Sudan University of Science and Technology

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Evaluation of the Quality Parameters for Some Laser-Irradiated Edible Oils

تقييم معاملات الجودة لبعض زيوت الطعام المشععة بالليزر

A Thesis submitted for fulfilment requirement of the degree of Ph.D. in Laser Applications in Physics

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الآيـة

قال تعالى:

((لِلَّهِ مَا فِي السَّمَوَاتِ وَمَا فِي الْأَرْضِ وَإِنْ تُبْدُوا مَا فِي أَنْفُسِكُمْ أَوْ تُخْفُوهُ يُحَاسِبْكُمْ بِهِ اللَّهُ فَيَغْفِرُ لِمَنْ يَشَاءُ وَيُعَذِّبُ مَنْ يَشَاءُ وَاللَّهُ عَلَى كُلِّ شَيْءٍ قَدِيرٌ (284) آمَنَ الرَّسُولُ بِمَا أُنْزِلَ إِلَيْهِ مِنْ رَبِّهِ وَالْمُؤْمِنُونَ كُلٌّ آمَنَ بِاللَّهِ وَمَلَائِكَتِهِ وَكُتُبِهِ وَرُسُلِهِ لَا نُفَرِّقُ بَيْنَ أَحَدٍ مِنْ رُسُلِهِ وَقَالُوا سَمِعْنَا وَأَطَعْنَا غُفْرَانَكَ رَبَّنَا وَإِلَيْكَ الْمَصِيرُ (285) لَا يُكَلِّفُ اللَّهُ نَفْسًا إِلَّا وُسْعَهَا لَهَا مَا كَسَبَتْ وَعَلَيْهَا مَا اكْتَسَبَتْ رَبَّنَا لَا تُؤَاخِذْنَا إِنْ نَسِينَا أَوْ أَخْطَأْنَا رَبَّنَا وَلَا تَحْمِلْ عَلَيْنَا إِصْرًا كَمَا حَمَلْتَهُ عَلَى الَّذِينَ مِنْ قَبْلِنَا رَبَّنَا وَلَا تُحَمِّلْنَا مَا لَا طَاقَةَ لَنَا بِهِ وَاعْفُ عَنَّا وَاغْفِرْ لَنَا وَارْحَمْنَا أَنْتَ مَوْلَانَا فَانْصُرْنَا عَلَى الْقَوْمِ الْكَافِرِينَ(286)))

صدق الله العظيم

سورة البقرة الايات (284-286)

Dedications

I dedicate my research to my family and my friends. A special feeling of gratitude to my loving mother and to my father, whose words of encouragement and pressure for perseverance ring in my ear.

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List of Abbreviation

Abbreviations	**Terminology**
AA	Arachidonic Acid
ALA	Alpha-linoleic Acid
AOAC	Association of Official Agricultural Chemists
AV	Acid Value
BRC	Bureau of Radiation Control
CAC	CodexAlimentarius Commission
CDs	Compact Discs
CW	Continues Wave
DGLA	Dihomo-Gamma-Linolenic Acid
DIN	Deutsches Institut für Normung
DVDs	Digital Video Discs
EFA	Essential Fatty Acid
EPA	Eicosapentaenoic Acid
ESR	Electron Spin Resonance
EVOO	Extra virgin olive oil
FAO	Food and Agriculture Organization
FAs	fatty acids
FFA	Free Fatty Acid
FTIR	Fourier Transform Infrared
IAEA	International Atomic Energy Agency
ISO	International Organization for Standardization
KOH	Potassium hydroxide
LED	light-emitting diode
LIDT	Laser-induced damage threshold
MPE	maximum permissible exposure
MS	Measuring System
MUFA	monounsaturated fatty acid
PUFA	polyunsaturated fatty acid
PUFAs	poly-unsaturated fats
PV	Peroxide Value
RBO	Rice Bran Oil
RHCO	Repeatedly heated cooking oil
RI	Refractive Index

RLSS	Research Laboratory & Safety Services
ROS	Reactive oxygen species
SCBA	Sugar cane bagasse ash
SFAs	School food authorities
SV	Saponification Value
TGs	Triglycerides
TPUFA	Total Polyunsaturated Fatty Acid
TSFA	Total Saturated Fatty Acids
UV	UV-Vis Spectroscopy
WHO	World Health Organization

Abstract

This work focuses on the laser food irradiation; mainly on investigating of the influence of green laser (532nm) radiation on some physicochemical properties of sesame oil before and after storage period of 15 consecutive days, and the effect of heating and reheating on sunflower oil properties using carbon dioxide laser compared with the electrical heater was also studied.

The samples of sesame oil were irradiated using diode laser beam with wavelength 532nm and 1 W output power, to duration times of 10, 20, 30, 40, 50 and 60 minutes, and kept for a storage period of 15 consecutive days along with an untreated control at ambient conditions. While two samples of sunflower oil were heated and twice heated with carbon dioxide laser beam up to 50°c it takes 30 minutes; another two samples of sunflower oil were heated and twice heated up to 250°c using electrical heater in two minutes, along with an unheated control sample at ambient conditions.

Physicochemical properties like acid value, ester value, free fatty acids, peroxide value, density, refractive index, viscosity, and moisture of sesame oil and of sunflower oil samples were studied. Fourier transform infrared spectroscopy was used to evaluate the degree of oxidation after irradiation processes of sesame oil and sunflower to differentiate between the chemical changes in the oil samples. The properties of sesame oil were compared at the 1st and 15th day of storage. Color measurements of sunflower oil samples were studied too.

Study reveals that green laser irradiation increases ester value, saponification value, acid value, free fatty acids, peroxide value, viscosity and moisture content of sesame oil; while, it slightly change refractive index and density. Study also indicates that the storage period of 15 days decreases the ester value, saponification value, and moisture content; while, it accelerate acid

value, free fatty acid, peroxide value, density viscosity, and slightly increases the refractive index. Meanwhile, FTIR spectra of the stored samples revealed a notable difference due to the green laser irradiation and storage.

The results also, demonstrated that when the same sunflower oil is reheated, the chemical reactions enhance foaming, darkening of oil color, increased viscosity, and off-flavor. Hence, repeated heating of the oil can lead to degradation of the cooking oil, both chemically and physically.

These results suggest that exposing sesame oil to the green laser irradiation influences their oxidation stability and quality. It was also found that the long time of heating using laser rising temperature up to 50°c catalyzed chemical reactions that resulted in effects in the oil samples characteristics greater than the effects of the electrical heater in a few minutes with temperature 250°c.

مستخلص

يركز هذا العمل على تشعيع الطعام بالليزر. بشكل رئيسي على تقصي تأثير إشعاع الليزر الأخضر (532nm) على بعض الخواص الفيزيائية والكيميائية لزيت السمسم قبل وبعد التخزين لفترة 15 يوماً متتاليا ، كما تمت دراسة تأثير التسخين وإعادة التسخين على خصائص زيت عباد الشمس باستخدام ليزر ثاني أكسيد الكربون مقارنة بالسخان الكهربائي.

تم تشعيع عينات زيت السمسم باستخدام شعاع ليزر ديود بطول موجة يبلغ 532 نانومتر وطاقة خرج تبلغ 1 واط ، إلى أوقات 10 و 20 و 30 و 40 و 50 و 60 دقيقة ، وتم الاحتفاظ بها لفترة تخزين 15 يوما متتاليا جنبا إلى جنب مع عينة مرجعية غير معالجة بالليزر في الظروف المحيطة. تم تسخين عينة من زيت زهرة الشمس و أخرى تم تسخينهما مرتين باستخدام شعاع ليزر ثاني أكسيد الكربون حتى 50 درجة مئوية ، يستغرق الأمر 30 دقيقة; تم تسخين عينة من زيت زهرة الشمس وعينة أخرى تم تسخينهما مرتين حتى 250 درجة مئوية باستخدام سخان كهربائي في دقيقتين إلى جانب عينة مرجعية غير مسخنة في الظروف المحيطة.

تمت دراسة الخصائص الفيزيائية والكيميائية مثل القيمة الحمضية وقيمة الإستر والأحماض الدهنية الحرة وقيمة البيروكسيد والكثافة ومعامل الإنكسار واللزوجة والرطوبة لزيت السمسم وزيت زهرة الشمس. تم استخدام التحليل الطيفي لتحويل فورييه بالأشعة تحت الحمراء لتقييم درجة الأكسدة بعد عمليات التشعيع لزيت السمسم وللتمييز بين التغيرات الكيميائية في عينات زيت زهرة الشمس. تمت مقارنة خصائص زيت السمسم في اليوم الأول وبعد 15 يوماً من التخزين. كما تمت دراسة قياسات اللون لعينات زيت زهرة الشمس.

وتكشف الدراسة أن شعاع الليزر الأخضر يزيد من قيمة الأستر و قيمة التصبن و قيمة حمض و الأحماض الدهنية الحرة و قيمة بيروكسيد و اللزوجة ومحتوى الرطوبة من زيت السمسم. تشير الدراسة أيضا إلى أن فترة التخزين البالغة 15 يوما تقلل من قيمة الإستر وقيمة التصبن ومحتوى الرطوبة ؛ في حين أنه يزيد القيمة الحمضية والأحماض الدهنية الحرة وقيمة البيروكسيد ولزوجة الكثافة ويزيد قليلا من معامل الإنكسار. وفي

الوقت نفسه ، كشفت أطياف الأشعة تحت الحمراء للعينات المخزنة عن اختلاف ملحوظ بسبب الليزر الأخضر والتخزين.

أظهرت النتائج أيضا أنه عندما يتم إعادة تسخين زيت زهرة الشمس ، فإن التفاعلات الكيميائية تعزز الرغوة وتغميق لون الزيت وزيادة اللزوجة والنكهة. وبالتالي، فإن التسخين المتكرر للزيت يمكن أن يؤدي إلى تدهور زيت الطهي ، كيميائياً وفيزيائياً.

تشير هذه النتائج إلى أن تعريض زيت السمسم لإشعاع الليزر الأخضر يؤثر على استقرار الأكسدة وجودتها. كما وجد أن زمن التسخين الطويل باستخدام الليزر إلى درجة حرارة عالية تصل إلى 50 درجة مئوية حفز التفاعلات الكيميائية التي نتج عنها تأثيرات في خصائص عينات الزيت أكبر من تأثيرات السخان الكهربائي في بضع دقائق بدرجة حرارة 250 درجة مئوية.

Chapter One

Introduction

CHAPTER ONE

INTRODUCTION

1.1. Background

Electromagnetic radiations and electrons are used in food irradiation process, whereas, extensive damage was caused to food when treated with some other types of radiations like neutrons, deuterons and α-rays (Rodis, 1995). Electromagnetic radiation caused various chemical and physical changes when it interacts with materials(Ahmmedand Marouf,2017).Light (Laser)plays a photochemical initiator role and is capable to inducing photochemical reactions when exposed to food (Zeb,*et al.*,2008). The mechanism of the photochemical oxidation of vegetable oils has been extensively studied (Choe and Min, 2006). Several researchers studied the impact of laser matter interaction; which could happen with metal, tissue, ceramic or food (Haimid,*et al.*,2019; Awadala, *et al*., 2020).

Irradiation technology usage in food preservation grew a large interest, because of its efficiency and itis potential applications. Various benefits are obtained with the use of irradiation such as protecting meats from pathogenic microorganism and increase their shelf lifetime. Likewise, irradiation produces structural changes in many nutrients (Nawar, 1996). In addition, irradiation of milk and yoghurt improves their shelf life (Marouf and Sara, 2017;Marouf and Siddiq, 2018).

Changes in nutritional and organoleptic characteristics of foods are due to the carbonyl compounds produced from the interaction of the free radicals formed by irradiation of unsaturated fatty acids and oxygen. Irradiation plays an important role in the storage life of food and enhances the safety by reducing pathogenic and spoilage microorganisms. Various chemical and physical parameters of edible oil are utilized to monitor the comp (Niki,*et al.,* 1984) positional quality of oils (Ceriani,*et al*., 2008; Khaneghah,*et al., 2012).* The chemical changes in the oil lead to physical changes like viscosity and density, darken color, production of (Ku,*et al*., 2014).

Laser heating has been recognized since the 1970s, with focusing on the CO_2 laser. Utilizing

CO_2 laser allows non-metallic materials to be heated directly. CO_2 laser with wavelength 10.6 μm equates to photon energy of 943 cm^{-1}, which is of the same order as lattice phonons in covalent crystals, and is absorbed by existing lattice vibrations. This absorption technique also provides the advantage of more uniform heating the materials concerned are generally transparent to 10.6 μm. It meaning that heating is provided via this process throughout the entire thickness of the sample, contrarily in metallic materials where the laser beam is absorbed primarily at the surface (Smith, *et al*., 2018).

Several studies were done in different applications of the laser heating techniques such as in thermo luminescence imaging of spatial dose distributions of ionizing radiation and in the measurement of two-dimensional temperature distributions in high-power laser dielectric coating (Bräunlich, *et al*., 1981). Some researchers studied laser-heating in combustion of agricultural wastes (Awad,*et al*., 2020; Gawbah et al., 2018; Gawbah*et al*., 2017). Other researchers used it in enhancing of the ceramic mechanical properties (Awadala et al., 2020).

Numerous studies were done in food irradiations such as milk pasteurization (Marouf and Sara, 2017), production of yogurt (Marouf and Esra , 2018) and irradiation of bee honey(Ahmmed and Marouf, 2017).

Edible oils are a vital component of our daily diet, which provide energy, essential fatty acids and serve as a carrier of fat-soluble vitamins. It is the oil extracted from the natural origins of plants, the liquid is less dense than water and does not mix with it often without the addition of intermediates, mostly chemical, which are also esters of three fatty acids and glycerol and are called triglycerides, which are produced from the interaction between glycerol and three types of similar or dissimilar fatty acids such as sesame oil, olive oil, sunflower oil, corn oil and other oils. Edible oils such as sunflower (*Helianthus Annuus*) oil are continuously used for deep fat frying. Oil is used continuously for deep frying fat. Use the same frying oil Again and again is a common practice in the family or in restaurants to save costs. The Methods used to assess the quality of cooking oil and its disposal when monitoring foam, The color turns dark or when it smells badodor(Phiri, *et al*.,2006). Chemical reactions such as thermal Polymerization, hydrolysis and oxidation occur when cooking oil is heated during depth Frying process(Stevenson,1984). Using reheated edible oil is unhealthy. Edible oil is exposed to high Prolonged temperatures in the process of frying food; this practice produces fat Peroxide products, which is harmful to human health (Lapointe, *et al*.,2006).

Another example is sesame oil (*Sesamum Iindicum L*.) Is a source of excellent vegetable oil; it is one of the highest oil contents (35% - 63%) among oil crops (Mohammed and Hamza, 2008; Gebremeskel, *et al*., 2021). The presence of some natural antioxidants such as sesame oil makes it one of the most stable vegetable oils in the world (Umar, *et al*., 2014). Sesame seed oil is very resistant to rancidity especially after hydrogenation due to the presence of natural antioxidants (Fapohunda, *et al*., 2012).

1.2. Research Problem

Until now, to the best of our knowledge, researchers studied the effects of different portions of electromagnetic radiation such as gamma, X-ray, sunlight and infrared on the physicochemical characteristics of edible oils; but they have not studied the effect of the single wavelength. In this research, we tried to determine the photochemical effect of green diode laser (532 nm) irradiation on the physicochemical characteristics of sesame oil before and after storage period of 15 consecutive days. And to investigate the thermal effect of carbon dioxide laser (10600 nm) heating and reheating on the chemical and physical characteristics of sunflower oil compared to the effect of electrical heater heating and reheating, in order to determine the most healthy heating method for cooking.

1.3. Previous Studies

In 2011 ,Abiona , et al investigate the frying process on the fatty acids of vegetable oil and palm oil. The growing popularity of fried food in Nigeria, as well as the prolong usage of such oils for frying as made it important for us to investigate the effect of frying time on the fatty acid profile of vegetable oil and palm oil. The frying experiment was conducted at 180ºC for six consecutive days, Changes in fatty acid profile was used to evaluate the alteration during frying. the highest component of FAs observed during the period of frying with palm oil are palmitic acid, oleic acid, linoleic acid and stearic acid which ranges are: (35.11-42.06 %), (30.91-41.75%), (9.11-9.23%) and (6.21-8.13%) respectively, while with vegetable oil,the following FAs palmitic (29.24-40.16%), stearic (14.72-21.94%), linoleic (13.69-28.89%) and oleic (13.69-28.89%) are the highest components. New fatty acid components were observed in the palm oil and vegetable oil during the frying period. With continuous usage, palmatic acid, oleic acid and stearic acid increases in vegetable oil while linoleic acid, oleic acid and stearic acid increases in palm oil. The total polyunsaturated fatty acids (PUFAs) in the vegetable oil decrease with length of usage while it increases in palm oil. In both frying medium, MUFA increases while TSFA decreases with length of usage. The ratios of C18:2/C16:0 in vegetable oil decreases with length of usage but increases in palm oil. Increased usage of vegetable oil andpalm oil in deepfat frying decreases the quality of oil in terms of stability against lipid oxidation, addition of more SFAs.Trans fatty acid (vegetable oil), reduction in the value of PUFAs (vegetable oil). Conversely, palm oil throughout the frying duration in this study, has a lower TSFAs, shows increase in TPUFA, has higher MUFA and TUFA, increase in the C18:2/C16:0 ratio and absent of trans FAs. It is therefore the best choice for deep fat frying(Abiona, *et al*.,2011).

In 2012, Ali and Mohammed investigate recovery of Fried Sunflower Oil by Using SugarCane Industry Waste and Hot Water. The main goal of the current investigation was to use sugar cane bagasse ash (SCBA) and to compare its adsorption efficiency with Magnesia XL as synthetic adsorbents to regenerate the quality of fried sunflower oil. In addition, to evaluate the effect of water washing process on the quality of fried and fried – treated sunflower oil. The metal patterns of sugar cane bagasse ash and Magnesia XL were determined. Some physical and chemical properties non-fried, fried and fried–treated, sunflower oil were determined. Sunflower oil sample was heated at 180 °C + 5 °C, then frozen French fries potato were fried every 30 min. during a continuous period of 20 h. Oil samples were taken every 4 h. The filter aids were added individually to the fried oil at levels 1, 2 and 3 % (w / v), then mechanically stirred for 60 min at 105 ºC. The results indicate that all the filter aids under study were characterized by high levels of Si and variable levels of other minerals. The highest level of Si was recorded for sugar cane bagasse ash (SCBA) was 76.79 wt. %. Frying process caused significant ($P \leq 0.05$) increases in physic+

chemical properties of fried sunflower oil. The treatments of fried oil with different levels of SCBA and Magnesia XL caused significant ($P \leq 0.05$) increase in the quality of fried-treated oil, however the soap content of fried – treated oil was increased also, therefore, and the effects of water washing process on the quality of fried and fried – treated sunflower oil was evaluated. The values of soap and polar after water washing process were about 4.62 and 7.27 times as low as that for fried sunflower oil treated with 3 % sugar cane bagasse ash (SCBA)(Ali and Mohammed,2012).

In 2012, Debnath ,et al investigate effect of frying cycles on physical, chemical and heat transfer quality of rice bran oil during deep-fat frying of poor: An Indian traditional fried food.The present work deals with the effect of frying cycles on physical, chemical and heat transfer quality of rice bran oil (RBO) during the preparation of poor (an Indian traditional fried food) by deep-fat frying. The frying was carried out in intermittent mode (5 batches each for 3min in a day without any time lag) and repeated for 6 frying cycles. Result indicated that in first two cycles, free fatty acid content, peroxide value and total polar materials increased, while radical scavenging activity decreased. Further increase in frying cycles did not result in any significant changes in these parameters ($p > 0.05$). Similar trends were observed for these parameters in case of heating. Relative amounts of total saturated fatty acids increased due to marginal decrease in total unsaturated fatty acids content during frying cycles, however, no significant change was observed during heating. The convective heat transfer coefficient was found to decrease with an increase in frying cycle due to increase in kinematic viscosity of RBO for every frying or cycles. Despite the marginal changes in physical and chemical properties, there was no significant difference ($p > 0.05$) in the sensory characteristics of poor prepared in oil subjected to different cycles of frying (Debnathb, *et al.*,2012).

In 2013 Angaye, et al, Investigate effect of Heat on the Physicochemical Properties of Groundnut Oil. Oil was extracted from fresh groundnut seeds (used as control) and groundnut seeds heated at 90^0C and $120^0\ C$, and market roasted groundnut seeds (fried and bottled seeds purchased from a local market) using n-hexane. The various oil samples were analyzed for their relative densities, viscosities, refractive indices, iodine values and peroxide values. Results obtained show that oxidative rancidity of the oil samples increased with increase in temperature the seeds were subjected to. Relative density increased from 0.9115 ± 0.0005 (for oil from fresh groundnuts) to 0.9241 ± 0.0002 (for oil from the market roasted sample). The viscosities of the various samples also showed an increase from $33.02\ M_2S_1$ (for oil from fresh groundnuts) to $61.6\ M_2S_1$ (for oil from the market roasted seeds). The oil sample obtained from the market roasted seeds recorded the highest level of oxidative deterioration(Angaye, et al.,2013).

In 2015 ,Leong , et al investigate effects of repeated heating of cooking oils on antioxidant. Reusing cooking oil in food preparation especially during deep-frying, is a Common practice to save costs. Repeated heating of the oil accelerates oxidative degradation of lipids, forming hazardous reactive oxygen species and depleting the natural antioxidant contents of the cooking oil. Long-term ingestion of foods Prepared using reheated oil could severely compromise one's antioxidant defense network, leading to pathologies such as hypertension, diabetes and vascular animation. The detrimental effects of reheated oil consumption extend beyond mere oxidative assault to cellular antioxidant shield. In this review, we have examined the experimental and clinical effects related to the intake of reheated oil on antioxidant contents, membrane lipid peroxidation and endothelial function. Understanding the mechanisms underlying the pathology Associated with intake of repeatedly heated oil will help to set a reference for assessing the safety of cooking oil. Finally, considering the potential hazard of repeatedly heating oil, this article aims to further increase awareness of the general public regarding the health risks associated with these oils(Leong, *et al.*, 2015).

In 2015 ,Goswami, *et al*., investigate oxidation of cooking oils due to repeated frying and human health.Food becomes tastier on deep frying, and some of food items can only be eaten, once are deep fried. Reusing this deep fried oil repeatedly for frying purposes is responsible for many health hazards in human population. Increased viscosity and darkening in colour are some of the physical changes which can alter the fatty acid composition of the cooking oil on repeated frying. Oxidation, hydrolysis and thermal polymerization are the chemical reactions occurring as a result of repeated heating of cooking oil for low and cheap food production. In this paper the degradation of the quality of cooking oil on repeated frying, it’s effects on human health and some ways to treat this cooking oil so as to make it more cost effective and less dangerous for human consumption are undertaken with special reference to Rajasthan(Goswami, et al.,2015).

In 2016,Venkata and Subramanyam investigate evaluation of the deleterious health effects consumption of repeatedly heated vegetable oil. Consumption of repeatedly heated cooking oil (RHCO) has been a regular practice without knowing the harmful effects of use. The present study is based on the hypothesis that, heating of edible oils to their boiling points results in the formation of free radicals that cause oxidative stress and induce damage at the cellular and molecular levels. Peroxide value of heated oil, histopathological alterations, antioxidant enzyme levels and blood biochemistry were determined in Wistar rats treated with the RHCO. RHCOrevealed higher peroxide value in comparison to oil that has been unheated or singly heated. Histopathological observation depicted significant damage in jejunum, colon and liver of animals that received oil heated repeatedly for 3 times. The altered antioxidant status reflects an adaptive response to oxidative stress. Alteration in the levels of these enzymes might be due to the formation of reactive oxygen species (ROS) through auto oxidation or enzyme catalyzed oxidation of electrophilic components within RHCO. Analysis of blood samples revealed elevated levels of glucose, creatinine and cholesterol with declined levels of protein and albumin in repeatedly heated cooking oil group. Hematological parameters did not reveal any statistically significant difference between treated and control groups. Results of the present study confirm that the thermal oxidation of cooking oil generates free radicals and dietary consumption of such oil results in detrimental health effects(Venkata and Subramanyam 2016).

In 2016,Biliaed, et al investigate the effects of frying on the thermal behaviour of some vegetable oils. Five different oils were selected for frying process, thermal behavior (temperature changes). The data obtained indicates that after one minute of frying time, the temperature decreased from $180^{\underline{o}}c\ to\ 140, 150, 160, 140\ and\ 140^{\underline{o}}c$ for olive, corn, and blend, sunflower and flaxseed oils respectively. The temperature continued to decrease according to the type of oil and reached the minimal the temperature at $125, 126, 135,\ 128$, and 130ºc for same studied oils respectively. The obtained data from thermal behavior carves showed that the calculated convection area percentages for olive ,corn, blend, flaxseed and sunflower oils were 27.7, 40.08, 42.8, 14.6 and 37.01 % respectively meanwhile the calculated conduction area percentages for the same oils were 72.2, 59.9, 57.1, 85.3 and 62.9 respectively of total heating area. Where the greater the conduction area percentage was the greater the oil potential for frying and the better the fried food. The results from thermal behavior carves of the five oils during frying process at 180°c after heating for one hours .showed that the initial time required reaching 180°c increased with increasing the heating time (frying cycles) . This time was stable (7, 8 ,9 minutes) for olive oil ,flaxseed and sunflower oil and did not change as compared to that recorded at zero heating time(control) and it increased to (9.5 and 14 minutes) for corn and blend oils respectively. The data obtained also revealed that the percentages of convection area increased and loss of water from 27.7, 40.0, 42.8, 14.6 and 37 after (one hours) to 33.7, 43.0, 55.8 , 33.3 and 39.0 after 2 hours of heating for olive, flaxseed, sunflower, corn and blend oil respectively . Meanwhile, the percentage of conduction area decreased from 72.2, 59.9, 57.1, 85.3 and 62.9 after frying for 1hr to 66.2,56.9,44.1,66.6 and 60.9 for the same studied oils. Eight Frying time did not change for both flaxseed and sunflower oils and was constant at 10.9 and 9 minutes while at increased to 20 minutes for corn and blend oil.(Biliaed, et al.,2016).

In 2017,Ying investigate degradation of edible oil during deep-frying process by electron spin resonance spectroscopy and physicochemical appreciation. During frying process of edible oil, lipid oxidation occurs, which is a complex process and involves free radical chain reactions. The impacts of oil with different fatty acid composition on free radicals were evaluated. An ESR study was performed to identify and quantify the formed radicals, along with the assessment of physicochemical parameters including peroxide value, oxidative stability, fatty acid composition, and volatile profile. Results showed an increase of formed free radicals in frying oils over frying time. Besides, frying oils with higher content of unsaturated fatty acids were more prone to be oxidized, as well as physicochemical parameters evidencing this phenomenon. Volatile compounds produced by β-scission hemolytic cleavage of peroxide group during thermal oxidation in frying oils were detected by GC-MS/MS. Results implied major aldehyde volatile compounds were derived from hydro peroxide and oil with higher proportions of unsaturated fatty acids are more likely to produce volatile oxidation products(Ying,2017).

In 2018,Alzaa , et al investigate evaluation of chemical and physical changes in different commercial oils during heating. Heating when cooking oils are exposed to heat, oil degradation occurs, and by-products are produced (free fatty acids, secondary products of oxidation, polar compounds). Some by-products of oil degradation have adverse effects on health. The smoke point of an oil is believed to be correlated with the safety and stability under heat, although technical evidence to support this is limited. The aim of this study was to assess the correlation between an oil's smoke point and other chemical characteristics associated with stability/ safety when heating. Analysis was undertaken in an ISO17025 accredited laboratory. Extra virgin olive oil (EVOO) and other common cooking oils were heated up to 240o C and exposed to 180o C for 6 hours, with samples assessed at various times, testing smoke point, oxidative stability, free fatty acids, polar compounds, fatty acid profiles and UV coefficients. EVOO yielded low levels of polar compounds and oxidative by-products, in contrast to the high levels of by-products generated for oils such as canola oil. EVOO's fatty acid profile and natural antioxidant content allowed the oil to remain stable when heated (unlike oils with high levels of poly-unsaturated fats (PUFAs) which degraded more readily). This study reveals that, under the conditions used in the study, smoke point does not predict oil performance when heated. Oxidative stability and UV coefficients are better predictors when combined with total level of PUFAs. Of all the oils tested, EVOO was shown to be the oil that produced the lowest level of polar compounds after being heated closely followed by coconut oil. Literature on the harmful effects of repeatedly heated vegetable oils. On antioxidant activity, lipid peroxidation and endothelial function. During the frying process, cooking oil is exposed to an extremely high temperature in the presence of air and moisture. Under such conditions, a complex series of chemical reactions takes place, resulting in loss of both quality and nutritional values of the cooking oil. Repeatedly heating the cooking oils initiates a series of chemical reactions, modifying the fat constituents of cooking oil through oxidation, hydrolysis, polymerization, and isomerization, eventually resulting in lipid peroxidation (Alzaa, et al.,2018).

1.4. Research Objectives

1.4.1. General Objective:

To study the photochemical effect of laser on sesame oil, and to the thermal effect of laser on the heating on the oils.

1.4.2. Special Objective:

1. Study the photochemical effect of the green laser on sesame oil.
2. Investigate the effects of laser irradiation of sesame oil on its physicochemical properties.
3. Determine the conservation quality of irradiated sesame oil.
4. Investigate the effects of laser irradiation of sesame oil on its physiochemical properties after a storage.
5. Evaluate the degree of oxidation in sesame oil after irradiation processes.
6. Identify the thermal effect of CO_2 on the physicochemical properties of sunflower oil.
7. Determine the preservation quality of laser-heated sunflower oil.
8. Investigate the effects of carbon dioxide laser heating and reheating on the chemical and physical characteristics of sunflower oil.
9. Compare the effect of effects of carbon dioxide laser with the effect of electrical heater heating and reheating.
10. Determine the most healthy heating method for cooking.

1.5. Methodology

To study the photochemical effect of the green laser sesame oil; seven samples of sesame oil will be irradiated using diode laser with a wavelength 532 nm and output power 1 Watt, continuous at mode. To investigate the effects of laser irradiation of sesame oil on its physiochemical properties after a storage period of 15 days; sesame oil samples will kept for a storage period of 15 consecutive days along with an untreated control sample at ambient condition. To identify the thermal effect of CO_2 on the physicochemical properties of sunflower oil; two samples of sunflower oil will be heated and twice heated with carbon dioxide (CO_2) laser beam with wavelength 10600nm and output power 30 Watts at continuous mode; another two samples of sunflower oil will be heated and twice heated using electrical heater, along with an unheated control sample at ambient conditions. Physicochemical properties of sesame oil and sunflower oil samples will be studied. Fourier transform infrared spectroscopy will used to evaluate the degree of oxidation after irradiation processes of sesame oil and to differentiate between the chemical changes in the sunflower oil samples. The properties of sesame oil will be compared at the 1st and 15th day of storage. Color measurements of sunflower oil samples will be studied and sesame oil samples too.

1.6. Thesis Layout

This thesis is consist of four chapters, chapter one contains introduction and previous studies, chapter two consist the basic concepts of laser, edible oils and laser-matter interaction, chapter three regard with the experimental part (materials, devices and method), chapter four consist of results and discussion, conclusion, recommendations, the list of references and finally appendixes.

Chapter Two

Theoretical Background

CHAPTER TWO

THEORETICAL BACKGROUND

2.1. Laser

The laser can be considered as one of the most important inventions in the twentieth century (Uchida, 2012). Laser is a device that emits a narrow beam of singlewavelength, coherent light as a result of stimulated emission. The term laser began as an acronym for “LightAmplification by Stimulated Emission of Radiation.” The output power of laser light can vary from a few mile watt (in the case of laser pointers) to several thousand watts (industrial laser cutters).

Every laser contains anactive medium, which is the source of atoms that will undergo cycles of excitation and de-excitation to release photons that will form the laser beam. Depending on the type of laser, the active medium can be a solid, liquid, or gas. Let’s walk through the necessary conditions for laser operation, all of which build upon the principles of energy levels, excitation, fluorescence, and phosphorescence that we covered earlier in this section (Nelson,2019).

Lasers have been regularly used to measure, cut, drill, weld, read, write, send messages, solve crimes, burn plaque out of arteries, and perform delicate eye operations. Over and over again the laser has proved to be an extremely practical tool. Nevertheless, lasers have also proved their usefulness in non-practical applications, especially in the realm of art and entertainment. Lasers are involved in almost all aspects of these fields, from “light shows” to Compact Discs (CDs) and Digital Video Discs (DVDs), to special effects in the movies. Some other commonplace application of lasers are as Laser pointers, barcode scanners, laser printers, etc. Still, much of the important modern day celebrated applications lie in the fiber-optic communication, laser machining and fabrication, trace element detection, laser metrology and medical imaging(Gai,2015).

2.1.1. Fundamentals of Laser

The word laser is an acronym for the most significant feature of laser action: light Amplification by Stimulated Emission of Radiation. There are many different kinds of laser, but they all share a crucial element: Each contains material capable of amplifying radiation. This material is called the gain medium because radiation gains energy passing through it. The physical principle responsible for this amplification is called stimulated emission and was discovered by Albert Einstein in 1916. It was widely recognized that the laser would represent a scientific and technological step of the greatest magnitude (Milonni and Eberly, 2010).The main characteristics of lasers, which determine the scope of their applications when compared to ordinary light, are: monochromaticity, directionality, and coherence. Figure 2.1 illustrated the basic elements of laser.

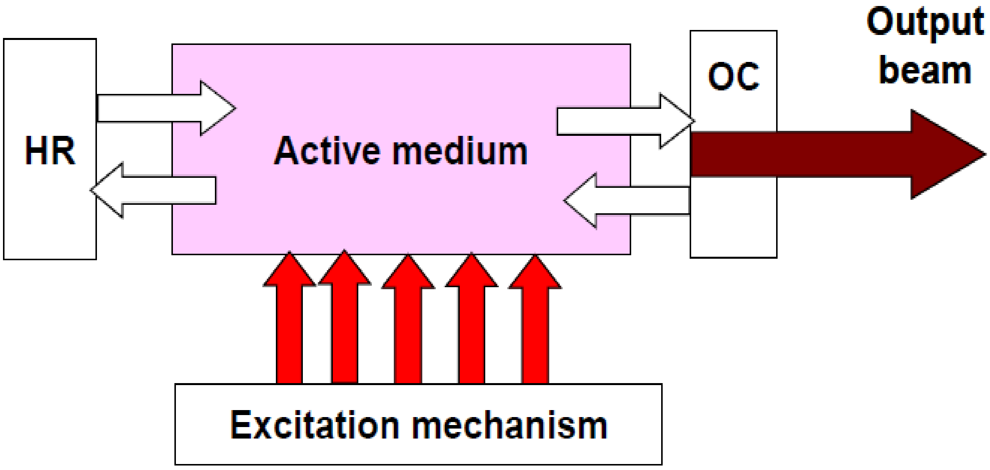


Figure 2.1: Basic elements of laser

2.1.2. Elements of Laser

The generating of the laser depends on three basic components .we use processes to increase or amplify light signals after those signals have been generated by stimulated emission and optical feedback (mirrors). These are:

I. Active Medium

Is an important part of the laser elements it may be consist of gas, liquid or solid the laser may be named according to their medium.

II. Pumping Source

It is an external energy source or pumping source or excitation mechanism that excites the atoms in the active medium from a lower energy state to a higher energy state in order to produce a population inversion.

III. The Resonator

A system of mirrors that reflects undesirable (off-axis) photons out of the system and reflects the desirable (on-axis) photons back into the excited population where they can continue to be amplified(Svelto, 2010).

2.1.3. Laser Classification

Lasers are divided into a number of classes depending upon the power or energy of the beam and the wavelength of the emitted radiation.

Lasers can be classified according to:

2.1.3.1. Laser Hazardous

Lasers are divided into classes based on the potential for causing immediate injury to the eye or skin and/or potential for causing fires from direct or indirect exposure to the beam. Lasers are classified using power, wavelength, and other physical parameters of the laser. Exposure duration is also considered

1. Class 1

Lasers in this class are incapable of causing eye damage because emissions are less than the maximum permissible exposure (MPE) limit. The MPE is defined as the level of laser radiation to which a person may be exposed without hazardous effect or adverse biological changes in the eye or skin. Therefore, Class 1 lasers are exempt from regulation and any labeling requirements and Class 1 users are not required to complete RLSS training.

2. Class 2

Lasers in this class operate in the visible portion of the electromagnetic spectrum (400-700 nanometers) and cannot exceed 1 millwatt total power. Eye damage occurs only if the beam is stared at directly for longer than the normal human aversion response to bright light stimulant (0.25 second). This means a person would naturally turn away from the beam before any damage is done. This laser class is normally not regulated by RLSS and users are normally not required to complete RLSS training unless the use includes intentionally overcoming the aversion response or includes the use of collecting optics (e.g., lenses, telescopes, microscopes, endoscopes, etc.). If collecting optics will be used, controls such as filters, attenuators, interlocks, or the use of cameras to prevent exposures above the MPE would be required. If these types of engineering controls are not practical, standard operating procedures must be developed to address the hazard.

3. Class 3

I. **Class 3a** lasers emit visible or invisible beams and are capable of causing eye damage from short duration (<0.25 second) viewing of the direct beam. A Class 3a laser produces accessible radiation that is between 1 and 5 times the Class 1 emission limit for invisible beams, or between 1 and 5 times the Class 2 emission limit for visible beams. Most laser pointers are 3a lasers. It is possible to be injured by a Class 3a laser if there is more than brief exposure, but injuries are very uncommon. The more likely hazard associated with a Class 3a laser is flash blindness or “dazzling” that interferes with normal vision and can cause an accident due to the temporary interference with normal vision.

This laser class is not regulated by the RLSS and users are normally not required to complete RLSS training unless conditions exist where injury can occur, such as intentionally overcoming the aversion response or using viewing optics (e.g., microscopes, telescopes, or endoscopes).

II. **Class 3b** lasers have an output power greater than that defined for Class 3a, but at levels less than Class 4 output levels. Class 3 laser output cannot exceed 0.5 watts for periods $>$ 0.25

seconds or 0.125 joule for periods <0.25 seconds. Danger labels are required and safety controls are necessary to prevent direct or indirect exposure to the beam. Class 3b laser beams are usually not a fire hazard or skin hazard, but are capable of producing acute eye injury. Class 3b lasers are regulated by the Bureau of Radiation Control (BRC) and are required to be registered with RLSS.

4. Class 4

lasers are high powered and capable of causing severe eye damage with short-duration exposure to the direct, indirect, or scattered beam. Class 4 lasers also present a skin hazard and fire hazard. Class 4 laser output exceeds 0.5 watts for periods $>$ 0.25 seconds and 0.125 joule for periods $<$ 0.25 seconds. Class 4 lasers are regulated by the BRC and are required to be registered with RLSS.(Svelto,2010).

2.1.3.2. Laser Spectral Region

Lasers can be described by which part of the electromagnetic spectrum is represented:-

- Ultraviolet (180-400 nm)
- Visible (400-700 nm)
- Infrared (700 nm–1 mm)

2.1.3.3. Laser Active Medium

But depending of the nature of the active media, lasers are classified into three main categories, namely, solid, liquid, and gas. Scientists and researchers have investigated a wide variety of laser materials as active media in each category since 1958, when lasing action was observed in ruby crystal. Here we will introduce one type of laser from each group as an example (Singh, 2012).

i. Gas

Gas laser can be divided in to three type atoms, molecules, lions.

The molecules in a gas-active-medium environment become excited when an electric current is passed through the gas.

Carbon dioxide is the most efficient molecular gas laser material that exhibits for a high power and high efficiency gas laser at infrared wavelength. It offers maximum industrial applications including cutting, drilling, welding, and so on. It is widely used in the laser pyrolysis method of nanomaterial processing. Carbon dioxide is a symmetric molecule (O=C=O) having three (i) symmetric stretching [i00],(ii) bending [0j0], and (iii) antisymmetric stretching[00k] modes of vibrations , where I, j, and k are integers(Singh, 2012).CO_2 lasers emit light with a wavelength of 10.6μm and have electrical efficiencies of approximately 10-15%.The laser gas mixture used in CO_2 lasers consists mainly of helium to ensure the dissipation of heat. It also contains carbon dioxide, the laser active medium, and nitrogen in which a gas discharge create the energy necessary for excitation (Emmelmanand Authors, 2000). For the CO_2 lasers currently employed for material processing, the heat dissipation (approx.85-90% of the input electrical energy) is usually achieved by using heat exchangers through which the gas is pumped via turbines, roots blowers or cross-flow fans. For axial flow and cross-flow lasers, a continuous supply of fresh laser gas has to be connected to the gas circuit during operation to maintain the efficiency necessary for optimum operation. Non-flow lasers, in which the laser gas mix in the discharge cavity is cooled only by heat conduction methods, have so far only been implemented with relatively low power beams.

Diffusion cooled, high power CO_2 laser, which has meanwhile been developed to industrial maturity, the so-called CO_2 Slab laser offers a number of advantages and already replaces flowing gas lasers in many application fields. The same design is used for low-power CO_2 Slab lasers, with the only difference of a sealed gas charge tube (Emmelman and Authors, 2000).

ii. Solid State Lasers

Another important family of lasers contains solid crystalline as an active medium, Ruby and neodymium are two common examples of solid state laser with widespread industrial applications, Ruby is crystalline aluminum oxide in which some of the aluminum ions in the crystal lattice have been replaced by chromium ions. These chromium ions are the active elements in the Ruby laser.

In early lasers the ruby was machined into a rod about 4 cm in length and 0.5 cm in diameter. It produces intense of light ($\lambda = 694.3$ nm)(Barin,2007). Another type of solid state laser is Nd:YAG lasers with chemical formula Nd: $Y_3Al_5O_{12}$ stand for neodymium yttrium aluminum garnet some of the aluminum in the YAG replaced by triply ionized neodymium (Nd^{3+}) a rare earth element, whose electrons goes to excited state and gives emission at 1064nm ,glass is also used as a host for neodymium lasers (Michael,2003). This system offers the advantage of being a four Level laser, which makes it much easier to achieve population inversion than with the ruby laser. Nd: YAG laser has a very high radiant power output at 1064 nm, which is usually frequency doubled to give an intense line at 532 nm. This radiation is often used for pumping tunable dye lasers. In order to bring 1064nm to visible region the frequency is doubled thus and wavelength becomes half, i.e. 532 nm (in visible region) (Aldeen, 2015).Due to the excellent properties of Nd:YAG laser it is extensively used in many industrial applications like drilling in solid objects ,welding and also in medical applications like eye surgery, etc(Narsimhachary, 2014).

iii. Liquid Lasers

The active medium in dye lasers is a fluorescent organic dye, dissolved in a liquid solvent. As in solid-state lasers, the only reasonable excitation technique is optical pumping with a flash lamp or (more often) with an external laser. The low efficiency of laser pumping is tolerable because it offers better quality output. The main attraction of the dye laser, is its tunable output wavelength and ability to produce ultra-short pulses or ultra-narrowline width, are so important for many applications that its low overall efficiency is entirely acceptable. Laser action has been demonstrated from dyes in the vapor phase, of embedded in a solid host, but such lasers have not proved practical (Rothbart, 2004).

iv. Semiconductor

Layers of semiconductor crystal material are used as an active medium in semiconductor lasers. Semiconductor lasers: Practical semiconductor lasers are excited when current carriers in semiconductor recombine at the junction of regions doped with n- and p- type donor materials.

This occurs when current is flowing through a forward-biased diode made from certain semiconductor materials. At low current densities, recombination at the diode junction generates excited states which spontaneously emit light, and the device operates as incoherent light-emitting diode (LED). If the current density is high, and if the semiconductor device includes reflective facets to provide optical feedback, the diode can operate as a laser (Rothbart,2004).

Diode laser

A laser pointer or laser pen is a small handheld device with a power source (usually a battery) and a laser diode emitting a very narrow coherent low-powered laser beam of visible light, intended to be used to highlight something of interest by illuminating it with a small bright spot of colored light.

The small width of the beam and low power of typical laser pointers make the beamitself invisible in a clean atmosphere, only showing a point of light when striking an opaque surface. Laser pointers can project a visible beam via scattering from dust particles or water droplets along the beam path. Higher-power and higher-frequency green or blue lasers may produce a beam visible even in clean air because of Rayleigh scattering from air molecules, especially when viewed in moderately-to-dimly lit conditions. The intensity of such scattering increases when these beams are viewed from angles near the beam axis. Such pointers, particularly in the green-light output range, are used as astronomical object pointers for teaching purposes.

Laser pointers make a potent signaling tool, even in daylight, and are able to produce a bright signal for potential search and rescue vehicles using an inexpensive, small and lightweight device of the type that could be routinely carried in an emergency kit.

There are significant safety concerns with the use of laser pointers. Most jurisdictions have restrictions on lasers above 5 mW. If aimed at a person's eyes, laser pointers can cause temporary visual disturbances or even severe damage to vision. There are reports in the medical literature documenting permanent injury to the macula and the subsequently permanent loss of vision after laser light from laser pointer being shone at a human's eyes. In rare cases a dot of light from a red laser pointer may be thought to be due to a laser gun sight (Safety ,2011).When pointed at aircraft at night, laser pointers may dazzle and distract pilots, and increasingly strict laws have been passed to ban this.

The low-cost availability of infrared (IR) diode laser modules of up to 1000 mW (1 watt) output has created a generation of IR-pumped, frequency doubled, green, blue,and violet diode-pumped solid-state laser pointers with visible power up to 300 mW. Because the invisible IR component in the beams of these visible lasers is difficult to filter out, and also because filtering it contributes extra heat which is difficult to dissipate in a small pocket "laser pointer" package, it is often left as a beam component in cheaper high-power pointers. This invisible IR component causes a degree of extra potential hazard in these devices when pointed at nearby objects and people.

Early laser pointers were helium–neon (HeNe) gas lasers and generated laser radiation at 633 nanometers (nm), usually designed to produce a laser beam with an output power under 1 milliwatt (mW). The least expensive laser pointers use a deep-red laser diode near the 650 nm wavelength. Slightly more expensive ones use a red-orange 635 nm diode, more easily visible because of the greater sensitivity of the human eye at 635 nm. Other colors are possible too, with the 532 nm green laser being the most common alternative. Yellow-orange laser pointers, at 593.5 nm, later became available. In September 2005 handheld blue laser pointers at 473 nm became available. In early 2010 "Blu-ray" (actually violet) laser pointers at 405 nm went on sale.

The apparent brightness of a spot from a laser beam depends on the optical power of the laser, the reflectivity of the surface, and the chromatic response of the human eye. For the same optical power, green laser light will seem brighter than other colorsbecause the human eye is most sensitive at low light levels in the green region of the spectrum (wavelength 520–570 nm). Sensitivity decreases for longer (redder) and shorter (bluer) wavelengths.

The output power of a laser pointer is usually stated in milliwatts (mW). In the U.S. lasers are classified by the American National Standards Institute (Sam's,2013) and Food and Drug Administration (FDA)—see Laser safety Classification for details. Visible laser pointers (400–700 nm) operating at less than 1 mW power are Class 2 or II, and visible laser pointers operating with 1–5 mW power are Class 3A or IIIa. Class 3B or IIIb lasers generate between 5 and 500 mW; Class 4 or IV lasers generate more than 500 mW. The US FDA Code of Federal Regulations stipulates that "demonstration laser products" such as pointers must comply with applicable requirements for Class I, II, IIIA, IIIB, or IV devices(Sam's,2013).

Trails by a 15 mW green laser pointer in a time exposure of a living room at night Green laser pointers appeared on the market around 2000 and are the most common type of DPSS lasers (also called diode-pumped solid-state frequency-doubled, DPSSFD). They are more complex than standard red laser pointers, because laser diodes are not commonly available in this wavelength range. The green light is generated through a multi-step process, usually beginning with a high-power (typically 100–300 mW) infrared aluminium gallium arsenide (AlGaAs) laser diode operating at 808 nm. The 808 nm light pumps a neodymium doped crystal, usually neodymium-doped yttrium orthovanadate (Nd:YVO4) or neodymium-doped yttrium aluminium garnet (Nd:YAG), or, less commonly, neodymium-doped yttrium lithium fluoride (Nd:YLF)), which lases deeper in the infrared at 1064 nm. This lasing action is due to an electronic transition in the fluorescent neodymium ion, Nd(III), which is present in all of these crystals.

Some green lasers operate in pulse or quasi-continuous wave (QCW) mode to reduce cooling problems and prolong battery life.

An announcement in 2009(Green diode,2009) of a direct green laser (which does not require doubling) promises much higher efficiencies and could foster the development of new color video projectors.

Because even a low-powered green laser is visible at night through Rayleigh scattering from air molecules, this type of pointer is used by astronomers to easily point out stars and constellations. Green laser pointers can come in a variety of different output powers. The 5 mW green laser pointers (classes II and IIIa) are the safest to use, and anything more powerful is usually not necessary for pointing purposes, since the beam is still visible in dark lighting conditions.

2.1.3.4. Laser Mode

Lasers can also be described by the length of time the beam is active:

- Continuous wave - the laser is continuously pumped and continuously emits light.
- Pulsed - lasers which emit light in the form of optical pulses, resulting in high-energy light.
- Ultra-short pulsed – pulsed lasers with pulse duration of at most a few tens of picoseconds (1 trillionth of a second).

2.1.4. Laser Applications

2.1.4.1. Laser Industrial Applications

Lasers are employed across the manufacturing industry as tools capable of delivering intense cutting or welding power with high precision. Their ability to manipulate and transform materials makes them ideal for the automobile, computer and clothing industries – to name but a few. In fact, it is difficult to find a modern consumer product that has not seen a laser during its manufacturing.

I. Laser Cutting

Most laser cutting is carried out using CO_2 or Nd:YAG lasers. The general principles of cutting are similar for both types of laser although CO_2 lasers dominate the market. The basic mechanism of laser cutting is extremely simple and can be summarized as follows:

i. A high intensity beam of infrared light is generated by a laser.

ii. This beam is focused onto the surface of the work piece by means of a lens.

iii. The focused beam heats the material and establishes a very localized melt (generally smaller than 0.5mm diameter) throughout the depth of the sheet.

iv. The molten material is ejected from the area by pressurized gas jet acting coaxially with the laser beam as shown in figure 2.2.

v. This localized area of material removal is moved across the surface of the sheet thus generating a cut. (Powell,*et al*., 2004).

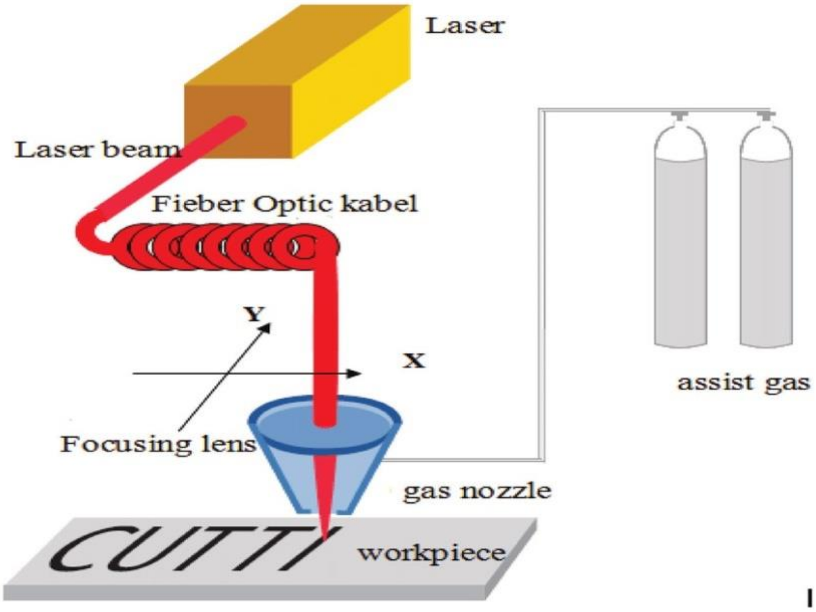


Figure 2.2 A schematic of laser cutting

II. Laser Heat Treatment

Heat treatment using lasers is a process that consists of heating metals and certain other materials for some time to harden them against wear with little or no distortion of other parts of the work piece. Tools and automobile parts, for instance, are almost heat-treated. For example, the ring grooves of the automobile piston can be hardened with a laser, without any distortion to the rest of the piece. To obtain proper hardening, the temperature of the work piece surface is raised above a critical temperature, followed by a particular cooling down stage.

During heat treatment, it is important to control of the time evolution and intensity of the laser beam. The optimal irradiance distribution is determined by the thermodynamics of the laser-material interaction and by the shape of the object, but typically, intensities between $5x10^2 - 5x10^3 W/cm^2$are used for thermal annealing.

In general, $\mathrm{CO_2}$ lasers are used for laser heating of metals. As they emit in the infrared (λ=10.6µm) and can operate either in the CW or pulsed mode, they are the laser of choice for heat treatment. However, since the reflectance of metals is very high in the infrared (λ=10.6µm), to reduce the reflectance and achieve adequate absorption, the surface of the work piece is covered with an absorptive coating, such as black spray paint or graphite (Ready ,1997).

1.1.4.2. Military Applications of Lasers

Lasers appear also in various military applications, the most widely utilized being the so called laser range finders , an analogy to the ground laser radar, that can measure with great accuracy the target' s distance and thus obtain the optimal trajectory of a missile and higher real ability of the hit. For this purpose, the Nd:YAG lasers seem to be best. However, much less sophisticated are the laser markers used in, e.g., guns, to identify the target at a distance of up to 20 m. In this case small diode lasers are used.

On the other hand, for intercontinental ballistic missiles to be destroyed, it is necessary to use a high-power laser, i.e., of the $\mathrm{CO_2}$ or chemical type, and mirrors placed in space due to minimal free-space path loss, the beam is transmitted without any loss to the next mirror focusing the beam and homing it to the target, e.g., a rocket .To achieve best homing and hit, the mirrors can be moved round according to the rocket' s parameters. The laser can be located at a ground station or on a trajectory (Jelínek and Kluiber,1991).

2.1.4.3. Medical Applications

Surgical removal of tissue with a laser is a physical process similar to industrial laser drilling. Carbon-dioxide lasers operating at 10.6 micrometers can burn away tissue as the infrared beams are strongly absorbed by the water that makes up the bulk of living cells. A laser beam cauterizes the cuts, stopping bleeding in blood-rich tissues such as gums. Similarly, laser wavelengths near one micrometer (Neodymium-YAG Laser) can penetrate the eye, welding a detached retina back into place, or cutting internal membranes that often grow cloudy after cataract surgery . Less-intense laser pulses can destroy abnormal blood vessels that spread across the retina in patients suffering from diabetes, delaying the blindness often associated with the disease.

Ophthalmologists surgically correct visual defects by removing tissue from the cornea, reshaping the transparent outer layer of the eye with intense ultraviolet pulses from Exciter Lasers. Laser light can be delivered to places within the body that the beams could not otherwise reach through optical fibers similar to the tiny strands of glass that carry information in telephone systems. One important example involves threading a fiber through the urethra and into the kidneys that the end of the fiber can deliver intense laser pulses to kidney stones.

The laser energy splits the stones into fragments small enough to pass through the urethra without requiring surgical incisions. Fibers also can be inserted through small incisions to deliver laser energy to precise spots in the knee joint during arthroscopic surgery . Another medical application for lasers is in the treatment of skin conditions. Pulsed lasers can bleach certain types of tattoos as well as dark red birthmarks called port-wine stains. Cosmetic laser treatments include removing unwanted body hair and wrinkles (Jones,*et al.*,2000).

2.1.4.4. Metrological and Geophysical Applications

Surveyors and construction workers use laser beams to draw straight lines through the air. The beam itself is not visible in the air except where scattered by dust or haze, but it projects a bright spot on a distant object. Surveyors bounce the beam off a mirror to measure direction and angle.

The beam can set an angle for grading irrigated land, and a rotating beam can define a smooth plane for construction workers installing walls or ceilings. Pulsed laser radar can measure distance in the same manner as microwave radar by timing how long it takes a laser pulse to bounce back from a distant object. For example, in 1969 laser radar precisely measured the distance from the Earth to the Moon. Laser range finding is now widely used for remote sensing.

Instruments flown on aircraft can profile the layers of foliage in a forest, and the Mars Global Surveyor used a laser altimeter to map elevations on the Martian surface(Sugioka and Cheng, 2014).

2.1.4.5. Laser Applications in Agricultural

In agriculture land leveling System laser had controlled it. This kind of technology decreases the cost 6.3% up to 15.4% of production of most crops such as wheat, rice, cotton, soybean as well as corn. laser benefits in agriculture includes, (1) Save water where laser controlled land leveling, (2) Save fertilizers by increasing and flatness, (3) Increasing emergency of the seedling, (4) Control land leveling technology to control the ridges, (5) Making fingerprint for pesticides and fertilizer, (6), Estimate the purity of fertilizer and pesticides, (7) Laser can be use to kill the weeds without hurting the surrounding plants or main crop, (8) Detection of the following toxic materials that may be mixed with imported foodstuffs, (9) Grain yield estimation and their size was estimated, (10) Measure things bruising on fruits and vegetables, (11) Laser enhancing plant growth and chemical constituents. Farmers frequently fertilize their crops. Lasers can now scan the density of the soil. The idea is that less dense soil is probably more fertile because it is packed with organic matter. This can help a farmer determine what they need to do to prepare the soil for new crops before they ever plant or how much fertilizer he needs to apply once they are already growing. These results recorded by Subrata and Atanu,2013. There are studies that prove that lasers can be used to kill weeds without hurting the surrounding plants. Laser technology is currently very expensive, but it can save large farms money in the long run (Laser, 2013). Unleveled field become less productive compared to leveled fields. Unleveled fields also give rise to salinity and water-logging problems. In this context, leveling of fields is essential to maintain soil fertility and productivity and to save irrigation water. Further, (Taylor,*et al.*, 2004) recorded that, using agriculture nozzles, the spray quality generated is important for the pesticide application processes by affecting spray deposits and drift-ability. These results hold true with (Klein and Johnon, 2002; Wolf, 2002).

2.2. Laser Matter Interaction

When laser radiation strikes a target surface part of it is absorbed, the energy that is absorbed begging to heat the surface, under some conditions there are important effects due to absorption of energy such as plasma formation, vaporization, ablation, etc(Peter and Dieter,2010).

Whenever the material is irradiated in the laser radiation, the energy will be firstly transformed into electronic excitation energy, and then transformed to lattices of material through collisions between electrons and lattices. The laser energy converted into mechanical, thermal electronic as well as into anther electromagnetic wave with different configuration. The deposition of laser energy will produce a series of effects depending on the amount of such as a temperature rise, gasification and ionization, which are depend on the incident radiation and properties of the material (Jinghua and yaguo, 2011).

There are two different phenomena result of interaction of laser with matter, pyro lytic and photolytic processes. In both cases short pulses are applied in order to remove material in a controlled way. Pyrolytic process is based on breaking of chemical bonds by photon energy; there is very little thermal interaction with work piece itself. Photolytic processes are based on a rapid thermal cycle heating, melting and partly evaporation of the heated volume(Krizystof,2005).

2.2.1. Basic Phenomena of Laser Matter Interaction

In figure 2.3 typical situations shown, where light beam is incident on a slice of matter, there are many principle effects existence as follows:

Reflection, refraction, Absorption, transmission and Scattering

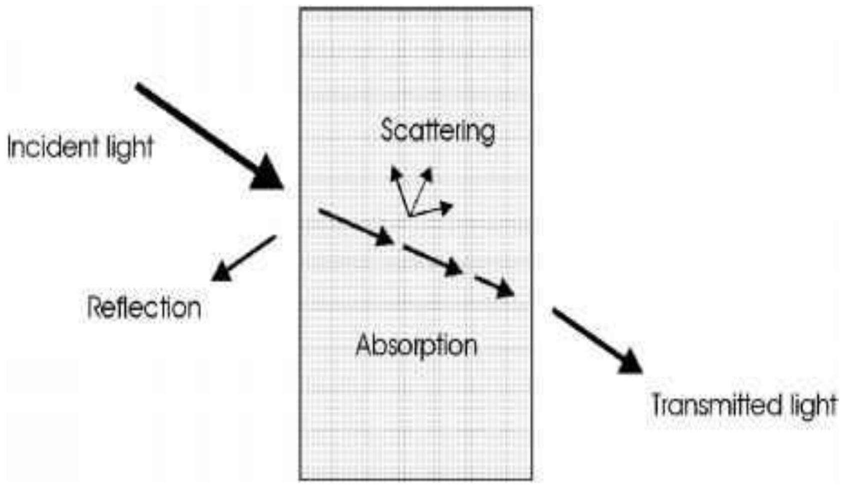


Figure2.3 Geometric of Reflection, refraction, absorption and scattering

Reflection, absorption or scattering are dominated primarily depends on the type of material and the incident wavelength. The wavelength is a very important parameter indeed it determines the index of refraction as well as the absorption and scattering coefficients (Markolf, 2007).

2.2.1.1. Reflection and Refraction

Reflection is defined as the returning of electromagnetic radiation by surfaces upon which is the physical boundary between two materials of different indexes of refraction such as air and matter. The simple law of reflection requires the normal of the incident and reflected beams and the normal of the reflecting surface to lie within one plane, called the plane of incidence.(Markolf,2007). The surface itself is assumed to be smooth, with surface irregularities being small compared to the wavelength of radiation. In contrast, i.e. when the roughness of the reflecting surface is comparable or even large than the wavelength of radiation, diffuse reflection occurs. Then, several beams are reflected which do not necessarily lie within the plane of incidence. Diffuse reflection is a common phenomenon of must materials because it none provided with highly polished surfaces such as optical mirrors.

Refraction usually occurs when the reflecting surface separated two media of different indices of refraction. It originates from a change in speed of the light wave. Reflectivity of surface is measure of the amount of reflectance radiation. Reflectivity and reflectance depend on the angle of incidence, the polarization of radiation, and the indices of reflection of the materials forming the boundary surface (Markolf,2007).

2.2.1.2. Absorption and Transmission

Absorption is important physical phenomena that occur during the laser-matter interaction. Absorption due to partial conversion of light energy into heat motion or certain vibrations of molecules of the absorbing material (Peter and Dieter,2010).Generally, when the laser is focused on the material surface, it is partially absorbed the absorptivity depends on the surface structure, material defects, power density and wavelength of laser light (Krizystof,2005).The absorbed amount of light by the media at certain wavelength is described by the Beer Lambert law equation:

$$I = I_0 \, exp \, (-\alpha \, z) \qquad (2.1)$$

Where:-

$I$is the transmitted intensity I_0 is the incident intensity

$\alpha$is absorption coefficient of the material z is thickness of material.

The absorption coefficient (α) depends on the medium, wavelength of radiation , intensity, temperature, and plasma formation above the target. This indicated that with increasing depth the absorbed radiation decreases in corresponds with equation (2.1)(Rubahn, 1999).Substance is said to show general absorption if it reduces the intensity of all wavelength in the considered spectrum by a similar fraction. Selective absorption, on the other hand , is the absorption of certain wavelengths in preference to others the existence of colors actually originates from selective absorption .

Usually , body colors and surface colors are distinguished. Body color is generated by light which penetrates a certain distance into the substance. By backscattering, it is then deviated and escapes backward from surface but only after being partially absorbed at selected wavelength. In other hand, surface color originates from reflection at the surface itself it mainly depends on the reflectance which are reflected to the wavelength of the incident radiation.

The ability of a medium to absorb electromagnetic radiation depends on a number of factors mainly the electronic constitution of its atoms and molecules, the wavelength of radiation, the thickness of the absorbing layer, and internal parameters such as temperature or concentration of absorbing agents (Markolf, 2007). Transparent medium permits the passage of light without any absorption, e.g. the total radiation energy entering into and emerging from such a medium is the same. In contrast media in which incident radiation is reduced practically to zero are called opaque.

The terms “transparent“ and “opaque” are relative, since they certainly are wavelength dependent (Markolf,2007). Only no reflected and non-absorbed or forward scattered photons are transmitted by the slice and contribute to the intensity detected behind the slice. The ratio of transmitted and incident intensities is called transmittance (Krizystof, 2005).

2.2.1.3. Scattering

When elastically bond charged particles are exposed to electromagnetic waves, the particles are set into motion by the electronic field. If the frequency of the wave equal the natural frequency of free vibrations of a particle, resonance occurs being accompanied by considerable amount of absorption. Scattering, on other hand , takes place at frequencies not corresponding to those natural frequencies of particles(Markolf ,2007). Elastic and inelastic scattering are distinguished, depending on whether part of incident photon energy is converted during the processes of scattering. in the following paragraphs, we will first considers elastic scattering, where “incident” and scattered phonon have the same energy a special kind of elastic scattering is “Rayleigh scattering” it’s only restriction is that the scattering particles be smaller than the wavelength of incident radiation(Markolf,2007). in particular, we will find a relationship between scattering intensity and index of refraction, and that scattering is inversely proportional to the fourth power of wavelength. the latter statement is also known as Rayleigh’s law and it expressed by :

$$I_s = \frac{1}{\lambda^4} \tag{2.2}$$

One important type on inelastic scattering is known as Brillouin scattering it arises from acoustic waves propagating through a medium. Thereby inducing in homogeneities of the refraction index. Brilloun scattering of light higher (or lower) frequencies occurs because scattering particles are moving toward (or away from) the light source (Markolf ,2007).

2.2.2. Interaction Mechanisms

The laser irradiation-induced effects on materials refer to numerous aspects, including optical, electromagnetic, thermodynamic, biological changes in material properties. The laser-matter interaction is an interdisciplinary and complicated subject (Jillian,2012).When the material is irradiated with lasers, the laser energy will be firstly transformed into electronic excitation energy and then transferred to lattices of materials through collisions between electrons and lattices.The deposition of laser energy will produce a series of effects, such as temperature rise, gasification and ionization. The physical processes of interactions between lasers and matters can be grouped into linear and nonlinear responses of materials to laser pulses, namely thermal effects, nonlinear interactions, laser plasma effects and so forth (Wood,2003**).** The variety of interaction mechanisms that may occur when applying laser light to materials is manifold. These types can classify into many categories such as: thermal interaction, photochemical interaction, photo ablation interaction (Jillian,2012).Each of this types can discus in some detail.

2.2.2.1. Thermodynamics

Laser ablation entails complex thermal processes influenced by different laser parameters, inclusive of laser pulse energy, laser wavelength, power density, pulse duration. According to the response of material to incident laser, the responses can be categorized into two groups: thermal and mechanical effects. Thermal effects refer to melting, vaporization (sublimation), boiling, and phase explosion while mechanical response involves deformation and resultant stress in materials.

Different thermal processes will induce different mechanical responses, which will be detailed in the following:

a. Thermal Interaction

It is effect of deposition of energy into the material, it cause increase in local temperature (Jillian,2012).Photo thermal explanation of the observations implies that the absorbed photon energy is transferred directly into excitations of lattice vibration; a "hot spot" is generated, which result in thermally activated fragmentation on the material (Rubahn, 1999). Thermal effects can be induced by either CW or pulsed laser radiation (Markolf,2007).Depending on the duration and peak value of the matter temperature achieved, different effects like coagulation, vaporization, carbonization, and melting may be distinguished (Markolf,2007).

If sufficient energy is absorbed then the vibration of molecules becomes so intense and the molecular bonding is stretched so far that it is no longer capable of exhibiting mechanical strength and the material is said to have melted on further heating the bonding is further loosened due to the strong molecular vibrations and the material is said to have evaporated.The vapor is still capable of absorbing the radiation but only slightly since it will only have bound electrons.

The exception occurs if the gas is sufficiently hot so that electrons are shaken free and that gas is then said to be plasma (Krizysto,2005). The vaporization process can be described by the heat flow theory where the surface temperature of the target at the end of the laser pulse is determined by the light absorption and thermal diffusivity (Schneider and Lippert,2010).Materials subjected to laser irradiation will absorb the incident laser energy, raising the temperature and causing material expansion and thermal stress in materials. When the stress exceeds a certain value, the material may fracture and/or deform plastically.

Material expansion will induce various changes in refractive index, heat capacity, etc.The deposition of the laser energy can heat the materials and raise the temperature of materials. Given that laser beam is perpendicular to the surface of materials (flat surface), the temperature with respect to time t and depth x will be:

$$\Delta T(x,t) = 2(1-R)\alpha I_0 \frac{t}{\pi k\rho C} ierfc \frac{x}{2\sqrt{\frac{kt}{\rho C}}} \tag{2.3}$$

Where; t is the laser irradiation time,R is the reflectivity, α is the absorptivity,I_0 is the spatial distribution of laser intensity, k is thermal conductivity,c is light velocity,ρ is the density of irradiated materials.When $x > 4\sqrt{\frac{kt}{\rho C}}$, the surface temperature will be simplified as:

$$\Delta T(t) = \frac{2\alpha I_0\sqrt{t}}{\sqrt{\pi k\rho C}} \tag{2.4}$$

The temperature rise may alter physical and optical properties of materials. The influence of temperature rise will be discussed in more detail.

1.1.Analysis of Damage Threshold

If the laser energy level at which the irradiated materials start to melt is referred to as the damage threshold (LIDT) of the materials, it is clear that the LIDT is directly proportional to $\sqrt{t}$ as shown in eq. (2.4). A number of experiments evidence that for laser pulses that $t > 10ps$, the proportional relationship is applicable to vast majority of semiconductor materials, metals, and dielectric thin films coated on optical components, etc.

However, the damage threshold increases with decreasing pulse duration for the laser pulses <10ps. The variation is due to different damage mechanisms of materials when subjected to ultra-short laser pulses(Stuart , *et al.*,1996).

1.2. Thermal Distortion and Stress in Solid-State Lasers

Materials can absorb the energy of the incident laser, a part of which will be converted into heat. Non-uniform temperature distribution will appear because of the uneven heat diffusion.

Consequently, expansion and contraction will lead to laser-induced thermal stress. The stress can limit the average workable power of solid-state lasers .Thermo-aberration can seriously affect the uniformity of the output laser field and therefore induce the phase distortion.

1.3. Frequency Doubling

The deposition of laser energy can result in thermal depolarization in optical crystals for doubling/tripling frequency and also degrade the efficiency of frequency doubling. Self-thermal effect resulting from pump loss will influence the harmonic conversion of the incident laser.

During the process of harmonic conversion, crystals inevitably absorb the energy of fundamental frequency light and frequency-doubled/tripled light. Part of the absorbed energy will convert into heat leading to uniform temperature rise in crystals, which will give rise to a refractive index ellipsoid and disturb phase matching. Furthermore, harmonic conversion efficiency will drop and the quality of output beam will deteriorate (Wang and Weiner,2003).

b. Melting and Solidification

With the increase of laser energy, materials will absorb more laser energy and the deposited energy will cause the material to melt in the case that materials temperature exceeds the melting point. Melting followed by solidification will change the atomic structure of materials and can realize the mutual transformation between crystalline and amorphous state.

c. Ionization and Gasification

Laser-induced gasification can be divided into surface gasification and bulk gasification. As the temperature continues to increase to the vaporization point, part of the absorbed laser energy is converted into the latent heat of evaporation, the kinetic energy of gasification and the quality of spray steam. With increasing the laser intensity, the melted materials will be gasified and/or ionized. The gasification is discussed based mainly on liquid-gas equilibrium. Gaseous particles with the Maxwell distribution will splash out from the molten layer.

The gasified particles are ejected several microns away from the surface. The space full of particles is the so-called Knudsen layer. The ionization will greatly enhance the absorption and deposition of the laser energy. After ionization is completed, the inverse bremsstrahlung absorption dominates the absorption of plasma. Re-crystallization of the ionized materials may cause changes in material structure.

d. Phase Explosion

Phase explosion is another important thermal effect. The occurrence of phase explosion follows the stages: the formation of super-heated liquid owing to laser energy deposition; then the generation and growth of nucleation in super-heated liquid and explosion of nucleation.

The physical process is depicted . Upon the irradiation of laser, the temperature of materials will rise and the deposited energy diffuses into the bulk of materials to a certain depth; the temperature of melted materials sharply increase to over the boiling point due to the heavy deposition of laser energy; nevertheless, the boiling does not start and the liquid is super-heated because of the absence of nucleation; the disturbance will bring about nucleation and the super-heated liquid thickens as the size and the number of bubbles grow; the startling boiling will arise once the size of bubbles is sufficiently large and afterwards the super-heated liquid and particles will be ejected.

This way, the phase explosion takes place. In order to generate phase explosion, three requirements must be met: 1. the fast creation of super-heated liquid, the temperature of which should at least be (0.8-0.9) T_{cr} (T_{cr} is the critical temperature)(Bleiner and Bogaerts,2006);2. the thickness of super-heated liquid is large enough to accommodate the nuclear, usually on the order of tens of microns; 3. sufficient time t_c during which the size of nucleation reaches the critical size r_c, generally several hundreds of picoseconds. All the three factors are indispensable (Yoo,*et al.*,2000).The generation of phase explosion requires specific laser pulses and material properties. The power density of laser pulses should be more than the threshold of materials (~1010W/cm^2).

The phase explosion can be generated not only by single pulse but also by high-repetition rate pulses (Han, *et al.*,2010).

2.2.2.2. Photochemical Effects

During photochemical interaction, light induce chemical effects and react within macromolecules or tissues, It can occur at very low power densities approximately about (1w/cm^2) and long exposure times ranging from seconds to continuous wave (Markolf , 2007).

In this case photons interaction with bound and free electrons in the material structure, which raises them to the higher energy levels. Energy conversion takes place through various collision process involving electrons, lattice phonons ionized impurities and defects structures (Krizystof,2005). Thermal energy has been used, the rate of chemical reaction increase with heating in metals, the laser energy absorbed by free electrons, these electrons vibrate and react without disturbing, the solid atomic structure as electromagnetic wave-front arrives at surface of the matter. Then all of the free electrons in the surface vibrate in the phase with incoming beam creating (electron gas), this electron gas within the metal structure means that the radiation is unable to penetrate metals to any significant depth, only one or two atomic diameters.

Thus metals are opaque and they appear shiny. (Krizystof,2005). When the excitation time is shorter than the thermalization time in the material, non-thermal photomechanical ablation mechanisms can occur. for example, with ultra-fast pulsed , direct ionization and the formation of dense electron–hole plasmas can lead to a thermal phase transformations, direct bond- breaking ,and explosive disintegration of the lattice through electronic repulsion (Mathew and Craig,2010).

2.2.2.3. Photo Ablation Interaction

Laser ablation is a removable of material from a substrate by direct absorption of laser energy. Laser ablation methods have the huge advantage of being "self-developing", there for no wet chemistry is necessary in order to removes the irradiated material (Schneider and Lippert,2010).In the type of reaction we can use pulsed or CW laser (Mathew and Craig,2010). The ablation process depending on the absorption properties of the target material. The presence of defects. And on laser parameters such as wavelength, pulse duration (Bauer, 2000).

When a photon interacts with matter, the photon energy is coupled to the lattice through electronic processes. The photon absorption by a material occurs in general over an optical depth of several nanometers, where energy in metals is transferred to the electronic system directly and in nonmetallic system typically to the lattice (Schneider and Lippert,2010).

Many types of pulsed lasers have been used for the ablation of solid materials, and ruby lasers were among the first to be applied. At present, laser ablation systems are equipped with either Nd: YAG or exciter lasers. Solid state Nd:YAG systems are employed because they are relatively inexpensive, require little maintenance, there is hardly any divergence of the laser beam, and they can be easily incorporate into small commercial ablation systems (Schneider and Lippert, 2010).

In all cases, material removal is accompanied by highly directed plume ejected from the irradiated zone. The dense vapor plume may contain solid and liquid clusters of material. At high intensities, a significant fraction of the species may become ionized, producing plasma (Wood, 2003).The lasers temporal pulse length can have a significant effect on the dynamics of the ablation process. In general, as the pulse length is shortened, energy is more rapidly deposited into the material leading to more raped material ejection the volume of material that is directly excited by laser has less time to transfer energy to the surrounding material before being ejected. Therefore the ablation volume becomes more precisely defined by the lasers spatial profile and optical penetration depth and the remaining material has less residual energy which reduces the hazard (Brown and Arnold, 2010).

The laser wavelength is an important parameter for the ablation process in general, with decreasing laser wavelength an increase in the ablation rate is expected for Nd:YAG lasers, the fundamental wavelength is in the near infrared at 1064nm.

A shorter wavelength offers a higher photon energy which is more suitable for an efficient vaporization and ionization of the solid sample. The absorption properties of the target material will finely decide which laser frequency can be employed (Schneider and Lippert, 2010) .

2.3. Fourier Transform Infrared (FTIR)

Fourier transform infrared spectroscopy (FTIR) is a largely used technique to identify the functional groups in the materials (gas, liquid, and solid) by using the beam of infrared radiations. An infrared spectroscopy measured the absorption of IR radiation made by each bond in the molecule and as a result gives spectrum which is commonly designated as % transmittance versus wavenumber (cm^{-1}). A diverse range of materials containing the covalent bond absorbed electromagnetic radiation in the IR region. The IR region is at lower energy and higher wavelength than the UV-visible light and has higher energy or shorter wavelength than the microwave radiations. For the determination of functional groups in a molecule, it must be IR active. An IR active molecule is the one which has dipole moment. When the IR radiation interacts with the covalent bond of the materials having an electric dipole, the molecule absorbed energy, and the bond starts back and forth oscillation. Therefore, the oscillation which caused the change in the net dipole moment of the molecule should absorbed IR radiations. A single atom doesn't absorb IR radiation as it has no chemical bond. Symmetrical molecules also do not absorbed IR radiation, because of zero dipole moment. For instance, H_2 molecule has two H atoms; both canceled the effect of each other and giving zero dipole moment to H_2 molecule. Therefore, H_2 molecule is not an IR active molecule. On other hand, H-F (hydrogen fluoride) is an IR active molecule, because when IR radiation interacts with H-F molecule, the charge transferred toward the fluorine atom and as a result fluorine becomes partial negative and hydrogen becomes partial positive, giving net dipole moment to H-F molecule. It should be noted here that a particular IR radiation (frequency) will be absorbed by a particular bond in the molecule, because every bond has their particular natural vibrational frequency. For instance, a molecule such as acetic acid (CH3COOH) containing various bonds (C-C, C-H, C-O, O-H, and C=O), all these bonds are absorbed at specific wavelength and are not affected by other bond. We can say that two molecules with different structures don't have the same infrared spectrum, although some of the frequencies might be same(Atkins and Paula,2009).

Electromagnetic Spectrum is interaction of matter with any part of the electromagnetic spectrum is called spectroscopy, which is an instrumentally assisted study between matter and electromagnetic radiation of any range. Electromagnetic spectrum is composed of various radiations containing different wavelengths, which is a type of radiant energy, ranging from gamma rays to X-rays via visible light to radio waves, each of which can be considered as a wave or particle traveling at the speed of light. Electromagnetic radiations have a broad range of spectrum starting from the highest energy cosmic rays via X-rays, UV, Vis, infrared, micro-, and radio waves . They propagate in vacuum with the speed of light. The electromagnetic radiations are described by the parameters like frequency, wavelength, and energy. Chemists used selectively the electromagnetic radiations to explore the complete structural, chemical, and physical properties of the molecules.

The IR region is lying between visible and microwave end of the electromagnetic radiation spectrum. It is basically divided into three main portions: near IR (14000– 4000 cm^{-1}), mid-IR (4000–400 cm^{-1}), and far IR (400–40 cm^{-1}). IR spectroscopy is an advanced and extensively used analytical tool that investigates the structural chemistry of the sample by irradiating with IR radiations. The molecules or sample absorbed the IR radiations and displayed an absorption spectrum. IR measured the amount of radiations absorbed by the molecule and their intensity. The absorption of the IR radiations causes various molecular motions in the molecule, which create a net dipole moment. Therefore, a molecule is said to be IR active if the molecule has a net dipole moment (e.g., CH_4, C_2H_6, NO_2, TiO_2), otherwise it will be IR inactive(e.g., H_2, O_2, etc.). One of main advantages of FTIR spectroscopy is its capability to identify functional groups such as C=O, C-H, or N-H. FTIR spectroscopy enables by measuring all types of samples: solids, liquids, and gases.

The plot of measured infrared light intensity (absorbance or % transmittance) versus its property, such as energy range expressed in wavenumber (cm^{-1}), is called an infrared spectrum. The infrared spectrum is conventionally plotted with high wavenumber to the left and low wavenumber to the right in its x-axis. Plots of the typical FTIR spectra should always follow this convention. However, in certain articles the contrary profile of FTIR spectra are also reported . The IR spectrum is recorded in absorbance mode, which measures the amount of light absorbed by a sample, and its intensity at y-axis is plotted in absorbance unit. the absorption band of each characteristic functional group in compound is pointed up, and their tops represent wavenumbers at which significant amounts of IR light were absorbed by the sample. The height or area of a peak in an absorbance spectrum is proportional to concentration; therefore Beer’s law can be used to determine the concentrations of molecules in samples.

When electromagnetic radiation interacts with a material, for example, UV-Vis light, the excitation of electrons occurs from the highest occupied molecular orbital (HOMO) to lowest unoccupied molecular orbital (LUMO). This movement of an electron from a lower energy level to a higher energy level is known as a transition. Generally, the infrared spectroscopy is principally similar as the other absorption spectroscopy. The lower energy radiation in the infrared (IR) region of the electromagnetic spectrum can interact with atoms, and molecules can produce changes within these entities. This type of radiation is not energetic enough to excite electrons, but it can cause the chemical bonds in molecules to vibrate in different ways. The absorption of IR radiation is also quantized like other absorption process. In a quantized process, the molecule absorbed only selected frequency. The energy changes in the infrared radiation are in the order of 8–40 KJ/mole (1.9–9.5 KCal/mol). The energy in this range covers the stretching and bending vibrations of various bonds in covalent molecules. During an absorption process, the molecule absorbed only those frequencies of IR radiation which matched with the natural vibrational frequency of the bonds and hence increases the amplitude of vibrational modes of the molecules. However, all the bonds in a molecule cannot absorb the IR radiation irrespective to the matching frequency of IR radiations, until and unless it has a net dipole moment(Shrivastava, *et al*., 2017).

2.4. Edible Oils

The importance of edible oils in industries such as foods, energy, cosmetics, pharmaceuticals,or lubricants has been well documented. Physical properties of vegetable oils depend primarily on composition (and hence on biological origin) and temperature(John and Julian, 1997). The estimation of the physicochemical properties of edible oils is essential in the design of unit processes such as distillation, heat exchangers, reactors and piping. On the other side, physicochemical properties are an important factor that determines the overall quality and stability of a food system. It has been shown that for vegetable oils, density decreases linearly with temperature increasing . (Esteban ,*et al*.,2012).Density, saponification value, iodine value, acid value, peroxide value are some of the important characteristics of a vegetable oil. Macronutrients like proteins, carbohydrates and lipids and micronutrients like vitamins, minerals and antioxidants are the major components of a healthy daily diet (SR EN ISO ,2005).Edible oils are an important part of human diet, being used as food or as an ingredient in food products.

2.4.1. Types of Edible Oils

I. Fruit oil

This is usually obtained by compressing the plant’s fruit part in cold press and then extracting all the excess moisture such that its purity is not disturbed. Olive oil and coconut oil are few among those.

II. Bran oil

Bran oil is usually extracted from the cereals and millets which has a bran and husk as their outermost surface. These bran oil like rice bran oil are researched to be healthier than the cooking oil obtained from oilseeds.

III. Oil from Oil Seeds

Sunflower, groundnut, mustard, rapeseed and sesame are the oilseed crops that are grown for extraction of various grades of cooking oil and spent meal. Mustard, rapeseed and sesame oils have stronger residual flavors when used in food and hence are limited in usage. That has made refined sunflower and groundnut oil one of the most popular oils in Asian and African cuisine.

IV. Oils from other Sources

Vegetable oils that are extracted from various plants (a blend of refined oils), and various animal fats like butter, ghee, lard etc fall into these categories. There are oils that are extracted from grape seed, avocado, pistachio etc also, but have various limitations like low smoking point, sparsely available for commercial extraction, strong flavors, colors, or triglycerides and not-so-good fat. (Suprabha,2016).

2.4.2. Vegetable Oil

Vegetable oil is very common, affordable and used by majority of people across the globe especially in the tropics. Its use as antidote to prevent some oxidative stress related diseases and a complication is advocated (Oguntibeju, *et al.*, 2010). Vegetable oils in particular are natural products of plant origin consisting of ester mixtures derived from glycerol with chains of fatty acid contain about 14 to 20 carbon atoms with different degrees of unsaturation (Emmanuel and Mudiakeoghene,2008). Vegetable oil is the oil extracted from natural plant origins, such a sesame oil ,olive oil, sunflower oil, corn oil, peanut oil and other oils. It a liquid less dense than water and often doesn’t mix with it without the addition of mostly chemical intermediates. man has known the production of vegetable oil for more than five thousand years from the plants. Food oils and fats are third essential component of human food after sugars and proteins. the first information about their chemical composition was known in 1823 .scientific progress has also made it possible to know the components of the extracted oil and its impact on human health (Peng and Stanley,2001)

2.4.3. Sesame Oil

Sesame (*Sesamum Indicum L.*), otherwise known as sesamum or benniseed, member of the family Pedaliaceae, is one of the most ancient oilseeds crop known to mankind. Sesame plays an important role in human nutrition. Most of the sesame seeds are used for oil extraction and the rest are used for edible purposes (Khie,*et al.*, 2008). Sesame is grown primarily for its oil-rich seeds. Before seeds were appreciated for their ability to add nutty flavor or garnish foods, they were primarily used for oil and wine (Ghandi, 2009). After the extraction of oil, the cake is mostly used for livestock feed or often as manure. Its color varies from cream-white to charcoal-black but it is mainly white or black. Other colors of some sesame seed varieties include, yellow, red or brown (Naturland, 2002). In Nigeria, the notable colors for sesame seed are white, yellow and black (Fariku, et al., 2007). The lighter varieties of sesame which are considered to be of higher quality are generally more valued in the West and Middle East, while both the pale and black varieties are prized in the Far East(Hansen,2011). There are numerous varieties and ecotypes of sesame adapted to various ecological conditions (Nzioku, et al., 2010). The major world producers include India, Sudan, China and Burma (who contribute about 60% of the total world production) (Khier, et al, 2008). It is also one of main commercial crops in Nigeria, Sudan and Ethiopia . Sesame is an important crop to Nigerian agriculture: it is quite extensively cultivated especially in Northern Nigeria. It yields in relatively poor climatic conditions, and it is widely used within Nigeria. Moreso, it is an important component of Nigeria’s agricultural exports (Chemonics, 2002). Sesame seed is rich in fat, protein, carbohydrates, fiber and some minerals. The oil seed is renowned for its stability because it strongly resists oxidative rancidity even after long exposure to air (Global , 2010). The oil fraction shows a remarkable stability to oxidation. This could be attributed to endogenous antioxidants namely lignin’s and tocopherols (Elleuch,*et al.*, 2007; Lee *et al.*, 2008).

The seed is rich in protein and the protein has disable amino acid profile with good nutritional value similar to soybean (Naerls, 2010). The chemical composition of sesame shows that the seed is an important source of oil (44-58%), protein (18-25%), carbohydrate (~13.5%) and ash (~5%) (Borchani *et al*., 2010). Sesame seed is approximately 50 percent oil (out of which 35% is monounsaturated fatty acids and 44% polyunsaturated fatty acids) and 45 percent meal (out of which 20% is protein) (Ghandi,2009; Hansen, 2011).Sesame seeds are small, almost oblate in shape and have a mild and delicious aroma and taste. Sesame seed is used whole in cooking and also yields sesame oil (Ram ,*et al.*,1990; Hansen, 2011). It has a rich nutty flavor (although such heating damages their healthful polyunsaturated fats) and is used mainly as a food ingredient in whole, broken, crushed, shelled, powdered and paste forms. Its use is country based, in the US; it is used as some form of whole seed product for the confection and baking industries. A small percentage percent of total production is however processed into oil, meal or flour (Hansen, 2011). In Nigeria, the seeds are consumed fresh, dried, fried or when blended with sugar. It is also used as a paste in some local soups. (Fariku, *et al*., 2007).

Sesame seeds are not only used for culinary purposes but also in traditional medicines for their nutritive, preventive and curative properties. Its oil seeds are sources for some phytonutrients such as omega-6 fatty acids, flavonoid phenolic anti-oxidants, vitamins and dietary fiber with potent anti-cancer as well as health promoting properties (Agriculturae,2010). Sesame oil is an edible vegetable oil derived from sesame seeds used in various countries. It is used asa cooking oil in South India and Asia and often as a flavor enhancer in Chinese, Japanese, Korean, and to a lesser extent Southern Asia cuisine (Ram ,et al.,1990).It is stable and free from undesirable nutrition or flavor component. Beniseed oil has anatural oxidant which prevents aging and is vital for the production of liver cells (Naerls, 2010; Weiss, 2000). The oil also contains oleic (35.9-47%), linoleic (35.6-47.6), palmitic (8.7- 13.8%), stearic (2.1-6.4%), as well as arachidic acids (0.1-0.7%) (Elleuch, *et al*., 2007; Borchani, *et al*., 2010).

2.4.4. Sunflower Oil

Oil crops are well distinguished for supplying protein and energy in human diet . Sunflower (*Helianthus annuus*) is a significant crop possessing 15-21% protein and 50% oil content (Ayeen, 1996). It holds second position in the world in edible oil manufacturing following soybean oil (Nandha, *et al.*, 2014) and grouped among preeminent plant oils for human diet due to its nutritional worth (Skoric, et al, 2008). Its seeds provide considerable amount of vitamins, minerals and tocopherols (Skoric, 2009) and found to be rich in minerals like magnesium, iron, copper, calcium, zinc, sodium, potassium, phosphorus, selenium and manganese (Nandha,*et al.* 2014). Regular sunflower oil contains 69% linoleic acid, 20% oleic acid and 11% saturated fatty acids, but a number of strategies have been adopted to present advance range of sunflower oils with elevated oleic acid, stearic acid, linoleic acid, palmitic acid and low saturated acid. Presence of Vitamin E content in sunflower oil makes it advantageous for human consumption. Defense system against ROS (reactive oxygen species) is strengthened by the presence of Vitamin E component i.e., α-Tocopherol facilitating oxidation of polyunsaturated fatty acids. Growth conditions have a large impact on the fatty acid organization of the sunflower oil. Warmer climatic conditions generate more monounsaturated fatty acid MUFA oleic acid and less n^{-6} polyunsaturated fatty acid (PUFA) and linoleic acid (an essential fatty acid, EFA) in comparison to colder climatic conditions (Morrison, *et al.*,1995).

Fatty Acid Composition of Sunflower Oil: Fatty acids that cannot be synthesized in our body but most required for human health are designated as Essential fatty acids (EFA) and grouped to the class polyunsaturated fatty acids (PUFAs). On one hand, where saturated oils coagulate, these stay fluid in cold climatic conditions. PUFAs can be grouped in to two categories, omega-3 and omega6. Linoleic acid and alpha-linoleic acid (ALA) is parent of omega-6 fatty acid and omega-3 fatty acid respectively (Singh, 2005). Although human body cannot synthesize either of these fatty acids from scratch, it can use them to synthesize other essential fatty acids. These essential fatty acids are crucial in cell membrane construction and for maintenance of body developmental activities. These two categories of EFA are functionally different from each other. Polyunsaturated fatty acids like Arachidonic acid (AA) (omega-6), Dihomo-gamma-linolenic acid (DGLA) (omega-6) and Eicosapentaenoic acid (EPA) (omega-3) are involved in the manufacture of signaling components, eicosanoids. Eicosanoids are involved in controlling immunological system, cell development and blood clotting. But they control these functions depending on their origin either from AA, EPA or DGLA. This is the basic reason why maintaining the intake of AA (omega6), DGLA (omega-6), and EPA (omega-3) is significant for our proper health. An intake of omega 6 and omega 3 in the ratio of 5 to 10 has been recommended by world health organization (WHO) (WHO, 2003).

2.4.5. Physical and Chemical Characteristics of Edible Oil

These physicochemical parameters including density, moisture content, boiling point, saponification value, peroxide value, acid value and iodine value which is used to assess the quality and functionality of the oil(Farhoosh, *et al.*,2008).A good type edible oil which can be used as cooking purpose must consider with a proved range of the physicochemical parameters. By understanding the properties we can evaluate the oil for human health consideration, industrial application etc. Low density oils are highly appreciable to consumers (Jinfeng, *et al.*, 2011).Oils having higher value of the moisture content can be used for food texturing, baking, and frying and industrially in the manufacture of soaps, detergents, cosmetics and oil paints (SON,2000) The higher the saponification value values, the shorter the average chain length of the fatty acids and the lower the average molecular weight of the fatty acids and vice versa (Birnin,2011). By the study of peroxide value, the quality and stability of oils can be indicated by measuring the rancidity reactions have occurred during storage. Increase in peroxide value indicates the rancidity of oils due to relative higher oxidation in oils (AOCS, 2016) .Higher acid value indicates that triglycerides of oil are converted into fatty acids and glycerol which cause rancidity of the oil (Zahir, *et al.*, 2014). So, the cooking oils must have lower acid value otherwise the oil can damage human health. Iodine value measures the degree of unsaturation in a fat or vegetable oil (Ekwu and Nwagu,2004).

He higher the unsaturation, the greater the possibility of the oils to go to rancid Sever (AOCS, 2016) al researchers studied the physicochemical parameters to assess the quality and functionality of the oil (Jinfeng,*et al*., 2011; AOCS, 1993)

2.4.5.1. Physical Characteristics of Oils

I. Refractive Index

The refractive index (RI) is the ratio of the speed of light in a vacuum to the speed of light through a given material (Mohammed and Ali, 2015; Jack, *et al*., 2013). The summary of the refractive indices of the investigated oils at 20 °C are depicted (Suzanne, 2010). This signifies that the above mentioned samples probably contain highly unsaturated or long chain fatty acids in their triglycerides. It explains that the viscosity and density increases with saturation and polymerization. From the nutritional point of view, edible oils rich in unsaturated fatty acids, especially with monounsaturated fatty acid, are more useful than oils with saturated fatty acids (Nawal, *et al.*, 2014; Amos, *et al.*, 2013).

The refractive index of the oil was determined by AOAC .The refract meter was first adjusted at 1.3330 at 200 C with pure distilled water as a blank reading. A drop of the oil was placed in the instrument and telescope was adjusted so that the crosshairs were distinct and in focus. The adjustment of the knob was rotated until the lower part of the field was dark and the upper part was light and a clear definite boundary appeared. The coarse adjustment knob was moved first and then the fine adjustment knob until the boundary line coincided with the intersection of the cross hair in the telescope. The instrument was read when temperature is stable. (Nawal, *et al.*, 2014)

II. Color

Refined oils have usually soft tastes, clear and transparent appearance. The color values for the seven edible oils are given (Suzanne, 2010). Moreover, overheating, dilution ratios, presence of dissolved impurities and other unfinished product could give color to oils (Nangbes, *et al*., 2013). Color was determined according to handbook of food analysis(AOAC,1984) . The sample liquid and filtered through a filter paper to remove any impurities and traces of moisture tillis sure that the sample was absolutely clear and free from turbidity. The glass cell of desired size cleaned with carbon tetrachloride and allowed to dry. The cell filled with the oil and placed in position in the tonometer. The color matched with sliding red, yellow and blue colors(Suzanne, 2010).

Report the color of the oil in terms of Lovebird units as follows:

$$\text{Color reading} = (aY + 5bR)\text{or}(aY + 10bR) \qquad (2.7)$$

Where :

a = sumtotal of the various yellow slides (Y) used

b = sumtotal of the various red (R) slides used

$Y + 5R$is the mode of expressing the color of light coloured oils; and

$Y + 10\ R$are for the dark-coloured oils

III. Viscosity

Triglycerides (TGs) are major components of edible oils. The nature and arrangement of the fatty acids on the glycerol backbone of the triglyceride determine viscosity. Therefore, the oil viscosity has a direct relationship with degree of unsaturation and chain length of the fatty acids in lipids. Its value increases with increasing degree of saturation (Fazal, *et al.*, 2015). Oils obtained from palm plant normally exhibits low unsaturated to saturated fatty acid ratio (1.0) while oils from sunflower seeds have highest ratio in the triacylglyceride chain (10.1) (Nangbes, *et al.*, 2013).

Viscosity was determined according to Lemuel M. Diamante and Tainting Lan. The absolute viscosities of the different vegetable oils were determined using a Lamy viscometer RM100 (Lamy, France), a rotating viscometer with coaxial cylinder. Approximately 25 mL of oil was placed in the Tube DIN 1 outer cylinder, and then the bob MK Din-9 was inserted. The radius of the tube (Ra) is 16.25 mm and the radius of the bob is (Ri) 15.5 mm. The length of the bob is 54 mm. The correct mode was set for the appropriate measuring system (MS 19) and the measurement time was fixed at 60 seconds. The torque of each sample at the different temperatures was recorded at a range of shear rate (Y) from 64.5 to 4835 s^{-1}. All viscometric measurements of the samples were carried out in triplicate. Every replicate was run twice the mean torque value of the two runs was recorded for each replicate at a given shear . The shear stress was obtained from (Fazal, *et al.*, 2015).

IV. Density

Oils with the density of lower values are highly appreciable to consumers (SON,2000).The density of sunflower oil, corn oil, rapeseed oil and peanut oil was experimentally determined for temperature ranging from 20 ^{o}C to 50 ^{o}C. The density of vegetable oils is dependent on their fatty acid composition, minor components and temperature (Fakhri and Qadir,2011) .This difference in the density of the oils may be due to the refined and respectively unrefined character of the studied oils. The oil density was determined according to (AOAC,1984)methods, using psychomotor. An empty stoppered psychomotor was weighed, filled with water and kept at constant temperature of 27°C in water bath for 30 minutes. The weight of water at 27°C was determined by subtracting weight of empty psychomotor from its weight when filled with water. The end of time stoppered psycho-meter was adjusted to proper level dried with a cloth and weighted. In the same manner, the weight of the oil at 27°C was determined(Fakhri and Qadir,2011). The density was calculated as follows:

$$\text{The density at } 27\ ℃ = \frac{W_1}{W_2} \qquad (2.8)$$

Where:

W_1= Weight of oil at 27 °C

W_2=Weight of water at 27°C

V. Moisture

The higher the value of the moisture content of the oil, the greater the value used for food texturing, baking, and frying and industrially in the manufacture of soaps, detergents, cosmetics and oil paints (Birnin and Garba,2011). Three crucibles were weighed and into each 10 g of the oil sample were added. The samples were dried to constant weights in an oven at 105°C, cooled in desiccators and weighed. The procedure was repeated thrice for each sample and the average value was determined (Zhou and Zhang, 2010).

2.4.5.2. Chemical Properties of the Oils

I. Acid Value (Av) and Free Fatty Acid (FFA) Content

The acid value (AV) is defined as the number of milligrams of potassium hydroxide required to neutralize the free acids present in one gram of oil. The acid value is determined by direct titration of the oil sample in an alcoholic medium, against standard potassium hydroxide solution, in the presence of phenolphthalein as indicator (SR EN ISO, 2009).The acid value is a measure of the free fatty acids content of the oil. Therefore, acid value is a good indicator of oil degradation caused by hydrolysis or enzymes (Othman and Ngaasapa, 2010). The variation of acid value in our samples could be due to difference in moisture contents as well as difference in the refining and deodorization technologies. High level of free fatty acids, especially linoleic acid, is undesirable in finished oils because they can cause off-flavors and shorten the shelf life of the oils (Mohammed, *et al*., 2015; Amos, *et al*., 2013).

High FFA value could be attributed to decomposition, poor extraction techniques, use of damaged seeds and incorrect or lengthy storage that can be accelerated by light and temperature. Nevertheless, the most common factor is being not refined which lead to remain higher in acidity (Rajko, *et al*., 2010; Fazal, *et al*., 2015).

The acid and free fatty acid (FFA) values are used to indicate the level of rancidity and edibility of oils (Amos, *et al*., 2013). Acid value represents the mg KOH required to neutralize the free fatty acid in 1 g of oil while free fatty acid is the percentage by weight of a specified fatty acid such as percent oleic acid in oil. Acid value was determined according to handbook of food analysis. The oil mixed thoroughly before weighing. About 5 of cooled oil sample accurately weighed in a 250 ml conical flask and 50 ml added to 100 ml of freshly neutralized hot ethyl alcohol and about one ml of phenolphthalein indicator solution. The mixture boiled for about five minutes and titrated while hot against standard sodium hydroxide shaking vigorously during the titration (Ngaasapa.

and Othman, 2001; Musa, *et al.*, 2012). The weight of the oil taken for the estimation and the strength of the alkali used for titration shall be such that the volume of alkali required for the titration does not exceed 10 ml (Nangbes, *et al.*,2013).

Calculation

$$\text{Acid value} = 56.1\ \text{VNW} \tag{2.9}$$

Where:

V = Volume in ml of standard sodium hydroxide used

N = Normality of the Sodium hydroxide solution

W = Weight in g of the sample

The acidity is frequently expressed as free fatty acid for which calculation shall be.

$$\text{Free fatty acids as oleic acid} = \frac{28.2\text{VN}}{\text{W}} \tag{2.10}$$

Per cent by weight

$$\text{Acid value} = \text{Percent fatty acid (as oleic)} \times 1.99. \tag{2.11}$$

II. Saponification value

The saponification value (SV) is defined as the weight of potassium hydroxide, in milligrams, needed to saponify one gram of oil. The method of saponification value determination is based on the oil sample saponification by refluxing with a known excess of alcoholic potassium hydroxide solution. The alkali required for saponification is determined by titration of the excess potassium hydroxide with standard hydrochloric acid, in the presence of phenolphthalein as indicator. A corresponding blank reagent is simultaneously prepared (SR EN ISO, 2005). Saponification value is a measure of oxidation during storage, and also indicates deterioration of the oils.

Saponification measures the average chain length of the fatty acid that makes up the oil. In other words, saponification values are useful in providing information as to the quantity, type of glycerides and mean weight of the acids in a given oil sample (Mohammed and Ali, 2015; Fazal, *et al.*, 2015). The lower the saponification value, the larger the molecular weight of fatty acids in the glycerides or the number of ester bonds is less and vice versa (Musa, *et al*., 2012). The shorter the average chain length of the fatty acids, the higher the saponification value and the lower the average molecular weight of the fatty acids and vice-versa (Ekwu and Nwagu, 2004).The lower saponification values suggests that the mean molecular weight of fatty acids is lower or that the number of ester bonds is less. This might imply that the fat molecules did not interact with each other (Firestone,1994).

Saponification value was determined according to handbook of food analysis. About 1.5 to 2.0 g sample were transferred into a 200 ml conical flask. A 30 mL of 0.5 N potassium hydroxide ethanol, and fix a cooling pipe to the flask. The flask gently heated and occasionally shake while adjusting the heat so that back flow ethanol will not reach the top of cooling pipe. After heated for 1 hour, immediately cooled, and titrated with 0.5 N Hcl before the test liquid is solidified. Blank test performed for 3 times to obtain mean value of titration volume of 0.5 N hydrochloric acid. The saponification was calculated as followed:

$$\text{Saponification value (mg/g)} = (\mathrm{BL_1} - \mathrm{EP_1}) \times \mathrm{TF} \times \mathrm{C_1} \times \mathrm{K_1} \,/\, \mathrm{SIZE} \qquad (2.12)$$

Where:

EP_1 : Titration volume (mL)

BL_1 : Blank level (25.029mL)

TF : Reagent (HCl) factor (1.006)

C_1 : concentration conversion coefficient (28.05 mg/mL)

K_1 : Unit conversion coefficient (1)

SIZE : Sample size (g).

III. Peroxide Value

Peroxide value is the most common indicator of lipid oxidation. The unrefined vegetable oils are characterized by greater PV values, compared to refined oil. Peroxide value (PV) measures the miliequivalents of oxygen (hydro peroxides) per 1000 gram of oil. The peroxide value is a measure of the concentration of substances that oxidize potassium iodide to iodine. The oil sample taken into a glass stopper iodine flask, is dissolved in chloroform (SR EN ISO,2005). The measured volume of glacial acetic acid reagent and potassium iodide is accurately added and after thorough mixing, is placed in the dark for exactly fifteen minutes. At the end of the specified time, the mixture is diluted with distilled water to prevent free iodine loss. The amount of free iodine is determined by titration with sodium thiosulfate, using starch as indicator. A corresponding blank reagent is simultaneously prepared. The peroxide value was measured as followed (AOAC,1984)

$$\text{Peroxide value}\left(\frac{\text{meq}}{\text{kg}}\right) = (\text{EP}_1 - \text{BL}_1) \times \text{TF} \times \text{R} / \text{SIZE} \qquad (2.13)$$

Where:

EP_1 : Titration volume (mL)

BL_1 : Blank level (0.00mL)

TF : Factor of reagent (1.006)

R : Constant (10)

SIZE : Sample size (g)

Peroxide value is used as a measure of the extent to which rancidity reactions have occurred during storage and it is used as a good criterion for the prediction of the quality and stability of oils (Nangbes, *et al.*, 2013). High peroxide value could be resulted from high degree of unsaturation and found to increase with the storage time, temperature, light and contact with atmospheric oxygen (Mohammed and Ali, 2015)

There is a successive increase in peroxide value indicates the rancidity of oils due to relative higher oxidation in oils. Peroxide value ranges are closely related to the standard value of 10 meq O_2/kg as specified (AOCS, 2016).

2.5. Food Irradiation

Introduction Food irradiation is a non- thermal technology often called “cold pasteurization” or “irradiation pasteurization” because it does not increase the temperature of the food during treatment (Cleland, 2005). The process is achieved by treating food products with ionizing radiation. Other common non- thermal processing technologies include high hydrostatic pressure, high- intensity pulsed electric fields, ultraviolet (UV) light, and cold plasma. Irradiation technology has been in use for over 70 years. It offers several potential benefits, including inactivation of common food borne bacteria and inhibition of enzymatic processes (such as those that cause sprouting and ripening); destruction of insects and parasites; sterilization of spices and herbs; and shelf life extension. The irradiation treatment does not introduce any toxicological, microbiological, sensory, or nutritional changes to the food products (packaged and unpackaged) beyond those brought about by conventional food processing techniques such as heating (vitamin degradation) and freezing (texture degradation) (Morehouse and Komolprasert, 2004). It is the only commercially available decontamination technology to treat fresh and fresh- cut fruits and vegetables, which do not undergo heat treatments such as pasteurization or sterilization. This is critical because many recent food borne illness outbreaks and product recalls have been associated with fresh produce due to contamination with Listeria , Salmonella , and Escherichia coli . ” (Maherani, *et al*., 2016).

Irradiation is generally defined as the exposure of a substance to radiation of various frequencies. food irradiation is the process in which a product or commodity is exposed to ionizing radiation to improve its safety and to maintain its quality. During irradiation, energy is transferred from a source of ionizing radiation into the treated product. Among the irradiation process parameters, the most important is the amount of ionizing energy absorbed per unit mass of the target material, which is termed ‘absorbed dose’ or simply ‘dose’. (IAEA, 2008).

Although it is little known by the general public, the irradiation process is used on a wide commercial scale across the world to enhance polymers and to sterilize single-use medical devices. The technology is also used to maintain the quality of food, improve its microbiological safety or reduce waste. The Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture estimates that the quantity of food irradiated in 2013 was approximately 700 000 tones.

One of the most significant changes in the radiation processing industry since 1995 has been the adoption of quality assurance procedures (IAEA,2008), a standard of the International Organization for Standardization (ISO) which sets out the requirements of a quality management system, has become a universal reference. For the radiation sterilization of healthcare products, many irradiation facilities across the world are now certified to Sterilization of Health Care Products (IAEA,2006), which contains the requirement for the development, validation and routine control of irradiation processes. For food irradiation, a similar standard Food Irradiation: Requirements for the Development, Validation and Routine Control of the Ionizing Radiation Process Used for the Treatment of Food (IAEA,2011)was developed and published for the first time in 2011. It builds on the international standards for food irradiation and a code of practice for food irradiation facilities that are enshrined in the Codex Alimentarius standards and guidelines, which underpin international trade. Understanding the requirements of such standards is not easy for non-specialists. In addition, the standards state what must be done but not how it must be done and an appreciation of how practices can best meet the standards can be demanding for non-specialists and specialists alike. It is recognized that the degree of implementation of quality management systems can be quite different in developed and developing countries. This difference sometimes results in a barrier for irradiated foods that are, or can be, the object of international trade.

2.5.1. The Potential of Food Irradiation Benefits and Limitations

Preservation of food has been a major anxiety of man over the centuries. Contamination with microorganisms and pests causes considerable losses of foods during storage, transportation and marketing (15% for cereals, 20% for fish and dairy products and up to40% for fruits and vegetables). Particularly, pathogenic bacteria are an important cause of human suffering and one of the most significant public health problems all over the world. The World Health Organization (WHO) stated, the infectious and parasitic diseases represented the most frequent cause of death worldwide (35%), the majority of which happened in developing countries in 1992 (Loaharanu, 1994). Numerous processing techniques have been developed to control food spoilage and raise safety. The traditional methods have been supplemented with pasteurization (by heat), canning, freezing, refrigeration and chemical preservatives (Agrios, 2005). Another technology that can beadded to this list is irradiation. Food irradiation is the process of exposing amount of energy in the form of speed particles or rays for improving food safety, eliminating and reducing organisms that destroy the food products. This is a very mild treatment, because a radiation dose of 1 kGy represents the absorption of just enough energy to increase the temperature of the product by 0.36°C. It means that, heating, drying and cooking cause higher nutritional losses. Moreover, heterocyclic ring compounds and carcinogenic aromatic produced during thermal processing of food at high temperatures were not identified in irradiated foods (Tomlins, 2008). More than one century of research has gone into the understanding of the effective use of irradiation as a safety method. It has been repeatedly considered and judged suitable on available evidence. The international bodies including the Food and Agriculture Organization (FAO), the International Atomic Energy Agency (IAEA), WHO and Codex Alimentarius Commission (CAC) investigate projects on food irradiation to verify the safety and quality of different irradiated products. It has shown that irradiation used on alone or in combination with other methods could improve the microbiological safety and extend shelf-life (IAEA, 2009). Furthermore, people are very confused to distinguish irradiated foods from radioactive foods. At no time during the irradiation process does the food come into contact with the radiation source and, it is not possible to induce radioactivity in the food by using gamma rays or electron beams up to 10 MeV (Farkas, 2004).

The differences in chemical and physical structure of organisms, environmental factors, moisture content, temperature during irradiation, presence or absence of oxygen and in their ability to recover from the radiation injury cause the distinctions in their sensitivity to radiation. According to long-term animal feeding studies, radiation-pasteurized or sterilized foods are safe and nutritious also for humans (Thayer and Boyd, 1999).

Irradiation is used for a variety of reasons, such as disinfesting food, reducing or eliminating food borne pathogens, shelf life extending and may serve as a quarantine treatment for many fruits, vegetables, nuts, cut flowers and animal origin products to facilitating international trade of foods (Marcotte,2005). But, not all foods are appropriate for irradiation. For instance, some fruits (such as cucumbers, grapes, and some tomatoes) are sensitive to radiation. Nowadays, over 60countries use irradiation for one or more food products. But, the misconceptions and irrational fear of nuclear technologies mostly caused the lack of acceptance of food irradiation. It should be noted that if irradiated products offer clear advantages, and the science-based information on the process is readily available, consumers would be ready to accept more irradiated products. Table 2.1 shows the different applications of irradiation to food and the indicative minimum dose for each purpose.

Table 2.1 The Different Applications of Irradiation to Food and the Indicative Minimum Dose for Each Purpose

Indicative dose range (kGy)	Effects	Examples
0.1–1	Sprouting inhibited. Ripening delayed. Insects unable to reproduce (phytosanitary treatment). Insects killed Parasites inactivated (helminths and protozoa).	Potatoes, onions, garlic and yams Banana and papaya Fresh produce Dried fish, dried fruit and legumes Meat products, fresh fruit and vegetables
1–10	Number of spoilage organisms reduced Shelf life extended Non-sporulating microorganisms inactivated Microbiological contamination reduced	Strawberries Refrigerated meats and fish, ready-to-eat meals. Refrigerated or frozen meats, fish and seafood, pre-cut fruit and vegetables. Spices and dried food ingredients
Above 10	Reduce microorganisms to the point of sterility	Hospital diets emergency rations and food for astronauts

2.5.2. Irradiation Facility Licenses

Any person or organization intending to build or operate an irradiation facility should notify the national regulatory authority for radiation safety and control of radiation sources, and should submit an application for authorization from the regulatory authority for sating, design, construction, acquisition, storage and operation of the irradiation facility (IAEA, 2010). Any subsequent modification that may have implications on radiation safety should also be made only after receiving approval from the regulatory body.

The operating organization is responsible for the safety of the facility and for operating the irradiation facility in accordance with the national regulatory requirements and radiation safety standards. In accordance with SSG-8 (IAEA,2010),the facility should, among other things:

1. Have personnel qualified in radiation protection matters, including a radiation protection officer;
2. Conduct safety assessments.
3. Conduct periodic verifications of safety.
4. Have a radiation protection program in place.

Inspections by other governmental bodies may also verify compliance with fire safety rules, conventional work safety standards and, increasingly, protection against malevolent or terrorist acts. An increasing number of irradiation facilities have voluntarily implemented quality systems using standards published by ISO as a reference and requested third parties to certify that they comply with these standards.

Chapter Three

Experimental Part

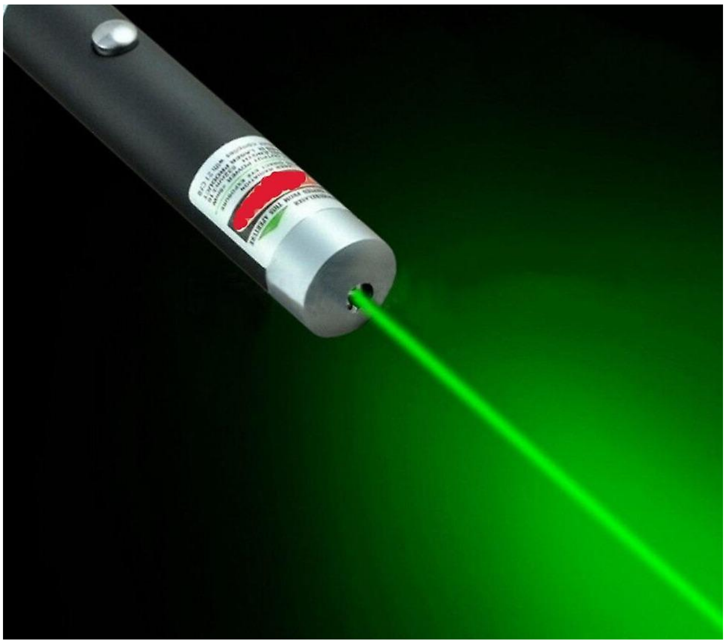
CHAPTER THREE

EXPERIMENTAL PART

3.1. Apparatus and Devices

3.1.1. Diode Laser

The laser system used to irritate sesame oil was Green Laser Pointer 303, China ,with a wavelength 532nm and CW output power 1 Watt.



Fig(3.1) Green Laser Pointer Laser

3.1.2. Carbon Dioxide Laser

The laser system used to irritate sunflower oil was $30W$ (maximum power) CO_2 laser Modle IB-601B supplied from (Beijing InnobriTechnology,China) with 10600nm Wavelength.

Fig(3.2) CO2 Laser

3.1.3. Electrical Heater

The capacity of the electric heater is 35.35 Watt in a time of two minutes , and its thermal energy is 4242.19 Joule.

Fig(3.3) Electrical Heater

3.1.4. Magnetic Stirrer

A magnetic stirrer or magnetic mixer is a laboratory device that employs a rotating magnetic field to cause a stir bar immersed in a liquid to spin very quickly, thus stirring it. It was used to distribute the laser heat for all molecules of oil while exposing.

Fig(3.4) Magnetic Stirrer

3.1.5. Fourier Transform Infra-Red Spectrometer

Fourier Transform Infra-Red Spectrometer (IR Spirit, Shimadzu, Japan). Fourier Transform Infrared Spectrometer (IR Spirit, Shimadzu, Japan).Model/Part number: FTIR-8400S ,Product Weight: 37 kg Package Weight: 47 kg , MPN: FTIR-8400S, Brand: SHIMADZU ,UPC : Does not apply

Fig(3.5)Fourier Transform Infra-Red Spectrometer

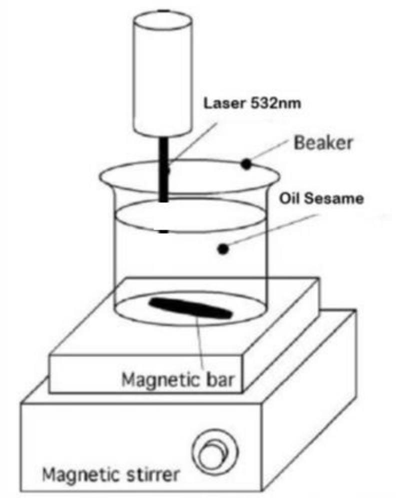
3.2. Materials

Sesame oil and Sunflower oil were obtained from the local market in Khartoum, Sudan. Seven samples of sesame oil were taken with amount of 50 ml for each sample.Five samples of sunflower oil were taken with amount of 50 ml for each sample. Then the following processes were done.

3.3. Methods

3.3.1. Laser Irradiation of Sesame Oil

The irradiation process of sesame oil was done using diode laser (Green Laser Pointer 303, China) with a wavelength 532nm,CW mode and output power 1 Watt, while the oil samples were placed in open crucible with a capacity of 100 ml on magnetic stirring at room temperature. The distance between the oil and the end of the laser was 1 cm. Six samples were exposed to the laser beam at different time durations, namely 10, 20, 30, 40, 50 and 60 minutes, the seventh one was left to be control sample with duration time of exposure equal to 0 minutes.



Fig(3.6) Laser Irradiation of Sesame Oil

3.3.2. Heating Process of Sunflower Oil

One of the five sunflower oil samples was heated to 250°c using electrical heater. The second sample was heated twice to the same degree (250°c) using electrical heater; it was left to cools to the room temperature then reheated; it takes two minutes in heating and in reheating. The third sample was heated to 50°c using laser irradiation. The fourth sample was heated twice to the same degree (50°c) using laser irradiation; it was left to cools to the room temperature then reheated. The irradiation process was done using carbon dioxide laser (CO_2)(Model IB-601B, Beijing Innobri Technology, China) with wavelength 10600nm and output power 30 Watt with CW mode, with duration time of exposure equal to 30 minutes. The sunflower oil sample was placed in open beaker with a capacity of 100 ml on magnetic stirring at room temperature. The distance between the oil and the end of the laser was 1 cm. The fifth one was left to be control sample.

Fig(3.7) Heating Process of Sunflower Oil

3.3.3. Physicochemical Properties Characterization

The characterization of the stored and non-stored sesame oil samples and the sunflower oil samples physicochemical properties: acid value, ester value, free fatty acids, peroxide value, density, refractive index, viscosity, and moisture; were measured according to the methods described below. All tests were performed in triplicate. The measurement of these parameters for sesame oil samples were repeated after two weeks storage at ambient conditions, for the irradiated and control samples, in order to determine their relative stability.

3.3.3.1. Refractive Index Measurements

The refractive index of the oil was determined by A.O.A.C. (AOAC, 1990).The refract meter was first adjusted at 1.3330 at 20˚C with pure distilled water as a blank reading. A drop of the oil was placed in the instrument and telescope was adjusted so that the cross hairs were distinct and in focus. The adjustment of the knob was rotated until the lower part of the field was dark and the upper part was light and a clear definite boundary appeared. The coarse adjustment knob was moved first and then the fine adjustment knob until the boundary line coincided with the intersection of the cross hair in the telescope. The instrument was read when temperature is stable.

3.3.3.2. Determination of Color

Color was determined according to handbook of food analysis (Nollet, 2004). The sample liquid and filtered through a filter paper to remove any impurities and traces of moisture till is sure that the sample was absolutely clear and free from turbidity. The glass cell of desired size cleaned with carbon tetrachloride and allowed to dry. The cell filled with the oil and placed in position in the tonometer. The color matched with sliding red, yellow and blue colors.

Report the color of the oil in terms of Lovebird units as follows:

$$\text{Color reading } = (a\,Y\ +\ 5\,b\,R)\text{or}(a\,Y\ +\ 10\,b\,R). \qquad (3.1)$$

Where a = sum total of the various yellow slides (Y) used

b = sum total of the various red (R) slides used

Y + 5R is the mode of expressing the color of light colored oils; and

Y + 10 R are for the darccolored oils.

3.3.3.3. Viscosity

Viscosity was determined according to Lempel M. Diamante and Tainting Lan. The absolute viscosities of the different vegetable oils were determined using a Lamy viscometer RM100 (Lamy, France), a rotating viscometer with coaxial cylinder. Approximately 25 mL of oil was placed in the Tube DIN 1 outer cylinder, and then the bob MK Din-9 was inserted. The radius of the tube (R_a) is 16.25 mm and the radius of the bob is (R_i) 15.5 mm. The length of the bob is 54 mm. The correct mode was set for the appropriate measuring system (MS 19) and the measurement time was fixed at 60 seconds. The torque of each sample at the different temperatures was recorded at a range of shear rate (Y) from 64.5 to 4835 s^{-1}. All viscometric measurements of the samples were carried out in triplicate. Every replicate was run twice the mean torque value of the two runs was recorded for each replicate at a given shear rate.

3.3.3.4. Density Measurements

The oil density was determined according to AOAC (1990) methods, using psycho-meter. An empty stoppered psycho-meter was weighed, filled with water and kept at constant temperature of 27°C in water bath for 30 minutes. The weight of water at 27°C was determined by subtracting weight of empty psycho-meter from its weight when filled with water. The end of time stoppered psycho-meter was adjusted to proper level dried with a cloth and weighted. In the same manner, the weight of the oil at 27°C was determined. The density was calculated as follows:

$$\text{The density at} 27^{\circ}C = \frac{W_1}{W_2} \qquad (3.2)$$

Where:

W_1= Weight of oil at 27 °C

W_2=Weight of water at 27°C

3.3.3.5. Acid Value Measurements

Acid value was determined according to handbook of food analysis (Nollet, 2004). The oil mixed thoroughly before weighing. About 5 of cooled oil sample accurately weighed in a 250 ml conical flask and 50 ml added to 100 ml of freshly neutralized hot ethyl alcohol and about one ml of phenolphthalein indicator solution. The mixture boiled for about five minutes and titrated while hot against standard sodium hydroxide shaking vigorously during the titration. The weight of the oil taken for the estimation and the strength of the alkali used for titration shall be such that the volume of alkali required for the titration does not exceed 10 ml.

Calculation

$$\text{Acid value} = \frac{56.1VN}{W} \qquad (3.3)$$

Where:

V = Volume in ml of standard sodium hydroxide used.

N = Normality of the Sodium hydroxide solution.

W = Weight in g of the sample.

The acidity is frequently expressed as free fatty acid for which calculation shall be.

$$\text{Free fatty acids as oleic acid} = 28.2\frac{VN}{W} \qquad (3.4)$$

Per cent by weight

$$\text{Acid value} = \text{Percent fatty acid (as oleic)} \times 1.99 \qquad (3.5)$$

3.3.3.6. Saponification Value Measurements

Saponification value was determined according to handbook of food analysis (Nollet, 2004). About 1.5 to 2.0 g sample were transferred into a 200 ml conical flask. A 30 mL of 0.5 N potassium hydroxide ethanol, and fix a cooling pipe to the flask. The flask gently heated and occasionally shaked while adjusting the heat so that back flow ethanol will not reach the top of cooling pipe. After heated for 1 hour, immediately cooled, and titrated with 0.5 N Hcl before the test liquid is solidified. Blank test performed for 3 times to obtain mean value of titration volume of 0.5 N hydrochloric acid.

The saponification was calculated as followed:

$$\text{Saponification value } (\frac{\text{mg}}{\text{g}}) = (BL_1 - EP_1) \times TF \times C_1 \times K_1 / SIZE \qquad (3.6)$$

Where:

EP_1 : Titration volume (mL).

BL_1 : Blank level (25.029mL).

TF : Reagent (HCl) factor (1.006).

C_1 : concentration conversion coefficient (28.05 mg/mL).
(Potassium hydroxide in Eq.:56.11×0.5).
K_1 : Unit conversion coefficient (1).
SIZE : Sample size (g).

3.3.3.7. Peroxide Value Measurements

Peroxide value was determined according to Handbook of food analysis (Nollet, 2004). Five grams of the sample were delivered into a conical flask with stopper. About 25 mL of solvent (15 ml acetic acid+10 ml chloroform) were added and gently shake to dissolve the sample completely. The air inside flask gently replace with nitrogen to remove remaining oxygen. One ml of saturated potassium iodide was added and immediately seals the flask and gently shakes it for one minute. The flask left at room temperature 15 to 20°C in a dark room. Thirty mL of pure water were added, and the flask sealed and stirred. Titration with 0.01mol/L sodium thiosulphate was performed to measure peroxide value.

The peroxide value was measured as followed:

$$\text{Peroxide value } \left(\frac{\text{meq}}{\text{kg}}\right) = (EP_1 - BL_1) \times TF \times R \,/\, SIZE \qquad (3.7)$$

Where:

EP_1 : Titration volume (mL).

BL_1 : Blank level (0.00mL).

TF : Factor of reagent (1.006).

R : Constant (10).

SIZE : Sample size (g).

3.3.4. FT-IR Characterization

FT-IR spectra of irradiated and non-radiated sesame oil samples after the storage and sunflower oil samples period were carried out using a Fourier Transform Infra-Red Spectrometer (IR Spirit, Shimadzu, Japan). It is used to compare the chemical structure of the irradiated and unirradiated sesame oils samples after the storage and sunflower oil samples period for monitoring the oxidation process in oils.

Chapter Four

Results and Dissections

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1. Sesame Oil

The exposure of oils to sunlight (with its all wavelengths) can induce photooxidation in the oils in two ways:

1. A direct photochemical reaction occurs with the photons from the high-intensity sunlight rays, being directly absorbed and causing a change in the oil's chemical composition (Sankar,*et al*., 2005). The oil undergoes a photochemical change, and the $C - C$ bond dissociates into radicals. The free radical state is unstable and readily reacts with atmospheric triplet oxygen, which initiates the oxidation of the oil (Choe and Min, 2006).
2. The second way in which oxidation could occur is referred to as photosensitized oxidation, which is triggered by the presence of photosensitizes in oils (Min and Boff, 2002). In this study, laser irradiation of sesame oil samples were investigated for the changes in the physicochemical properties like acid value, ester value, free fatty acids, peroxide value, density, refractive index, viscosity, and moisture content. The results of these properties were compared at the 1st and 15th day of storage.

4.1.1. Results and discussion of Physicochemical Properties of Sesame Oil

The variations in the effects of laser irradiation durations time on the physical and chemical characteristics of sesame oil are presented in figures 4.1–4.7.

4.1.1.1. Results and discussion of the Chemical Properties of Sesame Oil

Figure 4.1a, shows that the initial ester value of the sesame oil was 185.29 mg KOH/g; whereas, after irradiation to 10 minute using laser it slightly decreased to 185.10 mg KOH /g; when increasing of 532 nm laser irradiation time from 20 to 60 minutes, it gradually increased to the range from 191.52 to 196.63 mg KOH) /g. After storage period of 15 consecutive days; the ester value of the control sample raised to 187.91 mg KOH /g; whereas, the ester value of all irradiated sesame oil samples reduced and became close to the initial value of the sesame oil 185.29 mg KOH /g.

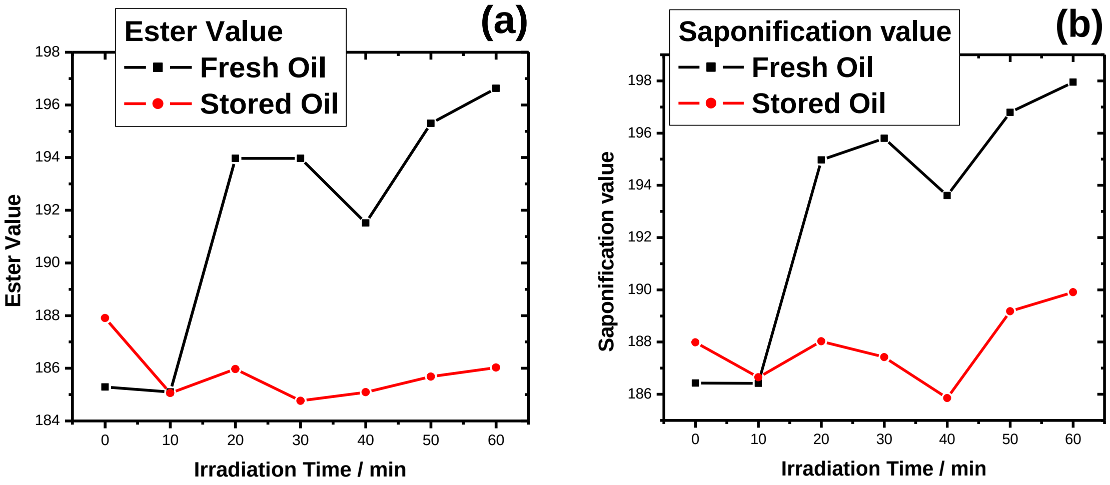


Figure 4.1. Effect of green laser irradiation time on (a) Ester value (b) Saponification value of fresh and stored sesame oil.

The obtained saponification value of the oil samples in figure 4.1b, showed 186.43 mg KOH/g for the control sample before storage and it ranged from 186.42 to 197.95mg KOH/g for the laser irradiated oil samples. These values are lying in the expected range of 186 – 195 mg KOH/g for sesame oil as specified by Codex Standard (2001) (Codex, 2001). The highest saponification value indicates a high content of triacylglycerol, consistent with the high ester value (Tesfaye and Abebaw 2016); this is why graph in figure 4.1a matching graph in figure 41b.

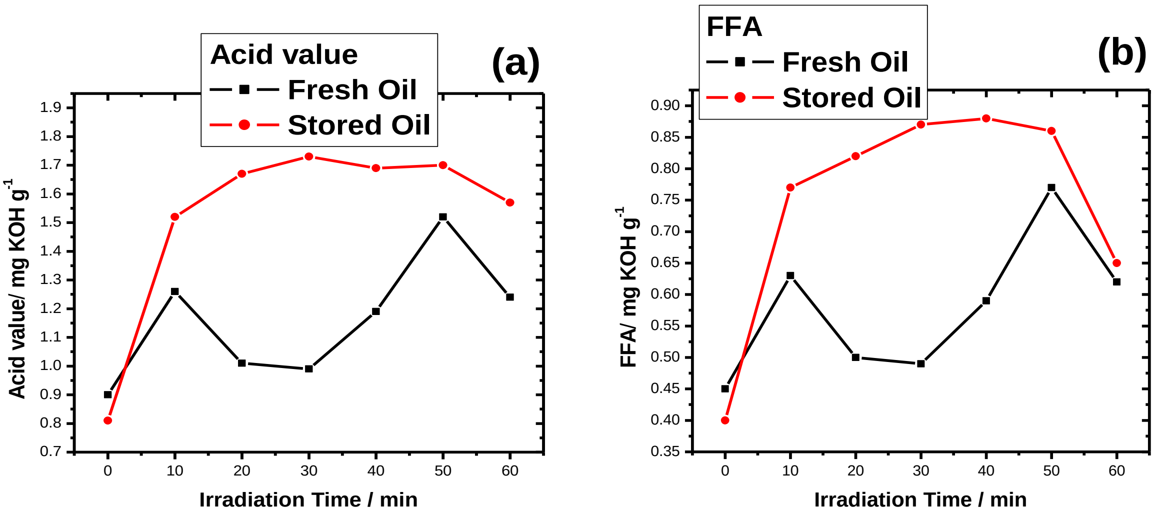


Figure 4.2. **Effect of green laser irradiation time on (a) Acid value (b) Free Fatty Acids (FFA) of fresh and stored sesame oil.**

Figure 4.2a, shows acid values of irradiated and non-irradiated sesame oil samples; it indicates that the acid value of the control sesame sample found to be 0.9 mg KOH/ g oil; which is lower than that reported for Brazzaville-Congo sesame oil (1.8%) (Kimbonguila,*et al*., 2010). While the irradiated samples, were very small between 0.99 and 1.26%. All other irradiated sesame oil exhibit acid values greater than the acid values of the control sample and lies within desirable limits 0.0–4.0 mg KOH/g oil (Mohammed and Hamza, 2008). After the storage period, the acid vale of the control sample slightly decreased, while the irradiated samples showed acid values higher than before storage period. It can be noticed that the acid value of irradiated sesame oil samples before and after storage period lie within the desirable range.

Free fatty acids of the irradiated and control samples before and aftter storage period are shown in figure 4.2b. Its value was 0.45% for the control sample and it ranged from 0.49 up to 0.77% for the irradiaed samples. While it decreased to 0.40% for the control sample and ranged from 0.65–0.88% for laser irradiated samples after a storage period of two weeks. The data indicates the increase in free fatty acids with the presense of radiation due to the photochemical iteraction that catlized by the visible green laser photons which convert triglycerides into free fatty acids. Free fatty acids are produced by the hydrolysis of fats in the presence of water or enzyme lipase during storage (Fazlullah,et al., 2004).

Acid value is the measure of percentage content of free fatty acids (FFAs) in a substance, and it is an important reference parameter to determine the conservation quality of fat and oils (Pereira, *et al.*,2018). Acid value of oil is an indicator of hydrolytic rancidity (Sharma,*et al*., (2019). The acid value is related with the lipase activity and hydrolysis of triacylglycerols in the seed (Khoddami, *et al*.,2014).

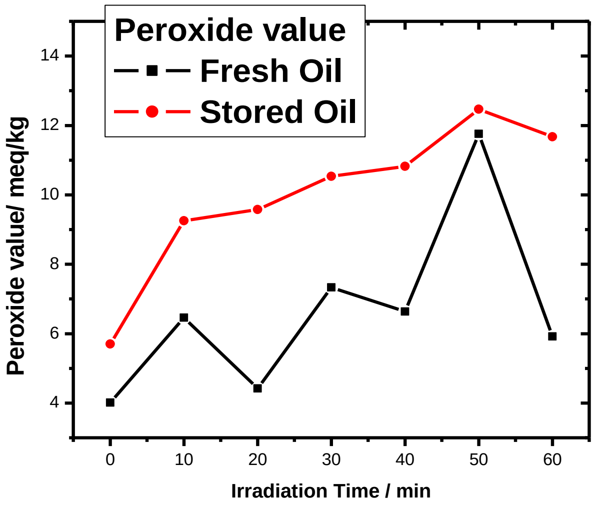


Figure 4.3. **Effect of green laser irradiation time on the peroxide value of fresh and stored sesame oil.**

Peroxide value is one of the most widely used tests for oxidative rancidity; it measured the concentration of peroxides and hydro peroxides formed in the initial stages of lipid oxidation.

The effect of increasing laser irradiation time on the peroxide value of sesame oil before and after storage period is presented in figure 4.3. It shows that the peroxide values ranged from 4.01 to 11.86 Meq/Kg in fresh oils and from 5.70 to 12.47 in the stored oils. It indicates that the laser initiate oxidation processes in sesame oil. Peroxide values of all oil samples after the storage period is higher than they were before the storage period. It is also clear from the data that the peroxide value increases with increasing radiation durations; this peroxide value increase can be due to the bond cleavage of sesame oil. Peroxide values for all oil samples studied showed levels value lower than the (20.0 meq O_2/kg) limitation in the standard for sesame oil. The storage of irradiated lipid food, in the presence of oxygen, accelerates the autoxidation(Diehl, 1983). It has been proved that irradiation accelerates the oxidation of unsaturated fatty acids and cholesterol during storage of foods(Heath, *et al.*, 1990; Hwangand Maerker, 1993). The chemical changes leads to physical changes as reported by Ku, *et al*., 2014(Ku, *et al.*, 2014).

4.1.1.2. Results of the Physical Properties of Sesame Oil

The chemical changes of the sesame oil samples due to laser radiation processing and during the storage time lead to some physical changes.

The effect of increasing laser irradiation time on the refractive index of sesame oil before and after storage period is presented in figure 4.4.

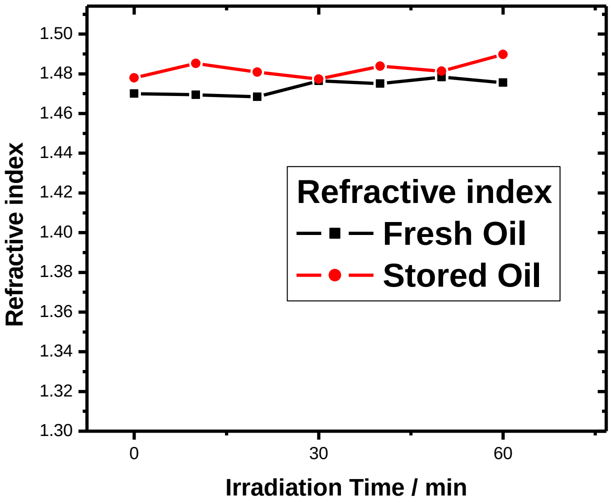


Figure 4.4. Effect of green laser irradiation time on the refractive index of fresh and stored sesame oil.

Refractive index is an important optical parameter to analyze the light rays traversing through materials medium. Refractive index can be used as a quality control technique to identify the adulteration of edible oils. From the obtained results it is found that laser irradiation slightly affect the refractive index of the fresh sesame oil samples in the range from 1.4684 to 1.4784. While, it is found that, refractive index of the stored sesame oil samples higher than it before storing ranged from 1.4773 to 1.4898, and most of them excesses the recommended codex standard of 1.469 – 1.479 for sesame oil.

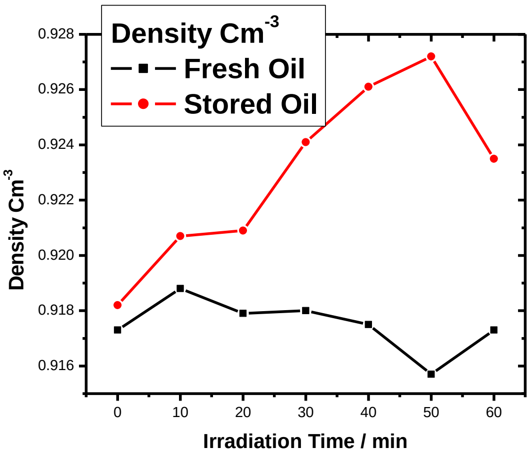


Figure 4.5. Effect of green laser irradiation time on the density of fresh and stored sesame oil.

The initial density of the fresh sesame oil was 0.9173 cm^{-1}; when it irradiated by laser, it ranged between 0.9188 and 0.9157 cm^{-1}, it fall in the recommended codex standard of 0.915- 0.924 cm^{-1} for sesame oil. While after the storage period it increases and ranged from 0.9182 to 0.9272 cm^{-1}, with two samples slightly excesses the recommended codex standard, see figure 4.5.

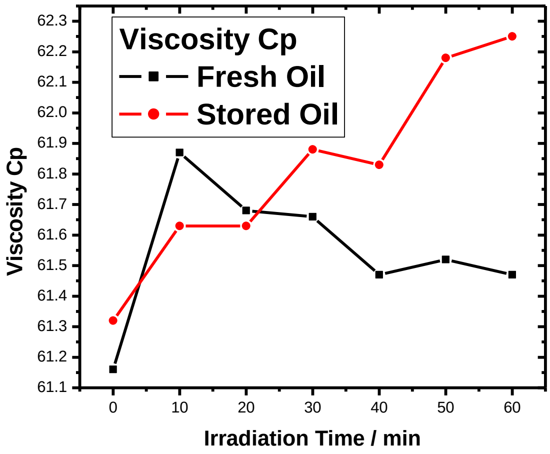


Figure 4.6. Effect of green laser irradiation time on the viscosity of fresh and stored sesame oil.

Figure 4.6; illustrate the effect of increasing 532 nm laser irradiation time on the sesame oil viscosity before and after storage. The viscosity of the control sample was 61.16 cP, after the stored period it increased to 61.32 cP. Meanwhile, the viscosity of the irradiated samples increased to range 61.87 to 61.47 cP. After the stored period it observed that there is a gradually increase in the viscosity with the increase of the irradiation time of all samples from 61.32 to 62.25cP.

Viscosity is related to the chemical properties of the oils. The increase in viscosity of emulsions when irradiated can be explained by unavoidable evaporative loss of volatile contents of oil. Formation of smaller oil micelles during irradiation not only has physical significance in terms of stability but also has effects on viscosity. Smaller micelles promote viscous emulsions due to more particle–particle interactions as a result of a larger interfacial area (Homer,*et al*., (2012).

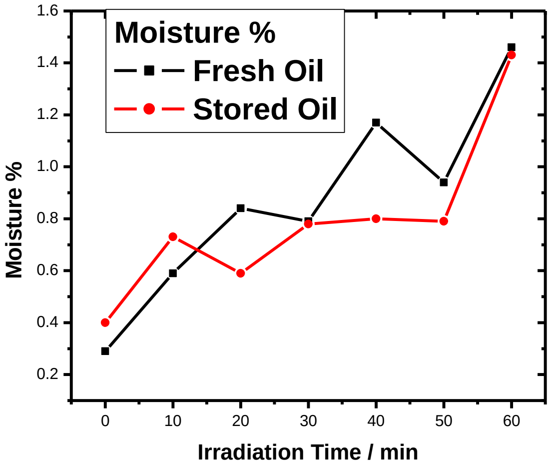
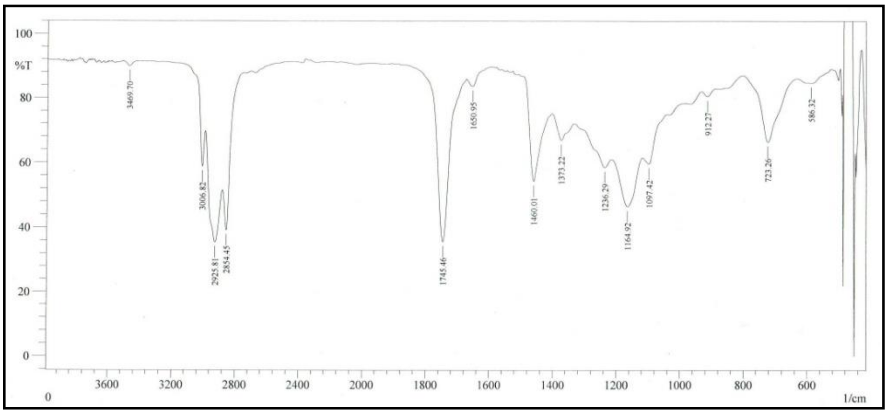


Figure 4.7. Effect of green laser irradiation time on moisture of fresh and stored sesame oil.

The effect of increasing 532 nm laser irradiation time on the sesame oil moisture, before and after the storage period is shown in figure 4.7. It is clear from the data that the moisture of the sesame oil increases with increasing irradiation time before and after storage. The moisture of the control sample was 0.29%, which increased gradually to 1.46% with increasing the irradiation time. Likewise, after the storage period the moisture increased gradually from 0.40% in the control sample up to 1.43% with increasing the irradiation time.

4.1.2. FTIR Results of Sesame Oil

FT-IR spectroscopy is a very good technique for analysis, as the intensities of the bands in the spectrum are proportional to concentration. FT-IR spectra of the control and laser irradiated samples after the storage period are presented in figure 4.8.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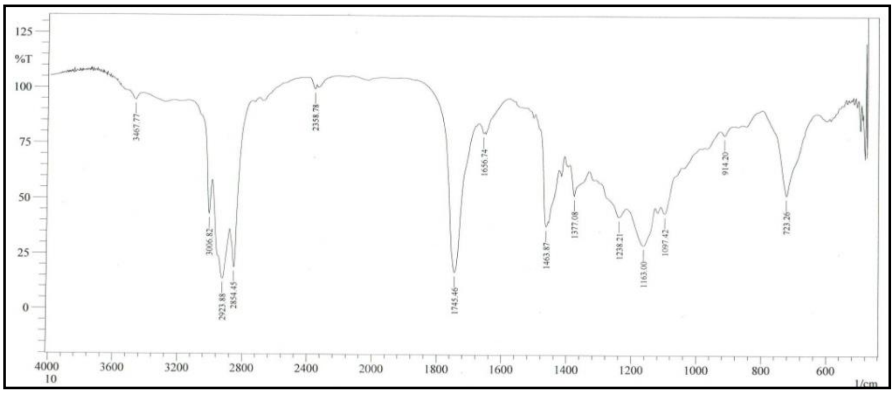
(a)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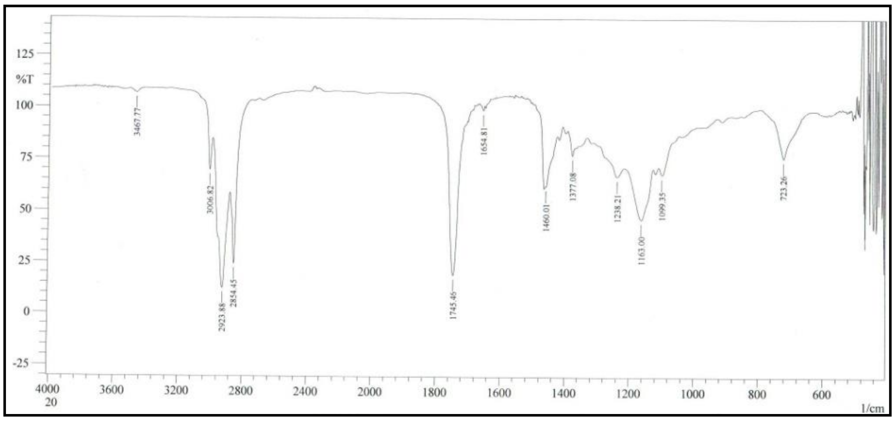
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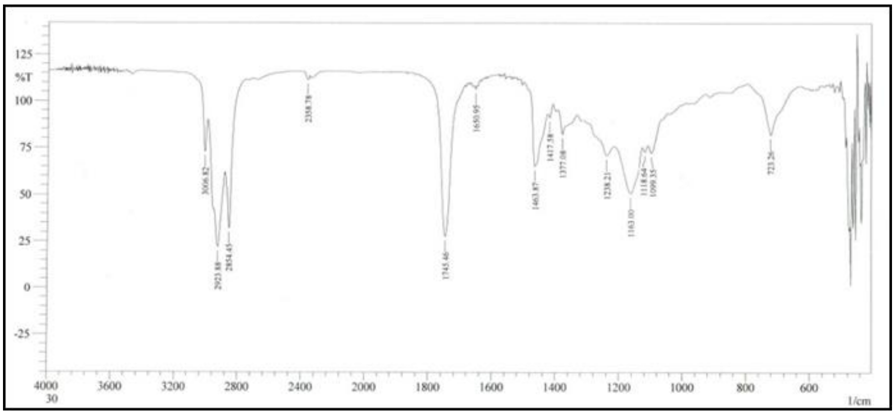
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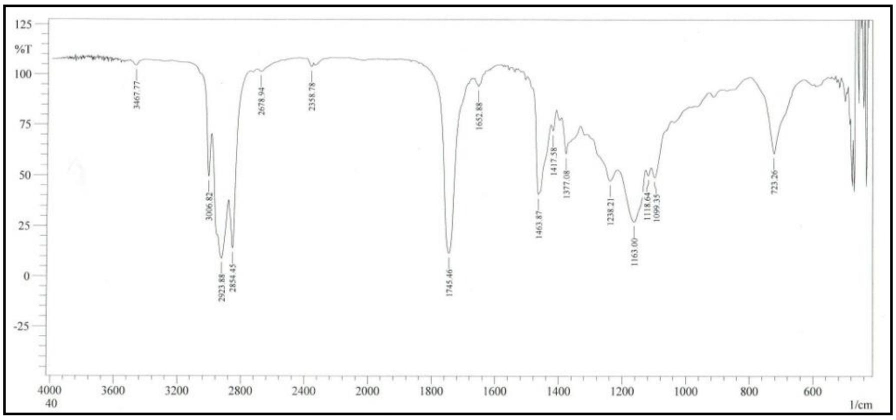
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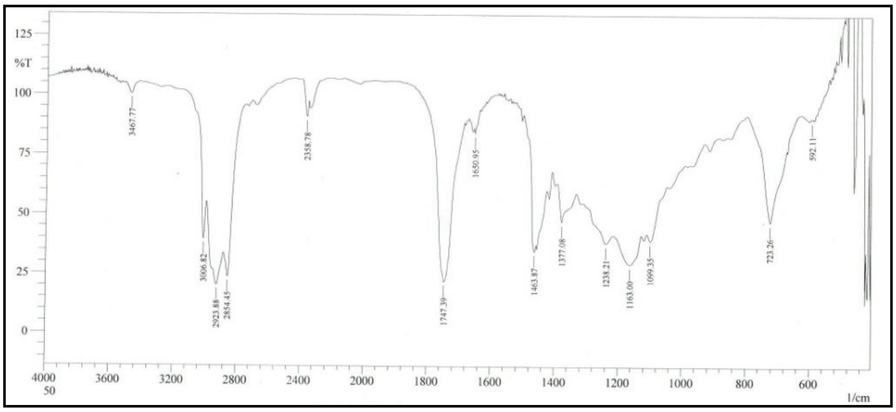
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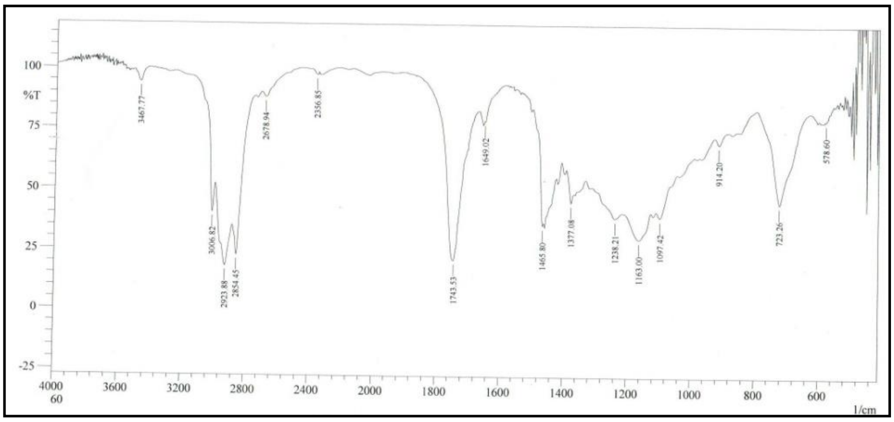


(g)

Figure 4.8. FT-IR spectrum of the irradiated sesame oil samples: (a) control (00 minutes) (b) 10 minutes, (c) 20 minutes, (d) 30 minutes, (e) 40 minutes, (f) 50 minutes, and (g) 60 minutes.

It showed that there exists a notable difference in the bands between the control and laser irradiated samples appeared during the storage period. The spectra of the stored irradiated oil samples containing several absorption bands, that are absent from the spectrum of the control sample. The first new band is occurs in 2688.94 cm^{-1} shift in the spectra of the high-irradiated samples (40, 50, and 60 minutes). The other new band is appears at 2358.78 cm^{-1}, 11417.58 cm^{-1}, and 1118.64 cm^{-1}.

4.2. Sunflower Oil

Due to repeated heating, the quality, color, smell and taste of edible oil changes due to the formation of polymers and polar compounds (Azman, et al., 2012).

In this study; the effects of heating and reheating of the sunflower oil samples using electrical heater and carbon dioxide laser were investigated for the changes in the physicochemical properties like acid value, ester value, free fatty acids, peroxide value, density, refractive index, viscosity, and moisture content. The results of these properties were compared with the unheated sample.

4.2.1. Physicochemical Properties of Sunflower Oil

The effects of heating and reheating on the physical and chemical characteristics of sunflower oil using laser and electrical heater compared with the unheated sunflower oil are presented in tables 1-3.

4.2.1.1. Results and Discussion of the Chemical Properties of Sunflower Oil

The obtained results of heating processes of the sunflower oil in table 4.1 showed increasing in the oil chemical properties. Reheating processes caused in more increasing in the oil chemical properties; these results agree with the results of Adriana (Aziz, *et al.*, 2018). It was observed that laser-heating process increased the oil chemical properties greater than the increasing of reheating processes by electrical heater.

Table 4.1: Values of the chemical properties of the five sunflower oil samples

Sample		Acid value	FFA	Peroxide value	Saponification value	Ester value
Control		0.56	0.30	3.98	186.01	184.10
Electrical heater	heated once	0.75	0.37	6.41	186.09	184.82
	heated twice	0.84	0.46	8.37	187.08	185.30
Laser-heating	heated once	1.27	0.65	9.37	187.43	186.97
	heated twice	1.52	0.76	10.43	189.12	186.58

This obtained results showed that the trend of chemical properties was increased crossover all tested samples starting from the once-heated electrically sample undergoing with the twice-heated electrically then once-laser-heated undergoing with twice-laser-heated sample. Chemical properties had increased with the increasing number of heating sessions. The increment of chemical properties of the oils is due to the development of hydro peroxides of unsaturated fatty acids because of the lipid oxidation process. Oxidation of oils is influenced by means of different factors which include the degree of unsaturation, heat, light, oil processing, antioxidants and transition metals (Aziz, *et al.*, 2018).

4.2.1.2. Results and discussion of the Physical Properties of Sunflower Oil

The chemical reactions in the oil leads to changing in some physical properties such as viscosity, density, darken color, or production of foam (Ku, *et al.*, 2014); these changes clearly showed in this study in the results presented in tables 4.2 and 4.3.

Table 4.2: Color measurements of the five sunflower oil samples

Oil sample		B	R	Y
control		0	1.2	10.2
Electrical heater	heated once	0	1.6	10.6
	heated twice	0	1.8	11.5
Laser-heating	heated once	0.1	2.3	11.7
	heated twice	0.2	2.5	11.9

Table 4.2 showed the changing of the oil color for the control, heated, and reheated samples. The blue color does not appear by heating and reheating using the electrical heater; while it was increased from 0 up to 0.2 by heating and reheating using the laser heating . The red color increased from 1.2 up to 1.8 by heating and reheating using the electrical heater; while it was increased from 1.2 up to 2.5 by heating and reheating using the laser heating . The yellow color increased from 10.2 up to 11.5 by heating and reheating using the electrical heater; while it was increased from 10.2 up to 11.9 by heating and reheating using the laser heating .It was observed that oil has darkened its color and emanated smoke during heating sessions, which indicates oil degradation. Chemical reactions, which influenced by heating processes; results in foaming production, darkening of oil color, and off-flavor. Increase in color of oil by heating was reported by (Soniaand Badereldeen, 1983), and was found also by (Augustin, *et al*., (1987) and (Mudawi, *et al.,* 2014).

Table 4.3: the other physical properties of the five sunflower oil samples

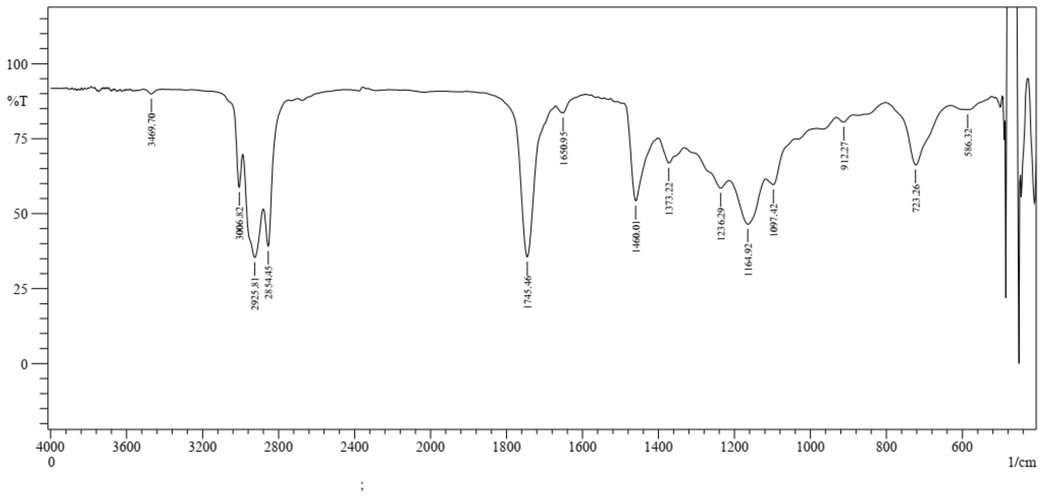
Sample		Moisture %	Density cm^3	Viscosity Cp	Refractive index
Control		0.34	0.9150	60.58	1.4698
Electrical heater	heated once	0.46	0.9178	60.83	1.4767
	heated twice	0.63	0.9173	60.85	1.4786
Laser-heating	heated once	0.79	0.9176	61.06	1.4794
	heated twice	0.91	0.9176	61.47	1.4866

Table 4.3 shows changes in moisture, density, viscosity and refractive index (RI) of all oil samples during heating and reheating. Moisture increased from 0.34% in control sample to 0.63% after reheating process using electrical heater, while it increased up to 0.91% after reheating process using laser. The density increased from 0.9150cm^3 in control sample to 0.9178 cm^3 after heating

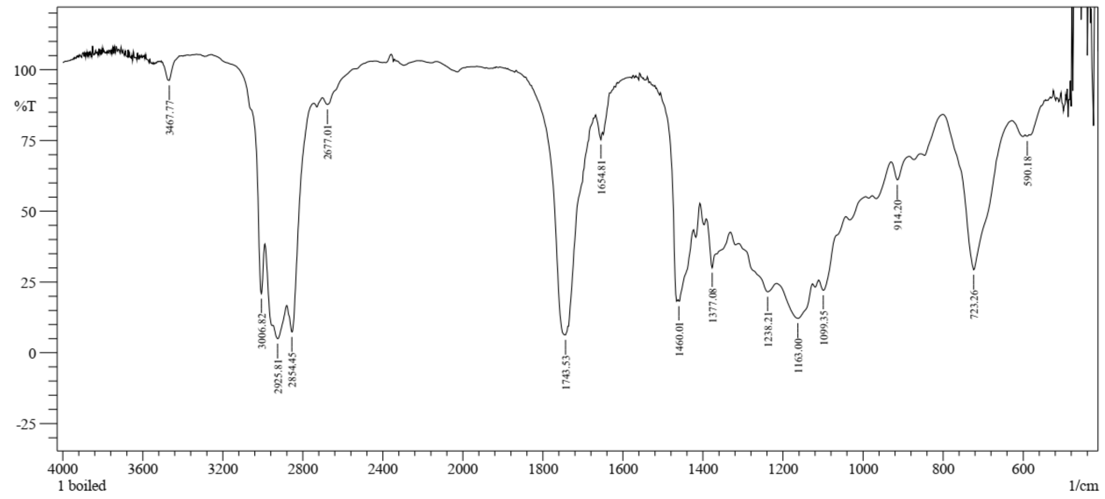
and it decreased to 0.9173cm^3 after reheating process using electrical heater, while it increased up to the same value 0.9176cm^3 after heating and reheating process using laser. While viscosity increased from 60.58 Cp in control sample up to 60.85 Cp after reheating process using electrical heater, whereas it increased up to 61.47 Cp after reheating process using laser. The refractive index increased from 1.4698 in control sample to 1.4786 after reheating process using electrical heater, while it increased up to 1.4866 after reheating process using laser. It is clear that all physical properties of the five sunflower oil samples increased by heating and reheating at 50°c. Physical properties temperature dependence is agree with the study of (Wiege, *et al.*, 2020).

4.2.2. FTIR Results of Sunflower Oil

FT-IR spectroscopy is a very good technique for analysis, as the intensities of the bands in the spectrum are proportional to concentration. FT-IR spectra of the control, heated and reheated sunflower oil samples are presented in figure 4.9.



(a)


100
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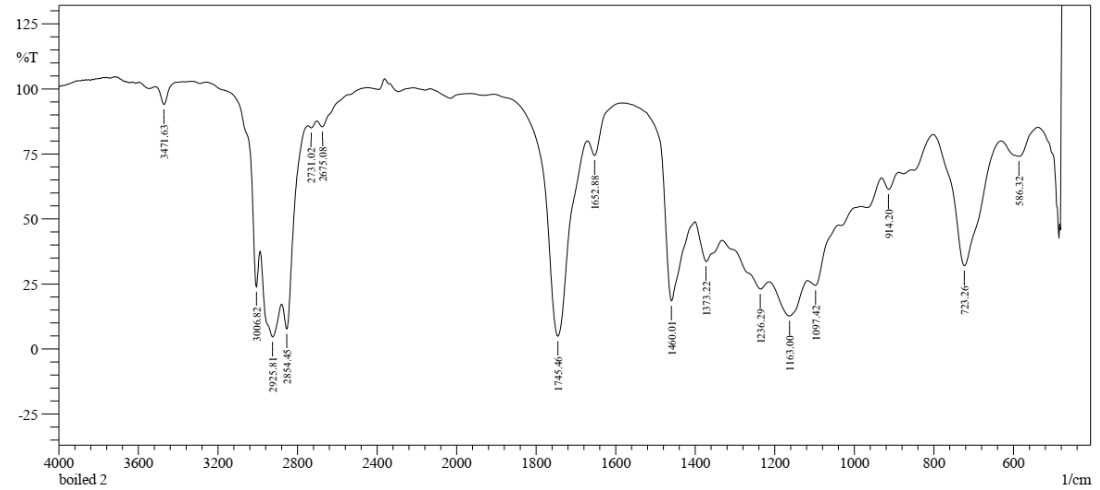
75
50
25
0
-25
3467.77
3006.82
2925.81
2854.45
2677.01
1743.53
1654.81
1460.01
1377.08
1238.21
1163.00
1099.35
914.20
723.26
590.18
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
600
1 boiled
1/cm

(b)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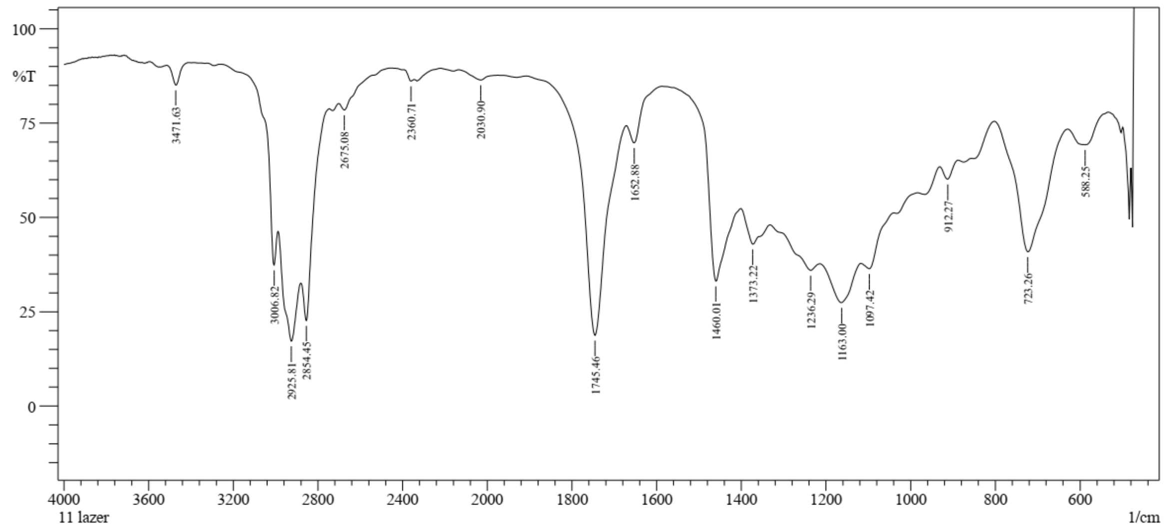
100
75
50
25
0
-25
3471.63
3006.82
2925.81
2854.45
2731.02
2675.08
1745.46
1652.88
1460.01
1373.22
1236.29
1163.00
1097.42
914.20
723.26
586.32
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
600
boiled 2
1/cm

(c)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1652.88
1460.01
1373.22
1236.29
1163.00
1097.42
912.27
723.26
588.25
4000
3600
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1800
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1400
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1000
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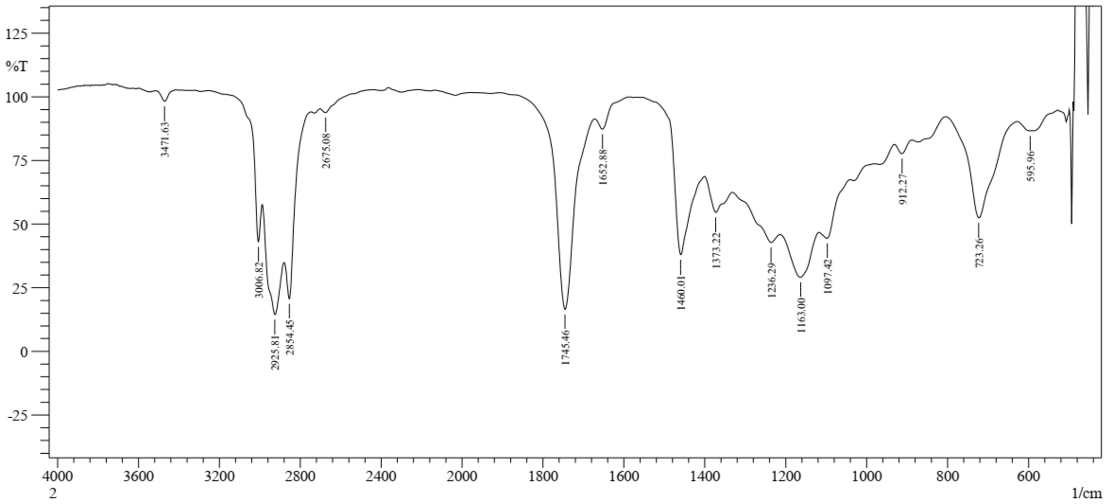
600
11 lazer
1/cm

(d)



(e)

Figure 4.9: FT-IR spectrum of the sunflower oil samples: (a) control (unheated) (b) Electrical heated once, (c) Electrical heated twice, (d) laser heated once, (e) laser heated twice.

FT-IR spectrum showed that each sample contains C=O (1745.46cm^{-1}) and C-O (1163.00 cm^{-1}) which may indicate possible existence of CO, ester functionality (oils), samples were rich in C-H (sp^3)(2923.88 cm^{-1} and 2854.45 cm^{-1}) indicating presence of CH_3 and CH_2as reinforced by bands in (1463.87 cm^{-1} and 1377.08 cm^{-1}). Weak band at 1650 cm^{-1} C=C with C=C-H 3006 cm^{-1} but not conjugated with C=O.

Table 4.4: Band Assignments of Sunflower Oil Samples.

FTIR shift/ cm^{-1}	Assign	Ref.
3469.70	O-H str	(Sudhakar et al., 2018)
3006.82	C-H st.vib.(sp^2)	(Poiana, et al., 2015)
2923.88 - 2854.45	C-H st.vib.(sp^3)	(Liu, and Kazarian, 2022)
2731.02	Aldehydic C-H Str.	(Hafeez, et al., 2019)
2360.71	C-H	(Evangelin and Gurulakshmi, 2020)
1745.46	C=O st.vib.	(Zhuang, 2020)
1654.81	C=O Str.	(Gupta, et al., 2011)
1650	C=C st.vib.	(Panicker, et al., 2009)
1463.87, 1377.08	CH_3, CH_2 bend.	(Priest, et al., 1999)
1163.00	C-O st.vib.	(Simonova, and Karamancheva, 2013)

The changed band assignment of sunflower oil samples were: 3469.70 cm^{-1}, 2677.01 cm^{-1} 2731.02 cm^{-1}, 2360.71 cm^{-1}, 1654.81 cm^{-1} and 585-595 cm^{-1}.It showed a notable difference depend on heating and reheating processes using laser and electrical heater.

4.3. Conclusions

In this research, the influence of green laser (532nm) radiation on some physicochemical properties of sesame oil before and after storage period of 15 consecutive days; and the effect of carbon dioxide laser (10600 nm) heating and reheating on the chemical and physical characteristics of sunflower oil compared to the effect of electrical heater heating and reheating were investigated. It was found that the green laser capable to occur changes in the physicochemical properties of sesame oil different from that occur by the visible light. Laser irradiation leads to photochemical interactions that change the chemical and physical properties of the oil. Moreover, storing to period of two weeks also caused changes in the physicochemical properties of sesame oil. The results also showed that when the same cooking oil is reheated, the chemical reactions enhance foaming, darkening of oil color, increased viscosity, and off-flavor. Hence, repeated heating of the oil can lead to degradation of the cooking oil, both chemically and physically.

All changes due to laser irradiation fall in the recommended codex standard (2001). Fourier transform infrared spectroscopy results confirm the physicochemical changes. Moreover, it was found that the long time of heating using laser rising temperature up to 50°c catalyzed chemical reactions that resulted in effects in the oil samples characteristics greater than the effects of the electrical heater in a few minutes with temperature 250°c.

4.4. Recommendations

1. It's recommend the usage of carbon dioxide laser with power greater than 30 watts so that the effect appears in less time .
2. It's recommend to study the effect of another single -wavelength on physical and chemical properties of other edible oil.

3. Increase the type of oils for computer ex(Corn – plam oil …).
4. Increase the strong time ex(2 month) .
5. Study the cooling on the heated oil change in(physics or chemical) properties.

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Appendix

Results of Physicochemical Properties of Sesame Oil

Sample	Acid value	FFA	Peroxide value	Moisture %	Density Cm^3	Viscosity Cp	Refractive index	Saponification matter	Ester Value
Control	0.65	0.32	4.05	0.24	0.9181	61.12	1.4767	186.22	185.11
	1.15	0.57	3.97	0.59	0.9175	61.18	1.4611	186.86	185.08
	0.90	0.45	4.01	0.41	0.9162	61.20	1.4727	186.22	185.67
Irradiated for 10 minutes	1.52	0.75	4.32	0.86	0.9181	61.97	1.9718	188.38	186.36
	1.00	0.50	8.62	0.66	0.9196	61.86	1.9667	185.99	184.67
	1.26	0.63	6.46	0.27	0.9188	61.80	1.9697	185.88	184.27
Irradiated for 20 minutes	1.08	0.54	3.87	0.88	0.9178	61.51	1.4718	196.27	193.36
	0.94	0.47	5.22	0.97	0.9182	61.88	1.4667	195.28	195.28
	1.02	0.50	4.17	0.66	0.9179	61.67	1.4667	193.36	193.28
Irradiated for 30minutes	1.06	0.53	8.65	0.55	0.9179	61.51	1.4781	195.67	193.36
	0.92	0.45	4.47	0.87	0.9182	61.81	1.4751	196.38	195.28
	0.99	0.49	8.88	0.96	0.9179	62.66	1.4762	195.36	193.28
Irradiated for 40minutes	1.12	0.56	9.42	1.11	0.9167	61.76	1.4718	187.67	185.76
	1.27	0.63	3.86	1.18	0.9182	61.52	1.4817	196.91	193.45
	1.19	0.59	6.64	1.22	0.9175	62.14	1.4718	196.27	195.36
Irradiated for50 minutes	1.50	0.75	11.66	0.99	0.9167	61.53	1.4781	195.21	194.28
	1.55	0.77	11.86	0.97	0.9117	61.67	1.4811	198.66	196.15
	1.52	0.79	16.75	0.87	0.9186	61.36	1.4761	196.50	195.48
Irradiated for 60minutes	1.15	0.57	3.73	1.36	0.9166	61.38	1.4721	197.27	196.51
	1.22	0.61	8.12	1.56	0.9170	61.66	1.4781	198.28	197.28
	1.36	0.68	5.93	1.47	0.9182	61.38	1.4767	198.29	196.11

Average values of physicochemical properties

sample	Acid value	FFA	Peroxide value	Moisture %	Density Cm^3	Viscosity Cp	Refractive index	Saponification matter	Ester value
control	0.9	0.45	4.01	0.29	0.9173	61.16	1.4701	186.43	185.29
Irradiated for 10minutes	1.26	0.63	6.46	0.59	0.9188	61.87	1.9694	186.42	185.10
Irradiated for 20minutes	1.01	0.50	4.42	0.84	0.9179	61.68	1.4684	194.97	193.97
Irradiated for 30minutes	0.99	0.49	7.33	0.79	0.9180	61.66	1.4764	195.80	193.97
Irradiated for 40minutes	1.19	0.59	6.64	1.17	0.9175	61.47	1.4751	193.61	191.52
Irradiated for 50minutes	1.52	0.77	13.42	0.94	0.9157	61.52	1.4784	196.79	195.30
Irradiated for 60minutes	1.24	0.62	5.92	1.46	0.9173	61.47	1.4756	197.95	196.63

Results of Physicochemical Properties of Sesame Oil store

sample	Acid value	FFA	Peroxide value	Moisture %	Density Cm^3	Viscosity Cp	Refractive index	Saponification matter	Ester value
control	0.86	0.43	5.86	0.36	0.9182	61.36	1.4681	188.36	187.27
	0.68	0.34	5.36	0.34	0.9175	61.27	1.4780	189.27	188.11
	0.88	0.44	5.88	0.51	0.9188	61.33	1.4880	186.36	188.36
Irradiated for 10 minutes	1.63	0.86	9.36	0.89	0.9201	61.86	1.4860	188.33	187.38
	1.56	0.78	9.52	0.85	0.9211	61.66	1.4820	185.29	183.51
	1.36	0.68	8.86	0.46	0.9210	61.36	1.4880	186.33	184.27
Irradiated for 20 minutes	1.58	0.74	8.97	0.76	0.9216	61.86	1.4767	187.51	186.27
	1.66	0.83	9.67	0.66	0.9211	61.66	1.4817	189.71	185.36
	1.76	0.88	10.11	0.36	0.9201	61.36	1.4841	186.86	186.27
Irradiated for 30minutes	1.78	0.89	10.86	0.86	0.9221	61.67	1.4672	186.52	184.27
	1.86	0.93	10.51	0.76	0.9281	61.86	1.4867	189.51	183.66
	1.56	0.78	10.22	0.71	0.9221	62.11	1.4781	186.22	186.36
Irradiated for 40minutes	1.67	0.88	10.36	0.86	0.9236	61.51	1.4867	186.77	185.27
	1.86	0.78	10.88	0.88	0.9272	61.86	1.4766	188.61	183.66
	1.56	0.98	11.22	0.66	0.9276	62.11	1.4882	182.17	186.36
Irradiated for50 minutes	1.86	0.93	12.36	0.91	0.9282	62.10	1.4811	188.51	187.71
	1.57	0.78	12.51	0.82	0.9267	62.18	1.4862	189.31	185.66
	1.67	0.88	12.55	0.66	0.9266	62.27	1.4767	189.71	183.66
Irradiated for 60minutes	1.45	0.27	11.81	1.66	0.9227	62.36	1.4866	189.67	186.51
	1.36	0.68	11.86	1.27	0.9288	62.51	1.4866	188.66	185.22
	1.89	0.99	11.33	1.36	0.9189	61.88	1.4961	182.41	186.36

Average values of physicochemical properties

sample	Acid value	FFA	Peroxide value	Moisture %	Density Cm^3	Viscosity Cp	Refractive index	Saponification matter	Ester value
control	0.81	0.40	5.70	0.40	0.9182	61.32	1.4780	187.99	187.91
Irradiated for 10minutes	1.52	0.77	9.25	0.73	0.9207	61.63	1.4853	186.65	185.06
Irradiated for 20minutes	1.67	0.82	9.58	0.59	0.9209	61.63	1.4808	188.03	185.97
Irradiated for 30minutes	1.73	0.87	10.53	0.78	0.9241	61.88	1.4773	187.42	184.76
Irradiated for 40minutes	1.69	0.88	10.82	0.80	0.9261	61.83	1.4838	185.85	185.09
Irradiated for 50minutes	1.70	0.86	12.47	0.79	0.9272	62.18	1.4813	189.18	185.68
Irradiated for 60minutes	1.57	0.65	11.67	1.43	0.9235	62.25	1.4898	189.91	186.03

Results of physicochemical Properties of Sunflower Oil

sample	Acid value	FFA	Peroxide value	Moisture %	Density Cm^3	Viscosity Cp	Refractive index	Saponification matter	Ester value
control	0.55	0.28	3.51	0.24	0.9146	60.51	1.4676	186.27	185.27
	0.66	0.38	3.86	0.36	0.9166	60.86	1.4667	186.30	183.66
	0.48	0.24	4.56	0.37	0.9138	60.38	1.4751	185.46	186.36
boiled once	0.78	0.39	6.36	0.45	0.9167	60.86	1.4785	186.33	185.27
	0.80	0.40	6.37	0.43	0.9182	60.77	1.4766	185.66	184.67
	0.66	0.33	6.51	0.50	0.9185	60.86	1.4751	186.27	184.51
Boiled twice	0.86	0.48	8.36	0.67	0.9281	60.97	1.4781	189.31	187.31
	0.82	0.42	8.52	0.66	0.9101	60.81	1.4797	186.27	185.32
	0.85	0.47	8.22	0.57	0.9137	60.78	1.4782	185.66	183.27
laser once	1.18	0.58	8.97	0.86	0.9167	60.86	1.4796	186.36	185.36
	1.27	0.68	9.27	0.66	0.9186	61.11	1.4784	187.67	187.27
	1.36	0.68	9.88	0.86	0.9176	61.22	1.4801	188.27	188.27
laser twice	1.67	0.83	10.36	0.95	0.9166	61.37	1.4852	189.27	186.31
	1.52	0.76	10.57	0.89	0.9175	61.66	1.4788	189.82	187.22
	1.36	0.68	10.36	0.88	0.9186	61.37	1.4867	188.28	186.20

Coluor:-

Sunflower oil sample	B	R	Y
control	0 0 0 Average :- 0	1.3 1.2 1.0 Average :- 1.2	10.3 10.2 10.1 Average:-10.2
boiled once	0 0 0 Average:-0	1.5 1.6 1.7 Average:-1.6	10.3 10.8 10.7 Average:-10.6
Boiled twice	0 0 0 Average:-0	1.9 1.8 1.6 Average:-1.8	11.2 11.8 11.5 Average:-11.5
laser once	0.1 0.1 0.1 Average:-0.1	2.8 2.1 2.1 Average:-2.3	11.7 11.9 11.5 Average:-11.7
laser twice	0.2 0.2 0.2 Average:-.0.2	2.7 2.5 2.3 Average:-2.5	11.8 11.9 11.9 Average:-11.9

Average values of physicochemical properties

sample	Acid value	FFA	Peroxide value	Moisture %	Density Cm^3	Viscosity Cp	Refractive index	Saponification matter	Ester value
control	0.56	0.30	3.98	0.34	0.9150	60.58	1.4698	186.01	185.10
boiled once	0.75	0.37	6.41	0.46	0.9178	60.83	1.4767	186.09	184.82
Boiled twice	0.84	0.46	8.37	0.63	0.9173	60.85	1.4786	187.08	185.30
laser once	1.27	0.65	9.37	0.79	0.9176	61.06	1.4794	187.43	186.97
Laser twice	1.52	0.76	10.43	0.91	0.9176	61.47	1.4866	189.12	186.58

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Impact of Single Wavelength (532 nm) Irradiation on the Physicochemical Properties of Sesame Oil

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Abstract

This paper investigates the influence of green laser (532 nm) radiation on some physicochemical properties of sesame oil before and after storage period of 15 consecutive days. The samples of sesame oil were irradiated with diode laser beam with wavelength 532 nm and 1 W output power, to duration times of 10, 20, 30, 40, 50 and 60 minutes, and kept for a storage period of 15 consecutive days along with an untreated control at ambient conditions. Physicochemical properties like acid value, ester value, free fatty acids, peroxide value, density, refractive index, viscosity, and moisture of sesame oil were studied. Fourier transform infrared spectroscopy was used to evaluate the degree of oxidation after irradiation processes. Their properties were compared at the 1st and 15th day of storage. Study reveals that green laser irradiation increases ester value, saponification, acid value, free fatty acids, peroxide value, viscosity and moisture content of sesame oil; while, it slightly changes refractive index and density. Study also indicates that the storage period of 15 days decreases the ester value, saponification value, and moisture content; while, it accelerates acid value, free fatty acid, peroxide value, density viscosity, and slightly increases the refractive index. Meanwhile, FTIR spectra of the stored samples revealed a notable difference due to the green laser irradiation and storage. These results suggest that exposing sesame oil to the green laser irradiation influences their oxidation stability and quality.

Keywords

Edible Oils, Food Irradiation, Physicochemical Characteristics, Quality Constants, Sesame Oil

1. Introduction

Electromagnetic radiations and electrons are used in food irradiation process, whereas, extensive damage was caused to food when treated with some other types of radiations like neutrons, deuterons and α-rays [1]. Electromagnetic radiation caused various chemical and physical changes when it interacts with materials [2]. Light plays a photochemical initiator role and is capable to inducing photochemical reactions when exposed to food [3]. The mechanism of the photochemical oxidation of vegetable oils has been extensively studied [4] [5]. Recently, several researchers studied the impact of laser matter interaction; which could happen with metal, tissue, ceramic or food [6] [7] [8] [9] [10].

Irradiation technology usage in food preservation grew a large interest, because of its efficiency and its potential application. Various benefits are obtained with the use of irradiation such as protecting meats from pathogenic microorganism and increase their shelf lifetime. Likewise, irradiation produces structural changes in many nutrients [11]. In addition, irradiation of milk and yoghurt improves their shelf life [12] [13].

Changes in nutritional and organoleptic characteristics of foods are due to the carbonyl compounds produced from the interaction of the free radicals formed by irradiation of unsaturated fatty acids and oxygen. Irradiation plays an important role in the storage life of food and enhances the safety by reducing pathogenic and spoilage microorganisms [14]. Various chemical and physical parameters of edible oil are utilized to monitor the compositional quality of oils [15] [16]. The chemical changes in the oil lead to physical changes like viscosity and density, darken color, production of foam [17].

Sesame (*Sesamum indicum L.*) is a source of excellent vegetable oil; it is one of the highest oil contents (35% - 63%) among oil crops [18] [19]. The presence of some natural antioxidants such as sesamum, sesamin, sesamolin, and sesamol in the sesame oil makes them one of the most stable vegetable oils in the world [20]. The oil of the sesame seed is very resistant to rancidity especially after hydrogenation due to the presence of natural antioxidants [21]. Until now, to the best of our knowledge, researchers studied the effects of different portions of electromagnetic radiation such as gamma, X-ray, sunlight and infrared on the physicochemical characteristics of edible oils; but they have not studied the effect of the single wavelength. In this work, we tried to determine the effect of green diode laser (532 nm) irradiation on the physicochemical characteristics of sesame oil before and after storage period of 15 consecutive days.

2. Materials and Methods

2.1. Material

Sesame oil was obtained from the local market in Khartoum, Sudan. Seven samples of sesame oil were taken with amount of 50 ml for each sample. Then the following processes were done.

2.2. Laser Irradiation

The irradiation process was done using diode laser (Green Laser Pointer 303, China) with a wavelength 532 nm and output power 1 Watt, while the oil samples were placed in open beaker with a capacity of 100 ml on magnetic stirring at room temperature. The distance between the oil and the end of the laser was 1 cm. Six samples were exposed to the laser beam at different time durations, namely 10, 20, 30, 40, 50 and 60 minutes, the seventh one was left to be control sample with duration time of exposure equal to 0 minutes.

2.3. Physicochemical Properties Characterization

The characterization of the stored and non-stored sesame oil samples physicochemical properties: acid value, ester value, free fatty acids, peroxide value, density, refractive index, viscosity, and moisture; was carried out according to the methods described in the [22]; All tests were performed in triplicate. The measurement of these parameters was repeated after two weeks storage at ambient conditions, for radiated and control samples, in order to determine their relative stability.

2.4. FT-IR Characterization

FT-IR spectra of irradiated and non-radiated sesame oil samples after the storage period were carried out using a Fourier Transform Infra-Red Spectrometer (IR Spirit, Shimadzu, Japan). It is used to compare the chemical structure of the irradiated and un-irradiated sesame oils samples after the storage period for monitoring the oxidation process in oils.

3. Results and Dissections

The exposure of oils to sunlight (with its all wavelengths) can induce photooxidation in the oils in two ways: 1) A direct photochemical reaction occurs with the photons from the high-intensity sunlight rays, being directly absorbed and causing a change in the oil's chemical composition [23]. The oil undergoes a photochemical change, and the C-C bond dissociates into radicals. The free radical state is unstable and readily reacts with atmospheric triplet oxygen, which initiates the oxidation of the oil [4]. 2) The second way in which oxidation could occur is referred to as photosensitised oxidation, which is triggered by the presence of photosensitisers in oils [24]. In this study, laser irradiation of sesame oil samples were investigated for the changes in the physicochemical properties like acid value, ester value, free fatty acids, peroxide value, density, refractive index, viscosity, and moisture content. The results of these properties were compared at the 1st and 15th day of storage.

3.1. Physicochemical Properties

The variations in the effects of laser irradiation durations time on the physical and chemical characteristics of sesame oil are presented in **Figures 1-7**.

3.1.1. Results of the Chemical Properties

Figure 1(a) shows that the initial ester value of the sesame oil was 185.29 mg

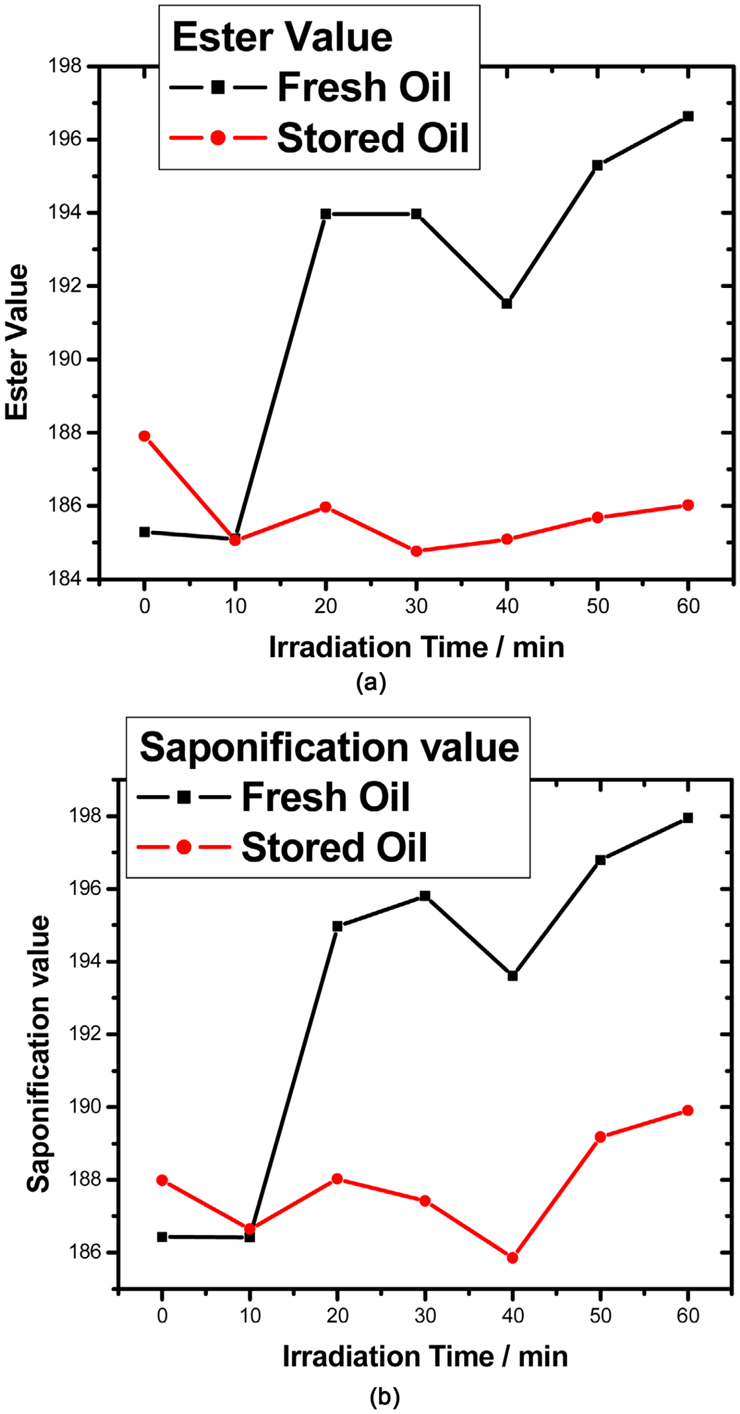
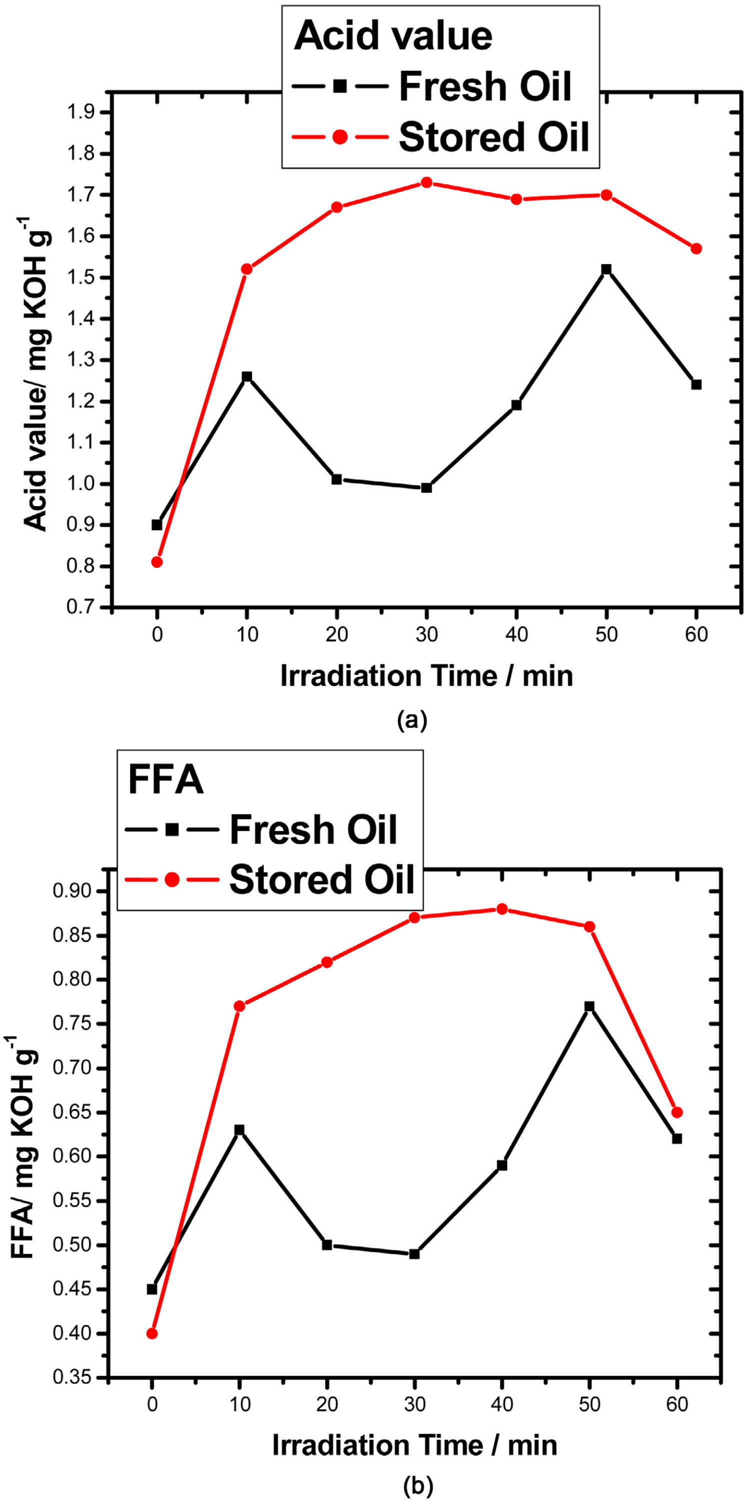


Figure 1. Effect of green laser irradiation time on (a) Ester value; (b) Saponification value of fresh and stored sesame oil.

KOH/g; whereas, after irradiation to 10 minute using laser it slightly decreased to 185.10 mg KOH/g; when increasing of 532 nm laser irradiation time from 20 to 60 minutes, it gradually increased to the range from 191.52 to 196.63 mg KOH)/g. After storage period of 15 consecutive days; the ester value of the control sample raised to 187.91 mg KOH/g; whereas, the ester value of all irradiated



(a)

(b)

Figure 2. Effect of green laser irradiation time on (a) Acid value; (b) Free Fatty Acids (FFA) of fresh and stored sesame oil.

sesame oil samples reduced and became close to the initial value of the sesame oil 185.29 mg KOH/g.

The obtained saponification value of the oil samples in **Figure 1(b)**, showed

186.43 mg KOH/g for the control sample before storage and it ranged from 186.42 to 197.95 mg KOH/g for the laser irradiated oil samples. These values are lying in the expected range of 186 - 195 mg KOH/g for sesame oil as specified by Codex Standard (2001) [25]. The highest saponification value indicates a high content of triacylglycerol, consistent with the high ester value [26]; this is why graph in **Figure 1(a)** matching graph in **Figure 1(b)**.

Figure 2(a) shows acid values of irradiated and non-irradiated sesame oil samples; it indicates that the acid value of the control sesame sample found to be 0.9 mg KOH/g oil; which is lower than that reported for Brazzaville-Congo sesame oil (1.8%) [27]. While the irradiated samples, were very small between 0.99 and 1.26%. All other irradiated sesame oil exhibit acid values greater than the acid values of the control sample and lies within desirable limits 0.0 - 4.0 mg KOH/g oil [18]. After the storage period, the acid vale of the control sample slightly decreased, while the irradiated samples showed acid values higher than before storage period. It can be noticed that the acid value of irradiated sesame oil samples before and after storage period lie within the desirable range.

Free fatty acids of the irradiated and control samples before and after storage period are shown in **Figure 2(b)**. Its value was 0.45% for the control sample and it ranged from 0.49 up to 0.77% for the irradiated samples. While it decreased to 0.40% for the control sample and ranged from 0.65% - 0.88% for laser irradiated samples after a storage period of two weeks. The data indicates the increase in free fatty acids with the presence of radiation due to the photochemical interaction that catalyzed by the visible green laser photons which convert triglycerides into free fatty acids. Free fatty acids are produced by the hydrolysis of fats in the presence of water or enzyme lipase during storage [28].

Acid value is the measure of percentage content of free fatty acids (FFAs) in a substance, and it is an important reference parameter to determine the conservation quality of fat and oils [29]. Acid value of oil is an indicator of hydrolytic rancidity [30]. The acid value is related with the lipase activity and hydrolysis of triacylglycerols in the seed [31].

Peroxide value is one of the most widely used tests for oxidative rancidity; it measured the concentration of peroxides and hydroperoxides formed in the initial stages of lipid oxidation.

The effect of increasing laser irradiation time on the peroxide value of sesame oil before and after storage period is presented in **Figure 3**. It shows that the peroxide values ranged from 4.01 to 11.86 Meq/Kg in fresh oils and from 5.70 to 12.47 in the stored oils. It indicates that the laser initiate oxidation processes in sesame oil. Peroxide values of all oil samples after the storage period is higher than they were before the storage period. It is also clear from the data that the peroxide value increases with increasing radiation durations; this peroxide value increase can be due to the bond cleavage of sesame oil. Peroxide values for all oil samples studied showed levels value lower than the (20.0 meq O_2/kg) limitation in the standard for sesame oil. The storage of irradiated lipid food, in the

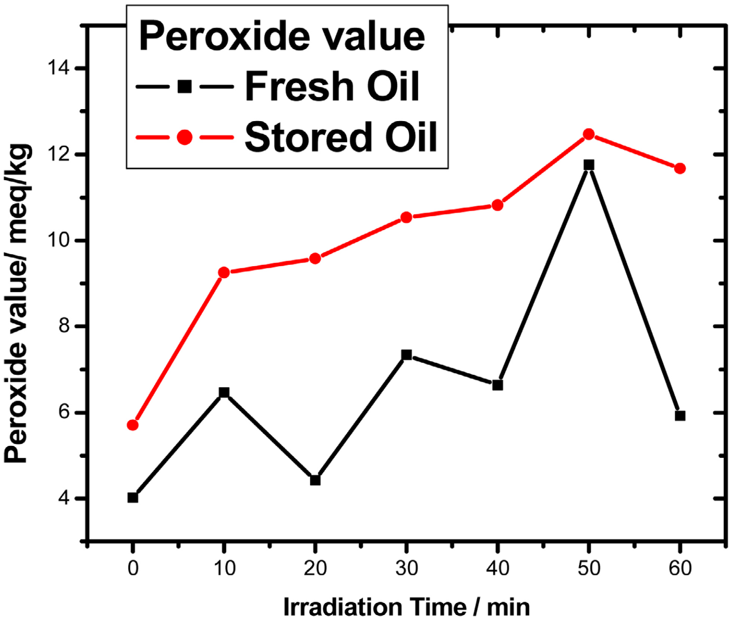


Figure 3. Effect of green laser irradiation time on the peroxide value of fresh and stored sesame oil.

presence of oxygen, accelerates the autoxidation [32]. It has been proved that irradiation accelerates the oxidation of unsaturated fatty acids and cholesterol during storage of foods [33] [34]. The chemical changes leads to physical changes as reported by Ku, *et al.*

3.1.2. Results of the Physical Properties

The chemical changes of the sesame oil samples due to laser radiation processing and during the storage time lead to some physical changes.

The effect of increasing laser irradiation time on the refractive index of sesame oil before and after storage period is presented in **Figure 4**.

Refractive index is an important optical parameter to analyze the light rays traversing through materials medium. Refractive index can be used as a quality control technique to identify the adulteration of edible oils. From the obtained results it is found that laser irradiation slightly affect the refractive index of the fresh sesame oil samples in the range from 1.4684 to 1.4784. While, it is found that, refractive index of the stored sesame oil samples higher than it before storing ranged from 1.4773 to 1.4898, and most of them excesses the recommended codex standard of 1.469 - 1.479 for sesame oil.

The initial density of the fresh sesame oil was 0.9173 cm^{-1}; when it irradiated by laser, it ranged between 0.9188 and 0.9157 cm^{-1}, it fall in the recommended codex standard of 0.915 - 0.924 cm^{-1} for sesame oil. While after the storage period it increases and ranged from 0.9182 to 0.9272 cm^{-1}, with two samples slightly excesses the recommended codex standard, see **Figure 5**.

Figure 6 illustrates the effect of increasing 532 nm laser irradiation time on the sesame oil viscosity before and after storage. The viscosity of the control

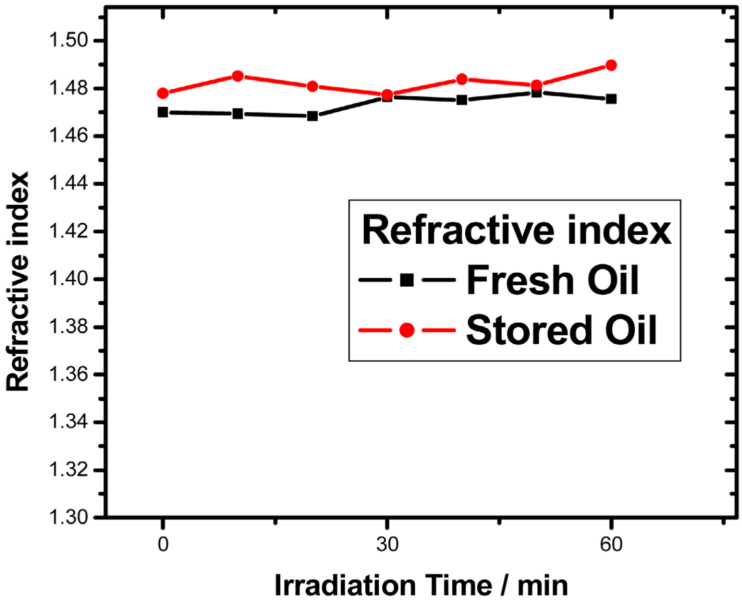


Figure 4. Effect of green laser irradiation time on the refractive index of fresh and stored sesame oil.

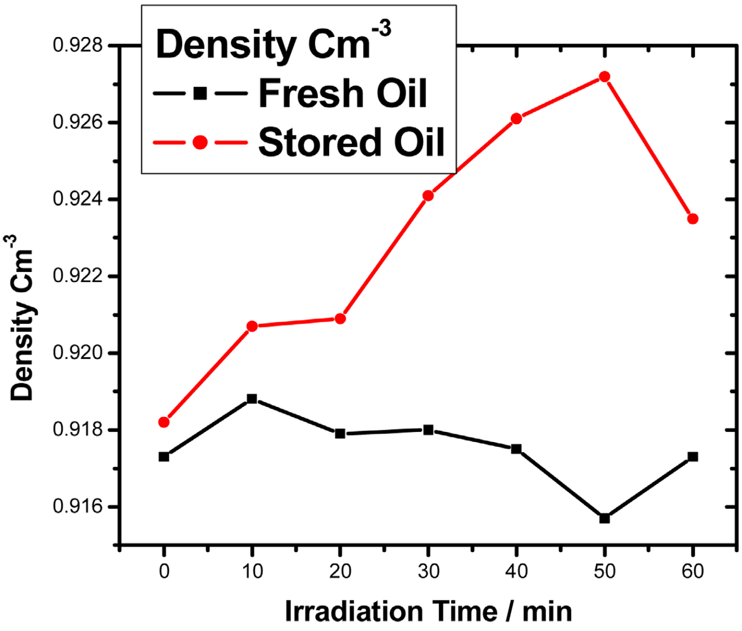


Figure 5. Effect of green laser irradiation time on the density of fresh and stored sesame oil.

sample was 61.16 cP, after the stored period it increased to 61.32 cP. Meanwhile, the viscosity of the irradiated samples increased to range 61.87 to 61.47 cP. After the stored period it observed that there is a gradually increase in the viscosity with the increase of the irradiation time of all samples from 61.32 to 62.25 cP.

Viscosity is related to the chemical properties of the oils. The increase in viscosity of emulsions when irradiated can be explained by unavoidable evaporative loss of volatile contents of oil. Formation of smaller oil micelles during irradiation not only has physical significance in terms of stability but also has effects on viscosity. Smaller micelles promote viscous emulsions due to more particle-particle interactions as a result of a larger interfacial area [35].

The effect of increasing 532 nm laser irradiation time on the sesame oil moisture, before and after the storage period is shown in Figure 7. It is clear from the data that the moisture of the sesame oil increases with increasing irradiation

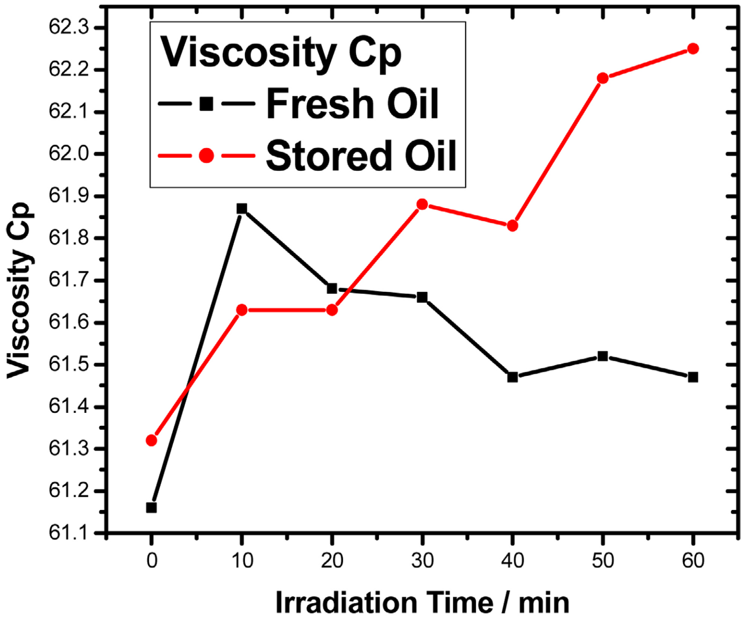


Figure 6. Effect of green laser irradiation time on the viscosity of fresh and stored sesame oil.

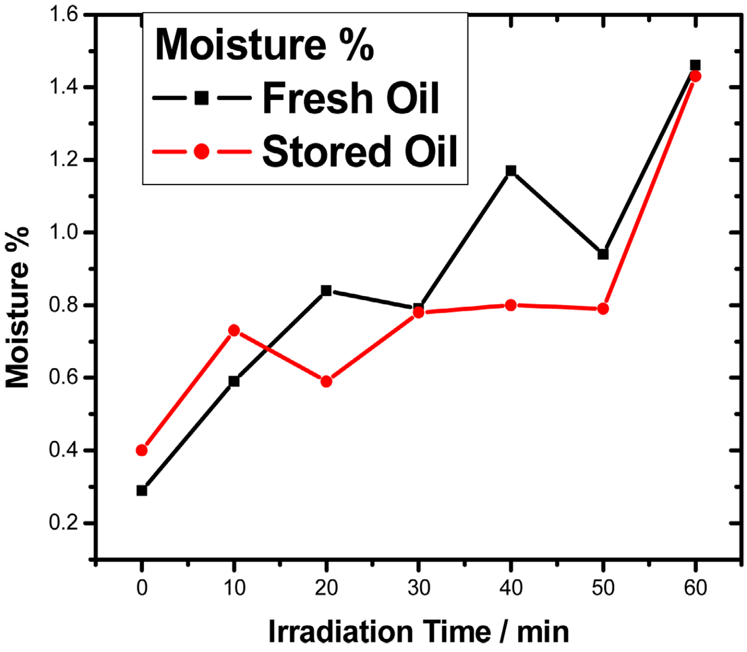
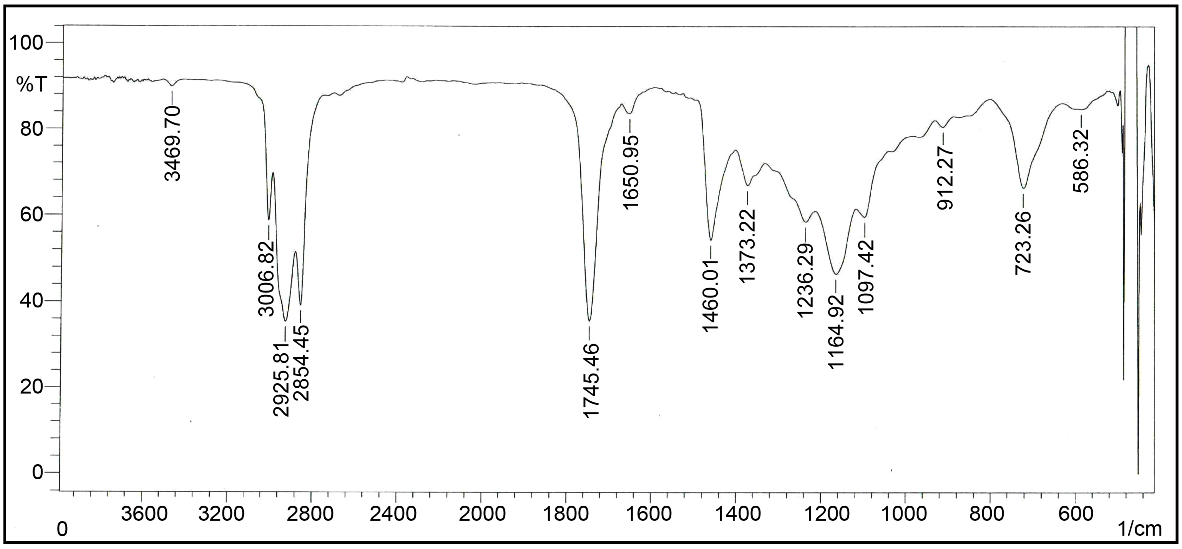


Figure 7. Effect of green laser irradiation time on moisture of fresh and stored sesame oil.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20
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3006.82
2925.81
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1236.29
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1097.42
912.27
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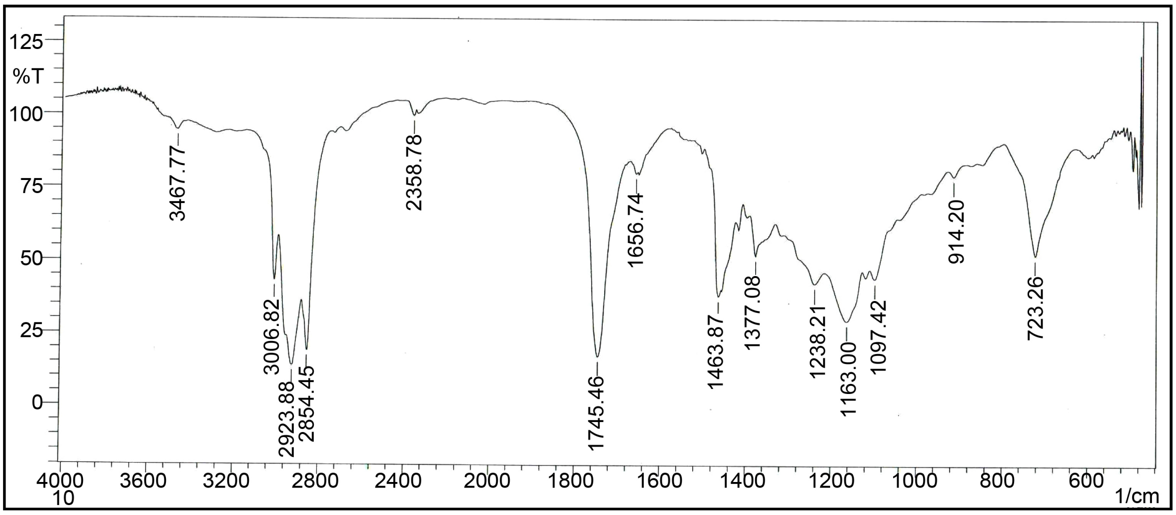
(a)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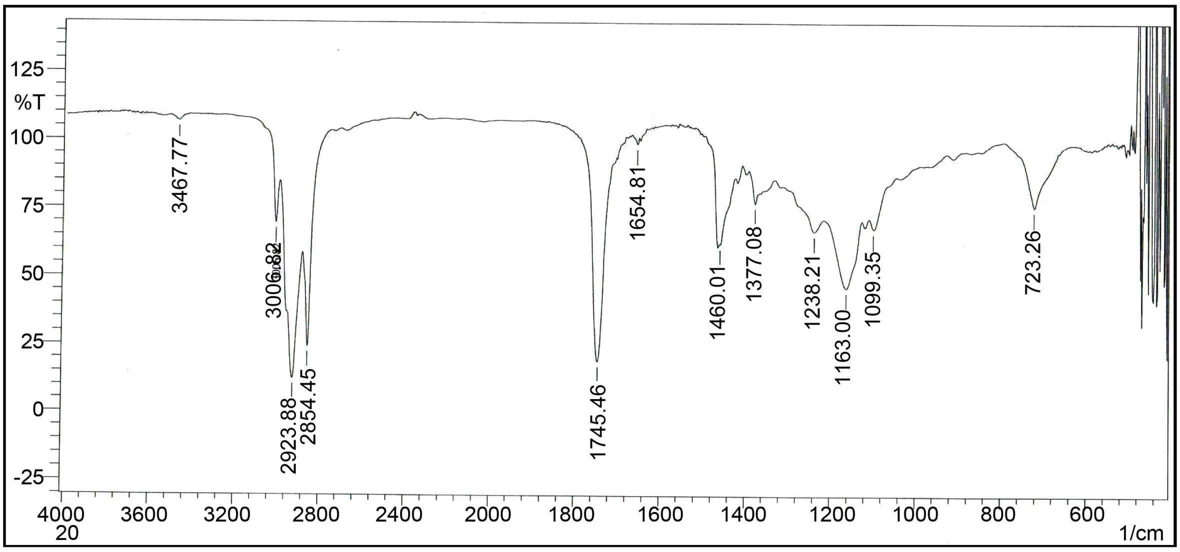
(b)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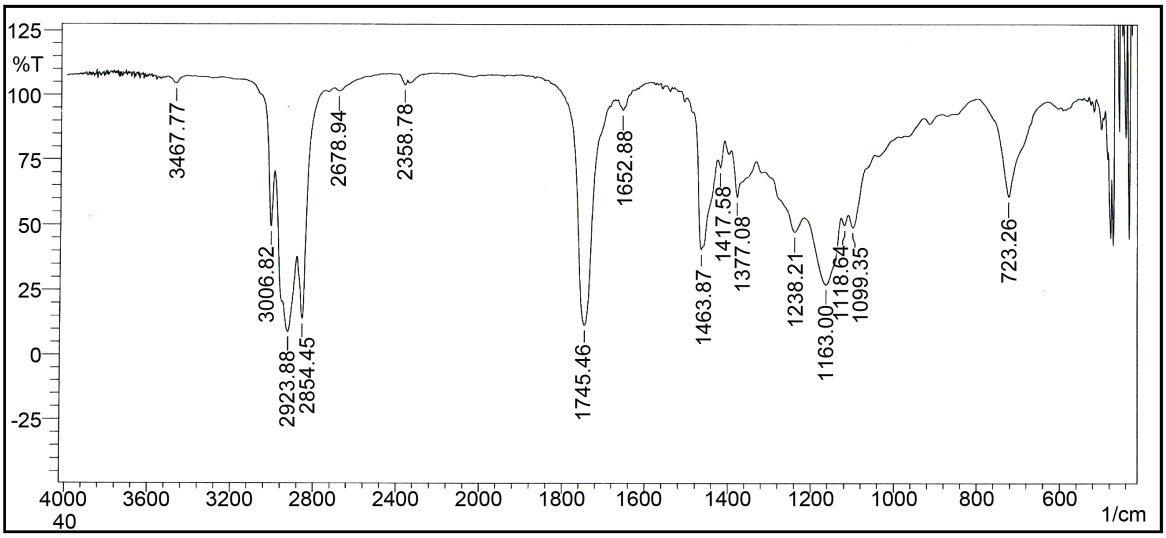
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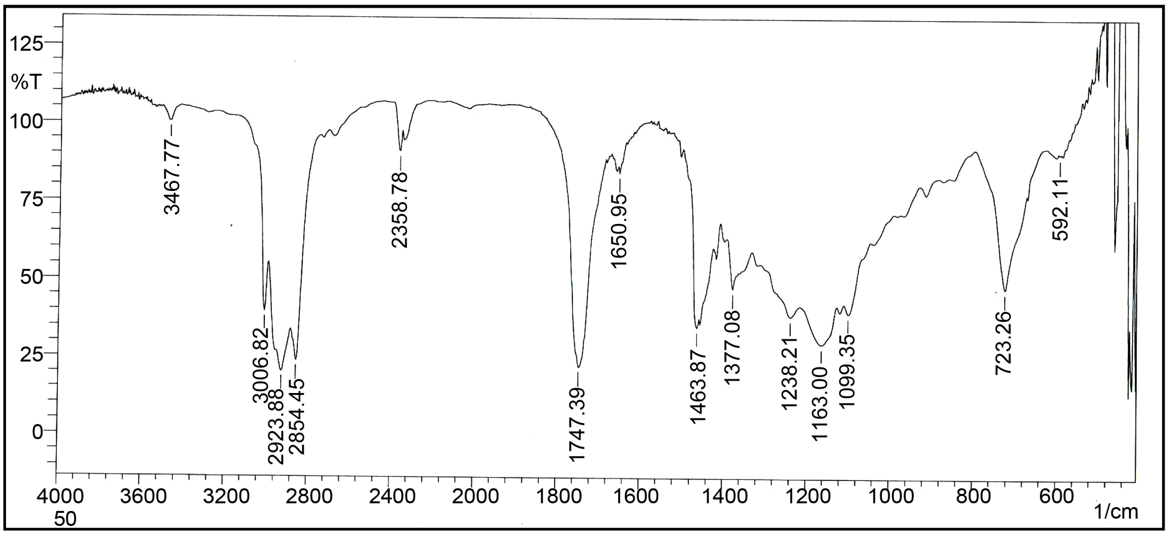
(d)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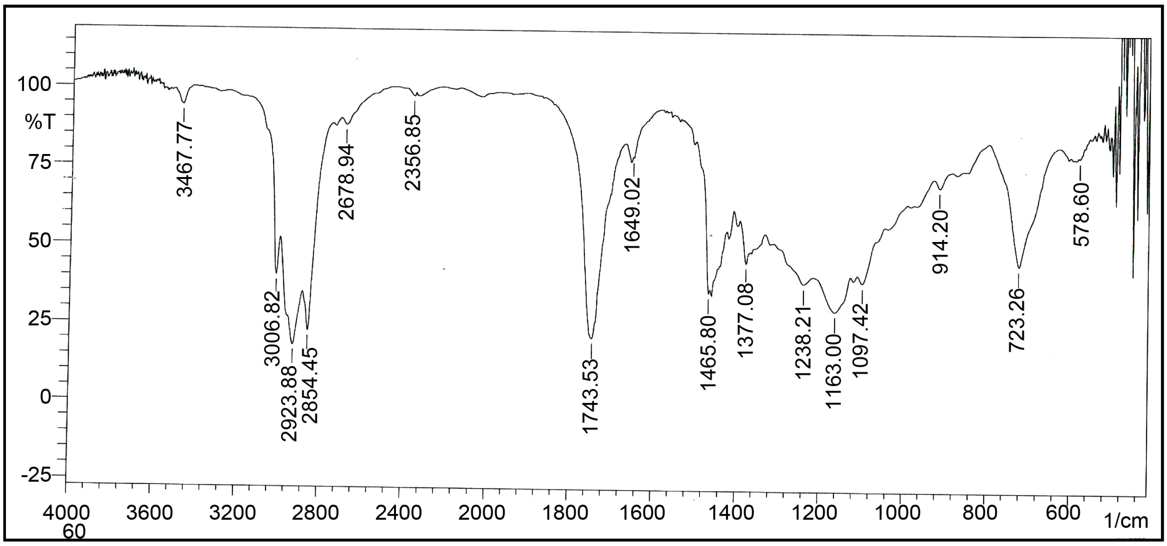
(e)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723.26
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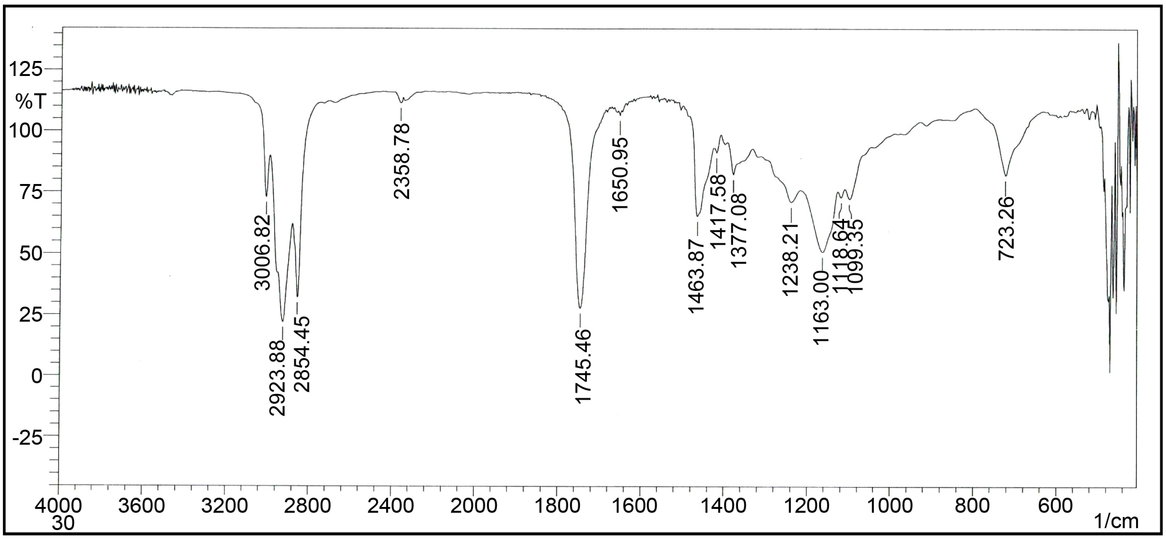
(f)



(g)

Figure 8. FT-IR spectrum of the irradiated sesame oil samples: (a) control (00 minutes); (b) 10 minutes; (c) 20 minutes; (d) 30 minutes; (e) 40 minutes; (f) 50 minutes; and (g) 60 minutes.

time before and after storage. The moisture of the control sample was 0.29%, which increased gradually to 1.46% with increasing the irradiation time. Likewise, after the storage period the moisture increased gradually from 0.40% in the control sample up to 1.43% with increasing the irradiation time.

3.2. FIR Results

FT-IR spectroscopy is a very good technique for analysis, as the intensities of the bands in the spectrum are proportional to concentration. FT-IR spectra of the control and laser irradiated samples after the storage period are presented in Figure 8.

It showed that there exists a notable difference in the bands between the control and laser irradiated samples appeared during the storage period. The spectra of the stored irradiated oil samples containing several absorption bands, that are absent from the spectrum of the control sample. The first new band is occurs in 2688.94 cm^{-1} shift in the spectra of the high-irradiated samples (40, 50, and 60 minutes). The other new band is appears at 2358.78 cm^{-1}, 11417.58 cm^{-1}, and 1118.64 cm^{-1}.

4. Conclusion

In this paper, the influence of green laser (532 nm) radiation on some physicochemical properties of sesame oil before and after storage period of 15 consecutive days was investigated. It was found that the green laser is capable to make changes in the physicochemical properties of sesame oil different from that occur by the visible light. Laser irradiation leads to photochemical interactions that change the chemical and physical properties of the oil. Moreover, storing to period of two weeks also caused changes in the physicochemical properties of sesame oil. All changes due to laser irradiation fall in the recommended codex standard (2001). Fourier transform infrared spectroscopy results confirm the physicochemical changes.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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Evaluation of Laser-Heating and Laser-Reheating of Sunflower (*Helianthus annuus*) Seed Oil Quality

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Abstract

This paper focuses on the difference between the effect of heating and reheating on sunflower oil properties by using carbon dioxide laser and electrical heater. Two samples of sunflower oil were heated and reheated with carbon dioxide laser beaming up to 50˚C, which takes 30 minutes; another two samples were heated and reheated up to 250˚C using electrical heater in two minutes, along with an unheated control sample at ambient conditions. Chemical properties like acid value, free fatty acids, peroxide value, saponification value and ester value beside physical properties such as moisture, density, viscosity, refractive index and color measurements of sunflower oil were studied. Fourier transform infrared spectroscopy was used to differentiate between the chemical changes in the samples. The results demonstrate that when the same cooking oil is reheated, the chemical reactions enhance foaming, darkening of oil color, increased viscosity, and off-flavor. Hence, repeated heating of the oil can lead to degradation of the cooking oil, both chemically and physically. It was found that the long time of heating using laser rising temperature up to 50˚C catalyzed chemical reactions that resulted in effects in the oil samples characteristics greater than the effects of the electrical heater in a few minutes with temperature 250˚C.

Subject Areas

Composite Material, Nanometer Materials

Keywords

Food Irradiation, Laser-Heating, Laser-Reheating, Physicochemical Characteristics, Quality Constants, Recycled oils, Thermal Oxidation

1. Introduction

Laser heating has been recognized since the 1970s, focusing on the CO_2 laser. Utilizing CO_2 laser allows non-metallic materials to be heated directly. CO_2 laser with a wavelength of 10.6 μm equates to the photon energy of 943 cm^{-1}, which is of the same order as lattice phonons in covalent crystals and is absorbed by existing lattice vibrations. This absorption technique also provides the advantage of more uniform heating for the materials that are generally transparent to the wavelength of 10.6 μm, meaning that heating is provided via this process throughout the entire thickness of the sample, contrarily in metallic materials where the laser beam is absorbed primarily at the surface [1].

Several studies were done in different applications of the laser heating techniques such as in thermoluminescence imaging of spatial dose distributions of ionizing radiation and the measurement of two-dimensional temperature distributions in high-power laser dielectric coating [2]. Some researchers studied laser-heating in the combustion of agricultural wastes [3] [4] [5]. Other researchers used it in enhancing the ceramic mechanical properties [6] [7]. Numerous studies were done by using lasers in food irradiations such as milk pasteurization [8] [9], production of yogurt [10], oil irradiation [11] and irradiation of bee honey [12]. Very promising results for applications involving non-destructive detection and classification of materials were obtained by Kassu *et al.* 2021 [13].

Edible oil is a vital component of our daily diet. It provides energy and essential fatty acids, and serves as a carrier of fat-soluble vitamins. Edible oils such as sunflower (*Helianthus annuus*) oil are continuously used for deep-fat frying. Usage of the same frying oil repeatedly is a common practice in the household or the restaurants to save costs. The methods used in assessing the quality of cooking oil and discarding it are observing foam, color turning dark or when it emits a bad odor [14]. Chemical reactions such as thermal polymerization, hydrolysis and oxidation occur when cooking oil is heated during the deep-frying process [15]. Using reheated edible oil is unhealthy. Edible oil is subjected to high temperatures for long periods in the process of frying food; this practice yields lipid peroxidation products, which is harmful to human health [16].

In this work, we tried to determine the effect of carbon dioxide laser (10,600 nm) heating and reheating on the chemical and physical characteristics of sunflower oil compared with the effect of electrical heater heating and reheating, in order to determine the most healthy heating method for cooking.

2. Materials and Methods

2.1. Material

Sunflower oil was obtained from the local market in Khartoum, Sudan. Five samples of sunflower oil were taken with amount of 50 ml for each sample. Then the following processes were done.

2.2. Heating Process

One of the five sunflower oil samples was heated to 250˚C using electrical heater.

The second sample was heated twice to the same degree (250˚C) using electrical heater; it was left to cools to the room temperature then reheated; it takes two minutes in heating and in reheating. The third sample was heated to 50˚C using laser irradiation. The fourth sample was heated twice to the same degree (50˚C) using laser irradiation; it was left to cools to the room temperature then reheated. The irradiation process was done using carbon dioxide laser (CO_2) (Model IB-601B, Beijing Innobri Technology, China) with wavelength 10,600 nm and output power 30 Watt, with duration time of exposure equal to 30 minutes. The sunflower oil sample was placed in open beaker with a capacity of 100 ml on magnetic stirring at room temperature. The distance between the oil and the end of the laser was 1 cm. The fifth one was left to be control sample.

2.3. Physicochemical Properties Characterization

Physicochemical properties such as acid value, ester value, free fatty acids, peroxide value, density, refractive index, viscosity, and moisture of the five sunflower oil samples were characterized according to the methods described in the A.O.A.C. 1990 [17]; All tests were performed in triplicate.

2.4. Determination of Oil Color

Color measurement of the oil samples was determined using a Lovibond Tintometer (Model E AF900 (The Tintometer Ltd.)) as units of red, yellow and blue according to the AOAC method [17]. Each visual measurement of every sample was taken in triplicate.

2.5. FT-IR Characterization

FT-IR spectra of the five sunflower oil samples were carried out using a Fourier Transform Infra-Red Spectrometer (Shimadzu, Japan). It is used to compare the chemical structure of the different five sunflower oils.

3. Results and Dissections

Due to repeated heating, the quality, color, smell and taste of edible oil changes due to the formation of polymers and polar compounds [18].

In this study; the effects of heating and reheating of the sunflower oil samples using electrical heater and carbon dioxide laser were investigated for the changes in the physicochemical properties like acid value, ester value, free fatty acids, peroxide value, density, refractive index, viscosity, and moisture content. The results of these properties were compared with the unheated sample.

3.1. Physicochemical Properties

The effects of heating and reheating on the physical and chemical characteristics of sunflower oil using laser and electrical heater compared with the unheated sunflower oil are presented in **Tables 1-3**.

Table 1. Values of the chemical properties of the five sunflower oil samples.

Sample		Acid Value	FFA	Peroxide Value	Saponification Value	Ester Value
Control		0.56	0.30	3.98	186.01	184.10
Electrical Heater	heated once	0.75	0.37	6.41	186.09	184.82
	heated twice	0.84	0.46	8.37	187.08	185.30
Laser-Heating	heated once	1.27	0.65	9.37	187.43	186.97
	heated twice	1.52	0.76	10.43	189.12	186.58

Table 2. Color measurements of the five sunflower oil samples.

Oil Sample		B	R	Y
Control		0	1.2	10.2
Electrical heater	heated once	0	1.6	10.6
	heated twice	0	1.8	11.5
Laser-heating	heated once	0.1	2.3	11.7
	heated twice	0.2	2.5	11.9

Table 3. The other physical properties of the five sunflower oil samples.

Sample		Moisture %	Density cm^3	Viscosity Cp	Refractive Index
Control		0.34	0.9150	60.58	1.4698
Electrical Heater	heated once	0.46	0.9178	60.83	1.4767
	heated twice	0.63	0.9173	60.85	1.4786
Laser-Heating	heated once	0.79	0.9176	61.06	1.4794
	heated twice	0.91	0.9176	61.47	1.4866

3.1.1. Results of the Chemical Properties

The obtained results of heating processes of the sunflower oil in Table 1 showed increasing in the oil chemical properties. Reheating processes caused in more increasing in the oil chemical properties; these results agree with the results of Adriana Abdul Aziz1 *et al.* [19]. It was observed that laser-heating process increased the oil chemical properties greater than the increasing of reheating processes by electrical heater.

This obtained results showed that the trend of chemical properties was increased crossover all tested samples starting from the once-heated electrically sample undergoing with the twice-heated electrically then once-laser-heated undergoing with twice-laser-heated sample. Chemical properties had increased with the increasing number of heating sessions. The increment of chemical properties of the oils is due to the development of hydroperoxides of unsaturated fatty acids because of the lipid oxidation process. Oxidation of oils is influenced by means of different factors which include the degree of unsaturation, heat, light, oil processing, antioxidants and transition metals [19].

3.1.2. Results of the Physical Properties

The chemical reactions in the oil leads to changing in some physical properties such as viscosity, density, darken color, or production of foam [20]; these changes clearly showed in this study in the results presented in **Table 2** and **Table 3**.

Table 2 showed the changing of the oil color for the control, heated, and reheated samples. The blue color does not appear by heating and reheating using the electrical heater; while it was increased from 0 up to 0.2. The red color increased from 1.2 up to 1.8 by heating and reheating using the electrical heater; while it was increased from 1.2 up to 2.5. The yellow color increased from 10.2 up to 11.5 by heating and reheating using the electrical heater; while it was increased from 10.2 up to 11.9. It was observed that oil has darkened its color and emanated smoke during heating sessions, which indicates oil degradation. Chemical reactions, which influenced by heating processes; results in foaming production, darkening of oil color, and off-flavor. Increase in color of oil by heating was reported by Sonia and Badereldeen [21], and was found also by Augustin *et al.* [22] and Mudawi *et al.* [23].

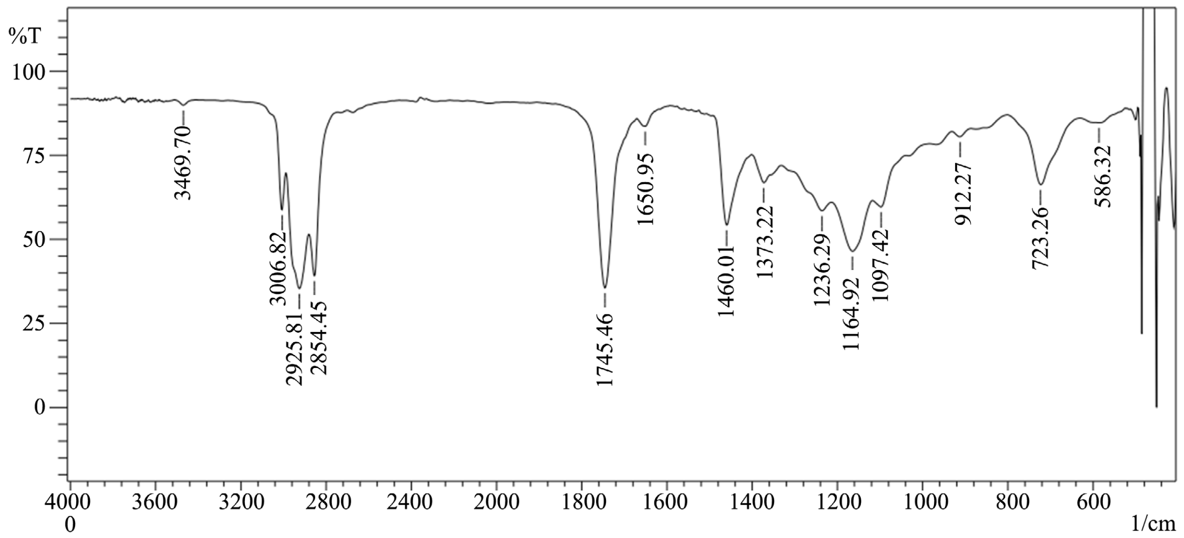
Table 3 shows changes in moisture, density, viscosity and refractive index (RI) of all oil samples during heating and reheating. Moisture increased from 0.34% in control sample to 0.63% after reheating process using electrical heater, while it increased up to 0.91% after reheating process using laser. The density increased from 0.9150 cm^3 in control sample to 0.9178 cm^3 after heating and it decreased to 0.9173 cm^3 after reheating process using electrical heater, while it increased up to the same value 0.9176 cm^3 after heating and reheating process using laser. While viscosity increased from 60.58 Cp in control sample up to 60.85 Cp after reheating process using electrical heater, whereas it increased up to 61.47 Cp after reheating process using laser. The refractive index increased from 1.4698 in control sample to 1.4786 after reheating process using electrical heater, while it increased up to 1.4866 after reheating process using laser. It is clear that all physical properties of the five sunflower oil samples increased by heating and reheating at 50˚C. Physical properties temperature dependence is agree with the study of Wiege *et al.* in 2020 [24].

3.2. FTIR Results

FT-IR spectroscopy is a very good technique for analysis, as the intensities of the bands in the spectrum are proportional to concentration. FT-IR spectra of the control, heated and reheated sunflower oil samples are presented in **Figure 1**.

Each sample contains C=O (1745.46 cm^{-1}) and C-O (1163.00 cm^{-1}) which may indicate possible existence of COO, ester functionality (oils), samples were rich in C-H (sp^3) (2923.88 cm^{-1} and 2854.45 cm^{-1}) indicating presence of CH_3 and CH_2 as reinforced by bands in (1463.87 cm^{-1} and 1377.08 cm^{-1}). Weak band at 1650 cm^{-1} C=C with C=C-H 3006 cm^{-1} but not conjugated with C=O.

As shown in **Table 4**, the changed band assignments of sunflower oil samples were: 3469.70 cm^{-1}, 2677.01 cm^{-1} 2731.02 cm^{-1}, 2360.71 cm^{-1}, 1654.81 cm^{-1} and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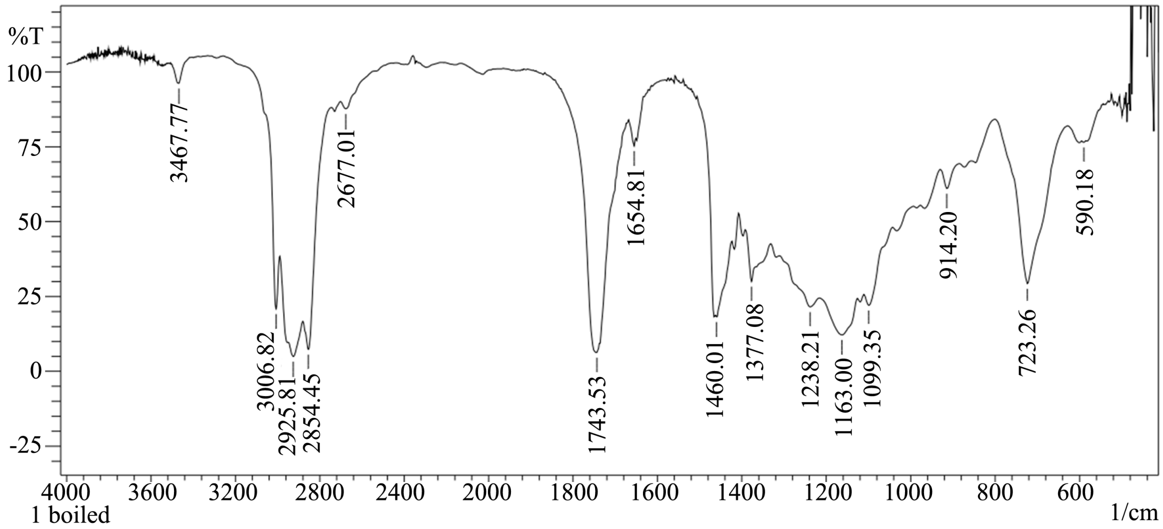
(a)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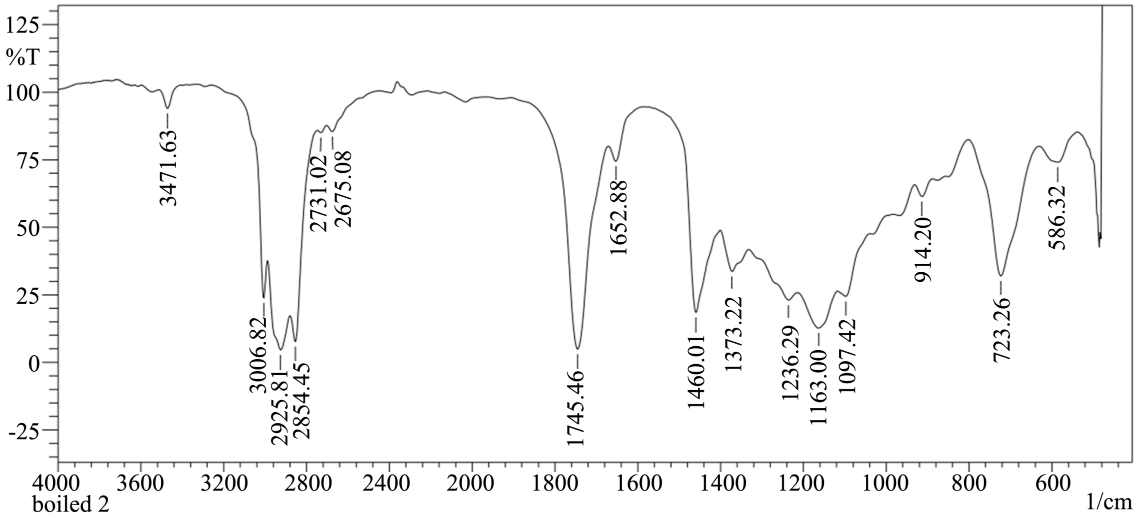
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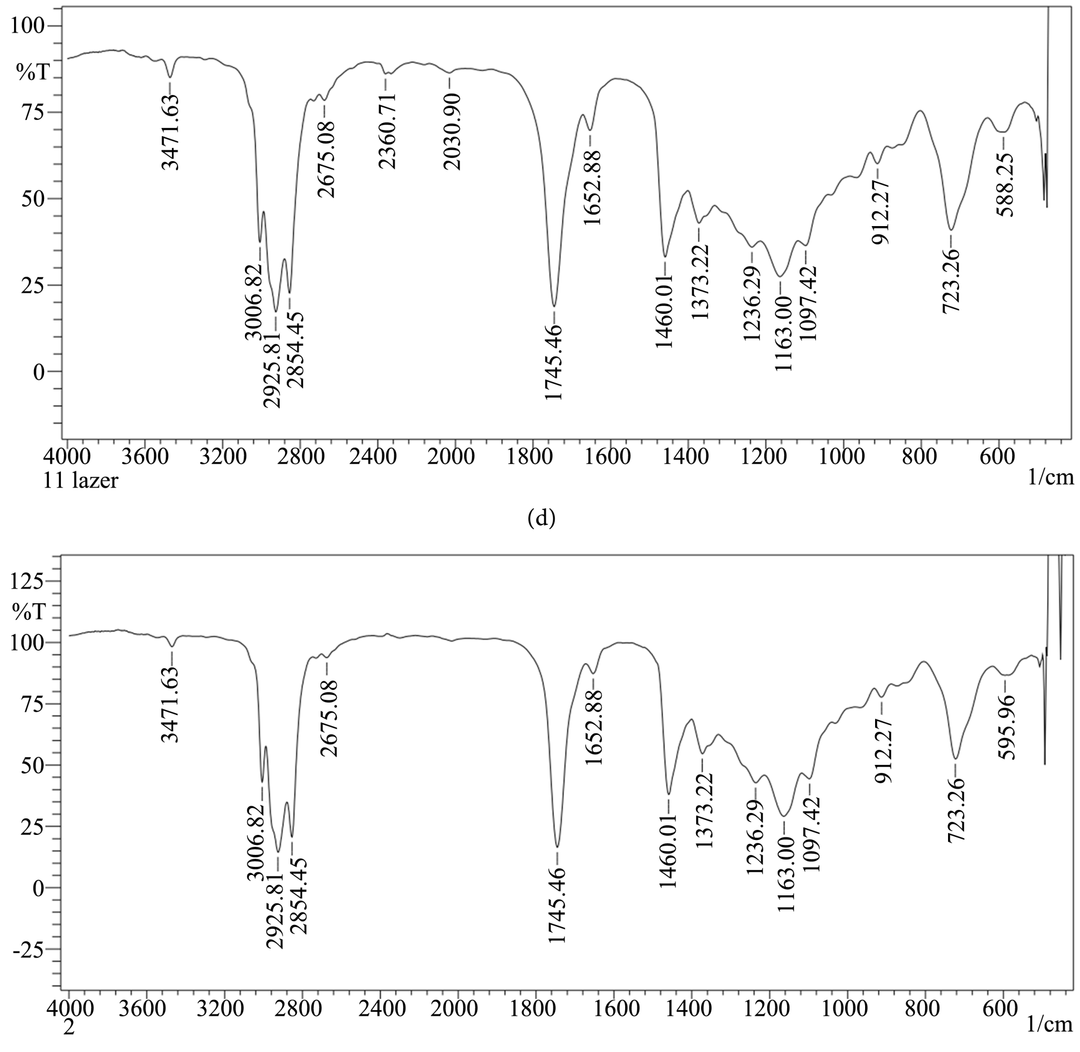

(d)

(e)

Figure 1. FT-IR spectrum of the sunflower oil samples: (a) control (unheated); (b) electrical heated once; (c) electrical heated twice; (d) laser heated once; (e) laser heated twice.

Table 4. Band assignments of sunflower oil samples.

FTIR shift/cm^{-1}	Assign	Ref.
3469.70	O-H str	Sudhakar *et al.*, 2018 [25]
3006.82	C-H st.vib. (sp^2)	Poiana, *et al.*, 2015 [26]
2923.88 - 2854.45	C-H st.vib. (sp^3)	Liu, and Kazarian, 2022 [27]
2731.02	Aldehydic C-H Str.	Hafeez, *et al.*, 2019 [28]
2360.71	C-H	Evangelin and Gurulakshmi, 2020 [29]
1745.46	C=O st.vib.	Zhuang, 2020 [30]
1654.81	C=O Str.	Gupta, *et al.*, 2011 [31]
1650	C=C st.vib.	Panicker, *et al.*, 2009 [32]
1463.87, 1377.08	CH_3, CH_2 bend.	Priest, *et al.*, 1999 [33]
1163.00	C-O st.vib.	Simonova, and Karamancheva, 2013 [34]

585 - 595 cm^{-1}. It showed a notable difference depend on heating and reheating processes using laser and electrical heater.

4. Conclusions

In this paper, the effect of carbon dioxide laser (10,600 nm) heating and reheating on the chemical and physical characteristics of sunflower oil compared with the effect of electrical heater heating and reheating were investigated. As a result, when the same cooking oil is reheated, the chemical reactions enhance foaming, darkening of oil color, increased viscosity, and off-flavor. Hence, repeated heating of the oil can lead to degradation of the cooking oil, both chemically and physically. It was found that the long time of heating using laser rising temperature up to 50˚C catalyzed chemical reactions that resulted in effects in the oil samples characteristics greater than the effects of the electrical heater in a few minutes with temperature 250˚C.

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Conflicts of Interest

The authors declare no conflicts of interest.

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