kelimeler: Menengiç, hazır kahve, kavurma, optimizasyon, duyusal özellikler.

ACKNOWLEDGEMENTS

First and above all, I praise God, the almighty for providing me this opportunity and granting me the capability to proceed successfully. I would also like to present my sincere and deepest thanks to my advisor Prof. Dr. Murat Özdemir for his supervision, continuous guidance and invaluable suggestions.

I am also thankful to all my committee members Prof. Dr. Hasan Sadıkoğlu, Prof. Dr. Alp Er Konukman, Assoc. Prof. Dr. Rezan Demir Çakan and Assoc. Prof. Dr. İbrahim Doymaz for their help, support and comments.

I would like to extent my sincere thanks to all the laboratory staff that I worked together in different departments at Gebze Technical University. I also thank my roommates Olcay Eren, Salih Özbay and Eda Hazal Baran for making the atmosphere of our room as friendly as possible.

A special note of appreciation goes to my parents for always believing in me, their continuous help and encouragement.

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LIST of ABBREVIATIONS and ACRONYMS

Abbreviations and Acronyms		Explanations
µg	:	Microgram
µm	:	Micrometer
µM	:	Micromolarity
cm	:	Centimeter
Hz	:	Hertz
h	:	Hour
min	:	Minute
ml	:	Milliliter
mm	:	Millimeter
MPa	:	Megapascal
N	:	Newton
nm	:	Nanometer
p	:	Power
s	:	Second
T	:	Temperature
t	:	Time
T_g	:	Glass Transition Temperature
W	:	Watt
x_i	:	Factors
y_n	:	Responses
β_k's	:	Constant Coefficients
ANOVA	:	Analysis of Variance
AOAC	:	Association of Official Analytical Chemists
CIE	:	International Commission on Illumination
DPPH	:	1,1-Diphenyl-2-picrylhydrazyl
DSC	:	Differential Scanning Calorimeter
FTIR	:	Fourier Transform Infrared
ISO	:	International Organization for Standardization
PCC	:	Pearson Correlation Coefficient

RCCS	:	Roast Color Classification System
RSM	:	Response Surface Methodology
SCAA	:	Specialty Coffee Association of America
SEM	:	Scanning Electron Microscopy
TG	:	Thermogravimetric
TPC	:	Total Phenolic Content
XRD	:	X-Ray Diffraction

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1. INTRODUCTION

Coffee is the second most important commodity of world trade after crude oil, and one of the most popular beverages consumed throughout the world [Yeretzian et al., 2002]. Coffee consumption is a routine part of daily life throughout the world [Homan and Mobarhan, 2006]. The characteristic aroma and flavor of coffee make it a unique beverage and more than 800 volatile compounds have been identified in roasted coffee [Baggenstoss et al., 2008].

With the increase of coffee consumption, people seek a caffeine-free coffee due to the negative effects of caffeine [Ozel et al., 2014]. To reduce negative effects of caffeine, ideal approach is to increase the consumption of decaffeinated coffee, but decaffeinated coffee is not completely caffeine free, and the color may change depending on the method used for decaffeination [Ramalakshmi and Raghavan, 1999]. Alternatively, some herbal coffees are present in the market to reduce negative side effects of excessive coffee consumption, but their consumption is very limited because their taste is different from the typical coffee taste. Since *Pistacia terebinthus* fruit after roasting has a similar aroma and flavor to conventional roasted coffee beans, it can be a promising caffeine-free alternative. In addition, *P. terebinthus* had a high antioxidant capacity, and it may show a preventive role in cancer risks by eliminating the attacks of free radicals [Kavak et al., 2010].

1.1. Hypothesis

Studies to investigate the effect of roasting conditions on the quality attributes of *P. terebinthus* and studies aiming to optimize the roasting conditions of green *P. terebinthus* beans are lacking. Therefore, roasting conditions of the *P. terebinthus* beans in a fluidized bed roaster and microwave oven can be optimized to obtain roasted *P. terebinthus* beans with desired quality characteristics from which instant *P. terebinthus* coffee can be obtained using proper production stages and processing conditions.

1.2. Objectives

The objectives of this study are to:

- develop predictive models for fluidized bed and microwave roasting of *P. terebinthus* beans,
- determine the optimal roasting conditions by using response surface methodology (RSM),
- investigate the effect of roasting conditions on the quality characteristics of the roasted *P. terebinthus* beans,
- reduce the oil content of *P. terebinthus* beans,
- obtain instant *P. terebinthus* coffee powder and instant *P. terebinthus* coffee beverage from the roasted *P. terebinthus* beans.

2. COFFEE and COFFEE PROCESSING

Coffee belongs to the botanical family Rubiaceae which has more than 90 different species [Davis, 2001]. However, only Coffea Arabica (Arabica), Coffea canephora (Robusta) and Coffea liberica are economically most important species [Schenker, 2000]. Commercial coffee beverage is made of Coffea arabica (Arabica) and Coffea canephora (Robusta) [Bertrand et al., 2003]. Coffea liberica is of minor interest in the market [Pablos et al., 1999]. Arabica beans are more expensive and more valuable because of their smooth, rounded flavor and produce a better tasting beverage [Defernez et al., 2017], [Zambonin et al., 2005]. Coffee is a sempervirent tree which grows 1-3 m tall. The taste of raw coffee beans changes after roasting [Vincze and Vatai, 2004]. Aroma, flavor and acidity of coffee beans may vary according to countries or regions [Davids, 2001]. These differences can originate not only from the coffee's growing region but also from processing conditions and genetic subspecies [Castle, 1991].

Coffee is the second most popular drink after water [Illy and Pizano, 2003]. Coffee is an important commodity in world trade because it ranks second after crude oil throughout the world [Esquivel and Jimenez, 2012]. Global coffee consumption has increased at an average annual rate of 1.9% over the past 50 years [Lee et al., 2015]. Economic importance of coffee originates from the coffee brew or beverage.

Coffee processing can be described as converting the raw fruit plant into the green coffee. Traditionally, green coffee is produced by two methods, wet and dry processing. In wet processing coffee beans are mechanically depulped while in dry processing the entire coffee beans are dried before all layers surrounding the seeds are removed in one husking step. High quality coffee is obtained by wet processing, but dry processing is an inexpensive process and easy, and it produces coffee with low quality [Mussatto, 2015].

Spent coffee and silverskin are generated during coffee processing. These are the two residues of coffee processing [Mussatto, 2015]. A schematic representation of spent coffee and silverskin generation during coffee fruit processing is given in Figure 2.1 [Mussatto, 2015].

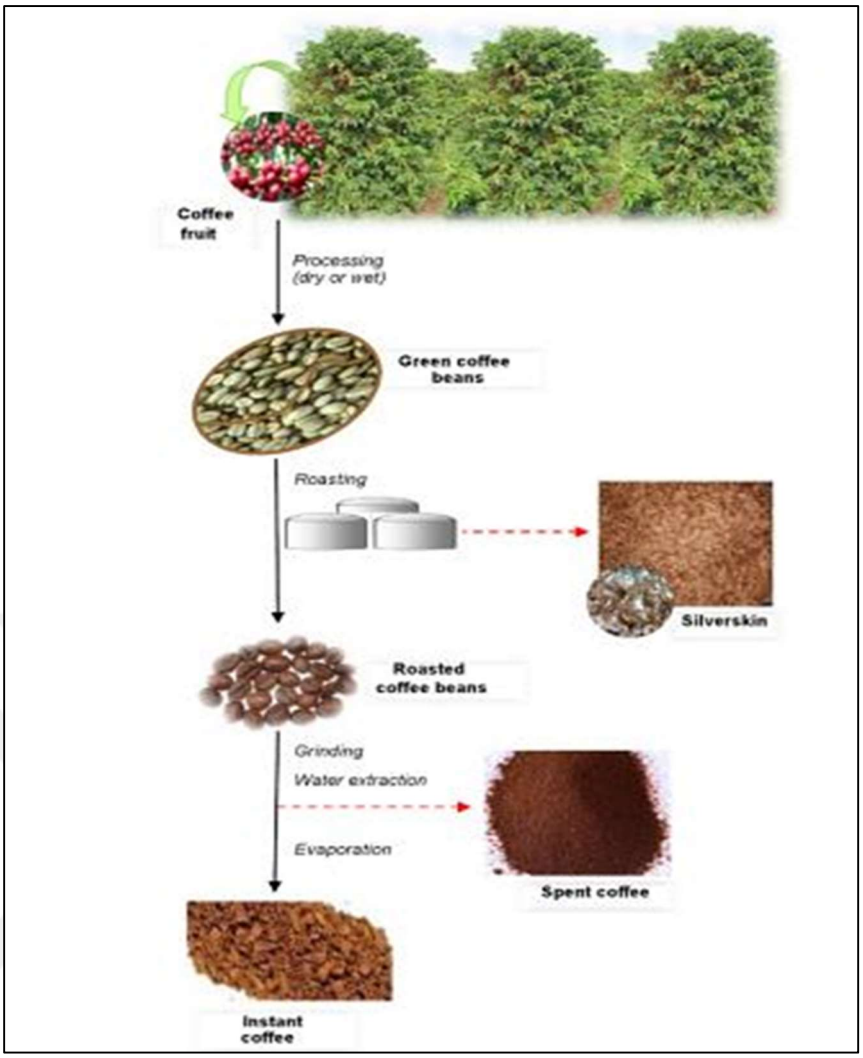


Figure 2.1: Schematic representation of spent coffee and silverskin generation during coffee fruit processing.

Soluble coffee, also called “instant coffee” and coffee powder production have risen significantly during the last decades [Pujol et al., 2013]. Practical use and neat processing used in preparing the beverages resulted in an increase in production of instant coffee and coffee powder production [Capek et al., 2013]. A typical composition of an instant coffee powder is given in Table 2.1 [Bhandari et al., 2013]

Table 2.1: Composition of an instant coffee powder.

Constituent	Content (% dry weight basis)
Minerals	7.6-14.6
Reducing sugars (glucose)	3.2-13.1
Galactomannan	2.4-10.5
Low molecular organic acids	12
Brown pigments	15-28
Caffeine	2.5-5.4
Trigonelline	1.56-2.65

The production of instant coffee mainly involves 5 stages (Figure 2.2) [Gerhartz et al., 1985].

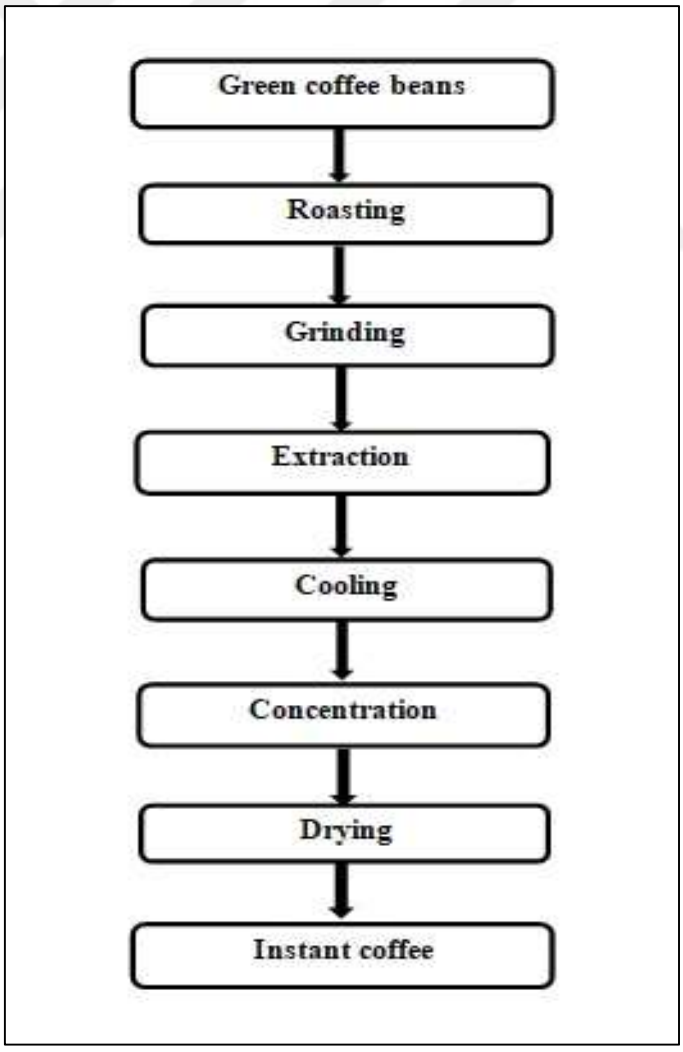


Figure 2.2: Schematic diagram of the production process of instant coffee.

2.1. Roasting of Coffee Beans

After the coffee beans are harvested, they are roasted. Since roasting produces marked chemical, physical, structural and sensorial changes, it is apparently the most important stage in coffee processing [Alessandrini et al., 2008]. The quality of coffee used for beverages is closely related to roasting conditions which produces the characteristic aroma and flavor of coffee resulted from a combination of hundreds of chemical compounds [Franca et al., 2005], [Jokanović, et al., 2012a]. Therefore, it is critical to determine the appropriate roasting method and roasting conditions for desirable roasted coffee aroma [Buffo and Cardelli-Freire, 2004].

2.1.1. Coffee Roasting Methods

Conventional roasting, pan roasting, microwave roasting, ultra-fast roasting, and fluidized bed roasting are the common methods used for coffee roasting. Conventional roasting is carried out at high temperatures with long times. Pan roasting is based on conduction heating and causes uneven roasting. In the conventional and pan roasting, outer surface of the beans can be over-roasted whilst the core of the beans is not roasted [Nebesny et al., 2007]. This causes non-homogeneous roasting, surface burns and undesirable aroma and flavor development. Although it is possible to obtain roasted beans rapidly using ultra-fast roasting, the desirable aroma and flavor components do not usually develop during that process. Traditional pan roasting is usually preferred due to its simplicity for the roasting, but this method does not provide uniformly roasted products. On the other hand, fluidized bed roasters operate based on high temperature short time principle, and fluidized bed roasting provides intense bean movement allowing uniform roasting of beans because heat from the air flow goes directly to the surface of each bean. This produces desirable aroma and flavor for the roasted beans. Use of microwaves offers advantages of speed of operation, energy savings, precise process control, and faster start up and shut-down times as compared with the conventional heat processes [Megahed, 2001]. Since microwaves operate directly in the core of the beans, the process of roasting is intensified throughout the whole interior of the bean. Nebesny et al. particularly coupled convective heating with microwave [Nebesny et al. 2007]. Microwaving considerably reduced the ultimate temperature

of roasted coffee beans. The application markedly decreased the time of roasting, and total amounts of volatile substances of roasted coffee increased. Nebesny and Budryn roasted robusta coffee beans convectively at 230 °C, microwaved at 700 W, and roasted by the coupled convective-microwave method. The best sensory properties were obtained by microwaving at 700 W [Nebesny and Budryn 2006].

2.1.2. Degree of Roasting

Roasted coffee commonly categorized as light, medium and dark roasted. A scale to rate the level of roasting in coffee products was developed by The Specialty Coffee Association of America (SCAA). SCAA values or more commonly known as Agtron numbers are precise industry standard generally used to determine the level of roasted coffee. The Agtron-SCAA Roast Color Classification System (Agtron-RCCS) was established with eight color disks (Figure 2.3), numbered in increments ranging from "very light" to "very dark" [Pugash, 1995].

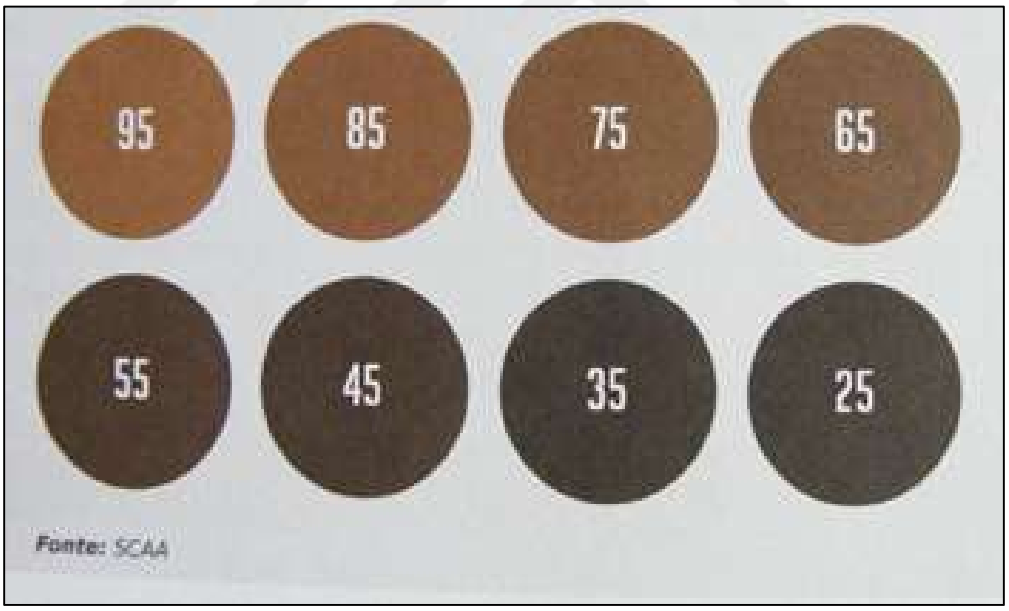


Figure 2.3: SCAA Agtron roast color kit.

2.2. Grinding of Coffee Beans

Since the pleasant aromas of roasted coffee beans released during the grinding, this stage is one of the important steps for instant coffee production [Akiyama et al., 2003]. The ‘mortar and pestle’, ‘the hand mill’, and ‘the electric grinder’ are three basic types of grinders. The uniformity of the product obtained after the grinding process affects the extraction of soluble solids [Perry and Caruso, 2004]. The grinding process facilitates the transfer of soluble substances into the brew and increases the interface between water and coffee [Andueza et al., 2003]. Coffee should not be ground more than 2 minutes before brewing due to possibility of oxidation.

2.3. Extraction of Coffee Beans

After the grinding, extraction is involved in order to produce instant coffee. The soluble solids and volatile compounds of coffee are extracted from the coffee beans using hot water in a multistage countercurrent extractor. Water is used to extract all the soluble solids from the coffee beans heated to about 175 °C under pressurized conditions. Batch and continuous extractors are used for extraction [Farah, 2012].

2.4. Concentration of the Coffee Extract

Coffee extract is concentrated up to 40% solids. Vacuum evaporation and freeze concentration are used to increase the soluble solids concentration of the extract. Since vacuum evaporation and freeze concentration ensure lower temperatures than normal to evaporate water, aroma and flavor compounds are kept and high quality product can be obtained [Auleda et al., 2011].

2.5. Dehydration of the Liquid Coffee

Being one of the most important stages of coffee production, dehydration converts the liquid coffee extract in to a dry form. Spray drying and freeze drying are two methods commonly used in coffee dehydration. Spray drying provides high

production capacities at low energy costs but it uses high temperatures which affect the taste of the final product. Freeze drying ensures the best quality in terms of aroma recovery but it requires more energy so it is an expensive process [MacLeod et al., 2006], [Sadikoglu et al., 2006].

2.6. Coffee and Health

Containing many potential health benefits, coffee is a medicinal alternative. The results of many investigations show that coffee consumption has positive effect on several chronic diseases. Coffee contains a lot of different chemicals, including carbohydrates, lipids, nitrogenous compounds, vitamins, minerals, alkaloids and phenolic compounds. Studies show that coffee consumption may help prevent several chronic diseases such as type-2 diabetes, liver diseases including hepatic injury, cirrhosis and hepatocellular carcinoma, Parkinson's disease and Alzheimer's disease. High content of phenolic compounds makes coffee one of the highest contributors to antioxidant intake in western diets [Bae et al., 2014], [Farah, 2009].

2.7. Caffeine and Decaffeinated Coffee

Although coffee has some health benefits, caffeine is not completely harmless. Moderate caffeine consumption may cause potential health risks such as anxiety, restlessness, insomnia and tachychardia [Benowitz, 1990], [James and Stirling, 1983], [Nehlig, 1999]. The investigations showed the negative effects of caffeine prompt to people seek a caffeine-free coffee. The coffee industry has searched for decaffeination methods which retain the pleasing aroma and flavor components of coffee. One of these methods is the extraction of caffeine by an organic solvent. Although the method is effective and currently being used commercially, solvents could be carcinogen and potential solvent contamination of the end-product could occur [Ernest, 1988]. Another method is based on the treatment by gaseous carbon dioxide under supercritical conditions to decaffeinate coffee. However, the process is expensive and requires intensive labor. Alternative methods have been investigated for decaffeination due to adverse effects of caffeine on health, but the coffee obtained from the decaffeinated process is not completely caffeine free and

the color may change depending on the method used for decaffeination [Ramalakshmi and Raghavan, 1999]. In addition, consumers have some negative perception about the decaffeinated coffee so they are not usually willing to consume it. People who are trying to eliminate caffeine from their diet seek a caffeine-free type of herbal coffee.

2.8. Herbal Coffee of *P. terebinthus*

Some herbal coffees are present in the market to reduce negative side effects of excessive coffee consumption, but their consumption is very limited because their taste is different from the typical coffee taste. Since *P. terebinthus* fruit after roasting has a similar aroma and flavor to conventional roasted coffee beans, it can be a promising caffeine-free alternative. Since *P. terebinthus* fruit after roasting has a similar aroma and flavor to conventional roasted coffee beans, and it has a high antioxidant capacity so it can be a promising caffeine-free alternative to coffee.

Ozel et al. roasted *P. terebinthus* fruit by either the traditional pan-roasting method or by coupled convection-microwave oven roasting. They analyzed volatile compounds using direct thermal desorption coupled with gas chromatography and found compounds specifically relating to coffee aroma (pyrazines, furans and furanones) [Ozel et al. 2014]. Gogus et al. analyzed volatile components of *P. terebinthus* pan roasted at 200 °C for 0, 5, 10, 15, 20 and 25 min. More than hundred volatile compounds were characterized by direct thermal desorption method coupled with comprehensive gas chromatography for *P. terebinthus* roasted for 25 min. As the roasting time increases, the formation of new compounds of pyrazines, furans and benzene derivatives which are typical components of coffee aroma increase. Pan roasted *P. terebinthus* beans were found to contain similar flavor compounds to typically roasted coffee beans suggesting that roasted *P. terebinthus* may provide an alternative for use in the coffee industry [Gogus et al. 2011].

3. *P. TEREBINTHUS* FRUIT

P. terebinthus L. is a member of the family Anacardiaceae [Couladis et al., 2003]. It is grown in the western regions of Morocco, Portugal and the Canary Islands and in south, southeast Anatolian and Mediterranean regions of Turkey [Özcan, 2004]. *P. terebinthus* plants grow well in all types of soil including rocky areas due to their drought resistance [Avanzato and Quatra, 2004].

Reddish-purple flowers of *P. terebinthus* appear between March and April in close compound clusters and grow from the ends of the previous year's shoots. The female racemes of *P. terebinthus* are relatively shorter than male racemes which achieves up to 80 mm during the blooming. The blooming begins from the middle of the rachis and goes to the top and the base during the second half of April [Gercheva et al., 2008]. Their beans are small and globular (or spherical) drupe red to black when it is ripe.

Small size of *P. terebinthus* beans (5-8 mm) is called "menengiç" while the bigger size of beans (8-12 mm) is called "bıttım". However, in different regions of Turkey it is called as "çitlenbik", "çıtlık", "çitemik" and "çedene" [Akan et al., 2005]. Almost 40% of the *P. terebinthus* fruit is oil, and this oil is rich in oleic acid (52.3%), palmitic acid (21.3%) and linoleic acid (19.7%) [Özcan, 2004]. *P. terebinthus* is also rich in minerals and vitamins including such as vitamin D_2 (2.5 $\mu g.g^{-1}$), vitamin D_3 (9.95 $\mu g.g^{-1}$), vitamin K_1 (21 $\mu g.g^{-1}$), α-tocopherol (15.35 $\mu g.g^{-1}$), δ-tocopherol (35.45 $\mu g.g^{-1}$), retinol (25.65 $\mu g.g^{-1}$) and α-tocopherol acetate (13.4 $\mu g.g^{-1}$) [Ciftci et al., 2009].

P. terebinthus beans are consumed as snack food, pastry inner material, in the baking of a specialty village bread, coffee like drink and soap made of *P. terebinthus* oil [Balbay, 2012], [Özcan, 2004].

4. OPTIMIZATION of ROASTING CONDITIONS of *P. TEREBINTHUS* BEANS

P. terebinthus beans were purchased from an herbalist in Pendik, Istanbul, Turkey. Beans that were uniform in size and defect-free were used in the roasting.

4.1. Roasting Process

4.1.1. Fluidized Bed Roasting

Optical roasting trials were performed in a fluidized bed optical roaster (Toper Company, Toper Optical 001, İzmir, Turkey). The roaster operates at a desired temperature between 0 °C and 250 °C, and time. The optical roaster is equipped with a digital temperature controller. The dimensions at which the optical roasting was carried out are 50 cm × 30 cm × 85 cm. *P. terebinthus* beans were roasted at 180 °C, 200 °C and 220 °C for 5, 20 and 35 minutes at each roasting temperature.

4.1.2. Microwave Roasting

Microwave roasting trials were performed in a digitally programmable commercial microwave oven (Arçelik MD 599, Arçelik A.Ş., Istanbul, Turkey) operating at 230 V, 50 Hz and a microwave frequency of 2450 MHz. The oven is capable of operating at five different microwave powers (180 W, 360 W, 540 W, 720 W and 900 W). Microwave output powers and roasting times were adjusted with a digital control unit equipped with the oven. The oven consists of a rotating glass plate with a diameter of 320 mm. Ninety grams of *P. terebinthus* beans were weighed, and 30 ± 0.5 g portions at a time were placed directly on the rotating plate of the microwave oven in the form of a hollow circle to minimize the differences in the degree of roasting and help ensure uniform roasting. Approximately 197-202 beans weighed approximately 30 ± 0.5 g. Roasting trials were performed in triplicate at microwave powers of 360 W, 540 W and 720 W for 5, 11 and 17 min at each roasting power in which microwave power was applied with 2 min increments and 1 min interval between each increment.

4.2. Measurements

4.2.1. Color Measurements

Color measurements of samples were performed using a Konica Minolta CR-400 (Konica Minolta, Sensing Inc., Osaka, Japan) chroma meter equipped with a D_{65} illuminant source and operating with CIE L*a*b* color space. The L* value represents lightness and changes from 0 (black) to 100 (white). The a* value changes from –a* (greenness) to +a* (redness) while the b* value is from –b* (blueness) to +b* (yellowness). The roasted beans were placed into the quartz cell of the chroma meter, ten measurements were randomly taken, and the results were given as the average of ten separate measurements. Calibration was performed with the white color calibration tile prior to the color measurements.

4.2.2. Moisture Content

Moisture content was gravimetrically determined based on the weight loss according to the procedure described in AOAC method 930.15 [AOAC, 2005]. Samples (2 g) were dried at 100 °C in an oven (Ecocell 55, MMM Medcenter Einrichtungen GmbH, München, Germany) until the constant weight was attained. Results of the compositional analysis were the mean of three replicates.

4.2.3. Density Measurements

Density (ρ) of the beans was measured according to Lerici et al. [1980] by using a pycnometer working based on volume displacement method using glycerine ($\rho_{20\ °C}$: 1.26 kg.dm^{-3}) at 20 °C. A sample-holding device was used to submerge the beans into the glycerine, and the weight of the sample-holding device was taken into account in the density measurements. Density measurements were done in triplicates, and the results were expressed as the mean value.

4.2.4. Determining of Breaking Force

A texture analyzer (Lloyd TA1, Lloyd Instruments Ltd., West Sussex, UK) was used to determine breaking force. Each bean was compressed by a cylindrical probe with a diameter of 6.30 mm at a constant deformation speed of 1 $mm.s^{-1}$ until failure occurred. A five kg load cell was used. Pre-test and post-test speeds were set to 1 $mm.s^{-1}$ Measurements were done in an environment at 23 ± 2 °C and 50 ± 5% RH. Ten replicates for each treatment were tested, and the mean value was given.

4.2.5. Sensory Acceptability

Sensory evaluation was carried out with 9 panelists (5 females and 4 males) by considering the guidelines in the norm ISO 8586 [2012]. The intensities of appearance, texture, odor, flavor and overall impression were evaluated using a five-point hedonic scale (1 = dislike very much, 2 = dislike, 3 = neither like/nor dislike, 4 = like and 5 = like very much) [Meilgaard et al., 2015]. Panelists were chosen from the staff members of Chemical Engineering Department in Gebze Technical University who have an experience in sensory evaluation. The panelists were trained prior to the acceptance tests. Training continued until consistent results were obtained among panelists and by each panelist individually. When the panelists were repeatable and consistent, individually and collectively, in their evaluations, they were considered ready to serve in a panel. Repeatability and reproducibility in evaluations were checked and verified in accordance with the method proposed by Rossi [2001]. Acceptance tests for the samples of roasted *P. terebinthus* beans were carried out by the panelists in isolated sensory booths illuminated with white fluorescent light in an environmentally controlled room (23 ± 2 °C and 50 ± 5% RH) under standardized conditions based on the norm ISO 8589 [2007]. Samples were placed into odor-free, disposable and white ceramic plates labeled with randomly coded three digit numbers and presented to the panelists in a randomized order. Each sample was presented to each panelist along with the appropriate questionnaire, one at a time, with a 3 min wait between the samples.

4.2.6. Statistical Analysis

Analysis of variance (ANOVA), a partial F-test for individual terms, and an analysis of residuals were performed to determine the significance of each factor. ANOVA tables were generated, and the effects and regression coefficients of individual linear, quadratic, and interaction terms were determined. Model diagnostics were done by generating normal probability plot, and the plots of residual versus predicted values, and observed versus predicted values. The degrees of significance of all terms in the polynomial were determined statistically by calculating the F-value at a probability (p) of 0.001, 0.01, or 0.05. Contour plots and surface plots were generated to see graphically how the responses change with respect to the factors and to obtain graphical representation of the response surface. All statistical analyses were performed using MINITAB® (Release 16.1, Minitab Inc, State College, PA, USA).

4.2.7. Experimental Design for Optimization of Roasting Conditions

A three-level two factor (3^2) full factorial design was used as the experimental design for fluidized bed roasting (Table 4.1) and microwave roasting (Table 4.2). The independent variables (factors or inputs) were roasting temperature (x_1) and roasting time (x_2) for fluidized bed roasting, and microwave power (x_1) and roasting time (x_2) for microwave roasting. The levels of factors were chosen based on the commercial roasting conditions of coffee in which three different roasting temperatures (180 °C, 200 °C and 220 °C) and three different roasting times (5, 20 and 35 min) were used for fluidized bed roasting and different microwave powers (360 W, 540 W and 720 W) and three different roasting times (5, 11 and 17 minutes at each microwave power with intervals for 2 minutes during roasting) were used for microwave roasting. The dependent variables (responses or outputs) were color parameters (L*, a* and b*), moisture content, density, breaking force and sensory properties (appearance, odor, texture, flavor and overall impression) of the roasted beans.

It was assumed that some mathematical functions (f_n), relating factors (x_i), and responses (y_n) existed:

$$y_n = f_n(x_1, x_2) \tag{4.1}$$

Since the exact nature of the true function(s) is either unknown or too complex, these functions were approximated by second order polynomials (Floros and Chinnan, 1988):

$$y_n = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2 \tag{4.2}$$

where, β_0, β_1, β_2, β_{12}, β_{11} and β_{22} are constant coefficients, and x_1 and x_2 are coded independent variables. Experimental design is presented in Table 4.1 for fluidized bed roasting and in Table 4.2 for microwave roasting.

Table 4.1: Experimental design including process variables and their levels for fluidized bed roasting*.

	Coded variables		Uncoded variables	
Treatments	x_1	x_2	Temperature, T (°C)	Time, t (min)
1	-1	-1	180	5
2	-1	0	180	20
3	-1	+1	180	35
4	0	-1	200	5
5	0	0	200	20
6	0	+1	200	35
7	+1	-1	220	5
8	+1	+0	220	20
9	+1	+1	220	35

*Experimental runs were performed in random order.

Table 4.2: Experimental design including process variables and their levels for microwave roasting*.

Treatments	Coded variables		Uncoded variables	
	x_1	x_2	Power, p (W)	Time, t (min)
1	-1	-1	360	5
2	-1	0	360	11
3	-1	+1	360	17
4	0	-1	540	5
5	0	0	540	11
6	0	+1	540	17
7	+1	-1	720	5
8	+1	+0	720	11
9	+1	+1	720	17

*Experimental runs were performed in random order.

4.3. Results and Discussion for Optimization of Roasting Conditions

4.3.1. Results for Optimization of Fluidized Bed Roasted *P. terebinthus* Beans

4.3.1.1. Effect of Roasting on Color

The models for L*, a* and b* values were obtained as a result of fitting Eq. (4.2) to experimental data shown in Table 4.3. These models were tested for adequacy and fitness by analysis of variance (ANOVA) (Table 4.4). Results of this analysis showed that the models developed for three responses (L*, a* and b*) were significant ($p \leq 0.001$) with no significant lack of fit suggesting that they adequately represented the relationship between the responses and factors. The temperature and time had the same effect on L*, a* and b* values of the roasted *P. terebinthus* beans whereas the interaction of temperature and time had a higher effect on L* value than a* and b* values of the roasted *P. terebinthus* beans. The quadratic effects of temperature and time had equally important on L*, a* and b* values. The model

coefficients or regression coefficients of the second-degree polynomial models are given in Table 4.5.

Table 4.3: Values (mean ± standard deviation) for L*, a* and b* for fluidized bed roasted *P. terebinthus* beans[a].

Roasting temperature (°C) and time (min)	L* value	a* value	b* value	Agtron description
180, 5	36.83 ± 0.05	-2.59 ± 0.07	1.80 ± 0.02	Medium
180, 20	32.48 ± 0.07	-1.64 ± 0.04	1.30 ± 0.03	Moderately dark
180, 35	25.84 ± 0.03	0.67 ± 0.02	0.84 ± 0.02	Dark
200, 5	26.56 ± 0.03	0.93 ± 0.05	0.98 ± 0.04	Dark
200, 20	24.10 ± 0.06	1.17 ± 0.04	0.68 ± 0.04	Dark
200, 35	19.22 ± 0.05	1.29 ± 0.06	0.56 ± 0.05	Dark
220, 5	19.93 ± 0.07	1.43 ± 0.07	0.63 ± 0.01	Dark
220, 20	17.71 ± 0.07	1.51 ± 0.05	0.40 ± 0.02	Very dark
220, 35	16.20 ± 0.04	2.06 ± 0.04	0.32 ± 0.04	Very dark

[a] Mean value is the average of three replications.

Table 4.4: Analysis of variance for the color values for fluidized bed roasted *P. terebinthus* beans.

Source	df	L* value	a* value	b* value
Regression model	5	768.679***	57.628***	5.340***
Linear	2	730.989***	47.779***	4.853***
Quadratic	2	11.299*	4.621*	0.119*
Cross-product	1	26.390***	5.227*	0.367*
Residual	21	10.028	10.419	0.719
Lack of fit	3	2.094	3.188	0.039
Pure error	18	9.934	7.231	0.680
Total	26	778.706	68.047	6.060
R^2		0.99	0.85	0.88

Significance level: ***p ≤ 0.001; ** p ≤ 0.01; *p ≤ 0.05.

Table 4.5: Regression coefficients of the second order polynomials for response parameters of L*, a* and b* values for fluidized bed roasted *P. terebinthus* beans.

Regression coefficients[a] βk	L* value k = 1	a* value k = 2	b* value k = 3
β_{k0}	274.763***	-108.385***	21.1373***
β_{k1}	-2.008***	0.971**	-0.1660***
β_{k2}	-1.337***	0.451**	-0.1435
β_{k11}	0.004**	-0.002**	0.0003***
β_{k22}	-0.003	-0.001	0.0002
β_{k12}	0.006***	-0.002**	0.0006**

[a] These are coefficients of Eq. (4.2), and subscripts 1 and 2 represent roasting temperature and roasting time, respectively.
Significance level: *** $p \leq 0.001$; ** $p \leq 0.01$; *$p \leq 0.05$.

The regression equations obtained were used to generate contour plots to better understand the relationship between the factors and responses (Fig. 4.1). L* and b* values decreased while a* value increased with increasing roasting temperature and roasting time. The increase in a* value represents an increase in the reddish color and the decrease in L* value represents a darker color of beans. During roasting, chemical reactions of phospholipid compounds of the beans enhance the development of brown pigments which give the roasted products darker color [Krysiak, 2011], [Ng et al., 2014]. Özdemir and Devres roasted hazelnuts using a forced air pilot scale roaster where L* values of the hazelnuts decreased and a* values increased by increasing the roasting temperature and time [Özdemir and Devres 2000]. Mendes et al. found similar behavior for the color values of the robusta coffee roasted using a laboratory scale electric rotary drum roaster [Mendes et al. 2001]. Our findings for the color change of the *P. terebinthus* beans during roasting are consistent with the results reported by Özdemir and Devres, and Mendes et al. [Özdemir and Devres 2000], [Mendes et al. 2001]. SCAA values or more commonly known as Agtron numbers are precise industry standard generally used to determine the level of roasted coffee. Therefore, the color values were transformed to Agtron values, and the roasting level of *P. terebinthus* beans were determined based on the Agtron scale. Measured L* values were compared with the visual SCAA roast coffee color standards ranging from very light to very dark, and the corresponding Agtron values were determined. The Pearson correlation coefficient

(PCC) between Agtron numbers and L* values were calculated. A high linear correlation (PCC = 0.99) was found between the Agtron values and L* values. It is stated that L* value is preferred for monitoring color development during nut roasting because the L* value is analogous to the color observation made by the operator [Moss and Otten, 1989], [Özdemir and Devres, 2000]. The Agtron descriptions corresponding to L* values of roasted *P. terebinthus* beans are given in Table 4.3.

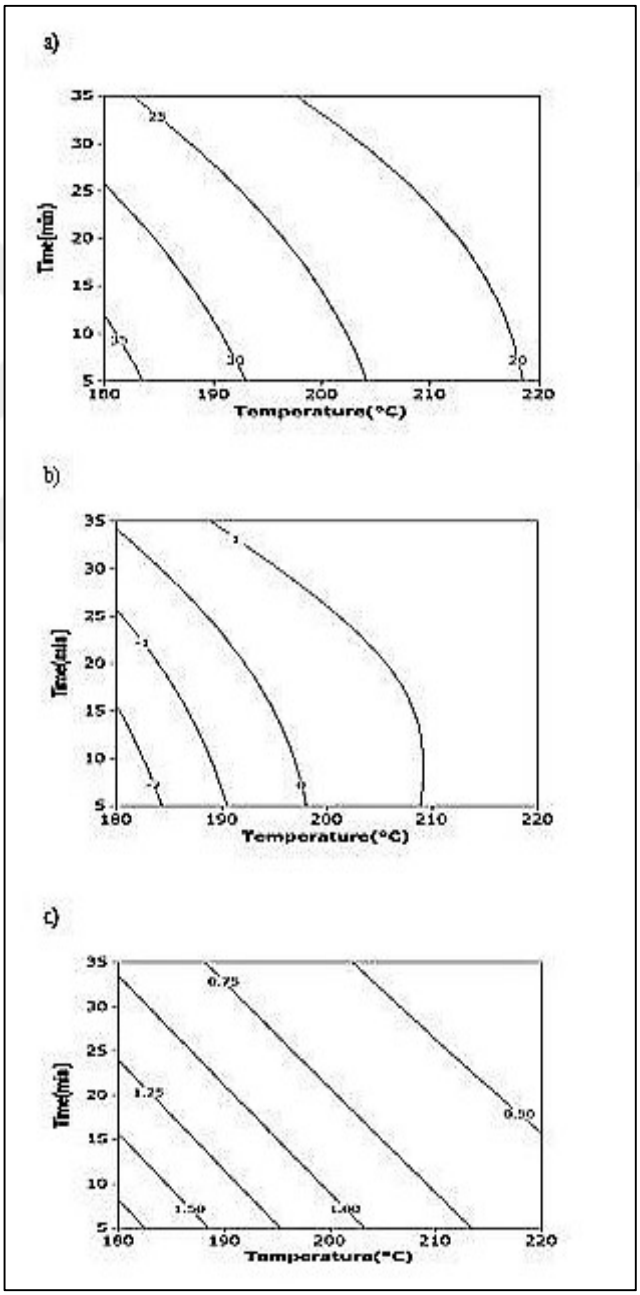


Figure 4.1: Contour plots for a) L*, b) a* and c) b* values of the fluidized bed roasted *P. terebinthus* beans.

4.3.1.2. Effect of Roasting on Moisture Content, Density and Texture

The criteria commonly used to evaluate the quality of roasted coffee beans include moisture, density and textural properties [Alessandrini et al., 2008]. The same criteria could be used to evaluate the quality attributes of roasted *P. terebinthus* beans. Mean values of the moisture content, density and breaking force for the roasted *P. terebinthus* beans are given in Table 4.6 in which the experimental data were fitted to Eq. (4.2) to develop models for the moisture content, density and breaking force. ANOVA (Table 4.7) showed that the models developed for the moisture content, density and breaking force were significant ($p \leq 0.001$) with no significant lack of fit suggesting that the models adequately represented the relationship between the responses and factors. Temperature and time, and their quadratic effects are more important than their interaction on the moisture content of *P. terebinthus* beans. Temperature and time, their interaction and their quadratic effects have the same level of importance on the density of *P. terebinthus* beans. Temperature and time, and their interaction were equally important on the breaking force of *P. terebinthus* beans whereas their quadratic effects were found to be statistically not significant ($p > 0.05$). The regression coefficients of the second-degree polynomial models are presented in Table 4.8.

Based on the regression equations obtained, the contour plots were developed (Fig. 4.2). During roasting, moisture content of *P. terebinthus* beans decreased with increasing the temperature and time. A decrease in density occurred with an increase in the roasting temperature and time. The decrease in density simultaneously occurs by the increase of the internal gas formation due to the heat-induced reactions that result in increase in bean volume and loss of volatiles that result in decrease in bean mass [Massini et al., 1990], [Ortolá et al., 1998], [Jokanović et al., 2012b].

The roasting process affected textural property of the beans as evidenced by the decrease in the breaking force with the increase in the roasting temperature and time. The lowest breaking force value was obtained at 220 °C for 35 minutes of roasting. The force needed to break the roasted beans depends on the moisture content of the beans. Less force is necessary to break the roasted beans with low moisture content because beans become more fragile when the moisture content is low. Pittia et al. also reported similar results for the effect of roasting conditions on

the breaking force of coffee beans in which roasted coffee beans with low moisture content have lower breaking force values [Pittia et al. 2007].

Table 4.6: Values (mean ± standard deviation) for moisture content, density and breaking force for fluidized bed roasted *P. terebinthus* beans[a].

Roasting temperature (°C) and time (min)	Moisture content ($g.kg^{-1}$)	Density ($kg.dm^{-3}$)	Breaking force (N)
180, 5	0.188 ± 0.02	0.841 ± 0.01	76.55 ± 0.08
180, 20	0.176 ± 0.01	0.722 ± 0.01	65.25 ± 0.06
180, 35	0.165 ± 0.02	0.608 ± 0.02	50.92 ± 0.08
200, 5	0.167 ± 0.05	0.628 ± 0.03	58.80 ± 0.07
200, 20	0.153 ± 0.01	0.490 ± 0.01	45.58 ± 0.07
200, 35	0.145 ± 0.03	0.409 ± 0.02	39.72 ± 0.06
220, 5	0.149 ± 0.03	0.444 ± 0.04	41.96 ± 0.06
220, 20	0.142 ± 0.05	0.378 ± 0.01	36.55 ± 0.04
220, 35	0.136 ± 0.04	0.315 ± 0.03	33.79 ± 0.08

[a] Mean value is the average of three replications.

Table 4.7: Analysis of variance for the moisture content, density and breaking force values for fluidized bed roasted *P. terebinthus* beans.

Source	df	Moisture content	Density	Breaking force
Regression Model	5	0.728***	0.723***	4918.44***
Linear	2	0.709***	0.703***	634.13***
Quadratic	2	0.011***	0.011***	55.60
Cross-product	1	0.007**	0.008***	228.71***
Residual	21	0.012	0.012	278.92
Lack of fit	3	0.002	0.002	31.31
Pure error	18	0.010	0.010	247.62
Total	26	0.741	0.735	5197.36
R^2		0.98	0.98	0.95

Significance level: *** $p \leq 0.001$; **$p \leq 0.01$; *$p \leq 0.05$.

Table 4.8: Regression coefficients of the second order polynomials for response parameters of moisture content, density and breaking force values for fluidized bed roasted *P. terebinthus* beans.

Regression coefficients[a] β_k	Moisture content k = 1	Density k = 2	Breaking force k = 3
βk_0	7.90178***	6.93904***	533.005**
βk_1	-0.05171***	-0.05253***	-3.757**
βk_2	-0.02566***	-0.02577***	-3.712***
βk_{11}	0.00010***	0.00011***	0.007
βk_{22}	0.00005	0.00005	0.005
βk_{12}	0.00009*	0.00009***	0.015***

[a] These are coefficients of Eq. (4.2), and subscripts 1 and 2 represent roasting temperature and roasting time, respectively.
Significance level: *** p ≤ 0.001; ** p ≤ 0.01; *p ≤ 0.05.

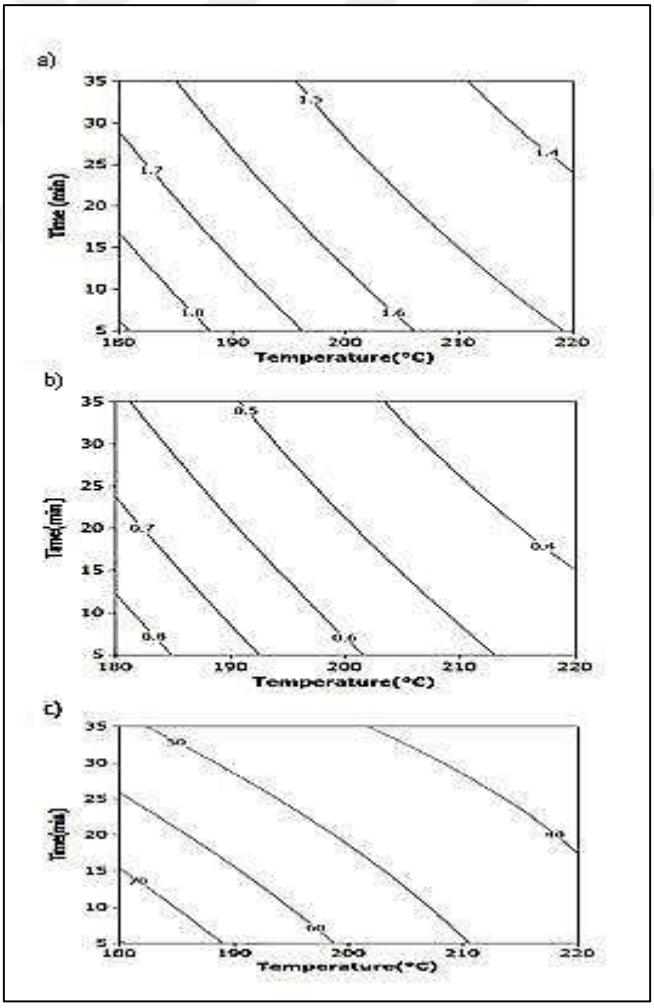


Figure 4.2: Contour plots for (a) moisture content, (b) density and (c) breaking force of fluidized bed roasted *P. terebinthus* beans.

4.3.1.3. Effect of Roasting on Sensory Acceptability

Sensory attributes of a food product determine its acceptability by the consumers. Sensory parameters such as appearance, odor, texture, flavor and overall impression were reported as the important quality attributes for roasted coffee beans [Chung et al., 2013], [Mendes et al., 2001]. Therefore, the appearance, odor, texture, flavor and overall impression of the *P. terebinthus* beans roasted at different roasting temperatures and times were scored by the panelists. Mean sensory scores for the roasted beans are given in Table 4.9. These data were fitted to Eq. (4.2) to generate models for appearance, odor, texture, flavor and overall impression. The models were tested for adequacy and fitness by ANOVA (Table 4.10). The models developed for five responses were significant ($p \leq 0.001$) with no significant lack of fit suggesting that the models adequately represented the relationship between the responses and factors. Coefficient of determination (R^2) values for the sensory data was higher than 0.80 which was considered sufficiently high in which the response variables were measured using a hedonic scale. It was reported that an R^2 value of at least 0.80 is sufficient to explain the variability of the regression model and variance of the sensory data [Prasad and Nath, 2002]. Therefore, the models developed for the sensory data were acceptable. Temperature and time, and their quadratic effects were equally important on the acceptability of the roasted *P. terebinthus* beans. Temperature and time, and their quadratic effects were found to be more important than the interaction of temperature and time on acceptability. Regression coefficients of the second-degree polynomial models were given in Table 4.11.

The regression equations obtained were used to generate contour plots (Fig. 4.3). The appearance, odor, texture, flavor and overall impression scores increased and then decreased with increasing roasting temperature and time. The scores were a function of the roasting temperature and time. Very light roasted and very dark roasted samples took low sensory scores by the panelists. Samples roasted at 220 °C for 35 minutes had the lowest sensory scores. Samples roasted at 200 °C for 20 minutes had the highest appearance, odor, texture, flavor and overall impression values. Our findings for sensory analysis are consistent with the results reported by Mendes at al. and Chung et al. in which sensory values for the roasted coffee beans changed with roasting temperature and time. Higher roasting temperatures and

higher roasting times produced coffee beans with low sensory characteristics [Mendes at al. 2001], [Chung et al. 2013].

Table 4.9: Sensory acceptability scores (mean ± standard deviation) for fluidized bed roasted *P. terebinthus* beans[a].

Roasting temperature (°C) and time (min)	Appearance	Odor	Texture	Flavor	Overall impression
180, 5	1.75 ± 0.09	1.47 ± 0.07	1.25 ± 0.04	2.90 ± 0.08	2.40 ± 0.01
180, 20	3.12 ± 0.09	3.20 ± 0.06	2.20 ± 0.08	2.95 ± 0.06	3.40 ± 0.01
180, 35	3.10 ± 0.08	3.10 ± 0.08	2.90 ± 0.03	2.25 ± 0.08	2.75 ± 0.03
200, 5	2.65 ± 0.07	2.60 ± 0.07	4.35 ± 0.02	4.25 ± 0.06	4.40 ± 0.01
200, 20	4.75 ± 0.07	4.70 ± 0.07	4.87 ± 0.02	4.90 ± 0.09	4.75 ± 0.02
200, 35	3.25 ± 0.08	3.65 ± 0.08	4.05 ± 0.06	3.87 ± 0.09	4.15 ± 0.02
220, 5	2.30 ± 0.08	2.90 ± 0.09	2.60 ± 0.05	4.00 ± 0.06	3.75 ± 0.03
220, 20	2.97 ± 0.06	3.00 ± 0.06	2.05 ± 0.04	1.80 ± 0.08	2.20 ± 0.01
220, 35	1.15 ± 0.09	1.25 ± 0.06	1.50 ± 0.03	1.25 ± 0.07	1.37 ± 0.02

[a] Mean value is the average of sensory acceptability scores of nine panelists.

Table 4.10: Analysis of variance for the sensory properties of fluidized bed roasted *P. terebinthus* beans.

Source	df	Appearance	Odor	Texture	Flavor	Overall impression
Regression Model	5	19.872***	21.669***	26.033***	22.182***	19.735***
Linear	2	0.924***	0.602***	0.034***	3.708***	2.118***
Quadratic	2	16.847***	17.216***	22.218***	18.293***	16.128***
Cross-product	1	2.101*	3.850*	3.781*	0.180*	1.487*
Residual	12	2.800	2.879	2.555	5.088	1.524
Lack of fit	3	0.587	0.373	0.564	0.808	0.317
Pure error	9	2.215	2.506	1.991	4.280	1.206
Total	17	22.672	24.549	28.589	27.271	21.259
R^2		0.88	0.88	0.91	0.81	0.93

Significance level: *** $p \leq 0.001$; ** $p \leq 0.01$; *$p \leq 0.05$.

Table 4.11: Regression coefficients of the second order polynomials for response parameters of sensory properties for fluidized bed roasted *P. terebinthus* beans.

Regression coefficients[a]	Appearance	Odor	Texture	Flavor	Overall impression
β_k	k = 1	k = 2	k = 3	k = 4	k = 5
βk_0	-177.648***	193.207***	-238.927***	-204.120***	-192.543***
βk_1	1.783***	1.924***	2.386***	2.097***	1.956***
βk_2	0.516***	0.604***	0.509***	0.140	0.361
βk_{11}	-0.004***	-0.005***	-0.006***	-0.005***	-0.005***
βk_{22}	-0.005***	-0.004**	-0.001	0.002	0.002*
βk_{12}	-0.002**	-0.002***	-0.002***	-0.001	-0.001**

[a] These are coefficients of Eq. (4.2), and subscripts 1 and 2 represent roasting temperature and roasting time, respectively.
Significance level: *** $p \leq 0.001$; ** $p \leq 0.01$; *$p \leq 0.05$.

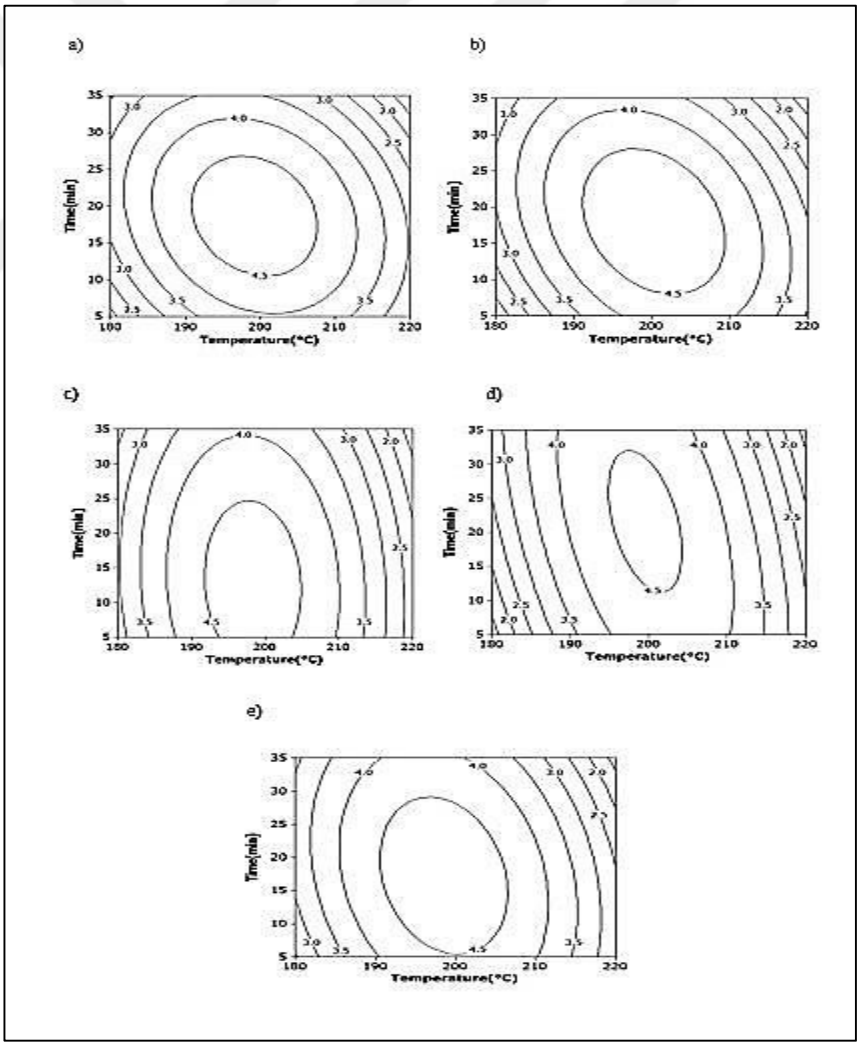


Figure 4.3: Contour plots for (a) appearance, (b) odor, (c) texture, (d) flavor and (e) overall impression of fluidized bed roasted *P. terebinthus* beans.

4.3.1.4. Determining Optimum Roasting Conditions and Model Validation

Optimal roasting conditions of temperature and time were obtained by superimposing the contour plots for the responses (Figure 4.4). Overall impression directly affects the consumers' preference, and it is the most important factor influencing the final decision of the consumers. Overall impression is a general assessment of a product because all the sensory attributes in a sensory test are considered by the overall impression [Chambers and Wolf, 1996]. Therefore, the overall impression score was considered in determining the optimum region. It was revealed that samples with the overall impression score less than 4 were not liked by the panelists. Therefore, an overall impression score of at least 4 (like) was chosen as the minimum value for the acceptability. The development of more intense aroma and flavor compounds that gives characteristic taste to coffee occurs at dark roast [Akiyama et al., 2008], [Bhumiratana et al., 2011]. Some of the popular designations for the dark roast include French Roast, Italian Roast, Espresso Roast, Continental Roast, New Orleans Roast and Spanish Roast. Many dark roasts are used for espresso blends because more balanced flavors and aromas develop as the roasting process proceeds, and the taste of the coffee becomes more intense. Therefore, the L* value, moisture content, density and breaking force corresponding to the dark roasting level based on the Agtron description were used as constraints to determine the optimum region. These constraints defined a set of lower and upper limits on the design variables such that $19.22 \leq L^* \leq 26.56$, $1.45\% \leq$ moisture content ≤ 1.67 $g.kg^{-1}$, 0.409 $kg.dm^{-3} \leq$ density ≤ 0.628 $kg.dm^{-3}$ and 39.72 N $\leq$ breaking force $\leq$ 58.80 N. The generated plots for the overall impression, L* value, moisture content, density and breaking force, and the criteria outlined above produced an optimum region (shaded area) in the superimposed plot (Figure. 4.4). The predicted optimum conditions of T = 205 °C and t = 15 min would yield a product with the Agtron description corresponding to the dark ($L^* \leq 26.56$), moisture content ≤ 0.167 $g.kg^{-1}$ density ≤ 0.628 $kg.dm^{-3}$, breaking force ≤ 58.80 N and overall impression score ≥ 4. Adequacies of the models at the predicted optimum conditions were tested by performing an independent experiment (Table 4.12). Three replications were done to experimentally determine L* value, moisture content, density, breaking force and overall impression for the *P. terebinthus* beans roasted at 205 °C for 15 min. The

predicted and experimental values were not statistically different at $p > 0.05$. The strong correlation between the experimental and the predicted values confirmed that the models developed were adequate describing the relationship between the factors and responses. The predicted values of L*, moisture content, density, breaking force and overall impression were found to be 25.94, 0.158 $g.kg^{-1}$, 0.528 $kg.dm^{-3}$, 48.56 N and 4.6, respectively.

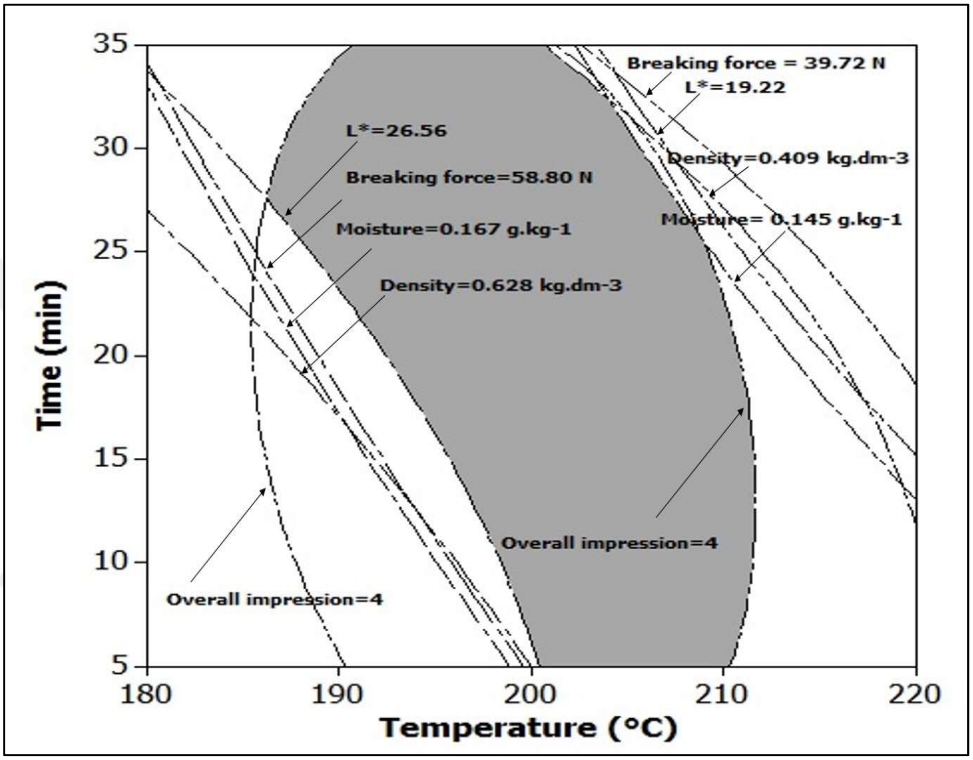


Figure 4.4: Optimum region obtained by superimposing contour plots of overall impression, L* value, moisture content, density and breaking force.

Table 4.12: Predicted and experimental values at optimum conditions for fluidized bed roasted beans.

Response	Predicted value	Experimental value[a]	
		Mean	Range
L*	25.94	25.44	24.22-26.32
Moisture content	0.156	0.155	0.152-0.158
Density	0.528	0.543	0.501-0.568
Breaking force	48.56	47.06	44.12-50.86
Overall impression	4.6	4.6	4.4-4.8

[a] Results are of three replications. The roasting conditions are 205 °C for 15 minutes.

4.3.2. Results for Optimization of Microwave Roasted *P. terebinthus* Beans

4.3.2.1. Effect of Roasting on Color

Color is one of the most important quality attributes of foods because it directly influences consumer acceptability. Color has been reported to play an important role in overall acceptability of roasted coffee and *P. terebinthus* beans [Baggenstoss et al., 2008], [Bolek and Ozdemir, 2017], [Franca et al., 2009]. Experimental data (Table 4.13) were fitted to Eq. (4.2) to obtain the models for L*, a* and b* values. The adequacy and fitness of the models were tested by ANOVA (Table 4.14). Results showed that the models developed for three responses (L*, a* and b*) were significant ($p \leq 0.001$) with no significant lack of fit suggesting that they adequately described the relationship between the responses and factors. ANOVA for L*, a* and b* values indicated that the linear terms for the microwave level and time were significant at $p \leq 0.001$ for L* value while they were significant at $p \leq 0.05$ for a* and b* values. The quadratic effects of power level and time for L* and a* values were found to be significant at $p \leq 0.001$ and $p \leq 0.05$, respectively, while they were not statistically significant ($p > 0.05$) for the b* value. The interaction of power level and time for L* value was statistically significant ($p \leq 0.05$) whereas the interaction of power level and time did not have any effect on a* and b* values. The model coefficients or regression coefficients of the second-degree polynomial models for L*, a* and b* values for microwave roasted *P. terebinthus* beans are given in Table 4.15.

The regression equations obtained were used to generate three-dimensional surface plots to better understand the relationship between the factors and responses (Fig. 4.5). L* and b* values decreased while a* value increased with increasing roasting power and roasting time. The increase in a* value represents an increase in the reddish color and the decrease in L* value represents a darker color of beans. Phenolic polymerization and Maillard reaction are the major reactions contributing to the formation of colored compounds, called melanoidins, which give darker color to roasted coffee [Nagaraju et al., 2016]. Similar results for color were also reported by Hojjati et al. who observed that microwave roasting significantly reduced L* values, but increased a* values of the pistachios [Hojjati et al., 2015].

Table 4.13: Values (mean ± standard deviation) for L*, a* and b* for microwave roasted *P. terebinthus* beans.

Roasting power (W) and time (min)	L* value	a* value	b* value	Agtron description
360, 5	41.66 ± 0.04	-3.59 ± 0.02	3.51 ± 0.05	Light medium
360, 11	37.76 ± 0.06	-2.74 ± 0.01	3.24 ± 0.06	Medium
360, 17	33.80 ± 0.07	-1.93 ± 0.01	2.92 ± 0.04	Moderately dark
540, 5	26.28 ± 0.04	-1.35 ± 0.02	2.53 ± 0.03	Dark
540, 11	25.12 ± 0.02	0.65 ± 0.03	2.22 ± 0.03	Dark
540, 17	20.33 ± 0.06	1.09 ± 0.04	1.59 ± 0.04	Dark
720, 5	18.81 ± 0.07	1.33 ± 0.02	1.29 ± 0.05	Very dark
720, 11	16.13 ± 0.08	1.75 ± 0.03	1.05 ± 0.02	Very dark
720, 17	14.32 ± 0.04	2.27 ± 0.03	0.79 ± 0.02	Very dark

[a] Mean value is the average of three replications.

Table 4.14: Analysis of variance for the color values for microwave roasted *P. terebinthus* beans.

Source	df	L* value	a* value	b* value
Regression model	5	1521.81***	72.21***	15.62***
Linear	2	1475.04***	70.19*	15.60*
Quadratic	2	41.09***	1.75*	0.01
Cross-product	1	5.68*	0.25	0.01
Residual	12	6.76	2.57	0.24
Lack of fit	3	4.36	1.42	0.12
Pure error	9	3.78	1.14	0.11
Total	17	1528.57	74.78	15.86
R^2		0.99	0.97	0.98

Significance level: ***p ≤ 0.001; ** p ≤ 0.01; *p ≤ 0.05.

Table 4.15: Regression coefficients of the second order polynomials for response parameters of L*, a* and b* values for microwave roasted *P. terebinthus* beans.

Regression coefficients[a] βk	L* value k = 1	a* value k = 2	b* value k = 3
β_{k0}	93.3913***	-15.4010***	6.1115***
β_{k1}	-0.1735***	0.0349***	-0.0069***
β_{k2}	-0.644**	0.3831*	0.0266
β_{k11}	0.0001***	-0.0001*	0.0001
β_{k22}	-0.0130	-0.0070	-0.0001
β_{k12}	0.0008*	-0.0002	0.0002

[a] These are coefficients of Eq. (4.2), and subscripts 1 and 2 represent roasting temperature and roasting time, respectively.

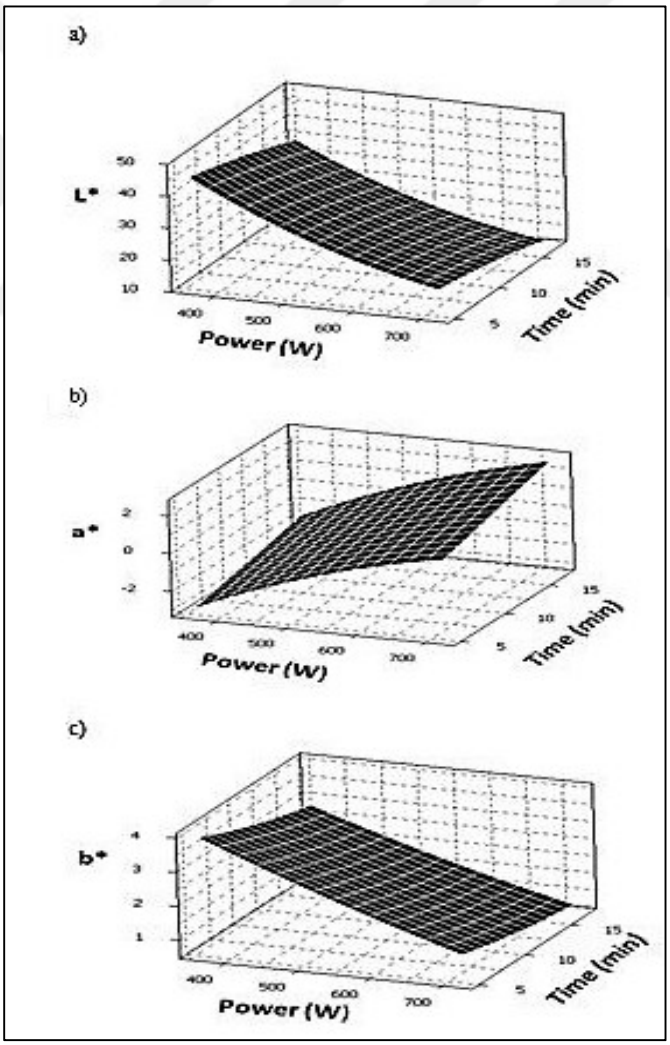


Figure 4.5: Surface plots for (a) L*, (b) a* and (c) b* values of the microwave roasted *P. terebinthus* beans.

4.3.2.2. Effect of Roasting on Moisture Content, Density and Texture

Moisture, density and textural characteristics are commonly used to evaluate the quality of roasted coffee beans [Alessandrini et al., 2008]. The same characteristics could be used to evaluate the quality attributes of microwave roasted *P. terebinthus* beans. Mean values of the moisture content, density and breaking force for the microwave roasted *P. terebinthus* beans are given in Table 4.16 in which the experimental data were fitted to Eq. (4.2) to develop models for the moisture content, density and breaking force. ANOVA (Table 4.17) showed that the models developed for the moisture content, density and breaking force were significant ($p \leq 0.001$) with no significant lack of fit suggesting that the models adequately represented the relationship between the responses and factors. Microwave power level and time, their interaction and their quadratic effects had equally important on the moisture content of microwave roasted *P. terebinthus* beans. Power level and time were more important than their interaction on the density and breaking force of the microwave roasted *P. terebinthus* beans. The regression coefficients of the second-degree polynomial models for the moisture content, density and breaking force are given in Table 4.17. Based on the regression equations obtained, three-dimensional surface plots for the moisture content, density and breaking force were generated (Fig. 4.6). There was an inverse relation between the factors (roasting power and roasting time) and the responses including moisture content, density and breaking force.

Moisture content of *P. terebinthus* beans decreased with increasing the roasting power and roasting time during the microwave roasting which caused the moisture to evaporate from the beans. At microwave power levels of 360, 540 and 720 W, increasing the roasting time from 5 to 17 min increased the moisture loss by 9.1%, 11.5% and 21.4%, respectively. This means that more moisture removal is possible at microwave roasting power of 720 W than microwave power levels of 360 and 540 W by solely increasing the roasting time. Anjum et al. also reported that increasing microwave roasting time from 5 to 15 min increased the moisture loss of microwave roasted sunflower seeds [Anjum et al. 2006]. A decrease in density occurred with an increase in the microwave roasting power and time. A decrease in density simultaneously occurs by the increase of the internal gas formation because

heat-induced reactions occur during roasting [Jokanović et al., 2012b], [Massini et al., 1990], [Ortolá et al., 1998].

Table 4.16: Values (mean ± standard deviation) for moisture content, density and breaking force for microwave roasted *P. terebinthus* beans[a].

Roasting power (W) and time (min)	Moisture content ($g.kg_{-1}$)	Density ($kg.dm^{-3}$)	Breaking force (N)
360, 5	0.186 ± 0.03	0.838 ± 0.03	74.42 ± 0.06
360, 11	0.176 ± 0.02	0.670 ± 0.02	62.37 ± 0.08
360, 17	0.169 ± 0.04	0.633 ± 0.01	59.10 ± 0.07
540, 5	0.165 ± 0.02	0.626 ± 0.03	56.24 ± 0.03
540, 11	0.153 ± 0.03	0.518 ± 0.01	48.82 ± 0.05
540, 17	0.146 ± 0.02	0.412 ± 0.03	40.32 ± 0.04
720, 5	0.140 ± 0.03	0.398 ± 0.02	38.43 ± 0.06
720, 11	0.121 ± 0.03	0.372 ± 0.01	34.62 ± 0.05
720, 17	0.110 ± 0.02	0.312 ± 0.01	31.22 ± 0.06

[a] Mean value is the average of three replications.

Table 4.17: Analysis of variance for the moisture content, density and breaking force values for microwave roasted *P. terebinthus* beans.

Source	df	Moisture content	Density	Breaking force
Regression model	5	0.661***	0.468***	3340.89***
Linear	2	0.658***	0.458**	3290.88**
Quadratic	2	0.002***	0.024	17.13
Cross-product	1	0.001***	0.007*	32.89*
Residual	12	0.002	0.014	76.52
Lack of fit	3	0.001	0.008	33.28
Pure error	9	0.001	0.006	43.24
Total	17	1.326	0.482	3417.40
R^2		0.99	0.97	0.98

Significance level: *** $p \leq 0.001$; **$p \leq 0.01$; *$p \leq 0.05$.

Table 4.18: Regression coefficients of the second order polynomials for response parameters of moisture content, density and breaking force values for microwave roasted *P. terebinthus* beans.

Regression coefficients[a] β_k	Moisture content k = 1	Density k = 2	Breaking force k = 3
βk_0	2.58770***	1.57706***	134.912***
βk_1	-0.00210***	-0.00190**	-0.158**
βk_2	-0.01579***	-0.03899**	-2.908**
βk_{11}	-0.00002***	0.00001	0.001
βk_{22}	0.00001	0.00046	0.038
βk_{12}	-0.00002***	0.00003*	0.002*

[a] These are coefficients of Eq. (4.2), and subscripts 1 and 2 represent roasting power and roasting time, respectively.
Significance level: *** $p \leq 0.001$; ** $p \leq 0.01$; *$p \leq 0.05$.

The microwave roasting power levels and roasting times used in this study produced almost the same decrease in moisture contents and densities of *P. terebinthus* beans roasted in a fluidized bed roaster where the roasting temperature and roasting time changed between 180 °C and 220 °C and 5 and 35 min, respectively [Bolek and Ozdemir, 2017].

Textural changes were used as an indicator of degree of roasting in coffee beans [Pittia et al., 2001], pistachios [Nikzadeh and Sedaghat, 2008], [Raei et al., 2010] and peanuts [Cea et al., 2015]. The roasting process affected breaking force of the beans as evidenced by the decrease in the breaking force with an increase in the roasting power and time. The breaking force of the microwave roasted *P. terebinthus* beans changed between 31.22 and 74.42 N depending on the microwave power level and roasting time. The lowest breaking force (31.22 N) was obtained at 720 W for 17 min of roasting *P. terebinthus* beans become more fragile during roasting because of the decreased water content and loosening of the structure resulting in increase in the bean's volume and porosity. Therefore, less force is necessary to break the dark roasted beans than the light roasted beans. A decrease in breaking force with an increase in roasting temperature and roasting time was observed for *P. terebinthus* beans roasted in a fluidized bed roaster [Bolek and Ozdemir, 2017], coffee beans [Pittia et al., 2007] and pistachio nuts [Nikzadeh and Sedaghat, 2008], [Raei et al., 2010], [Shakerardekani et al., 2011].

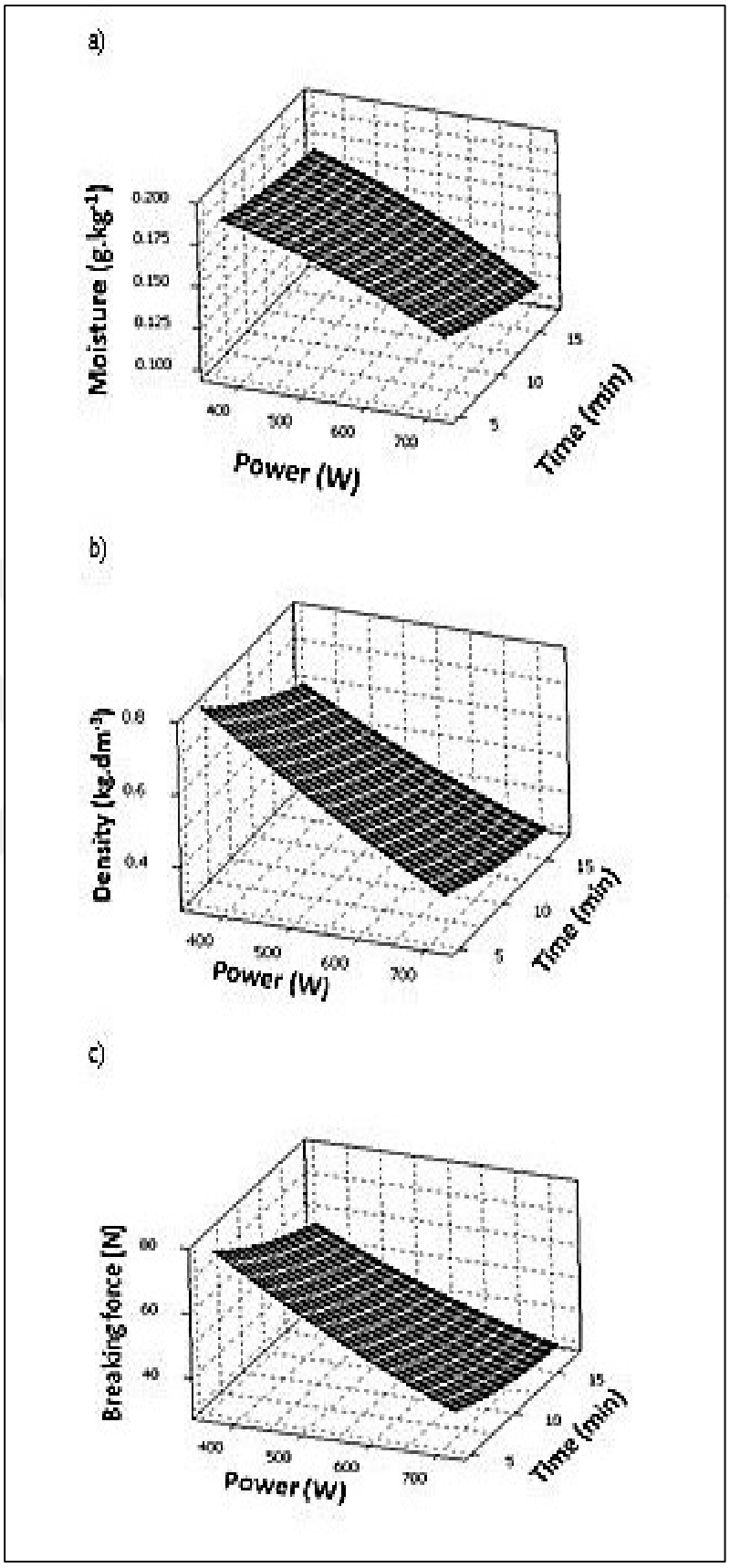


Figure 4.6: Surface plots for (a) moisture content, (b) density and (c) breaking force of microwave roasted *P. terebinthus* beans.

4.3.2.3. Effect of Roasting on Sensory Acceptability

Many thermal and chemical reactions occur during the roasting process which finally enhances the sensory quality. Roasting is the key unit operation and improves the characteristic aroma, color, texture and appearance of coffee beans [Schenker et al., 2002]. Appearance, odor, texture, flavor and overall impression were evaluated to assess the acceptability of the *P. terebinthus* beans because they were reported as the important quality attributes for roasted coffee beans [Chung et al., 2013], [Mendes et al., 2001]. Mean sensory scores for the microwave roasted beans are given in Table 4.19 and the data were fitted to Eq. (4.2) to generate models for the appearance, odor, texture, flavor and overall impression. The models were tested for adequacy and fitness by ANOVA (Table 4.20). The models developed for the five responses were significant ($p \leq 0.001$) with no significant lack of fit suggesting that the models adequately represented the relationship between the responses and factors. Coefficient of determination (R^2) is defined as the ratio of the explained variation to the total variation, and is a measure of the degree of fit in which R^2 values at least 0.80 is considered sufficiently high to explain variability of the regression model and variance of the sensory data [Prasad and Nath, 2002], [Sin et al., 2006]. R^2 values for the sensory data were found to be higher than 0.80, and the models developed for the sensory data were acceptable. Microwave power level and time, and their quadratic effects were significant at $p \leq 0.001$ for all the responses for the sensory. The interaction of microwave power level and time was significant at $p \leq 0.001$ for the odor, flavor, texture and overall impression while it was significant at $p \leq 0.05$ for the appearance. Regression coefficients of the second-degree polynomial models were given in Table 4.21.

Table 4.19: Sensory acceptability scores (mean ± standard deviation) for microwave roasted *P. terebinthus* beans[a].

Roasting Power (W) and time (min)	Appearance	Odor	Texture	Flavor	Overall impression
360, 5	1.75 ± 0.08	1.90 ± 0.03	1.10 ± 0.02	2.30 ± 0.04	1.60 ± 0.05
360, 11	2.65 ± 0.08	2.60 ± 0.06	1.65 ± 0.02	3.05 ± 0.04	2.55 ± 0.01
360, 17	3.05 ± 0.06	3.10 ± 0.04	2.55 ± 0.03	3.30 ± 0.06	2.65 ± 0.04
540, 5	4.05 ± 0.06	3.65 ± 0.04	4.20 ± 0.04	4.10 ± 0.03	4.15 ± 0.01
540, 11	4.35 ± 0.07	4.90 ± 0.05	4.58 ± 0.04	4.50 ± 0.03	4.25 ± 0.02
540, 17	4.10 ± 0.04	4.65 ± 0.05	4.10 ± 0.03	4.05 ± 0.05	4.05 ± 0.01
720, 5	4.00 ± 0.04	4.10 ± 0.03	4.00 ± 0.05	4.00 ± 0.02	3.85 ± 0.04
720, 11	1.35 ± 0.05	3.10 ± 0.07	1.75 ± 0.02	1.80 ± 0.02	1.75 ± 0.03
720, 17	1.10 ± 0.03	1.95 ± 0.06	1.25 ± 0.02	1.10 ± 0.02	1.00 ± 0.02

[a] Mean value is the average of sensory acceptability scores of nine panelists.

Table 4.20: Analysis of variance for the sensory properties of microwave roasted *P. terebinthus* beans.

Source	df	Appearance	Odor	Texture	Flavor	Overall impression
RegressionModel	5	23.287***	21.187***	30.710***	20.524***	23.779***
Linear	2	1.344***	0.363***	1.616***	2.220***	1.2167***
Quadratic	2	14.912***	13.218***	20.273***	10.892***	14.952***
Cross-product	1	7.031*	7.605***	8.820***	7.411***	7.6050***
Residual	12	5.177	0.470	2.371	2.795	3.917
Lack of fit	3	2.822	0.210	1.330	1.565	1.327
Pure error	9	2.355	2.600	1.041	1.230	2.590
Total	17	28.465	21.657	33.082	23.320	27.691
R^2		0.82	0.98	0.93	0.88	0.86

Significance level: *** $p \leq 0.001$; ** $p \leq 0.01$; *$p \leq 0.05$.

Table 4.21: Regression coefficients of the second order polynomials for response parameters of sensory properties for microwave roasted *P. terebinthus* beans.

Regression coefficients[a] β_k	Appearance k = 1	Odor k = 2	Texture k = 3	Flavor k = 4	Overall impression k = 5
βk_0	-17.0049***	-17.6005***	-21.5192***	-14.3778***	-17.8704***
βk_1	0.0731***	0.0714***	0.0870***	0.0631***	0.0742***
βk_2	0.3243	0.5199***	0.3588*	0.4146*	0.4245
βk_{11}	-0.0001***	-0.0001***	-0.0001***	-0.0001***	-0.0001***
βk_{22}	0.0042	-0.0019	-0.0058	0.0007	0.0005
βk_{12}	0.0009*	-0.0009***	-0.0010***	-0.0009***	-0.0008***

[a] These are coefficients of Eq. (4.2), and subscripts 1 and 2 represent roasting temperature and roasting time, respectively.
Significance level: *** $p \leq 0.001$; ** $p \leq 0.01$; *$p \leq 0.05$.

The regression equations obtained were used to generate three-dimensional surface plots (Fig. 4.7). Sensory evaluation is a useful tool to determine consumer perception and to ensure acceptability of a product by the consumers. The sensory scores of microwave roasted *P. terebinthus* beans first increased with an increase in power level and roasting time, and then showed a decrease due to the excessive roasting where higher roasting powers and roasting times are used. Chung et al. and Mendes et al. reported that higher roasting temperatures and higher roasting times produced coffee beans with low acceptability scores. Very light roasted and very dark roasted samples took low acceptability scores by the panelists [Chung et al. 2013], [Mendes et al. 2001]. Samples roasted at 720 W for 17 min had the lowest acceptability scores. Samples roasted at 540 W for 11 min took the highest appearance, odor, texture, flavor and overall impression scores from the panelists.

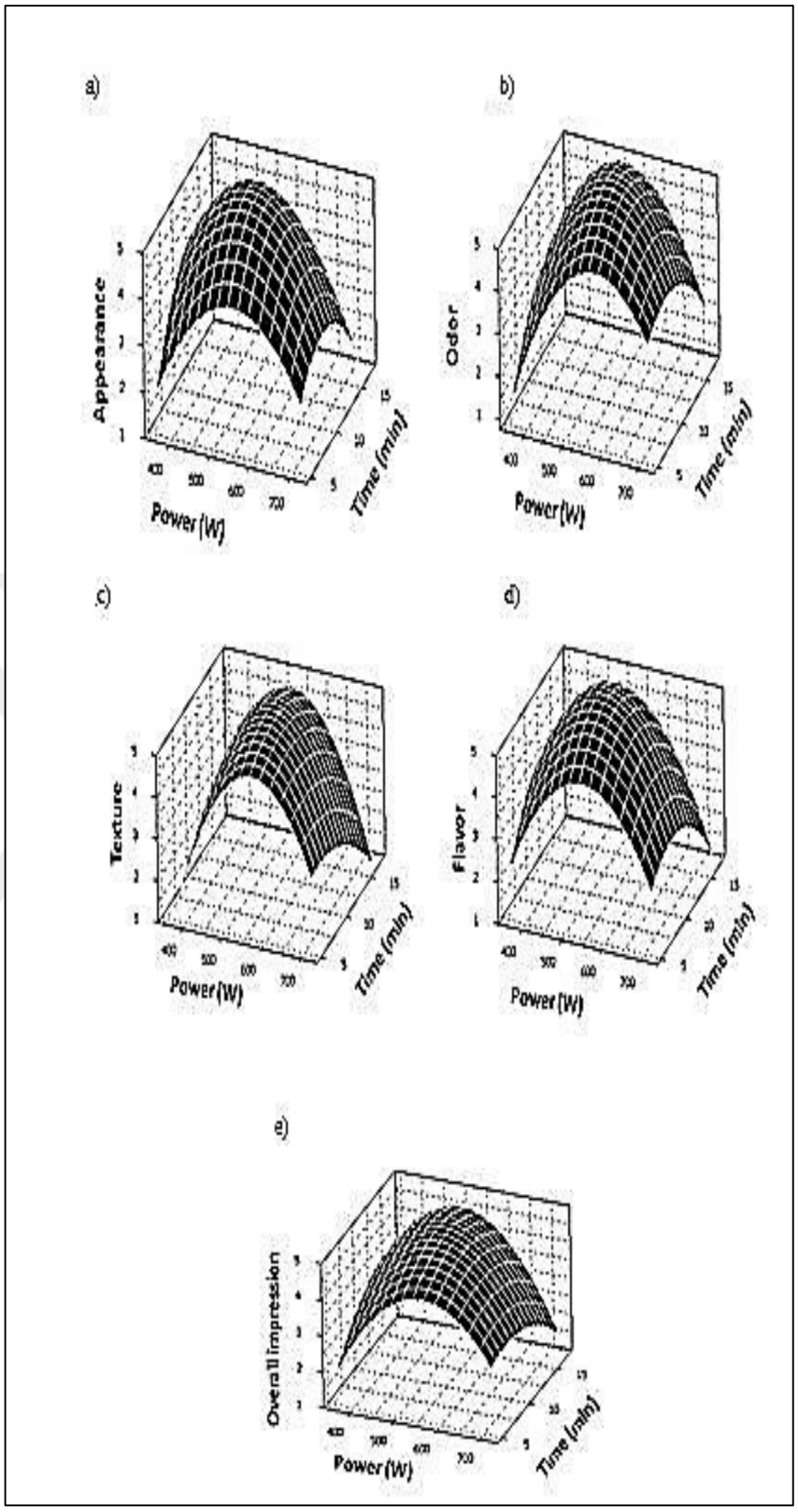


Figure 4.7: Surface plots for (a) appearance, (b) odor, (c) texture, (d) flavor and (e) overall impression of microwave roasted *P. terebinthus* beans.

4.3.2.4. Determining Optimum Roasting Conditions and Model Validation

RSM was used to determine optimum region of roasting conditions that provide *P. terebinthus* beans with desired quality characteristics. By superimposing the contour plots for the L* value, moisture content, density, breaking force and overall impression for the microwave roasted *P. terebinthus* beans, optimum region for the microwave roasting conditions in terms of roasting power and roasting time was determined (Fig. 4.8).

The optimum region was determined by considering the dark roasted beans because the development of more intense aroma and flavor compounds at dark roast gives characteristic taste to coffee [Akiyama et al., 2008], [Bhumiratana et al., 2011]. French Roast, Italian Roast, Espresso Roast, Continental Roast, New Orleans Roast and Spanish Roast are some of the popular designations for the dark roasted coffee beans used for espresso blends due to the development of intense taste with more balanced aroma and flavor in the final product. To determine the optimum region, the L* value, moisture content, density, breaking force and overall impression corresponding to the dark roasting level were used as constraints. These constraints defined a set of lower and/or upper limits on the design variables such that $20.33 \leq L^* \leq 26.28$, $0.146\ g.kg^{-1} \leq$ moisture content $\leq 0.165\ g.kg^{-1}$, $0.412\ kg.dm^{-3} \leq$ density $\leq 0.626\ kg.dm^{-3}$, $40.32\ N \leq$ breaking force $\leq 56.24\ N$ and overall impression score ≥ 4.

The generated plots for the L* value, moisture content, density, breaking force and overall impression, and the criteria outlined above produced an optimum region (shaded area) in the superimposed plot (Fig. 4.8). The predicted optimum conditions of T = 540 W and t = 14 min would yield a product with the Agtron description corresponding to the dark roast in which $L^* \leq 26.28$, moisture content $\leq 0.165\ g.kg^{-1}$ density $\leq 0.626\ kg.dm^{-3}$, breaking force $\leq 56.24\ N$ and overall impression score ≥ 4. Adequacies of the models at the predicted optimum conditions were tested by performing an independent experiment (Table 4.22). Three replications were done to experimentally determine L* value, moisture content, density, breaking force and overall impression for the *P. terebinthus* beans roasted at 540 W for 14 minutes. The predicted and experimental values were not statistically different ($p > 0.05$). The strong correlation between the experimental and the predicted values confirmed that

the models developed were adequate describing the relationship between the factors and responses. The predicted values of L*, moisture content, density, breaking force and overall impression were found to be 22.12, 0.149 $g.kg^{-1}$, 0.440 $kg.dm^{-3}$, 44.64 N and 4.10, respectively.

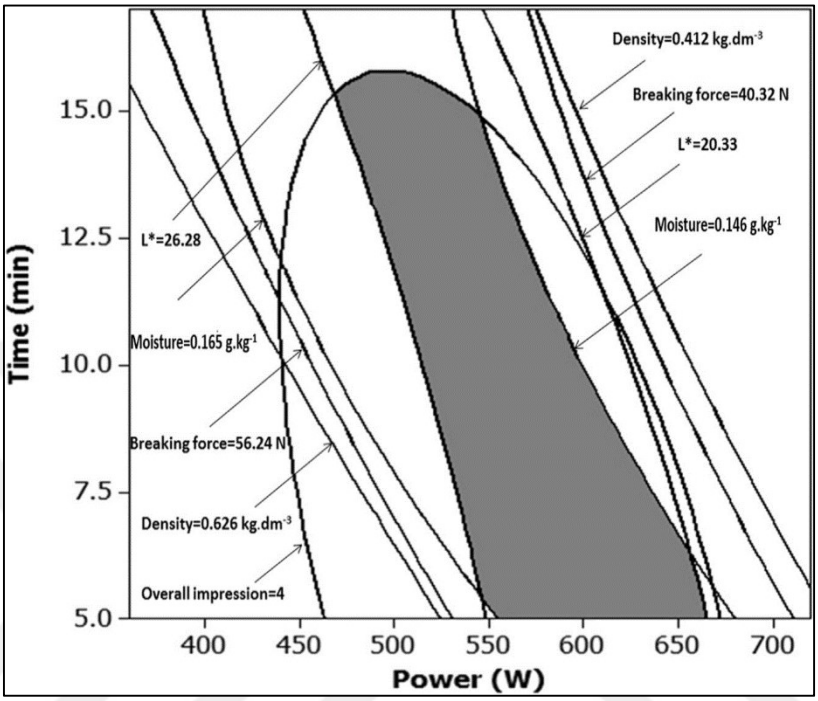


Figure 4.8: Optimum region obtained by superimposing contour plots of overall impression, L* value, moisture content, density and breaking force.

Table 4.22: Predicted and experimental values at optimum conditions for microwave roasted beans.

		Experimental value[a]	
Response	Predicted value	Mean	Range
L*	22.12	22.83	21.86-24.21
Moisture content	0.149	0.150	0.148-0.151
Density	0.440	0.449	0.414-0.516
Breaking force	44.64	44.36	42.12-46.74
Overall impression	4.10	4.12	4.05-4.15

[a] Results are of three replications. The roasting conditions are 540 W for 14 min.

5. CHEMICAL, STRUCTURAL and THERMAL ANALYSIS of ROASTED *P. TEREBINTHUS* BEANS

Methanol, 2,2-diphenyl-1-picrylhydrazyl (DPPH), Folin-Ciocalteu's reagent (10%, v/v) and gallic acid were supplied from Sigma-Aldrich (St. Louis, MO, United States). Na_2CO_3 was purchased from J. T. Baker Chemical Company, Phillipsburg, NJ, USA and n-hexane was obtained from Merck (Darmstadt, Germany). Chemicals used in the experiments were of analytical grade.

5.1. Chemical Analysis

5.1.1. Fourier Transform Infrared (FTIR) Analysis

FTIR spectra of fluidized bed roasted and microwave roasted *P. terebinthus* beans in the range of 4000–650 cm^{-1} were recorded on Perkin Elmer Paragon 1000 Fourier transform infrared spectrometer (Waltham, Massachusetts, USA) to characterize the presence of specific chemical groups.

5.1.2. Preparation of Extracts from *P. terebinthus* Beans

For each roasting condition, 2 g of roasted beans were ground and added to 20 ml distilled water. Each sample was stirred for 20 min under moderate heat (35 °C). The extract was separated from solid residues by centrifuging each sample (7000 × g, 10 min, 25 °C). Finally, each extract was separated into aliquots which were kept at -20 °C for future use.

5.1.3. Antioxidative Activity (DPPH) Assay

Antioxidative activity was evaluated by DPPH radical assay, as previously described by Singleton et al. [1999]. One ml freshly prepared methanolic solution of DPPH radical (100 μM) was mixed with the extract solution at various concentrations (0.5–100 $\mu g.ml^{-1}$). The contents were vigorously mixed and

incubated at room temperature in the dark for 20 min. The absorbance readings of the samples were measured against a blank solution with a UV-VIS spectrophotometer (UV Lambda 35, Perkin-Elmer Corp., Shelton, CT, USA) at 517 nm. The mean of three measurements was reported as the DPPH radical scavenging activity using the following equation:

$$DPPH\ Scavenging\ Activity\ (\%) = \frac{A_C - A_t}{A_C} \times\ 100 \tag{5.1}$$

- A_c: Absorbance of the control
- A_t: Absorbance of the sample solution

5.1.4. Determination of Total Phenolic Content (TPC)

TPC was determined by Folin and Ciocalteu method, and the results were expressed as gallic acid equivalent [Gutfinger, 1981]. Dilution of 0.1 ml extract was mixed with 0.4 ml methanol, and 5 ml of water was added to the mixture. Then 0.5 ml of Folin–Ciocalteu reagent was added, and the mixture was shaken vigorously. After waiting 3 min, 1 ml of saturated (35%, w/v) Na_2CO_3 solution was added. The content was mixed and diluted to 10 ml with water. Following 1 h of incubation at room temperature in the dark, the absorbance was measured with a UV-VIS spectrophotometer (UV Lambda 35, Perkin-Elmer Corp., Shelton, CT, USA) at 725 nm versus a blank containing Folin-Ciocalteu reagent and distilled water without the extract. Measurements were done in triplicate, and total phenolic content was expressed in mg of gallic acid equivalents (GAE) per g of extract.

5.1.5. Determination of Protein Content

The crude protein content of *P. terebinthus* beans was determined as described in the AOAC method 984.13 [AOAC, 2005]. The crude protein content is calculated according to formula given below:

$$Crude\ protein\ (g/kg) = Nitrogen\ (g/kg) \times\ 6.25 \tag{5.2}$$

5.1.6. Determination of Oil Content

The total oil content of ground *P. terebinthus* beans was determined according to the procedure given in the AOAC method 920.39 [AOAC, 2005]. A sample (10-15 g of sample) was weighed into an extraction thimble, and extraction was carried out in the Soxhlet apparatus with 250 ml n-hexane for 6 h. The solvent was evaporated by using a rotary evaporator (Heidolph Laboratory Digital 4000 rotary evaporator, Heidolph Instruments GmbH & Co., Schwabach, Germany). The flask containing oil was dried in a hot air oven, cooled in a desiccator and weighed. An empty flask as a reference was also heated, dried, cooled and weighed. The total oil content was calculated from the difference in weight between the empty flask and the flask containing the oil.

5.1.7. Determination of Ash Content

The ash content of *P. terebinthus* beans was determined according to the AOAC method 968.08 [AOAC, 2005]. Samples (2 g) were weighed into previously heated, dried, cooled and weighed porcelain crucibles and placed in a temperature-controlled furnace preheated to 580 °C. Samples were heated until they turned to a completely grey which took nearly 17 hours. Crucibles were transferred to a desiccator, cooled and weighed immediately thereafter. The ash content was calculated from the difference in weight between the empty crucible and the crucible with the ash residue. All measurements were conducted in triplicate and the average of three measurements was given.

5.1.8. pH Measurements

The pH of ground *P. terebinthus* beans was measured using a digital pH meter (SevenEasy S20-K, Mettler Toledo, Columbus, OH, USA). Two grams of ground *P. terebinthus* beans were accurately weighed into a 200 ml of glass bottle, and 100 ml of distilled water was added. The glass bottle with its contents was boiled for 10 min. Then the contents were filtered through Whatman #4 filter paper, and 50 ml of

the filtrate was cooled to room temperature and used in pH determinations. Measurements were performed in triplicate.

5.2. Structural and Thermal Analysis

Structural and thermal properties of *P. terebinthus* beans roasted in fluidized bed roaster at 180 °C, 200 °C and 220 °C for 5, 20 and 35 min at each roasting temperature, and in microwave oven at 360 W, 540 W and 720 W for 5, 11 and 17 min at each microwave power with 2 min increments and 1 min interval between each increment were analyzed.

5.2.1. Scanning Electron Microscope (SEM) Analysis

Roasted samples were coated with a thin layer of gold prior to analysis. The structure and morphology of the *P. terebinthus* beans were analyzed by a SEM (PhilipsXL30 FEG, Oregon, USA) operating at a voltage of 2 kV. Triplicate samples were used for each SEM analysis.

5.2.2. Differential Scanning Calorimeter (DSC) Analysis

DSC analysis were carried out to determine thermal behavior of roasted and unroasted *P. terebinthus* beans by using a DSC (Perkin Elmer Jade DSC, Shelton, CT, USA). Prior to DSC analysis, the instrument was calibrated for temperature and heat flow with indium. A sample of 2.5 ± 0.5 mg was placed in aluminum pan and the pan was sealed. An empty aluminum pan was used as a reference. DSC analysis were performed from ambient to 250 °C at a heating rate of 5 °C.min^{-1} under nitrogen with a flow rate of 25 ml.min^{-1} to avoid oxidation. All DSC measurements were done in triplicate.

5.2.3. Thermogravimetric (TG) Analysis

TG analysis was carried out using a thermogravimetric analyzer TGA/SDTA 851 (Mettler Toledo, Giessen, Germany) with a heating rate of 10 °C.min^{-1} from

ambient to 900 °C in a helium atmosphere with a flow rate of 25 ml.min^{-1}. A sample mass of 3 ± 1 mg was used, and the measurements were carried out in triplicate.

5.2.4. X-Ray Diffraction (XRD) Analysis

The samples were ground prior to XRD analysis. XRD patterns of roasted and unroasted *P. terebinthus* samples were determined with an X-ray diffractometer (Rigaku, D-max 2200PC, Tokyo, Japan) equipped with an X-ray source of Cu Kα radiation with a wavelength of 0.1542 Å. Data were collected from a diffraction angle (2θ) of 2 °C to 90 °C with a scanning rate of 3 °C.min^{-1}. The voltage and current of the X-ray tubes were 40 kV and 40 mA, respectively. All XRD measurements were done in triplicate.

5.3. Results and Discussion for Chemical Structural and Thermal Analysis

5.3.1. Results for Chemical Analysis

5.3.1.1. FTIR Analysis of Roasted *P. terebinthus* Beans

FTIR spectrum of unroasted *P. terebinthus* beans is given in Figure 5.1. FTIR spectra of fluidized bed roasted *P. terebinthus* beans are shown in Figures 5.2 to 5.4, and FTIR spectra of microwave roasted *P. terebinthus* are given in Figures 5.5 to 5.7 within the range of 4000-650 cm^{-1}. Similar FTIR spectra were observed for fluidized bed and microwave roasted *P. terebinthus* beans at different roasting conditions.

The broad band observed between 3200-3600 cm^{-1} corresponded to O–H stretching vibrations, arising mainly from water, protein and carbohydrates. The decrease in this band was due to dehydration during roasting (Figures 5.1 to 5.3). The bands located at ~2900 cm^{-1} and ~2855 cm^{-1} corresponded to methyl and methylene groups, respectively. The increase in this band for fluidized bed and microwave roasted beans implies that important changes in aliphatic hydrocarbons have occurred during the roasting process [El-Nabarawy et al., 1997], [Ng et al., 2014]. ~1800-1680 cm^{-1} region of the FTIR spectrum is carbonyl region that could

be used to correlate ester, aldehydes, ketones and acids [Lyman et al., 2003]. Absorbance at ~1745 cm^{-1} was attributed to the C=O stretching of lipid ester groups [Ng et al., 2014]. The band located at around 1639 cm^{-1} was attributed to the C=C stretching of the furan ring [Xu et al., 2011]. This band was not observed in FTIR spectrum of unroasted *P. terebinthus* beans (Figure 5.1). The band located at around ~1464 cm^{-1} was caused by N=C stretching of amino groups [Wang and Lim, 2012]. The decrease in %T as the roasting proceeded could be caused by Maillard reactions and denaturation of proteins.

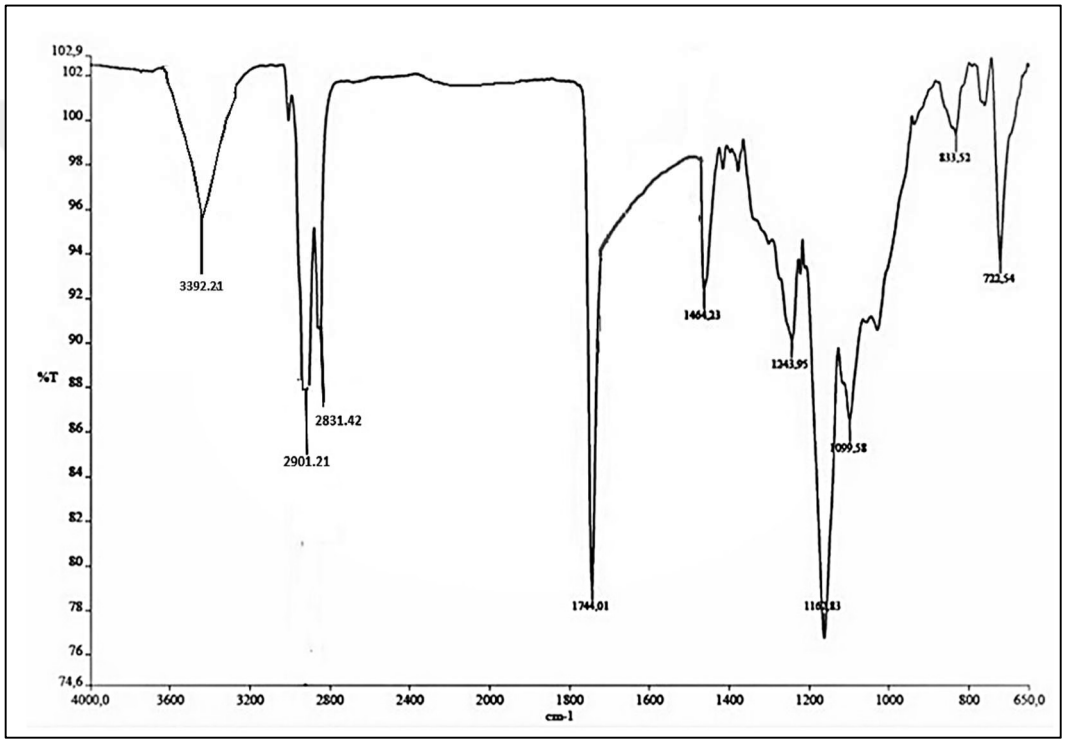
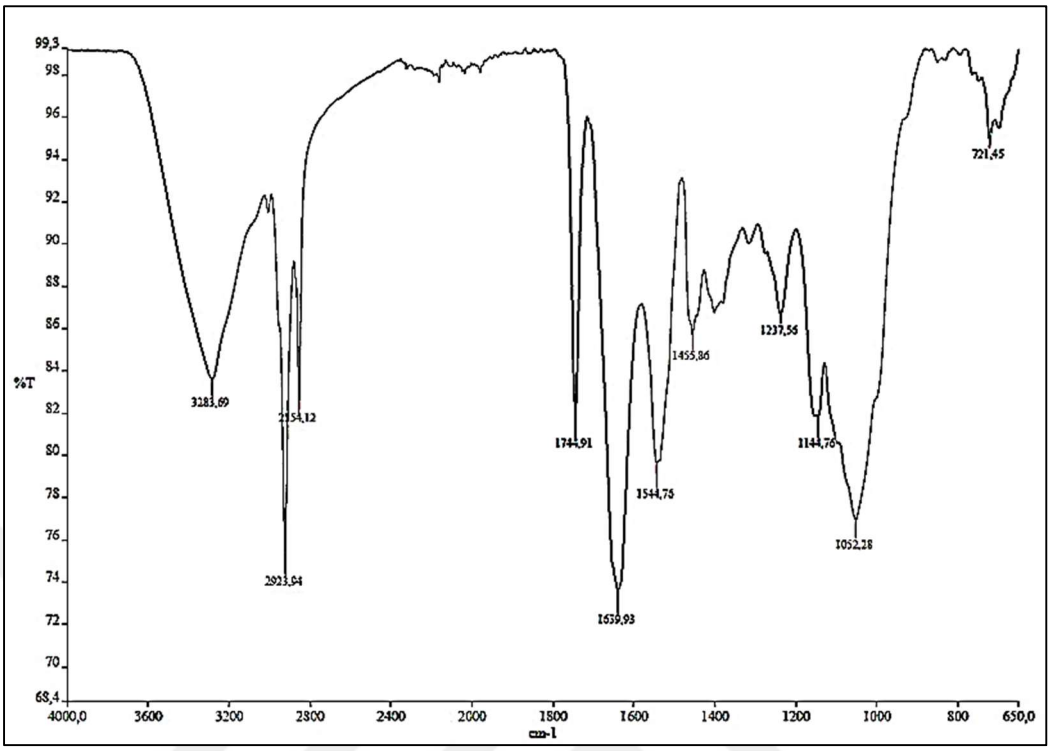


Figure 5.1: FTIR spectrum of unroasted *P. terebinthus* beans.

(a)



(b)

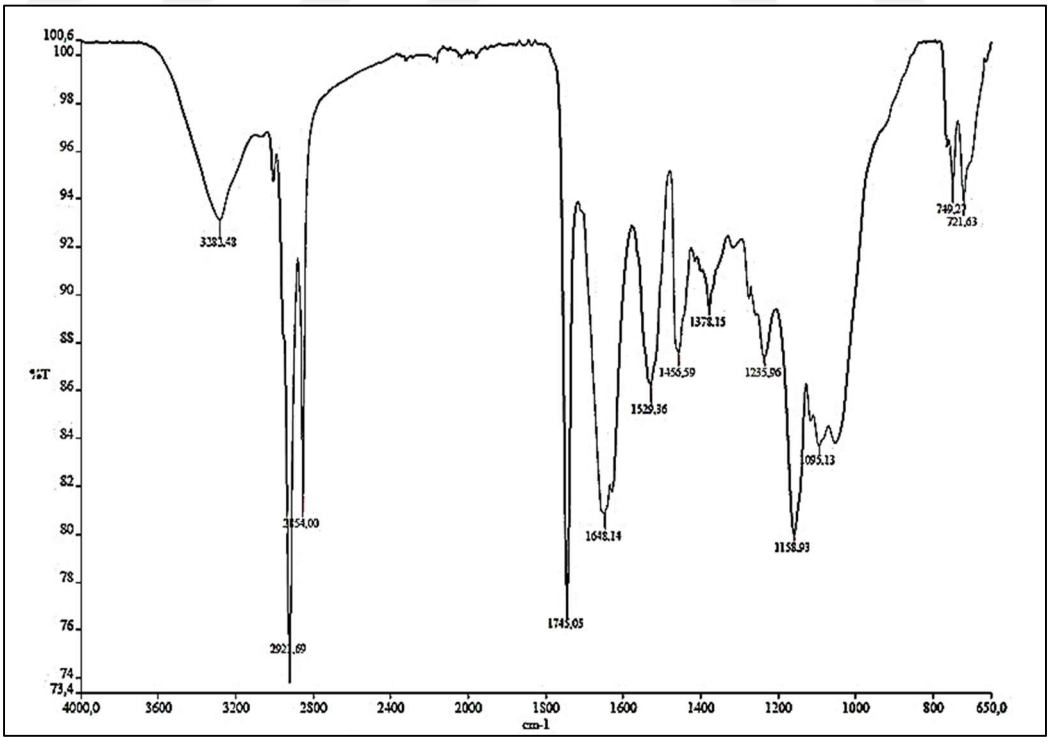


Figure 5.2: FTIR spectra of fluidized bed roasted *P. terebinthus* beans at 180 °C for a) 5 min, b) 20 min.

(c)

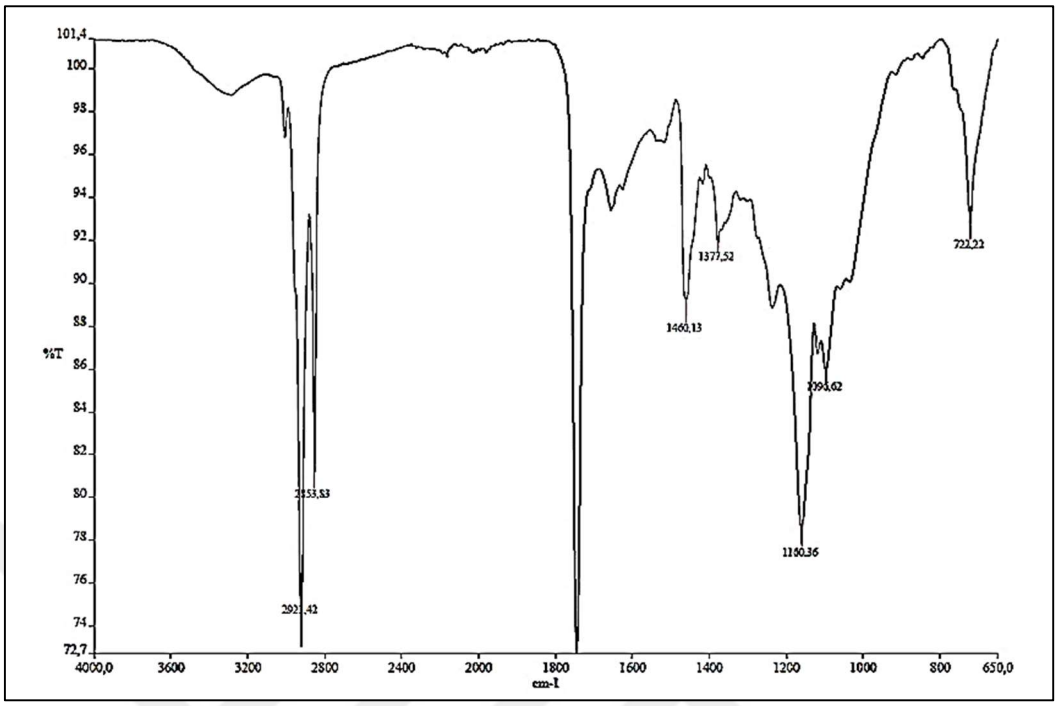


Figure 5.2 (Cont'd) : FTIR spectra of fluidized bed roasted *P. terebinthus* beans at 180 °C for c) 35 min.

(a)

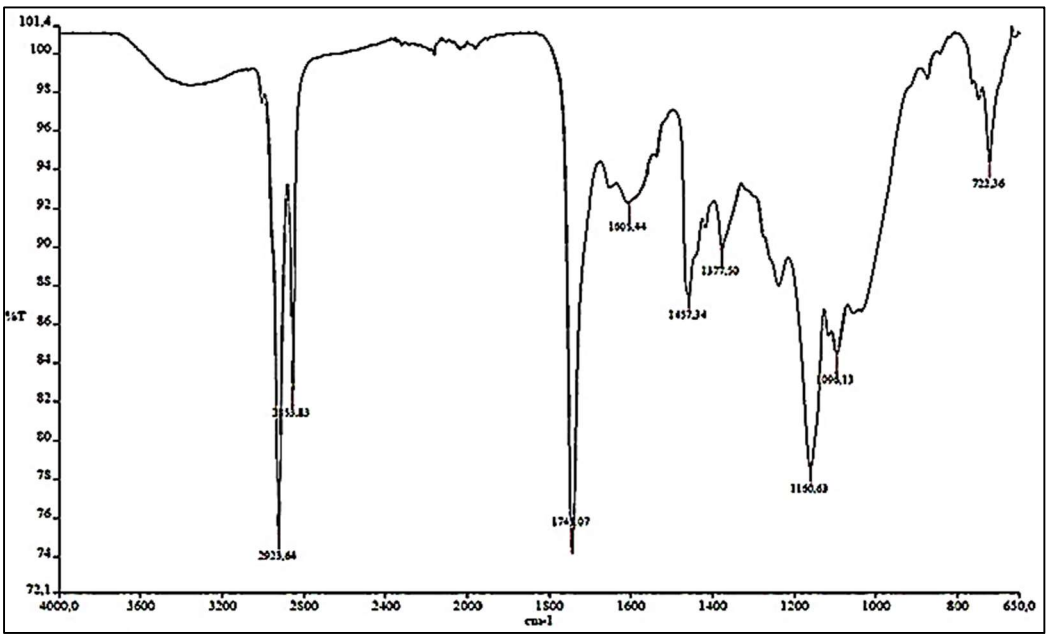
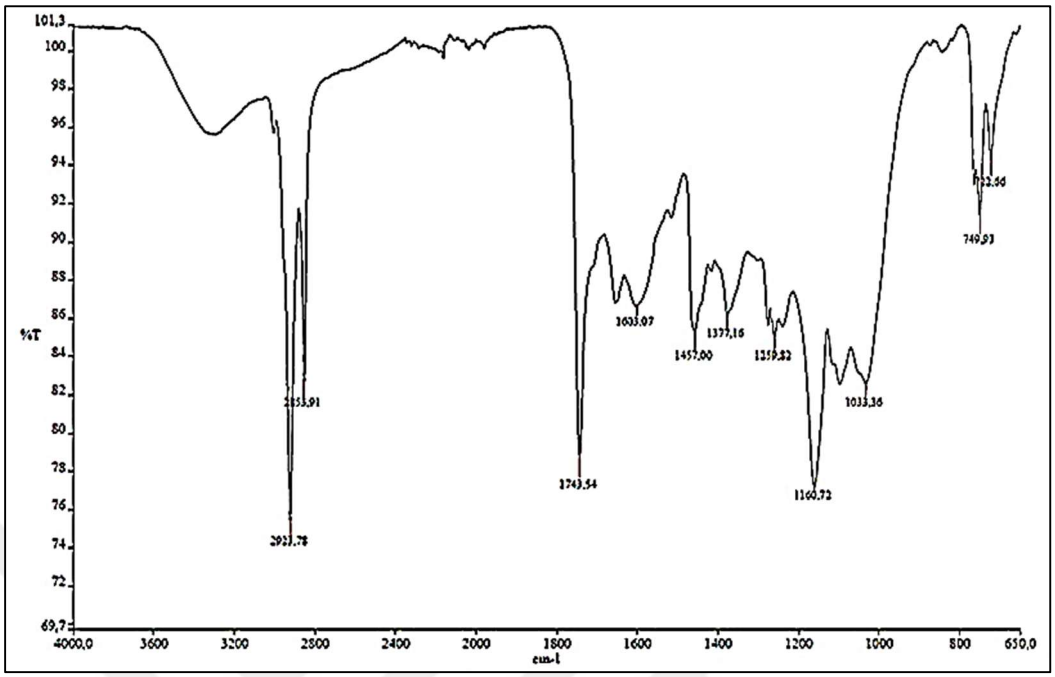


Figure 5.3: FTIR spectra of fluidized bed roasted *P. terebinthus* beans at 200 °C for a) 5 min.

(b)



(c)

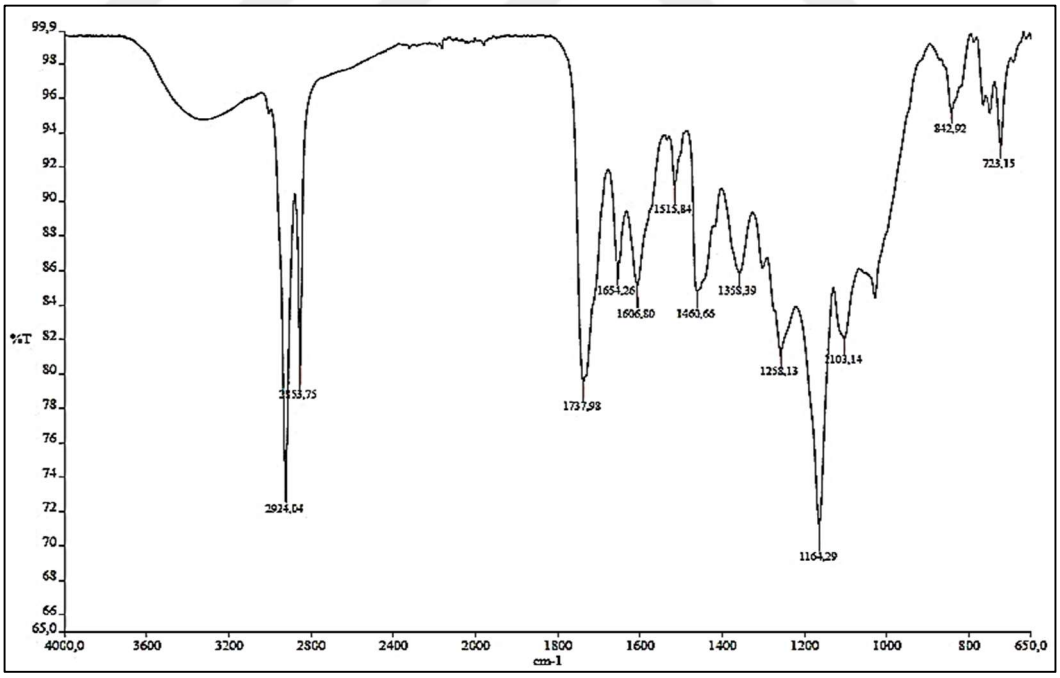
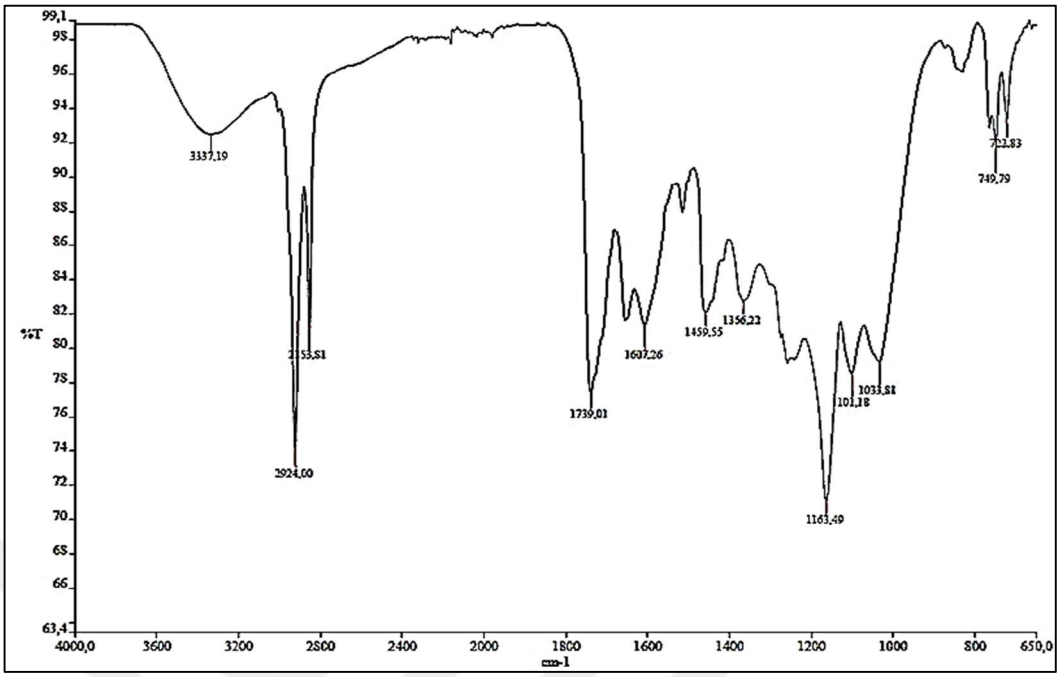


Figure 5.3 (Cont'd): FTIR spectra of fluidized bed roasted *P. terebinthus* beans at 200 °C for b) 20 min and c) 35 min.

(a)



(b)

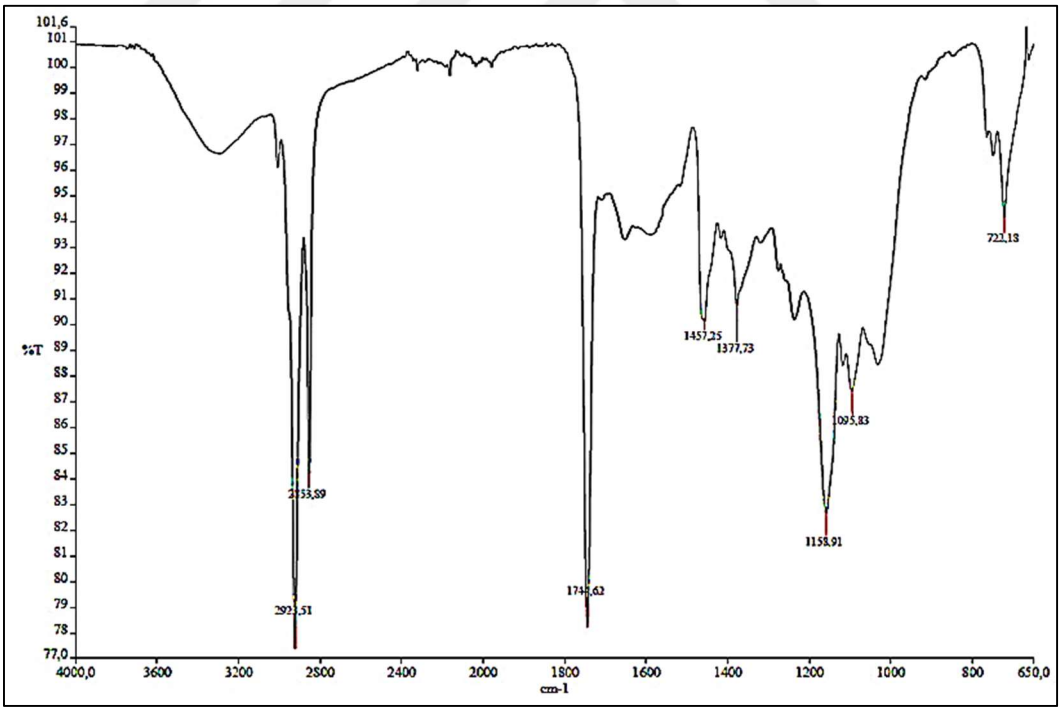


Figure 5.4: FTIR spectra of fluidized bed roasted *P. terebinthus* beans at 220 °C a) 5 min, b) 20 min.

(c)

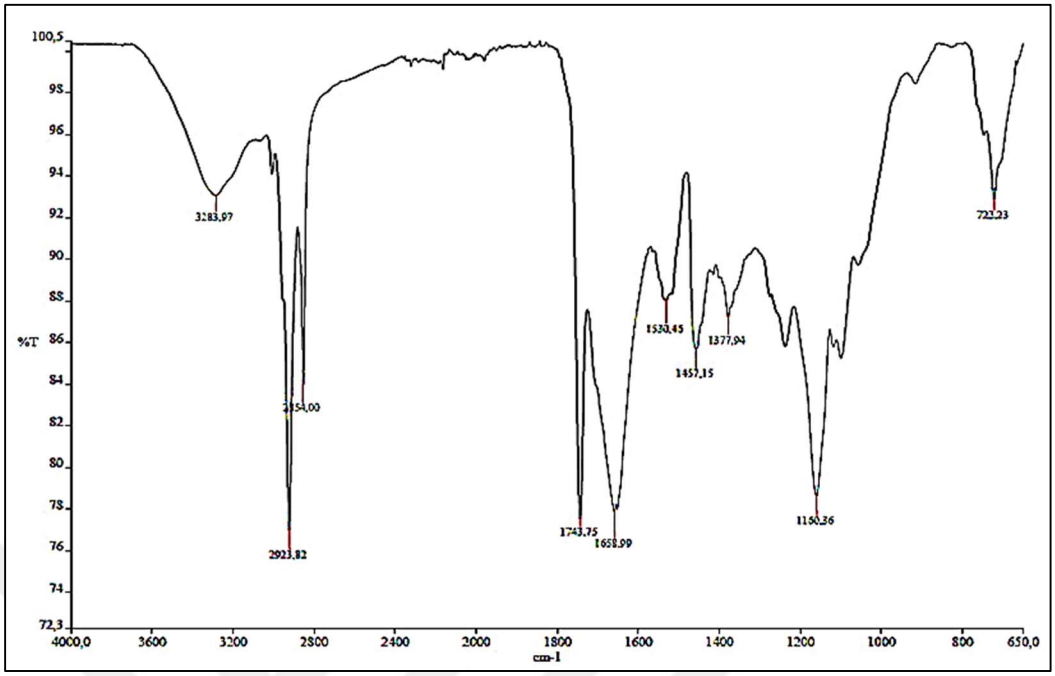


Figure 5.4 (Cont'd): FTIR spectra of fluidized bed roasted *P. terebinthus* beans at 220 °C c) 35 min.

(a)

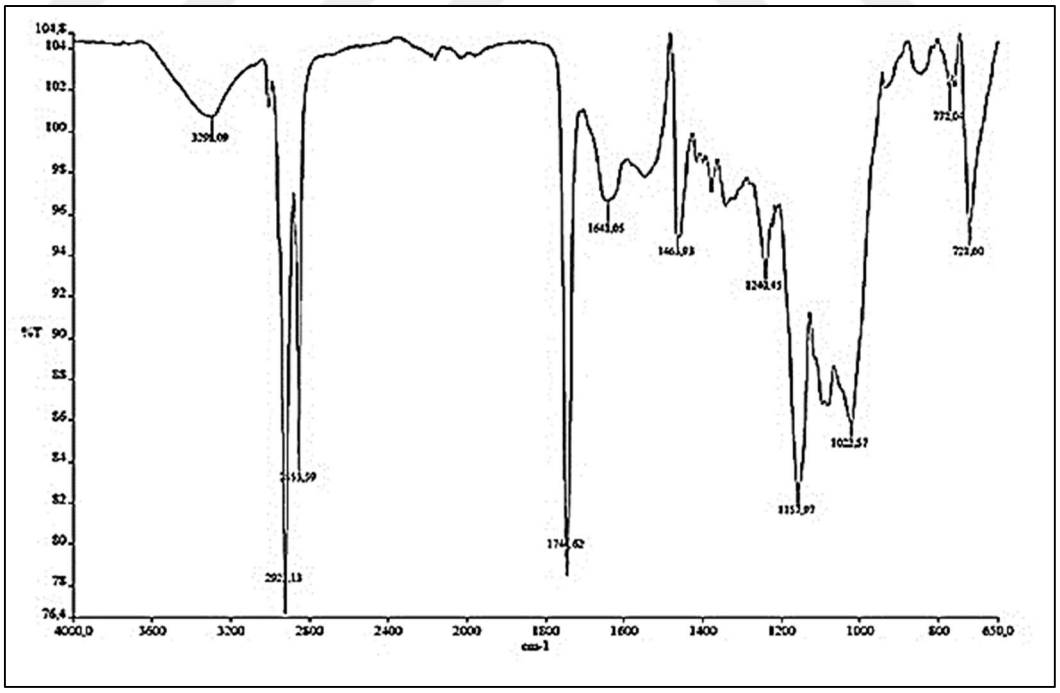
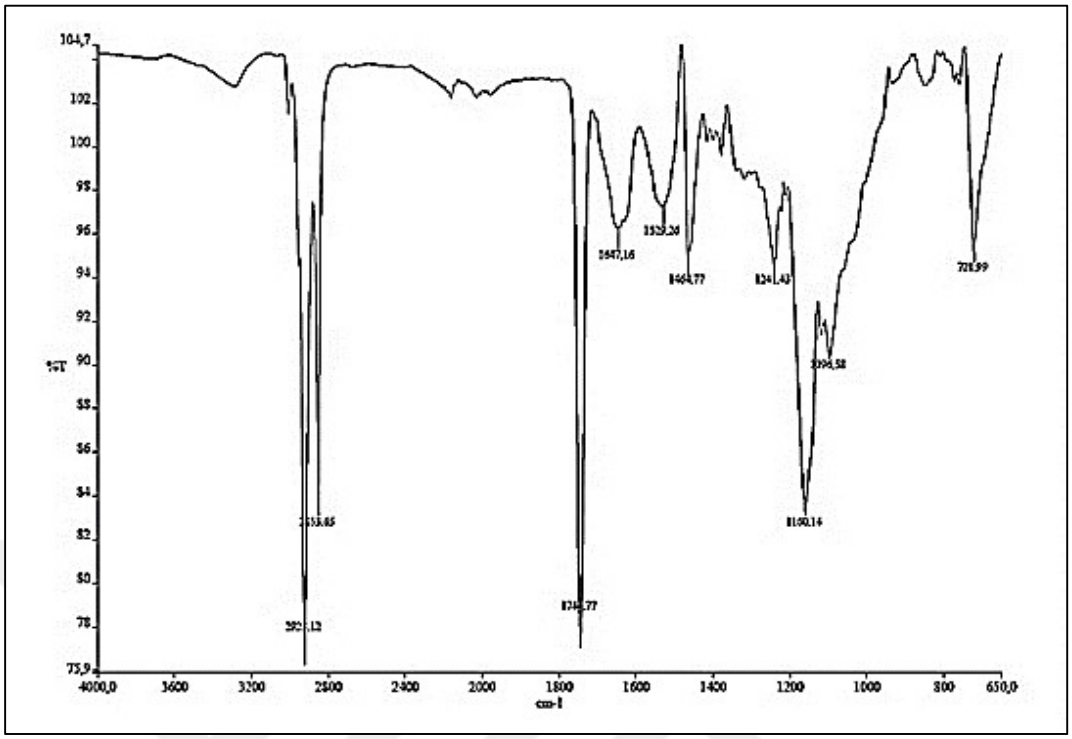


Figure 5.5: FTIR spectra of microwave roasted *P. terebinthus* beans at 360 W for a) 5 min.

(b)



(c)

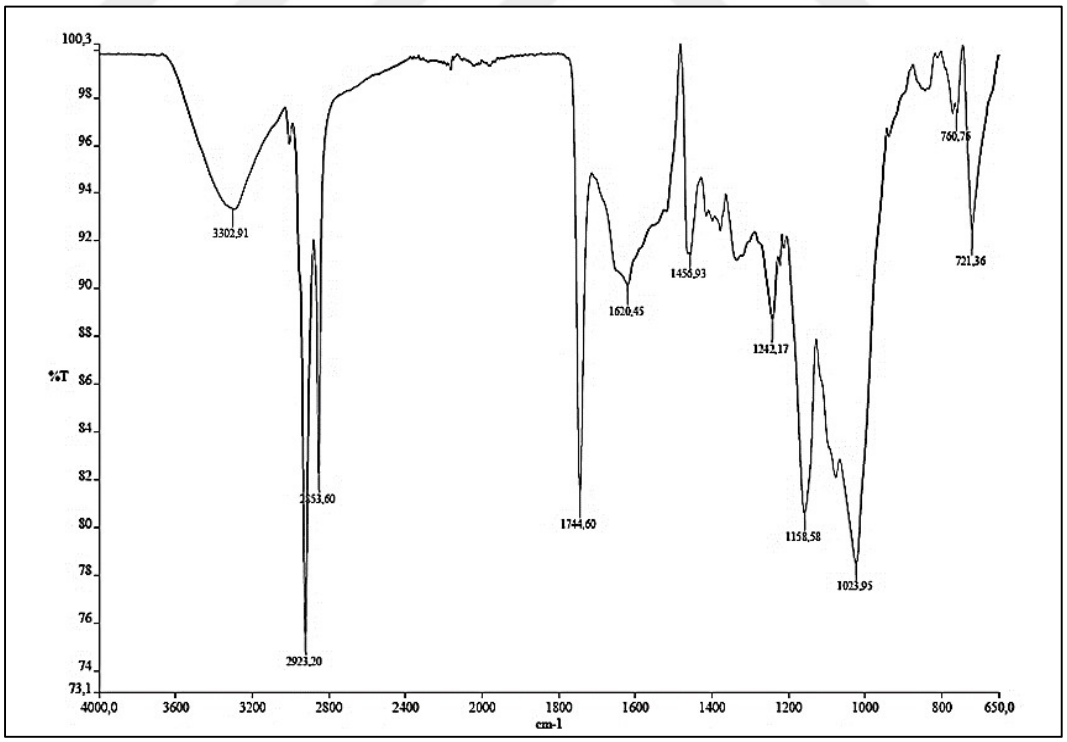
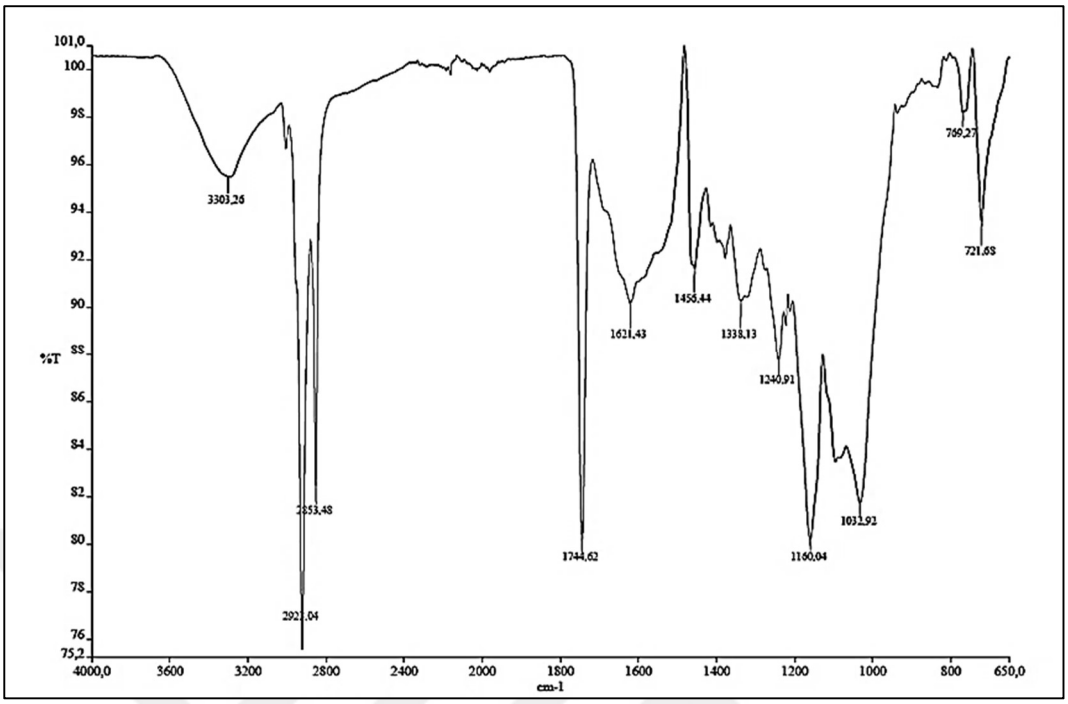


Figure 5.5 (Cont'd): FTIR spectra of microwave roasted *P. terebinthus* beans at 360 W for b) 11 min and c) 17 min.

(a)



(b)

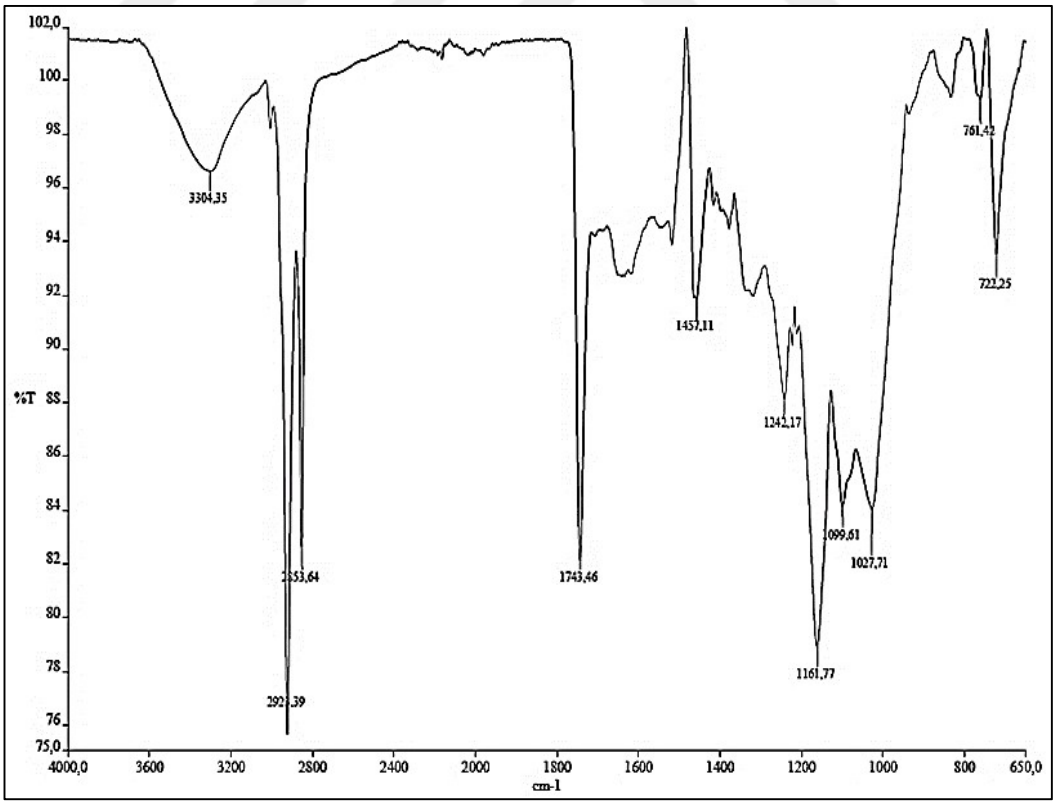


Figure 5.6: FTIR spectra of microwave roasted *P. terebinthus* beans at 540 W for a) 5 min, b) 11 min.

(c)

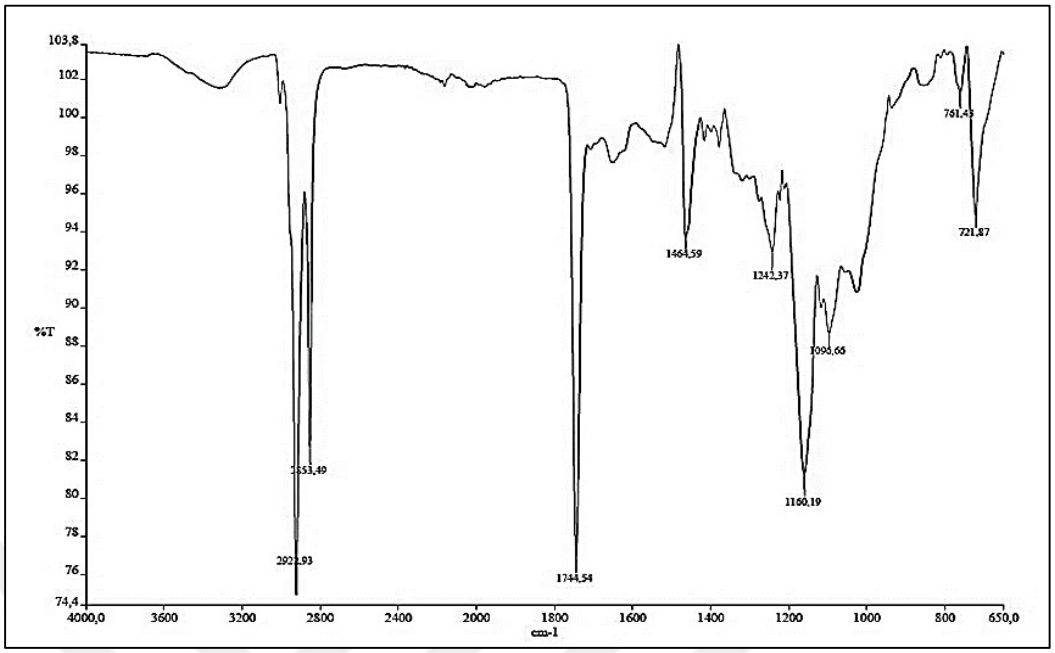


Figure 5.6 (Cont'd): FTIR spectra of microwave roasted *P. terebinthus* beans at 540 W for c) 17 min.

(a)

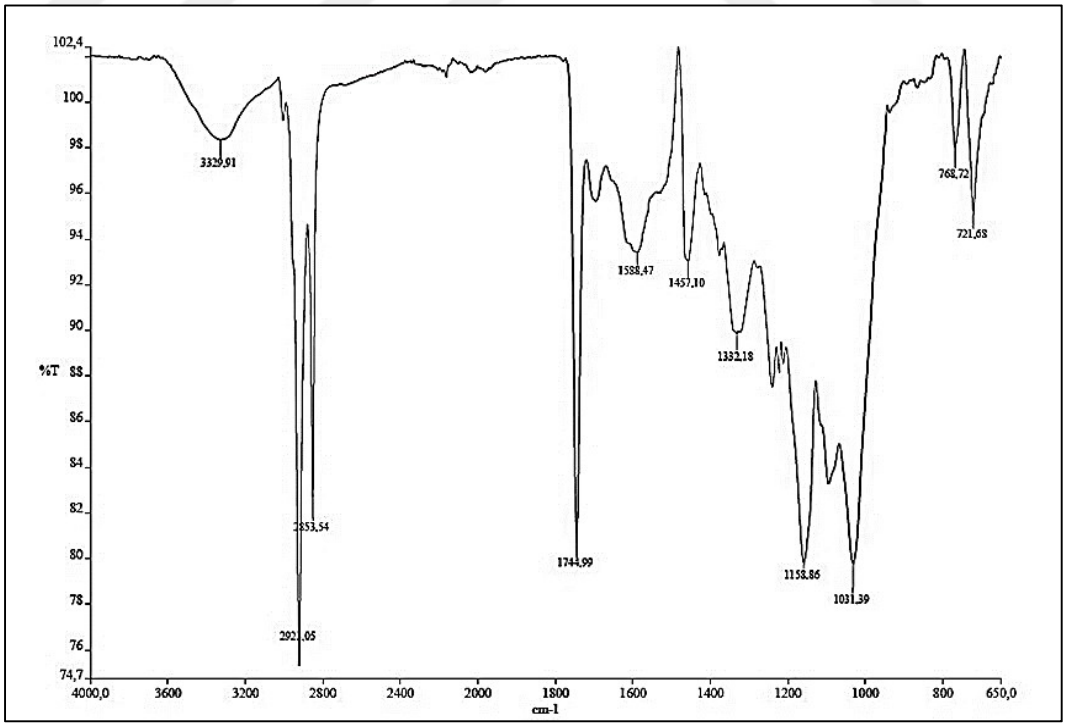
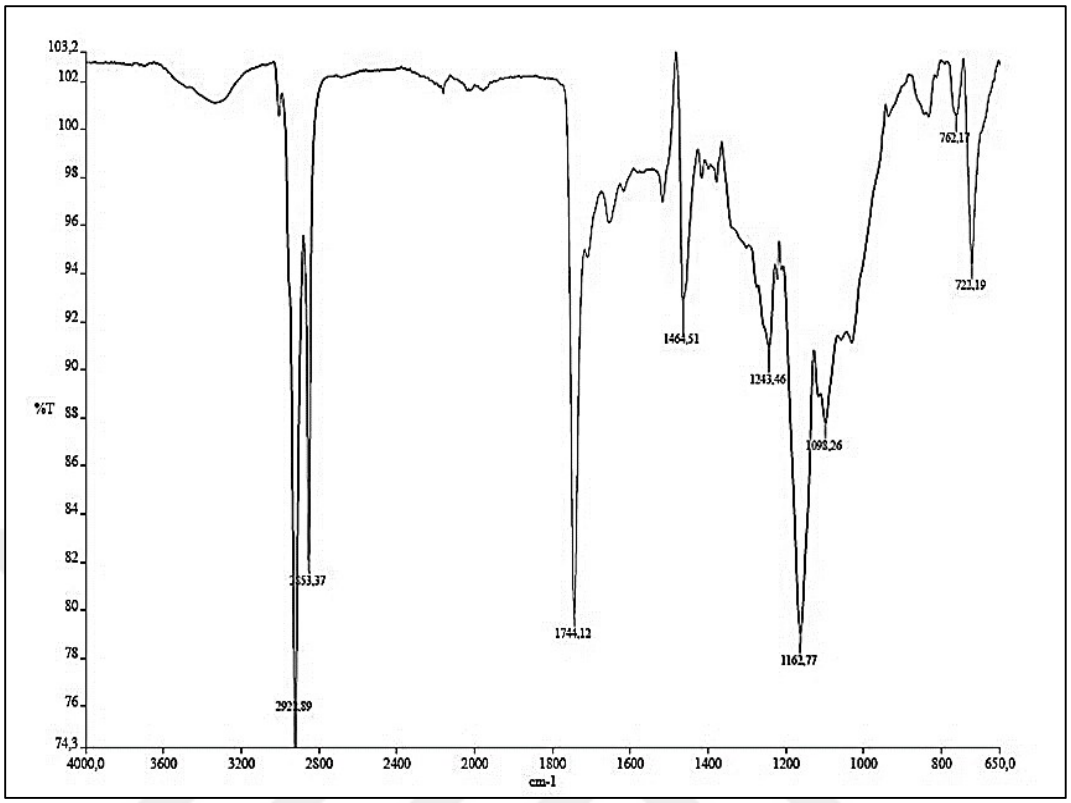


Figure 5.7: FTIR spectra of microwave roasted *P. terebinthus* beans at 720 W for a) 5 min.

(b)



(c)

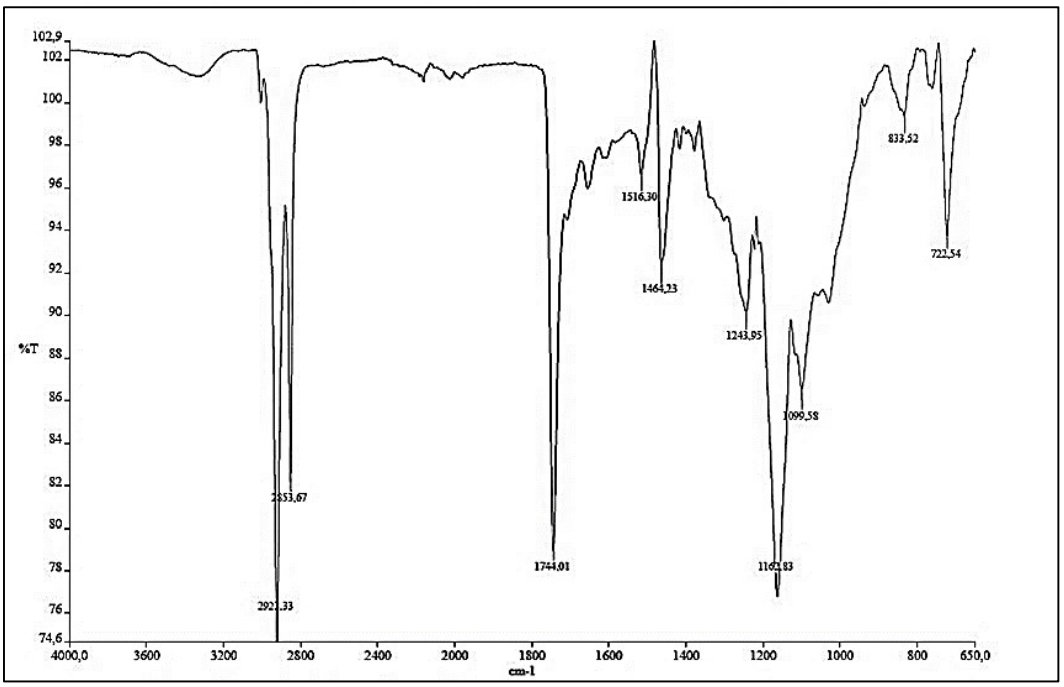


Figure 5.7 (Cont'd): FTIR spectra of microwave roasted *P. terebinthus* beans at 720 W for b) 11 min and c) 17 min.

5.3.1.2. Antioxidative Activity

Total antioxidant capacity reflects the presence of naturally occurring and newly formed antioxidant constituents in *P. terebinthus* beans during roasting or prior to roasting. Antioxidative activity of fluidized bed and microwave roasted *P. terebinthus* beans is presented in Tables 5.1 and 5.2, respectively. Results revealed that antioxidative activity of the *P. terebinthus* beans depends on the roasting temperature and roasting time for the fluidized bed roasted *P. terebinthus* beans, and microwave roasting power and roasting time for the microwave roasted *P. terebinthus* beans. Roasting caused an increase in the antioxidative activity of *P. terebinthus* beans as measured by the DPPH radical scavenging activity assay. Antioxidative activity of *P. terebinthus* beans gradually increased during roasting reaching to an apparent maximum at 200 °C for 20 min for fluidized bed roasted beans (Table 5.1) and 540 W for 14 min for microwave roasted beans (Table 5.2). There was a slight decrease in the antioxidant capacity of fluidized bed roasted *P. terebinthus* beans after a roasting temperature of 200 °C and a roasting time of 20 min while the decrease in the antioxidative capacity of microwave roasted *P. terebinthus* beans was noticeable after 540 W microwave power and 17 min of roasting. The behavior of antioxidative activity of *P. terebinthus* beans could be attributed to the importance of novel antioxidant substances that are formed during the roasting process such as Maillard reaction products, which are lost or undergone pyrolysis when the roasting proceeds under more severe conditions [Priftis et al., 2015], [Duarte et al., 2005].

EC_{50} (concentration required to obtain 50% antioxidant effect) is also used to express the antioxidative activity (Tables 5.1 and 5.2). The same trend observed in the DPPH radical scavenging activities of fluidized bed and microwave roasted *P. terebinthus* beans is also valid for EC_{50} values. These values are in close agreement with the DPPH radical scavenging activity results for fluidized bed and microwave roasted *P. terebinthus* beans. Durmaz and Gökmen extracted *P. terebinthus* oil from *P. terebinthus* beans and roasted the extracted oil at 180 °C for up to 40 min [Durmaz and Gökmen 2011]. Similar trend in the antioxidative activity of *P. terebinthus* oil was observed. del Castillo et al. roasted Colombian Arabica coffee beans at different roasting degrees. Maximum antioxidative activity was observed

for the medium roasted beans while lower antioxidative activity was obtained for dark roasted beans [del Castillo et al. 2002].

5.3.1.3. Total Phenolic Content (TPC)

TPC values were determined for the fluidized bed roasted (Table 5.1) and microwave roasted (Table 5.2) *P. terebinthus* beans. The maximum TPC (0.108 mg GAE/g extract) was attained at the roasting temperature of 200 °C and roasting time of 20 min for the fluidized bed roasted *P. terebinthus* beans while the maximum TPC (0.135 mg GAE/g extract) was obtained at 540 W for 17 min for the microwave roasted *P. terebinthus* beans. As similar to the antioxidative activity of roasted *P. terebinthus* beans, the TPC values were also dependent on the roasting temperature and roasting time for the fluidized bed roasted *P. terebinthus* beans, and microwave roasting power and roasting time for the microwave roasted *P. terebinthus* beans. The TPC of the fluidized bed and microwave roasted *P. terebinthus* beans increased during roasting reaching a maximum, followed by levelling off and a decreasing trend. This trend could be attributed to the formation of new phenolic compounds at lower roasting temperatures and lower microwave powers during the roasting [Vignoli et al., 2014] and the release of phenolic compounds from bound structures followed by chemical degradation of phenolics at higher temperatures and higher microwave powers [Veldsink et al., 1999], [Wijesundera et al., 2008]. The trend in TPC values of the fluidized bed and microwave roasted *P. terebinthus* beans observed in this work is in good agreement with the trend observed by Durmaz and Gökmen for TPC values of the *P. terebinthus* oil roasted at 180 °C for up to 40 min [Durmaz and Gökmen, 2011].

Table 5.1: Antioxidative activity and total phenolic content of fluidized bed roasted *P. terebinthus* beans*.

Roasting temperature (°C) and time (min)	% Inhibition (DPPH)	EC_{50} (mg sample/ml DPPH)	TPC (mg GAE/ 1000 g extract)
180, 5	20.12 ± 0.08	1.98 ± 0.02	54.04 ± 0.06
180, 20	22.26 ± 0.06	2.36 ± 0.04	65.42 ± 0.06
180, 35	24.42 ± 0.08	2.84 ± 0.02	76.73 ± 0.04
200, 5	25.67 ± 0.04	3.02 ± 0.01	82.42 ± 0.05
200, 20	28.45 ± 0.08	3.54 ± 0.02	108.46 ± 0.03
200, 35	27.53 ± 0.08	3.48 ± 0.01	106.24 ± 0.02
220, 5	27.10 ± 0.08	3.50 ± 0.01	102.23 ± 0.02
220, 20	25.32 ± 0.08	2.98 ± 0.01	85.62 ± 0.04
220, 35	20.40 ± 0.08	2.12 ± 0.02	78.48 ± 0.01

*Values are the means of three replications.

Table 5.2: Antioxidative activity and total phenolic content of microwave roasted *P. terebinthus* beans*.

Microwave power (W) and time (min)	% Inhibition (DPPH)	EC_{50} (mg sample/ml DPPH)	TPC (mg GAE/ 1000 g extract)
360, 5	22.64 ± 0.02	2.92 ± 0.01	84.25 ± 0.07
360, 11	24.76 ± 0.02	3.18 ± 0.01	95.62 ± 0.05
360, 17	28.65 ± 0.01	3.42 ± 0.01	104.88 ± 0.08
540, 5	26.67 ± 0.01	3.36 ± 0.02	98.42 ± 0.08
540, 11	29.53 ± 0.02	3.58 ± 0.02	129.45 ± 0.05
540, 17	34.45 ± 0.01	3.72 ± 0.01	135.46 ± 0.07
720, 5	32.49 ± 0.02	3.68 ± 0.01	131.23 ± 0.06
720, 11	32.42 ± 0.02	3.64 ± 0.02	126.62 ± 0.05
720, 17	28.36 ± 0.02	3.02 ± 0.02	102.48 ± 0.06

*Values are the means of three replications.

5.3.1.4. Protein Content

The change in crude protein contents of fluidized bed roasted and microwave roasted *P. terebinthus* beans is given in Tables 5.3 and 5.4, respectively. The crude protein content of fluidized bed roasted *P. terebinthus* beans changed between

0.909-0.926 $g.kg^{-1}$ while the crude protein content of microwave roasted *P. terebinthus* beans varied from 0.908 to 0.924 $g.kg^{-1}$. At each roasting condition, crude protein contents of both fluidized bed and microwave roasted *P. terebinthus* beans decreased slightly as the roasting time increased. This could be attributed to the denaturation of some of the proteins [Parliment, 2000] and Maillard reactions during roasting as the roasting conditions become more severe. The results presented in this work are compatible with the findings of de Maria et al. who determined that protein content of Arabica coffee decreased during roasting [de Maria et al. 1996].

5.3.1.5. Oil Content

Oil content of green *P. terebinthus* beans in this study was found to be 3 $g.kg^{-1}$ which was close to the oil content (approximately 4 $g.kg^{-1}$) of green *P. terebinthus* fruit reported by Özcan and Secilmis et al. [Özcan, 2004], [Secilmis et al., 2015]. Total oil contents of the fluidized bed roasted and microwave roasted *P. terebinthus* beans are given in Tables 5.3 and 5.4, respectively. *P. terebinthus* beans fluidized bed roasted at 220 °C for 35 minutes have the highest oil content (4.034 $g.kg^{-1}$) among the other fluidized bed roasted *P. terebinthus* beans while the highest oil content (4.068 $g.kg^{-1}$) was obtained at the microwave power of 720 W and roasting time of 17 min for microwave roasted *P. terebinthus* beans. At each roasting condition, a slight increase in oil contents of both fluidized bed and microwave roasted *P. terebinthus* beans was observed, and this could be due to the formation of substances during roasting which are soluble in oil. A slight increase in oil content of roasted Arabica coffee beans occurred with increasing the roasting time from 30 min to 2 h when the beans were roasted at 200 °C [Vasconcelos et al., 2007]. Budryn et al. roasted Robusta coffee beans from a roasting temperature of 190 to 216 °C between the roasting times 20 and 35 min. They observed that the oil content of Robusta coffee beans increased up to 9.8% depending on the roasting conditions [Budryn et al. 2012].

5.3.1.6. Ash Content

Ash content is an account of the inorganic matter found in *P. terebinthus* beans. The inorganic matter in roasted *P. terebinthus* beans varied from 0.462 $g.kg^{-1}$ to 0.522 $g.kg^{-1}$ (Tables 5.3 and 5.4). A slight decrease in ash contents was observed as the roasting proceeded. This result could be attributed to damaging of some inorganic materials during roasting.

5.3.1.7. pH Measurements

pH is important during storage of roasted beans because it affects stability and shelf life. The highest pH value (6.11) for the fluidized bed roasted *P. terebinthus* beans were observed for the beans roasted at 180 °C for 5 min (Table 5.3) while the highest pH (6.14) for the microwave roasted *P. terebinthus* beans was obtained at 540 W microwave power for 17 min of roasting time (Table 5.4). The pH decreased or increased depending on the roasting conditions used. The decrease in pH could be attributed to the formation of acids from the degradation of sugars in the *P. terebinthus* beans during roasting. On the other hand, the increase in pH could be interpreted as the destruction of organic acids formed and/or those that were present initially in the beans as the roasting conditions change. Wang and Lim roasted Brazilian coffee beans at 210, 220, 230, and 240 °C, and they reported a decrease and an increase in pH depending on the roasting conditions [Wang and Lim, 2012]. Similar trends in pH during roasting were also reported by Mwithiga and Jindal, Somporn et al. and Dutra et al. for Arabica coffee beans [Mwithiga and Jindal, 2007], [Somporn et al., 2011], [Dutra et al., 2001].

Table 5.3: Protein, oil and ash contents and pH values of fluidized bed roasted *P. terebinthus* beans.

Temperature (°C) and time (min)	Protein content ($g.kg^{-1}$)	Oil content ($g.kg^{-1}$)	Ash content ($g.kg^{-1}$)	pH
180, 5	0.926 ± 0.06	3.834 ± 0.08	0.520 ± 0.08	6.11 ± 0.03
180, 20	0.923 ± 0.05	3.872 ± 0.07	0.512 ± 0.08	6.02 ± 0.01
180, 35	0.921 ± 0.05	3.913 ± 0.07	0.53.2 ± 0.07	6.06 ± 0.02
200, 5	0.922 ± 0.06	3.902 ± 0.08	0.496 ± 0.07	6.03 ± 0.01
200, 20	0.918 ± 0.03	3.935 ± 0.08	0.488 ± 0.04	5.94 ± 0.01
200, 35	0.916 ± 0.03	3.972 ± 0.09	0.472 ± 0.04	5.98 ± 0.01
220, 5	0.917 ± 0.02	3.994 ± 0.08	0.470 ± 0.07	5.96 ± 0.01
220, 20	0.914 ± 0.06	4.020 ± 0.07	0.484 ± 0.06	6.02 ± 0.02
220, 35	0.909 ± 0.06	4.034 ± 0.06	0.492 ± 0.05	6.06 ± 0.02

*Values are means of three replications.

Table 5.4: Protein, oil and ash contents and pH values of microwave roasted *P. terebinthus* beans.

Microwave power (W) and time (min)	Protein content ($g.kg^{-1}$)	Oil content ($g.kg^{-1}$)	Ash content ($g.kg^{-1}$)	pH
360, 5	0.924 ± 0.04	3.852 ± 0.08	0.518 ± 0.01	6.07 ± 0.01
360, 11	0.922 ± 0.04	3.895 ± 0.08	0.522 ± 0.02	5.95 ± 0.01
360, 17	0.919 ± 0.02	3.936 ± 0.06	0.491 ± 0.01	6.01 ± 0.02
540, 5	0.921 ± 0.01	3.962 ± 0.06	0.488 ± 0.01	5.97 ± 0.01
540, 11	0.918 ± 0.02	3.984 ± 0.08	0.490 ± 0.02	5.91 ± 0.01
540, 17	0.916 ± 0.02	3.996 ± 0.04	0.462 ± 0.01	5.93 ± 0.01
720, 5	0.917 ± 0.01	4.023 ± 0.07	0.468 ± 0.02	5.92 ± 0.01
720, 11	0.912 ± 0.02	4.057 ± 0.05	0.476 ± 0.02	6.02 ± 0.02
720, 17	0.908 ± 0.04	4.068 ± 0.06	0.484 ± 0.01	6.14 ± 0.01

*Values are means of three replications.

5.3.2. Results for Structural and Thermal Analysis

5.3.2.1. SEM Analysis of Roasted *P. terebinthus* Beans

SEM micrographs revealed morphological changes on the external surface of fluidized bed roasted (Figures 5.8 and 5.9) and microwave roasted (Figures 5.10 and 5.11) *P. terebinthus* beans. Fluidized bed and microwave roasting conditions resulted in *P. terebinthus* beans to lose their structural integrity. Cracks, fractures and oil droplets on the external surface of the fluidized bed and microwave roasted *P. terebinthus* beans were observed. Oil secretion is noticeable in both fluidized bed and microwave roasted *P. terebinthus* beans as exemplified by the SEM micrographs. An increase in the roasting temperature and roasting time increased the oil secretion due to the severe disruption of intercellular structure of fluidized bed roasted *P. terebinthus* beans extensively (Figure 5.9). The oil secretion is more pronounced in microwave roasted *P. terebinthus* beans because more coalescence of oil droplets has occurred with increasing the microwave roasting power and microwave roasting time (Figure 5.11). Compared to the fluidized bed roasting, microwave roasting increased the oil diffusion from the bean core to the external surface.

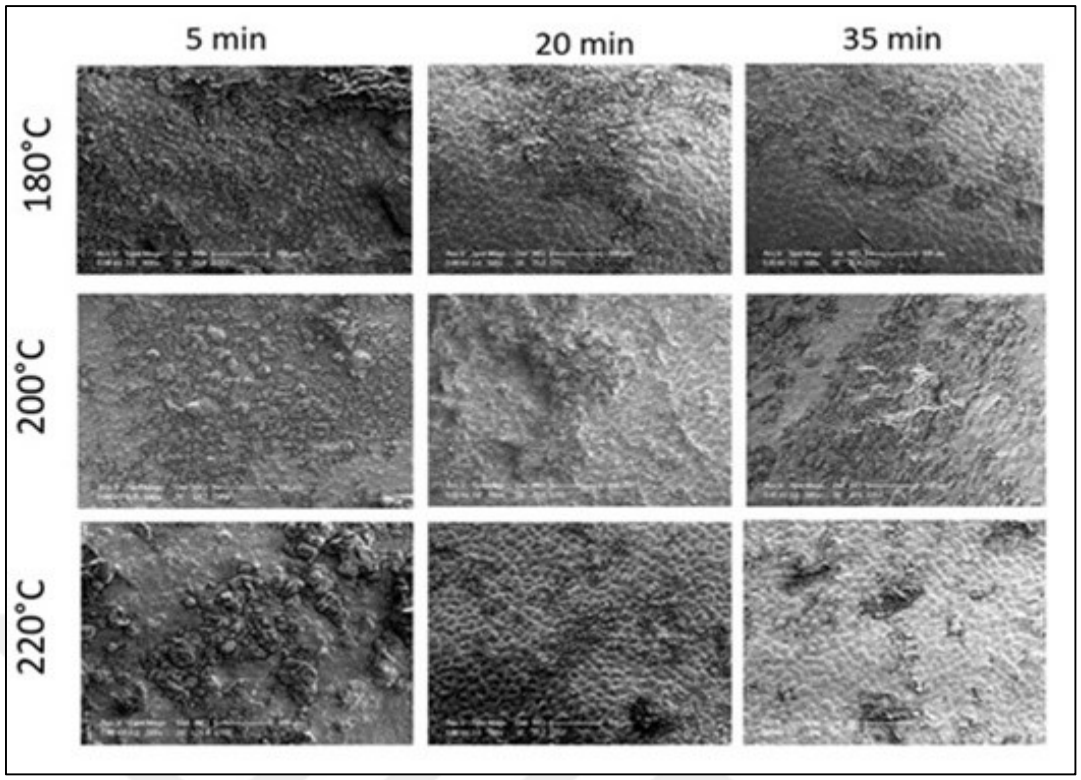


Figure 5.8: SEM micrographs of external surface of fluidized bed roasted *P. terebinthus* beans. Magnification is 500×. Scale bar = 100 µm.

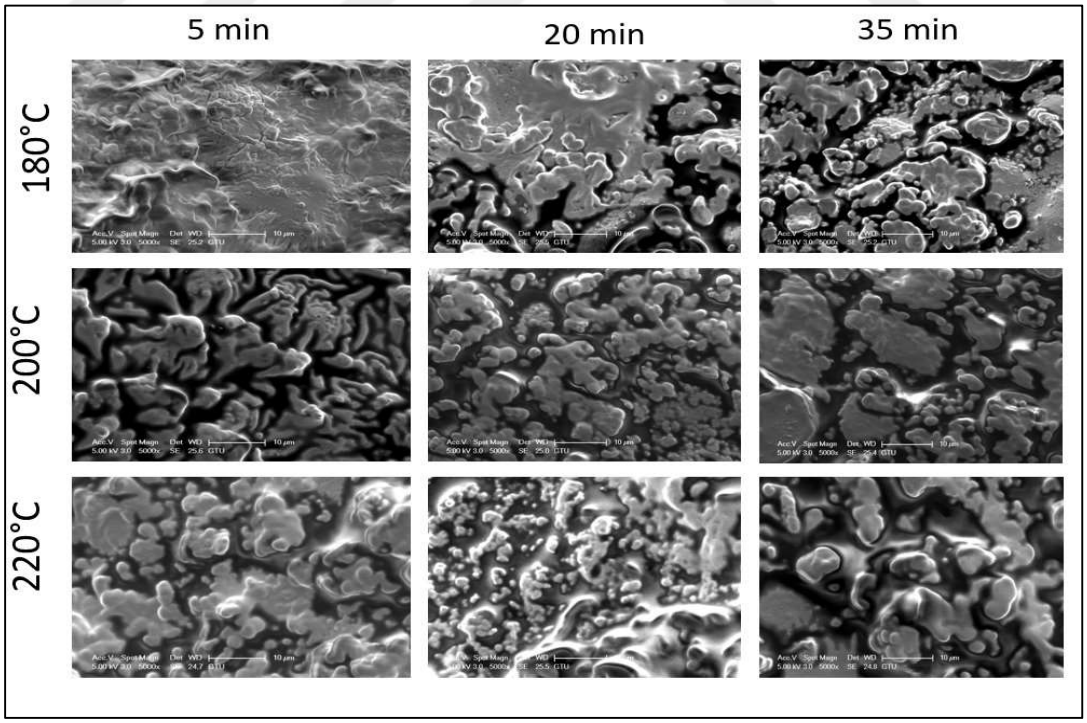


Figure 5.9: SEM micrographs of external surface of fluidized bed roasted *P. terebinthus* beans. Magnification is 5000×. Scale bar = 10 µm.

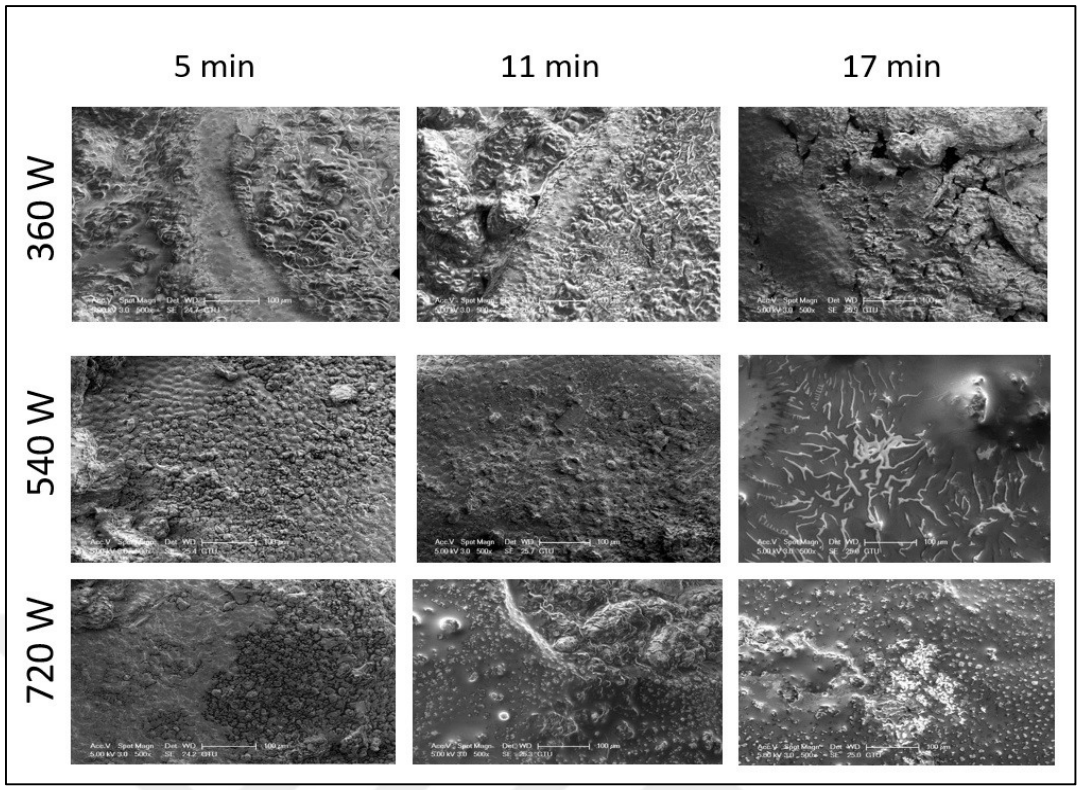


Figure 5.10: SEM micrographs of external surface of microwave roasted *P. terebinthus* beans. Magnification is 500×. Scale bar = 100 µm.

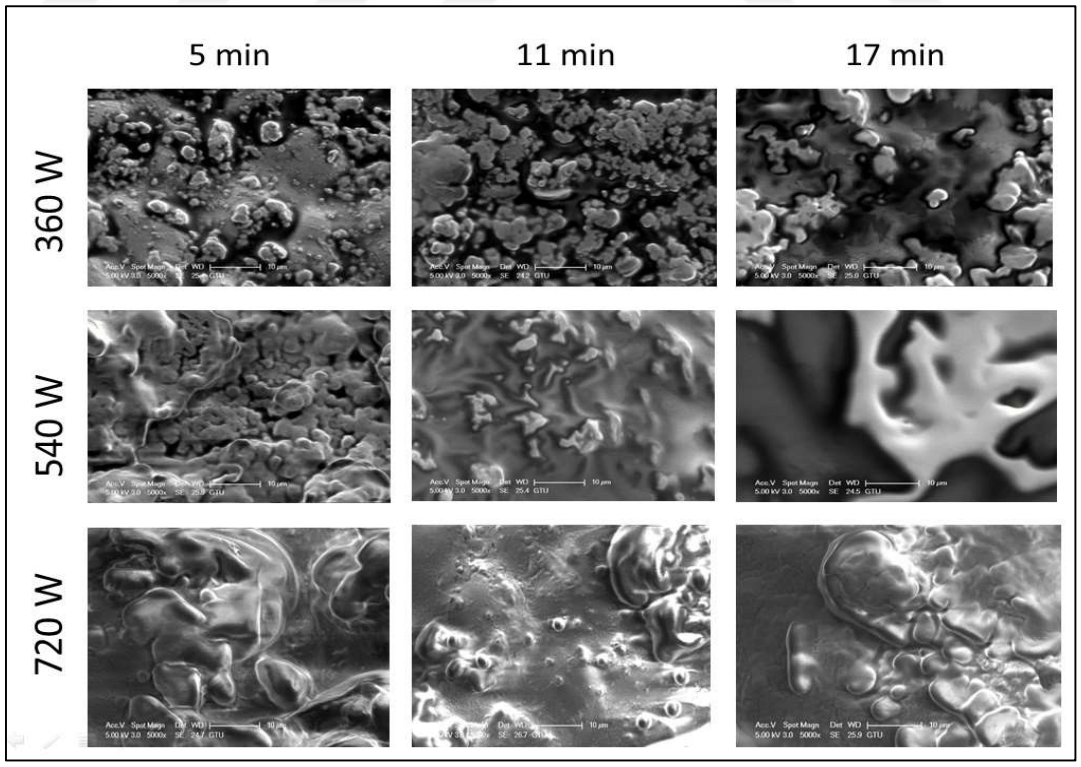


Figure 5.11: SEM micrographs of external surface of microwave roasted *P. terebinthus* beans. Magnification is 5000×. Scale bar = 10 µm.

5.3.2.2. DSC Analysis of Roasted *P. terebinthus* Beans

The evolution of the heat flow as a function of temperature gives information about the reversible and irreversible changes that a material has gone through. This information is important to determine the thermal phenomena or thermal transitions occurring during the roasting process. Pure substances can be characterized by unique and sharp thermal transition points, but the thermal transitions in DSC thermograms of foods may not be as clear as those of pure substances due to the chemical complexity and the presence of many ingredients like proteins, carbohydrates, fats and oils, flavors, aromas and numerous volatile compounds in foods.

DSC curve of unroasted *P. terebinthus* beans is given in Figure 5.12 in which unroasted *P. terebinthus* beans have an endothermic process of melting around 125 °C. DSC thermograms of fluidized bed roasted *P. terebinthus* beans at optimum conditions of 205 °C and 15 min and microwave roasted *P. terebinthus* beans at optimum conditions of 540 W and 14 min are presented in Figures 5.13 and 5.14, respectively. Basically, three thermal phenomena were observed in DSC thermograms of roasted *P. terebinthus* beans. An inflexion is associated to a glass transition around 57 °C for the fluidized bed and microwave roasted *P. terebinthus* beans. Rivera et al. determined a glass transition of 59 °C for roasted coffee beans of two different varieties, which is very close to the glass transition of the fluidized bed and microwave roasted *P. terebinthus* beans [Rivera et al., 2011]. A very wide and intense endothermic peak corresponding to the endothermic process of melting was determined around 136 °C and 155 °C for the fluidized bed and microwave roasted *P. terebinthus* beans, respectively. These endothermic peaks of melting are believed to originate from the melting of some constituents mainly proteins, carbohydrates and lipids present in *P. terebinthus* beans at the selected roasting conditions. Brondi et al. also observed a similar behavior in Arabica coffee beans roasted at temperatures of 235-250 °C for 15-25 min [Brondi et al., 2017]. The transitions at 230 °C for the fluidized bed roasted and at 238 °C for the microwave roasted *P. terebinthus* beans were determined. The thermal meaning of these transitions is associated with the decomposition process during roasting of *P. terebinthus* beans. Therefore, roasting process should be closely controlled to obtain *P. terebinthus* beans without severe thermal decomposition. Similar transitions and thermal

decomposition at 218 °C were reported by Rivera et al. for the Arabica coffee beans roasted at different temperatures ranging from 210 to 230 °C and roasting times up to 10 minutes [Rivera et al., 2011].

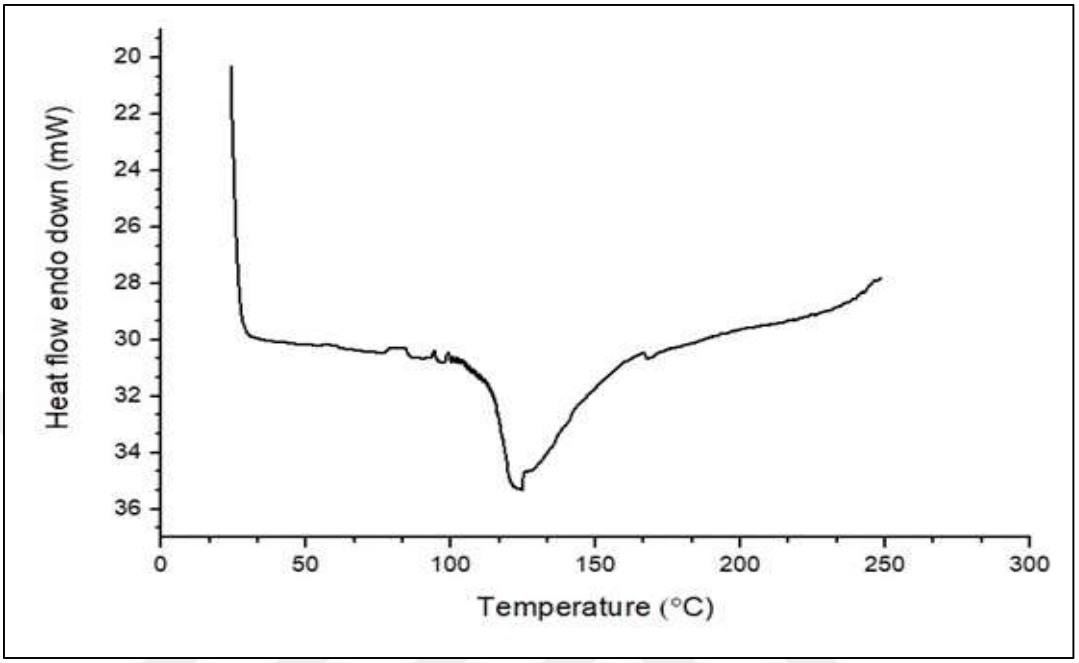


Figure 5.12: DSC curve of unroasted *P. terebinthus* beans.

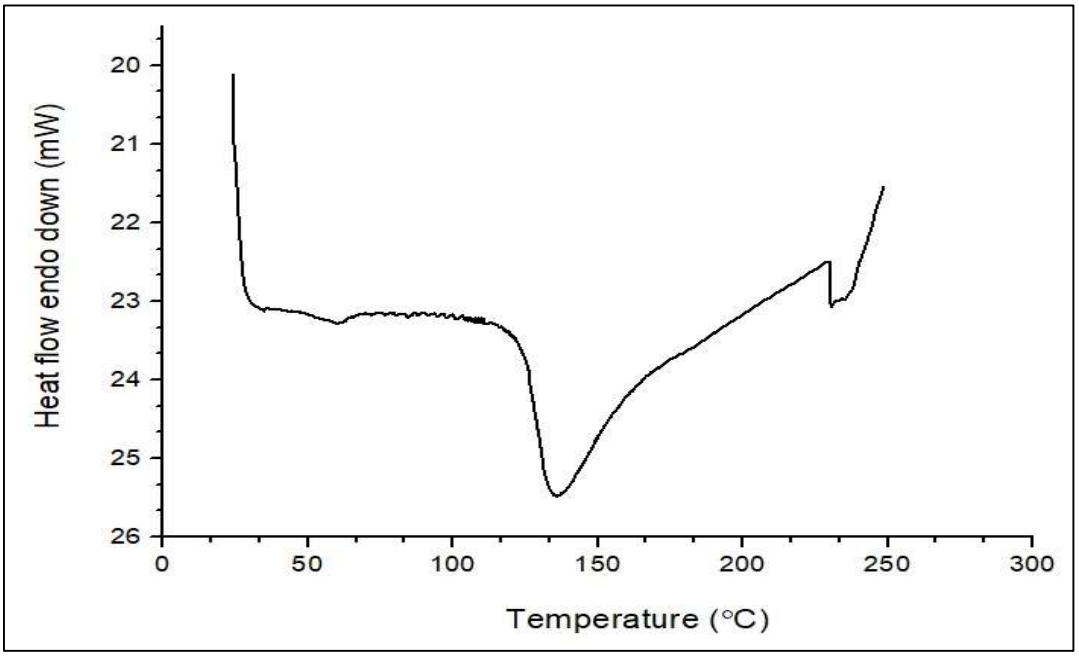


Figure 5.13: DSC curve of fluidized bed roasted *P. terebinthus* beans at 205 °C for 15 min.

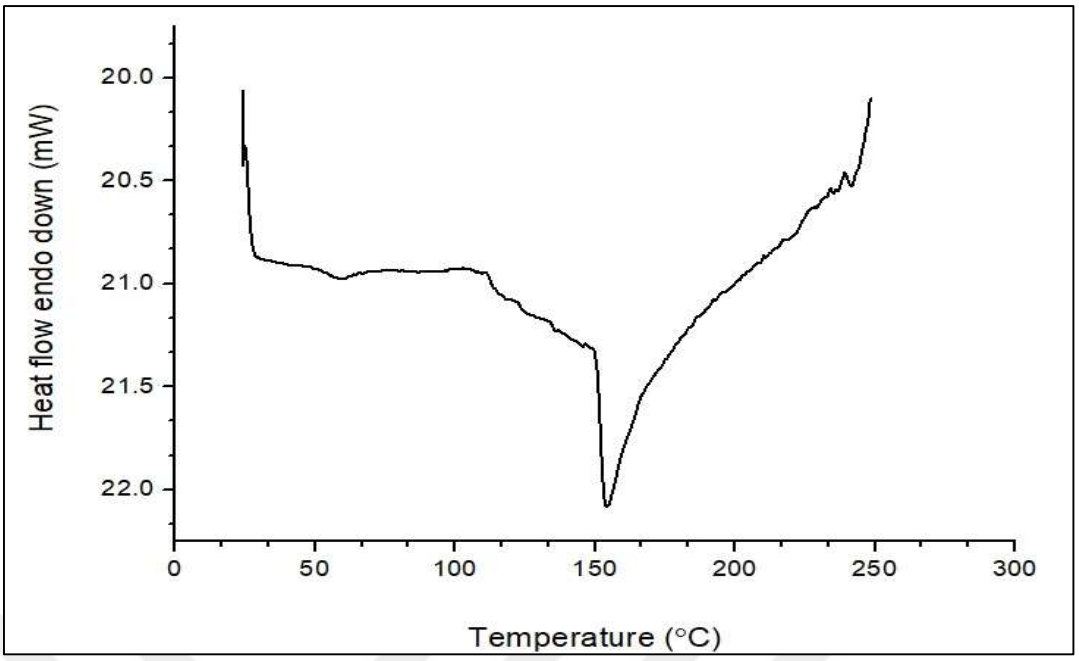


Figure 5.14: DSC curve of microwave roasted *P. terebinthus* beans at 540 W for 14 min.

5.3.2.3. TG Analysis of Roasted *P. terebinthus* Beans

TGA spectrum shows the changes of weight in relation to the change of temperature that a material has gone through. This information is valuable to determine the decomposition phenomena occurring during the roasting process.

TGA spectra of unroasted, fluidized bed roasted (at optimum conditions of 205 °C and 15 min) and microwave roasted (at optimum conditions of 540 W and 14 min) *P. terebinthus* beans are given from Figures 5.15 to 5.17, respectively. Unroasted, fluidized bed and microwave roasted *P. terebinthus* beans exhibited similar TGA curves with three decomposition stages. Stage I was ranging from approximately 180 to 235 °C for the fluidized bed roasted *P. terebinthus* beans while stage I was within the temperature range of approximately 165-245 °C for the microwave roasted *P. terebinthus* beans. At the stage I, 2% loss in volatiles occurred in the fluidized bed roasted *P. terebinthus* beans, but the loss in volatiles reached to 5% for the microwave roasted *P. terebinthus* beans. Rivera et al. found a loss between 6 and 10% for the Arabica coffee beans roasted at different temperatures ranging from 210 to 230 °C and roasting times up to 10 minutes. The results for weight losses at the stage I for roasted *P. terebinthus* beans were lower than the

weight loss values reported by Rivera et al. for the roasted coffee [Rivera et al. 2011]. The greatest decompositions and weight losses occurred during the stage II. At the stage II, ranging from approximately 235 to 450 °C for the fluidized bed roasted and from approximately 245 to 450 °C for the microwave roasted *P. terebinthus* beans, decompositions corresponding to 68 and 73% weight losses for the fluidized bed and microwave roasted *P. terebinthus* beans, respectively, are much more pronounced than those of the weight losses at the stage I. The reason of this pronounced decomposition at the stage II could be attributed to the decomposition of some oils and depolymerization of cellulose and hemicellulose in the beans [Chiou et al., 2015], [Buratti et al., 2018]. During the stage II, hemicellulose, cellulose and lignin were decomposed and released from the biomass, contributing approximately to 75% of weight loss for the spent coffee grounds [Li et al., 2014]. The last stage of the TGA curves was used to determine the % residues. Stage III corresponds to the temperatures between 450 and 900 °C where approximately 12% loss occurred and a char residue of around 18% were formed for the fluidized bed roasted *P. terebinthus* beans while approximately 8% loss was noticeable and 14% of char residue was obtained for the microwave roasted *P. terebinthus* beans. Li et al. found char residue of approximately 17.5% for the spent coffee grounds [Li et al., 2014], but roasted Arabica coffee beans yielded the char residue between 2.5 and 5.6% [Rivera et al., 2011] which is lower than the char residue of the roasted *P. terebinthus* beans.

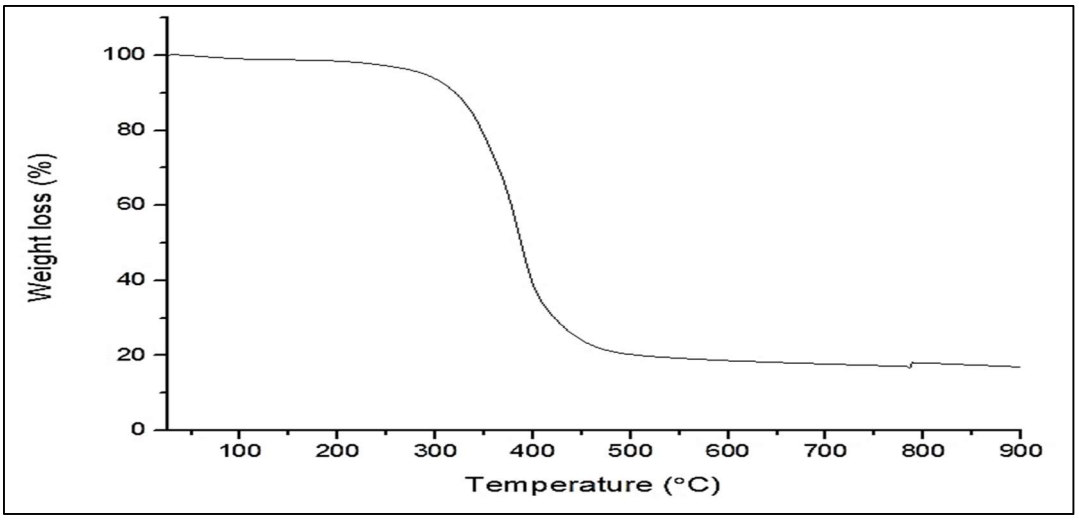


Figure 5.15: TGA spectrum of unroasted *P. terebinthus* beans.

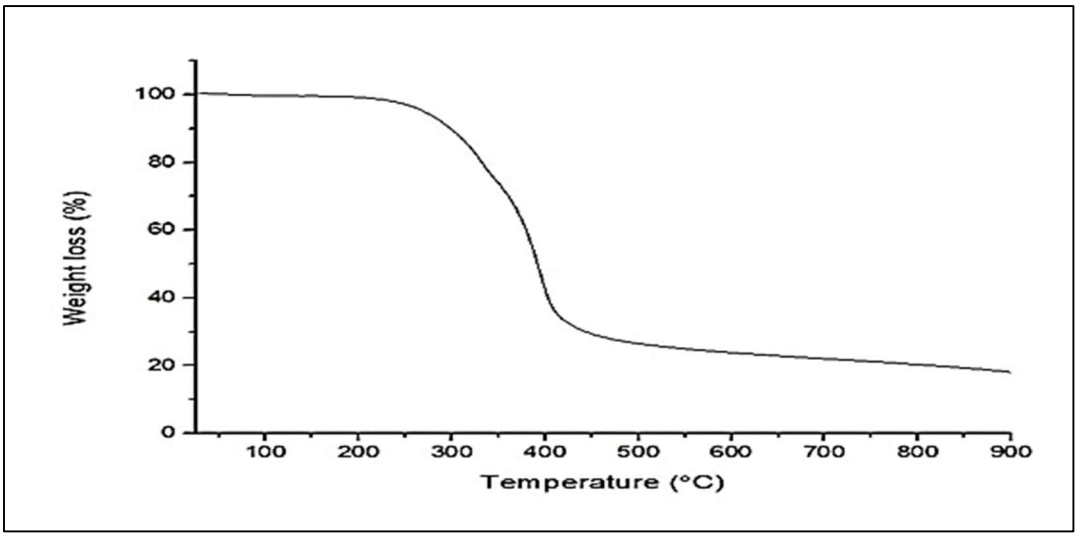


Figure 5.16: TGA spectrum of fluidized bed roasted *P. terebinthus* beans at 205 °C for 15 min.

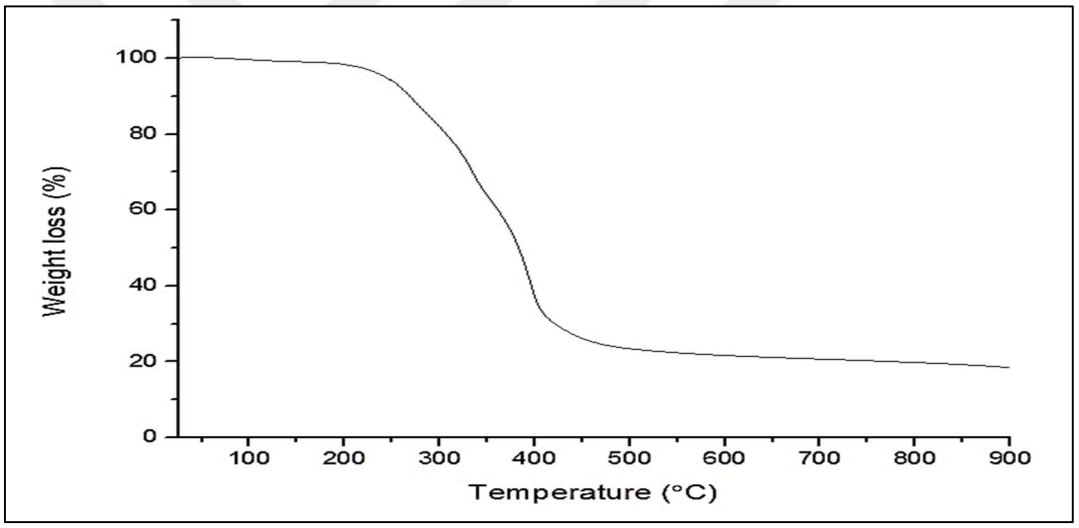


Figure 5.17: TGA spectrum of microwave roasted *P. terebinthus* beans at 540 W for 14 min.

5.3.2.4. XRD Analysis of Roasted *P. terebinthus* Beans

XRD technique allows the identification and characterization of the crystalline structures in a material. XRD pattern of unroasted, fluidized bed roasted (at optimum conditions of 205 °C and 15 min) and microwave roasted (at optimum conditions of 540 W and 14 min) *P. terebinthus* beans are given from Figures 5.18 to 5.20. Fluidized bed and microwave roasted *P. terebinthus* beans showed similar XRD patterns. Compared to the XRD curve of unroasted *P. terebinthus* beans, there are

two extra diffraction peaks at around 2θ = 30° and 2θ = 35° for the fluidized bed and microwave roasted *P. terebinthus* beans, respectively. This indicated a change in the crystalline regions in the structure of roasted *P. terebinthus* beans when the *P. terebinthus* beans were exposed to thermal treatment. Rivera et al. also observed a change in the crystallinity of Arabica coffee when it was subjected to roasting conditions ranging from 210 to 230 °C and roasting times up to 10 minutes [Rivera et al. 2011]. In addition, the diffraction peaks at 2θ = 4.2° and 2θ = 20° in the XRD diffractogram of unroasted *P. terebinthus* beans shifted to 2θ = 4.6° and 2θ = 20.4° for fluidized bed roasted, and 2θ = 5.5° and 2θ = 21° for microwave roasted *P. terebinthus* beans.

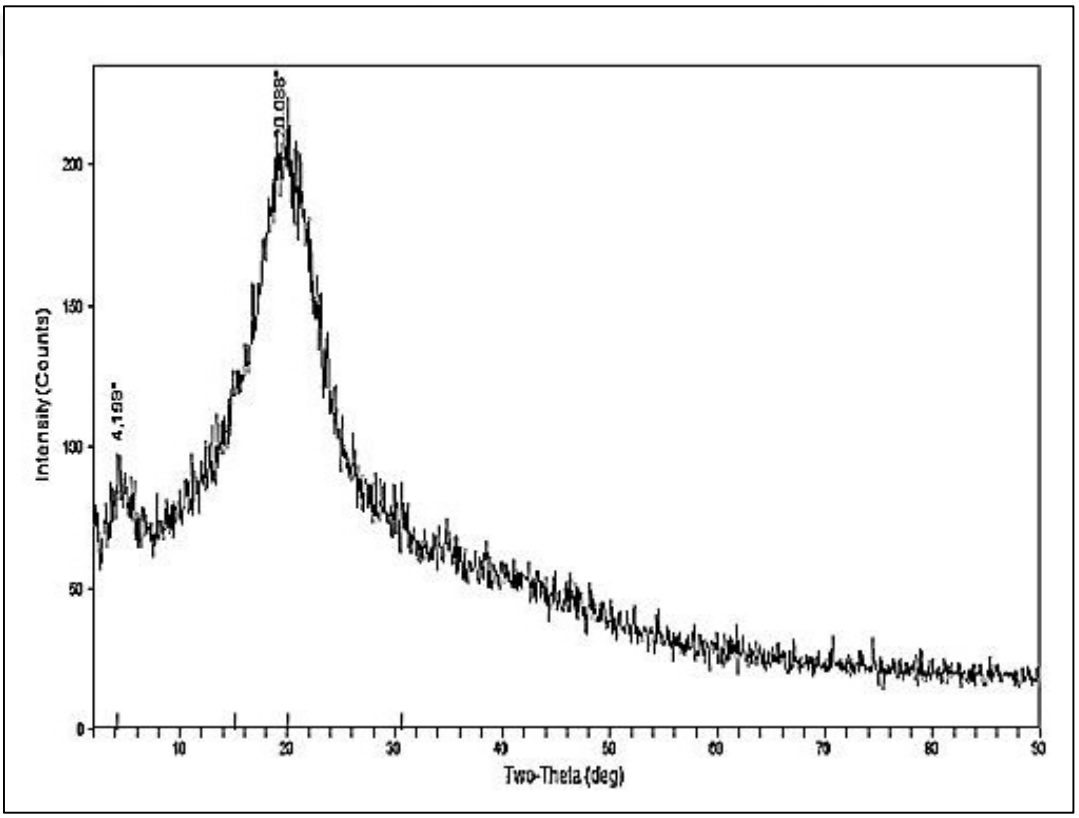


Figure 5.18: XRD pattern of unroasted *P. terebinthus* beans.

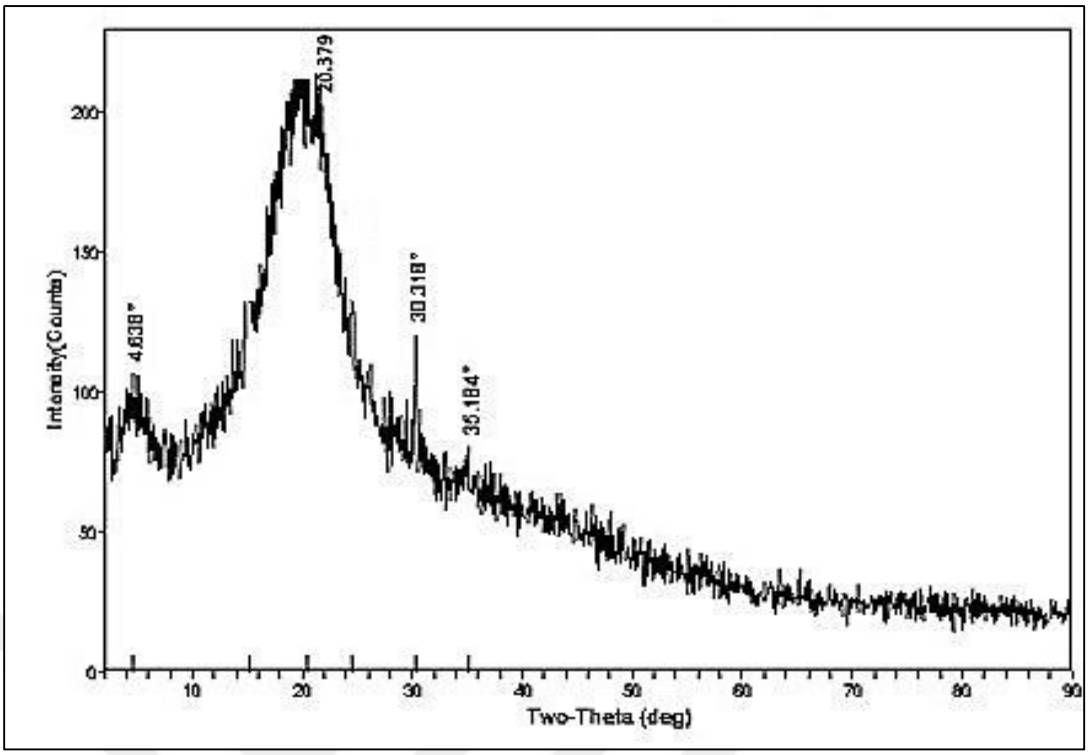


Figure 5.19: XRD pattern of fluidized bed roasted *P. terebinthus* beans at 205 °C for 15 min.

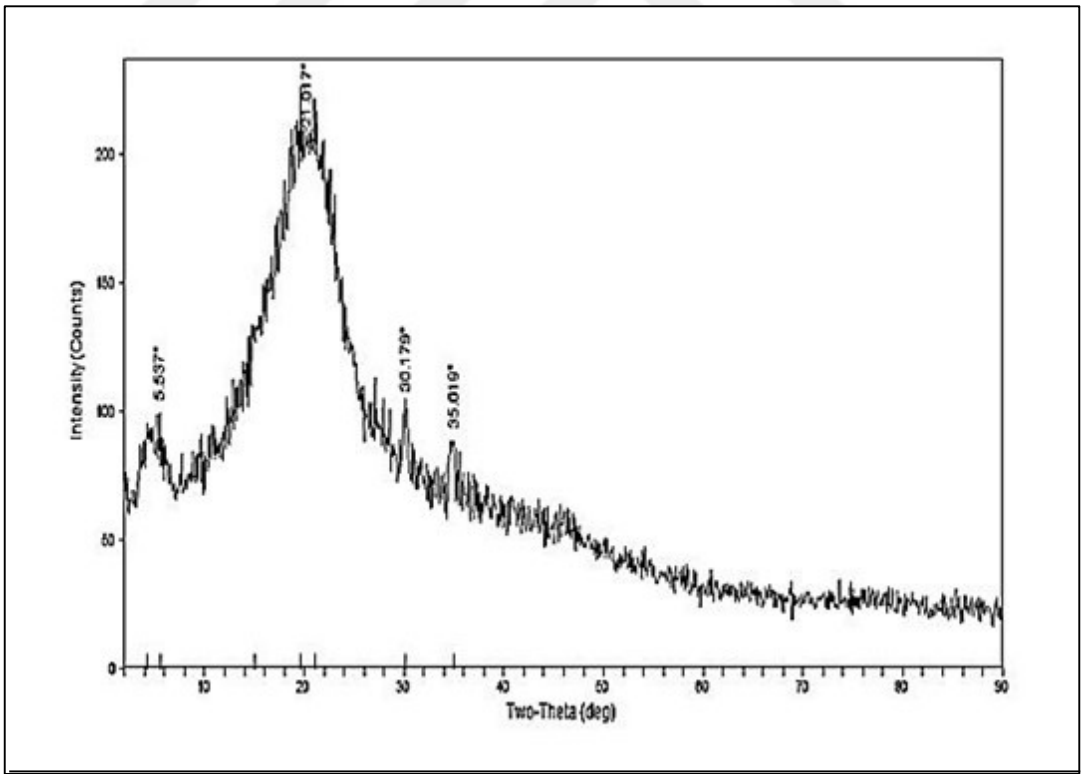


Figure 5.20: XRD pattern of microwave roasted *P. terebinthus* beans at 540 W for 14 min.

6. INSTANT *P. TEREBINTHUS* COFFEE

P. terebinthus beans roasted at the optimum conditions (fluidized bed roasted at 205 °C for 15 min and microwave roasted at 540 W for 14 min) were used for the production of instant *P. terebinthus* coffee. The production stages of the instant *P. terebinthus* coffee are given in Figure 6.1.

6.1. Production Stages for Instant *P. terebinthus* Coffee

6.1.1. Grinding, Extraction, Extract Concentration and Oil Removal

Roasted *P. terebinthus* beans (500 g each) were finely ground in a grinder (KG79, DeLonghi Appliances, Casula, NSW, Australia) for 30 s. Ground beans were subjected to hot water extraction in a batch extractor at 0.4 MPa. After extraction, water was removed with a rotary evaporator (Laborota 4000, Heidolph Instruments GmbH & Co., Schwabach, Germany) at 55 °C under moderate vacuum. The concentrated extracts were pressed with a laboratory type hydraulic hot press (Yılmaz Hidrolik, Gebze, Kocaeli, Turkey) operating at 65 °C for 10 min under 10 MPa to reduce the oil contents of the extracts.

6.1.2. Freeze Drying of Extracts

After hot pressing, *P. terebinthus* extracts (175 g each) were placed onto the tray of a freeze dryer (VirTis Ultra 25 Super XL, New York, NY, USA) and the temperature of the plates was set to its minimum value of 233 K for 4 h to completely freeze the extracts. The drying chamber pressure was set to 10 Pa and kept constant during the entire freeze-drying process. When the drying chamber pressure reached its set value of 10 Pa, the thermostat of the heating plates was set at 293.15 K for both primary and secondary drying stages. The temperature of the condenser was set to its minimum value of 203.15 K and kept constant during the entire drying process to enhance water vapor mass flux from the samples to ice condenser.

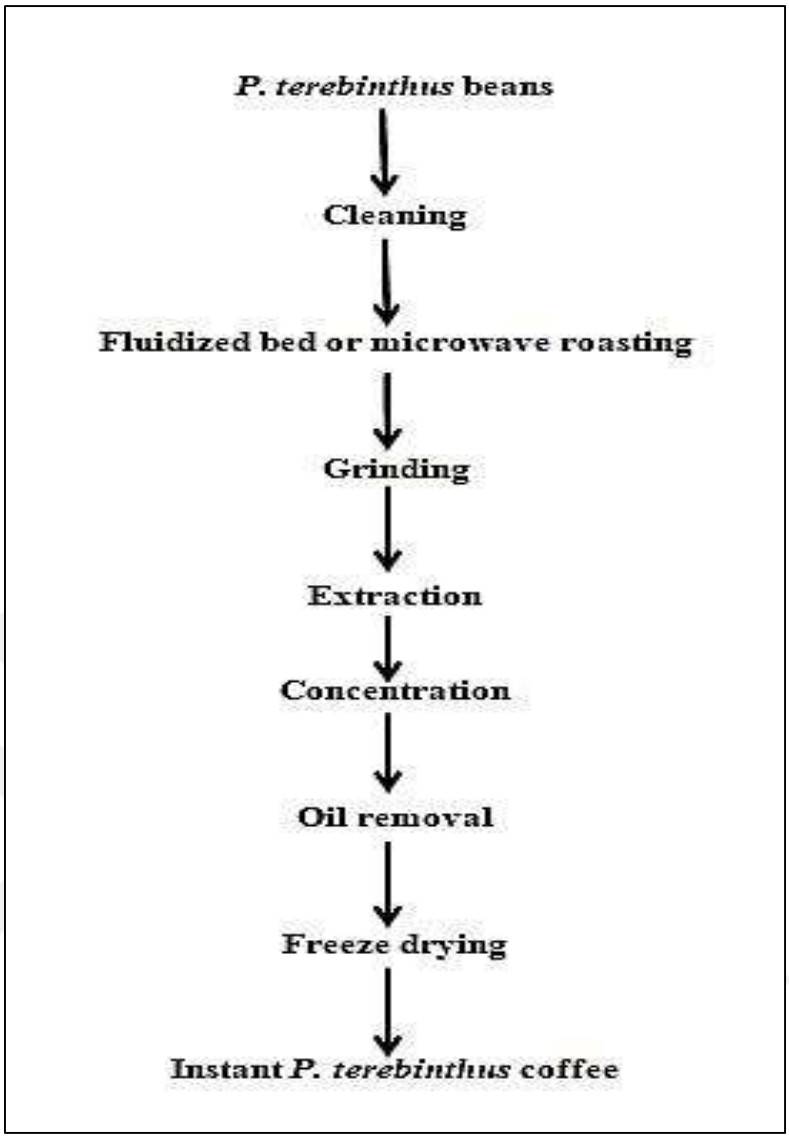


Figure 6.1: Production stages of *P. terebinthus* coffee.

6.2. Analysis of Instant *P. terebinthus* Coffee

6.2.1. Determination of Color

Color measurements of the instant *P. terebinthus* coffee samples were performed using a Konica Minolta CR-400 (Konica Minolta, Sensing Inc., Osaka, Japan) chroma meter equipped with a D_{65} illuminant source and operating with CIE L*a*b* color space. The L* value represents lightness and changes from 0 (black) to 100 (white). The a* value changes from –a* (greenness) to +a* (redness) while the b* value is from –b* (blueness) to +b* (yellowness). The instant *P. terebinthus* coffee samples were placed into the quartz cell of the chroma meter, ten

measurements were randomly taken, and the results were given as the average of ten separate measurements. Calibration was performed with the white color calibration tile prior to the color measurements.

6.2.2. Determination of Particle Size Distribution

Particle size determinations of the instant *P. terebinthus* coffee samples were done with a sieve shaker (Retsch AS 300 control, Retsch GmbH, Haan, Germany) in which the particulate material was divided into size fractions and weight of these fractions was determined by a digital balance (Mettler Toledo, PB03-S, Columbus, OH, USA) with an accuracy of ±0.001 g. The aperture sizes of sieves were 125, 250, 500 and 1000 μm. *P. terebinthus* powder (100 g) was put on the sieve's series and shaken and subjected to horizontal and vertical vibration at 60 Hz for 30 min. The amounts of particles above and below each sieve were weighed. Particle size range was reported as percent. Measurements were taken in triplicate for each sample.

6.2.3. Determination of Total Soluble Solids Content

Refractive index of the instant *P. terebinthus* coffee samples was measured at 20 °C (WYA Abbe Refractometer, 2WA-J, Greiner, Lemgo, Germany). Results were expressed as grams of soluble solids per 100 g *P. terebinthus* coffee powder according to the procedure given in AOAC method 932.12 [AOAC, 2005].

6.2.4. Determination of Moisture Content

Moisture content was gravimetrically determined based on the weight loss according to the procedure described in AOAC method 930.15 [AOAC, 2005]. Instant coffee samples (2 g) were dried at 100 °C in an oven (Ecocell 55, MMM Medcenter Einrichtungen GmbH, München, Germany) until the constant weight was attained. Results of the compositional analysis were the mean of three replicates.

6.2.5. Determination of Total Oil Content

Total oil content of the instant *P. terebinthus* coffee samples was determined according to the procedure given in the AOAC method 920.39 [AOAC, 2005]. A sample (10-15 g of sample) was weighed into an extraction thimble, and extraction was carried out in the Soxhlet apparatus with 250 ml n-hexane for 6 h. The solvent was evaporated by using a rotary evaporator (Laborota 4000, Heidolph Instruments GmbH & Co., Schwabach, Germany). The flask containing oil was dried in a hot air oven, cooled in a desiccator and weighed. An empty flask as a reference was also heated, dried, cooled and weighed. The total oil content was calculated from the difference in weight between the empty flask and the flask containing the oil.

6.2.6. pH Measurements

pH values of the instant *P. terebinthus* coffee samples were measured using a digital pH meter (SevenEasy S20-K, Mettler Toledo, Columbus, OH, USA). Two grams of instant *P. terebinthus* coffee were accurately weighed into a 200 ml of glass bottle, and 100 ml of distilled water was added. The glass bottle with its contents was boiled for 10 min. Then the contents were filtered through Whatman #4 filter paper, and 50 ml of the filtrate was cooled to room temperature and used in pH determinations. Measurements were performed in triplicate.

6.2.7. Determination of Antioxidative Activity

Antioxidative activity was evaluated by DPPH radical assay, as previously described by Singleton et al. [1999]. One ml freshly prepared methanolic solution of DPPH radical (100 μM) was mixed with the instant *P. terebinthus* coffee sample at various concentrations (0.5–100 $\mu g.ml^{-1}$). The contents were vigorously mixed and incubated at room temperature in the dark for 20 min. The absorbance readings of the samples were measured against a blank solution with a UV-VIS spectrophotometer (UV Lambda 35, Perkin-Elmer Corp., Shelton, CT, USA) at 517 nm. The mean of three measurements was reported as the DPPH radical scavenging activity using the following equation:

$$DPPH\ Scavenging\ Activity\ (\%) = \frac{A_C - A_t}{A_C} \times\ 100 \quad (6.1)$$

- A_c: Absorbance of the control
- A_t: Absorbance of the sample solution

6.2.8. Acceptability Tests

Acceptability tests were carried out with 40 panelists (20 females and 20 males) by considering the guidelines in the norm ISO 8586 [2012]. The intensities of appearance, odor, flavor, aftertaste and overall impression were evaluated using a five-point hedonic scale (1 = dislike very much, 2 = dislike, 3 = neither like/nor dislike, 4 = like and 5 = like very much) [Meilgaard et al., 2015]. Panelists were chosen from the staff members of Gebze Technical University. The panelists were trained prior to the acceptance tests. Training continued until consistent results were obtained among panelists and by each panelist individually. When the panelists were repeatable and consistent, individually and collectively, in their evaluations, they were considered ready to serve in a panel. Repeatability and reproducibility in evaluations were checked and verified in accordance with the method proposed by Rossi [2001]. Acceptance tests for the samples of *P. terebinthus* coffee were carried out by the panelists in isolated sensory booths illuminated with white fluorescent light in an environmentally controlled room (23 ± 2 °C and 50 ± 5% RH) under standardized conditions based on the norm ISO 8589 [ISO, 2007]. Just prior to the sensory evaluation, the instant *P. terebinthus* coffee beverages were prepared fresh by adding 2 g of instant *P. terebinthus* coffee to 150 ml of distilled water at 70 °C. *P. terebinthus* coffee beverages were immediately served to the assessors as soon as they were prepared to minimize the aroma and flavor loss.

6.2.9. Statistical Analysis

Data were subjected to one-way analysis of variance (ANOVA) using SPSS statistical software (Version 15, IBM Corporation, New York, NY, USA). Statistical differences between the treatment means were determined by post hoc analysis using Duncan's multiple range test. The differences between the means are regarded as statistically significant if the p-value ≤ 0.05.

6.3. Results for Analysis of Instant *P. terebinthus* Coffee

6.3.1. Color Analysis

The color values of instant *P. terebinthus* coffee powders obtained from the fluidized bed roasted beans at 205 °C for 15 min and microwave roasted beans at 540 W for 14 min are given in Table 6.1. The L*, a* and b* values of the instant *P. terebinthus* coffee powders obtained from two different roasting methods were not statistically different ($p > 0.05$). The L* values of the instant *P. terebinthus* coffee powders obtained from the fluidized bed and microwave roasted beans were lower than the L* values of the fluidized bed and microwave roasted *P. terebinthus* beans, which were 25.44 and 22.12, respectively. This result could be attributed to non-enzymatic browning reactions occurring during the extraction and oil removing steps of the instant *P. terebinthus* coffee production where roasted *P. terebinthus* samples were subjected to high temperatures.

Table 6.1: Color values of instant *P. terebinthus* coffee powders.

Roasting conditions	L* value	a* value	b* value
205 °C, 15 min	24.14 ± 0.04^{a}	1.19 ± 0.01^{a}	0.60 ± 0.01^{a}
540 W, 14 min	22.01 ± 0.02^{a}	1.32 ± 0.01^{a}	0.53 ± 0.02^{a}

Values are mean ± standard deviation (n = 3). Same letters show statistically no significant difference by Duncan's multiple range test at $p > 0.05$.

6.3.2. Analysis of Particle Size Distribution

The particle size distribution of the instant *P. terebinthus* coffee powder obtained from the fluidized bed roasted *P. terebinthus* beans was 3% < 125 µm, 125 µm < 82% < 250 µm, 250 µm < 8% < 500 µm, 500 µm < 5.5% < 1000 µm and 1.5% > 1000 µm (Figure 6.1). The particle size distribution of the instant *P. terebinthus* coffee powder obtained from the microwave roasted *P. terebinthus* beans was 2% < 125 µm, 125 µm < 81% < 250 µm, 250 µm < 10% < 500 µm, 500 µm < 4.5% < 1000 µm and 2.5% > 1000 µm (Figure 6.2). Instant coffee production involves grinding the beans to particle size of approximately 10-250 µm [Rao and Ramalakshmi, 2011]. In the present study, more than 80% of the instant *P. terebinthus* coffee powders obtained from the fluidized bed roasted and microwave roasted *P. terebinthus* beans were in the range of 125-250 µm. Results are in good agreement with the particle size distribution range given for instant coffee.

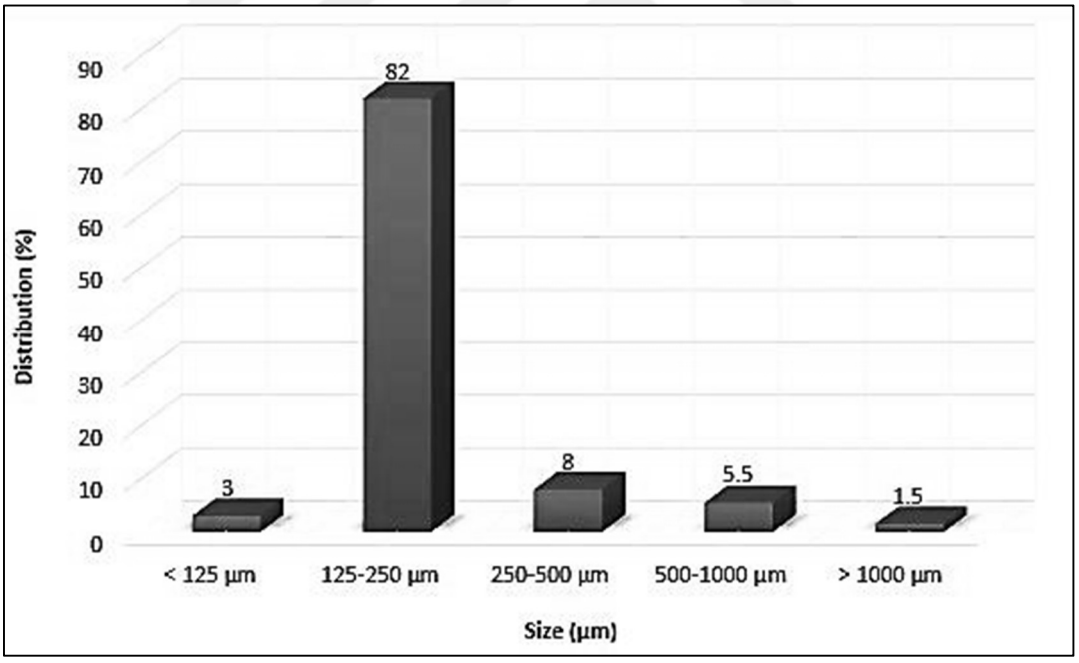


Figure 6.2. Particle size distribution of the instant *P. terebinthus* coffee powder obtained from the fluidized bed roasted *P. terebinthus* beans.

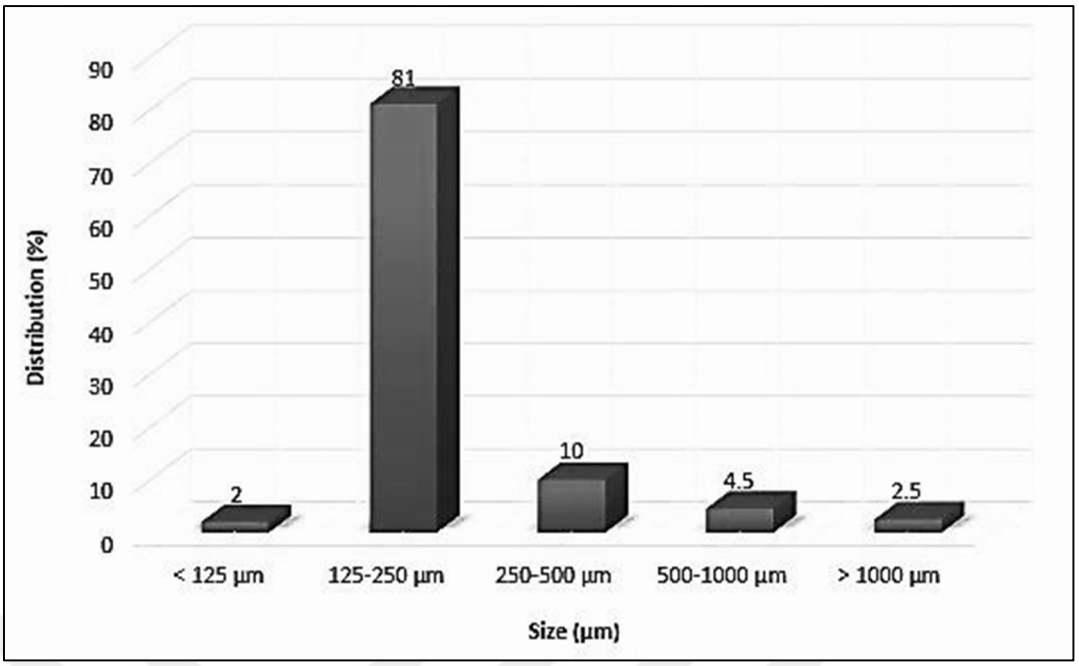


Figure 6.3. Particle size distribution of the instant *P. terebinthus* coffee powder obtained from the microwave roasted *P. terebinthus* beans.

6.3.3. Total Soluble Solids Content

The total soluble solids contents of the instant *P. terebinthus* coffee powders obtained from the fluidized bed roasted beans at 205 °C for 15 min and microwave roasted beans at 540 W for 14 min are given in Table 6.2. The total soluble solids contents of instant *P. terebinthus* coffee powders obtained from two different roasting methods were not statistically different ($p > 0.05$). Stokes et al. reported that total soluble solids contents of instant coffees obtained from different coffee bean species changed between 810 and 930 $g.kg^{-1}$ [Stokes et al., 2017]. Compared to the instant coffees, the total soluble solids content of instant *P. terebinthus* coffee powders were within the range of the total soluble solids content of instant coffees.

6.3.4. Total Oil Content

The oil content is important to obtain an instant coffee product with desirable quality characteristics. It is not practical to get an instant *P. terebinthus* coffee powder from the *P. terebinthus* beans with very high oil content (3.80 $g.kg^{-1}$). Therefore, the oil content needs to be reduced below 2.60 $g.kg^{-1}$ to obtain a powder

product [Secilmis et al., 2015]. The total oil contents of the instant *P. terebinthus* coffee powders obtained from the fluidized bed roasted beans at 205 °C for 15 min and microwave roasted beans at 540 W for 14 min are given in Table 6.2. There was no statistically significant difference ($p > 0.05$) between the oil contents of instant *P. terebinthus* coffees obtained from the fluidized bed roasted beans and microwave roasted beans. Secilmis et al. reduced the oil content of *P. terebinthus* beans by cold pressing with a laboratory scale press up to 2.15 $g.kg^{-1}$ [Secilmis et al., 2015], but the oil contents of instant *P. terebinthus* coffee powders were reduced up to 1.60 $g.kg^{-1}$ which is comparable with the oil contents of dark roasted coffee and Turkish coffee.

Table 6.2: Total soluble solids and total oil contents of instant *P. terebinthus* coffee powders.

Roasting conditions	Total soluble solids content ($g.kg^{-1}$)	Total oil content ($g.kg^{-1}$)
205 °C, 15 min	9.30 ± 0.01^{a}	1.75 ± 0.03^{a}
540 W, 14 min	9.10 ± 0.02^{a}	1.60 ± 0.01^{a}

Values are mean ± standard deviation (n = 3). Same letters show statistically no significant difference by Duncan's multiple range test at $p > 0.05$.

6.3.5. Moisture Content Analysis

The moisture contents of the instant *P. terebinthus* coffee powders obtained from the fluidized bed roasted beans at 205 °C for 15 min and microwave roasted beans at 540 W for 14 min are given in Table 6.3. There was no statistically significant difference ($p > 0.05$) between the moisture contents of instant *P. terebinthus* coffees obtained from the fluidized bed roasted beans and microwave roasted beans. Clarke stated that the moisture content of the instant coffee should be less than 0.4 $g.kg^{-1}$ to obtain a shelf-stable product [Clarke, 1987]. The moisture contents of the *P. terebinthus* coffee powders are less than 0.15 $g.kg^{-1}$ which is less than the maximum value reported for the moisture content of a shelf-stable instant coffee.

6.3.6. pH Analysis

The pH values of the instant *P. terebinthus* coffee powders obtained from the fluidized bed roasted beans at 205 °C for 15 min and microwave roasted beans at 540 W for 14 min are given in Table 6.3. No significant difference ($p > 0.05$) was determined between the pH value of the instant *P. terebinthus* coffee obtained from the fluidized bed roasted beans and the pH value of the instant *P. terebinthus* coffee obtained from the microwave roasted beans. The pH values of the instant *P. terebinthus* coffee powders are in a good agreement with the pH values of the ten regular instant coffees in which pH ranged from 4.98 to 5.12 [da Silveira et al., 2007].

6.3.7. Determination of Antioxidative Activity

The antioxidative activity of the instant *P. terebinthus* coffee powders obtained from the fluidized bed roasted beans at 205 °C for 15 min and microwave roasted beans at 540 W for 14 min are given in Table 6.3. There was no statistically significant difference ($p > 0.05$) in the antioxidative activities of the instant *P. terebinthus* coffee powders obtained from the fluidized bed roasted beans and microwave roasted beans. Results of DPPH radical scavenging activities and EC_{50} values indicated that instant *P. terebinthus* coffee powders obtained from two different roasting methods have similar antioxidative activities (Table 6.3). Ramadan-Hassanien analyzed the antioxidative activities of instant coffee, Turkish coffee and cappuccino using DPPH radical scavenging assay. Cappuccino had the highest antioxidative activity (66% DPPH), followed by the Turkish coffee (33.2% DPPH) and the last was the instant coffee (14% DPPH) [Ramadan-Hassanien, 2008]. Compared to the antioxidative activity of the instant coffee, instant *P. terebinthus* coffee powders have higher antioxidative activity than the instant coffee.

Table 6.3: Moisture content, pH and antioxidative activity of instant *P. terebinthus* coffee powders.

Roasting conditions	Moisture content ($g.kg^{-1}$)	pH	% Inhibition (DPPH)	EC_{50} (mg sample/ ml DPPH)
205 °C, 15 min	0.150 ± 0.02^a	5.1 ± 0.04^a	23.02 ± 0.01^a	3.05 ± 0.01^a
540 W, 14 min	0.142 ± 0.01^a	5.2 ± 0.02^a	24.80 ± 0.01^a	3.19 ± 0.02^a

Values are mean ± standard deviation (n = 3). Same letters show statistically no significant difference by Duncan's multiple range test at $p > 0.05$.

6.3.8. Sensory Acceptability

Appearance, odor, flavor, aftertaste and overall impression were evaluated to assess the acceptability of the instant *P. terebinthus* coffee beverages (Table 6.4). There was no statistically significant difference ($p > 0.05$) in the sensory scores of the instant *P. terebinthus* coffee beverages obtained from the fluidized bed roasted and microwave roasted beans except the odor scores. The instant *P. terebinthus* coffee beverage obtained from the microwave roasted beans took higher odor score than the instant *P. terebinthus* coffee beverage obtained from the fluidized bed roasted beans. The reason could be the continuous airflow during fluidized bed roasting that could result in more losses in odorous compounds in the *P. terebinthus* beans. A visual description of the sensory attributes was depicted in a cobweb diagram in which the mean sensory scores for each quality attribute of the instant *P. terebinthus* coffee beverages are shown (Figure 6.1). The instant *P. terebinthus* coffee beverages took overall impression scores of more than 4.5. The results of the sensory analysis showed that the instant *P. terebinthus* coffee beverages obtained from the fluidized bed roasted beans and microwave roasted beans were not different in terms of appearance, flavor, aftertaste and overall impression. Sensory acceptance tests revealed that the instant *P. terebinthus* coffee beverages are good enough to go to the market.

Table 6.4. Sensory analysis of instant *P. terebinthus* coffee beverages.

Roasting conditions	Appearance	Odor	Flavor	Aftertaste	Overall impression
205 °C, 15 min	4.70 ± 0.01^{a}	4.65 ± 0.02^{a}	4.80 ± 0.01^{a}	4.70 ± 0.02^{a}	4.60 ± 0.01^{a}
540 W, 14 min	4.20 ± 0.04^{a}	4.75 ± 0.01^{b}	4.40 ± 0.04^{a}	4.60 ± 0.03^{a}	4.50 ± 0.01^{a}

Values are mean ± standard deviation (n = 40). Same letters show statistically no significant difference by Duncan's multiple range test at $p > 0.05$.

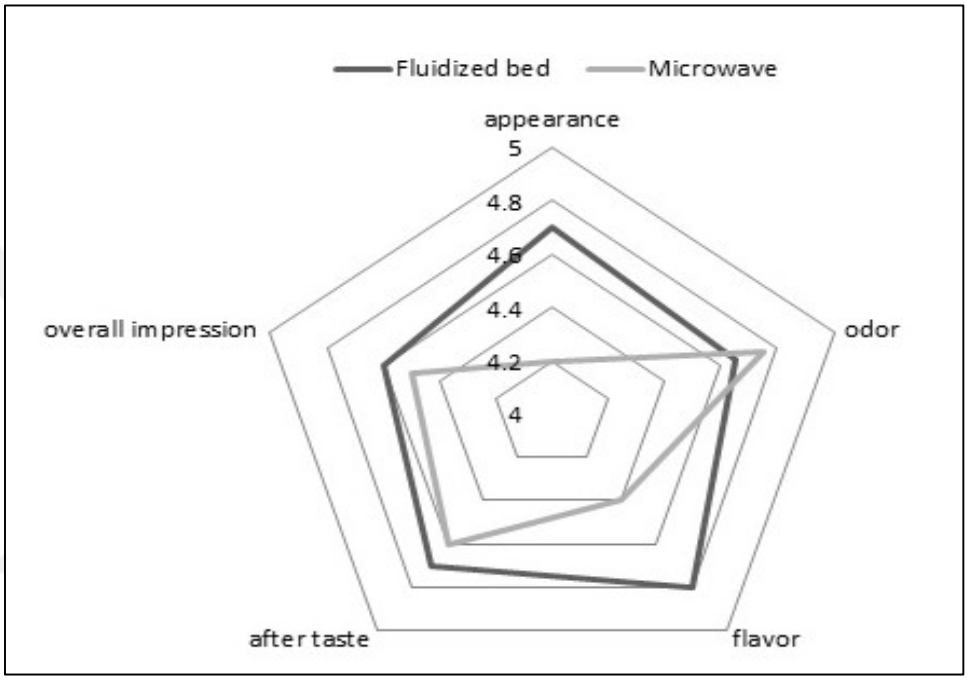


Figure 6.4: Cobweb diagram of the mean sensory scores.

7. CONCLUSION

Roasting temperature and roasting time are the important factors affecting the physical, chemical, structural and organoleptic properties of the fluidized bed roasted *P. terebinthus* beans. RSM was effective in producing the predictive models, establishing the relationships between the processing factors (roasting temperature and time) and the responses, and defining the optimum region for the roasting conditions of the fluidized bed roasted *P. terebinthus* beans. Predictive models, derived from second-degree polynomial, adequately described the roasting characteristics and sensory acceptability as a function of roasting temperature and exposure time. Moisture content, density and breaking force of the fluidized bed roasted *P. terebinthus* beans decreased with increasing the roasting temperature and time. Dark roasted *P. terebinthus* beans took the highest scores in sensory tests by the panelists. Fluidized bed roasting conditions yielding *P. terebinthus* beans with optimum acceptability were determined. Therefore, by using RSM, it was possible to define the optimum roasting conditions for the fluidized bed roasted *P. terebinthus* beans.

Three-dimensional surface plots and mathematical models have shown that microwave roasting power and roasting time were important factors affecting the color, moisture content, density, breaking force and quality attributes of the microwave roasted *P. terebinthus* beans. Predictive models, derived from the second-degree polynomial, adequately described the roasting characteristics and sensory acceptability as a function of microwave roasting power and roasting time. L* value, moisture content, density and breaking force of microwave roasted *P. terebinthus* beans decreased with increasing the microwave roasting power and roasting time. Dark roasted *P. terebinthus* beans took the highest acceptability scores in acceptability tests by the panelists. Optimal microwave roasting conditions for *P. terebinthus* beans were determined with the predictive models developed and using RSM.

FTIR spectra of roasted *P. terebinthus* revealed changes in chemical compositions during roasting. Similar FTIR spectra were observed for fluidized bed and microwave roasted *P. terebinthus* beans at different roasting conditions. Antioxidative activity of the *P. terebinthus* beans depends on the roasting

temperature and roasting time for the fluidized bed roasted *P. terebinthus* beans, and microwave roasting power and roasting time for the microwave roasted *P. terebinthus* beans. Roasting caused an increase in the antioxidative activity of *P. terebinthus* beans as measured by the DPPH radical scavenging activity assay. Further roasting caused partial degradation of some compounds contributing to antioxidative activity. EC_{50} value was also used to express the antioxidative activity. The same trend observed in the DPPH radical scavenging activities of fluidized bed and microwave roasted *P. terebinthus* beans was also valid for EC_{50} values. These values are in close agreement with the DPPH radical scavenging activity results for fluidized bed and microwave roasted *P. terebinthus* beans. As similar to the antioxidative activity of roasted *P. terebinthus* beans, the total phenolic content values were also dependent on the roasting temperature and roasting time for the fluidized bed roasted *P. terebinthus* beans, and microwave roasting power and roasting time for the microwave roasted *P. terebinthus* beans. The total phenolic content of the fluidized bed and microwave roasted *P. terebinthus* beans increased during roasting reaching a maximum, followed by levelling off and a decreasing trend. The crude protein content of both fluidized bed and microwave roasted *P. terebinthus* beans decreased slightly as the roasting time increased while the oil content increased slightly with roasting degree. A slight decrease in ash contents was observed as the roasting proceeded. The pH decreased or increased depending on the roasting conditions used.

SEM revealed considerable changes on the external surface microstructure of *P. terebinthus* beans. Fluidized bed and microwave roasting conditions resulted in *P. terebinthus* beans to lose their structural integrity. Cracks, fractures and oil droplets on the external surface of the fluidized bed and microwave roasted *P. terebinthus* beans were observed. Oil secretion is noticeable in both fluidized bed and microwave roasted *P. terebinthus* beans. Compared to the fluidized bed roasting, microwave roasting increased the oil diffusion from the bean core to the external surface. Three thermal phenomena were observed in DSC thermograms of roasted *P. terebinthus* beans which are an inflexion peak, a very wide and intense endothermic peak, and decomposition peak. Unroasted, fluidized bed and microwave roasted *P. terebinthus* beans exhibited similar TGA curves with three decomposition stages. The greatest decompositions and weight losses occurred during the stage II. Fluidized bed and microwave roasted *P. terebinthus* beans showed similar XRD

patterns. Compared to the XRD curve of unroasted *P. terebinthus* beans, there are two extra diffraction peaks at around 2θ = 30° and 2θ = 35° for the fluidized bed and microwave roasted *P. terebinthus* beans, respectively. This indicated a change in the crystalline regions in the structure of roasted *P. terebinthus* beans when the *P. terebinthus* beans were exposed to thermal treatment.

P. terebinthus beans roasted at the optimum conditions (fluidized bed roasted at 205 °C for 15 min and microwave roasted at 540 W for 14 min) were used for the production of instant *P. terebinthus* coffee. Color, particle size distribution, total soluble solids content, total oil content, moisture content, pH and antioxidative activity of the instant *P. terebinthus* coffee were determined. The L*, a* and b* values, the total soluble solids content, total oil content, moisture content, pH and antioxidative activity of the instant *P. terebinthus* coffee powders obtained from two different roasting methods were not different. More than 80% of the instant *P. terebinthus* coffee powders obtained from the fluidized bed roasted and microwave roasted *P. terebinthus* beans were in the range of 125-250 μm. Acceptability tests for the instant *P. terebinthus* coffee beverage were done. The instant *P. terebinthus* coffee beverages obtained from the fluidized bed roasted beans and microwave roasted beans were found to be not different in terms of appearance, flavor, aftertaste and overall impression.

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