EXTRACTION, CHARACTERIZATION AND THERMAL STABILITY STUDIES WITH TIGERNUT OIL

BY

OMOLUWA MERCY UFOMA

PSC1808710

PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMISTRY

IN PARTIAL FULFILMENT OF THE AWARD OF THE DEGREE OF BACHELOR OF SCIENCE (B.Sc.) HONOURS IN CHEMISTRY, FACULTY OF PHYSICAL SCIENCES, UNIVERSITY OF BENIN, BENIN CITY, EDO STATE, NIGERIA.

OCTOBER, 2023

CERTIFICATION

This is to certify that this project work with topic- **Extraction, characterization and thermal stability with Tigernut oil** was carried out by **OMOLUWA MERCY UFOMA** with matriculation number **PSC1808710** of the Department of Chemistry, Faculty of Physical Sciences, University of Benin, Benin City.

Omoluwa Mercy Ufoma	Date
(Student)	
Dr E.N. Dibie	Date
(Supervisor)	
Prof J.U. Iyasele	Date
(Head of Department)	

DEDICATION

This work is dedicated to God Almighty who has been my guide, support, help since the beginning of this journey even up till this moment. To my irreplaceable family who have been encouraging and supporting me in all areas and to my amazing friends who have contributed in one way or the other to the success of this work, I also dedicate this work.

ACKNOWLEDGEMENT

This work would not have been easy without the contributions of some wonderful individuals therefore I want to use this opportunity to applaud and appreciate everyone who in little or massive way acted as a contributor to the success of this work.

To my Supervisor, Dr E.N. Dibie, I want to say a very big "THANK YOU" for his encouragements, advices, selfless contributions, and for always making out time out of his busy schedule to listen to my complaints and correct my mistakes. I am extremely grateful for his patience in guiding and offering me helpful suggestions and I pray for God's blessings upon his life and that of his family. I wish to also express my profound gratitude to the HOD of the department- Prof. J.U. Iyasele, my course adviser- Dr. O.K. Ogbeide and to all lecturers in the department for guiding me through this journey.

I am particularly indebted to my friends and course mates Possible Esekhomo, Christabel Udezi, Michael Omogiate, Prudent Osifo and victory Abusomwan because without them I don't know how I would have coped thus far academically, financially and even emotionally, they are the real MVPs. Also to Stephen Omale and Sammy who have been of great help academically, I owe them my gratitude. My heartfelt appreciation goes to my other friends who supported from afar Ayomide, Bolaji, Tolani, John and Joy, they all never got tired of me. My roommates as well have been so thoughtful and helpful and for that I'm really grateful to them and to everyone whose names I couldn't mention.

This acknowledgement isn't complete without me appreciating my family: my father, siblings and most especially my mother Mrs Helen Omoluwa who has been my backbone supporting financially, spiritually and rendering words of advice to me. My elder siblings Blessing and Samuel Omoluwa I'm really thankful for all they have done so far. I love you all.

ABSTRACT

This work entails extraction, characterization and thermal stability studies with tigernut (*Cyperus esculentus*) oil. The Tigernut used in this work was obtained from an open market in Benin city. Oil extraction was carried out by soxhlet extraction using n-hexane as a solvent. Parameters studied were free fatty acid (FFA) content giving 0.48% as result, peroxide value (PV) with results of 1.96meq/kg and 7.80meq/kg for the raw oil and treated oil respectively, refractive index (RI) test- 69% and 69.5% brix for both raw and treated oil respectively and measurement of absorbance of the raw and treated oil at 234nm and 270nm. Additionally, Fourier-transform infrared spectroscopy (FTIR) was used to identify the functional groups present in the oil. Findings indicate that the oil is thermally stable when treated with heat. Due to the highly nutritional properties of this oil, it has found wide usefulness in culinary arts, pharmaceutical products and even cosmetics.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER ONE

INTRODUCTION AND LITERATURE REVIEW

1.1 INTRODUCTION

1.1.1 BACKGROUND OF STUDY

Tigernuts, scientifically known as *Cyperus esculentus*, have a rich history spanning centuries and are esteemed for their versatility in culinary and dietary contexts. These small tubers, native to regions across Africa, Europe, and Asia, have long been a dietary staple, offering a valuable source of energy, dietary fiber, vitamins, and essential minerals. However, it is the unique oil content of tigernuts that has recently attracted considerable attention from researchers and food scientists alike.

Tigernut oil, often referred to as chufa or earth almond oil, stands out as a distinctive vegetable oil with remarkable qualities. It has garnered recognition not only for its potential health benefits but also for its versatile applications in various culinary and industrial domains. The extraction, characterization, and evaluation of the thermal stability of tigernut oil represent critical avenues of research essential for unlocking its full potential.

The composition of tigernut oil sets it apart from other vegetable oils. Recent research has highlighted its richness in oleic acid, a monounsaturated fatty acid known for its heart-healthy properties. Additionally, tigernut oil boasts linoleic acid, palmitic acid, and a complex array of fatty acids, making it a unique blend with promising health advantages (Martinez-Ferez *et al.*, 2003).

Efficient oil extraction methods are fundamental in realizing the potential of tigernut oil. Two primary techniques, cold pressing and solvent extraction are commonly employed in the industry. Cold pressing, characterized by minimal heat exposure, is favored for its ability to

preserve the oil's quality and nutritional value. Conversely, solvent extraction methods offer higher oil yields, albeit with potential impacts on oil quality if not carefully controlled (Oladejo *et al.,* 2018).

Characterization of tigernut oil is pivotal in comprehending its properties and potential applications. Gas chromatography-mass spectrometry (GC-MS) serves as a widely employed technique for analyzing the fatty acid composition of tigernut oil, providing valuable insights into its chemical makeup (Lozano-Milo *et al.,* 2017). Furthermore, Fourier-transform infrared spectroscopy (FTIR) can identify functional groups within the oil, enhancing our understanding of its unique characteristics (Akubugwo *et al.,* 2007). Examining the thermal stability of tigernut oil, including its behavior at elevated temperatures, is crucial for determining its suitability in cooking and industrial processes (Sado Kamdem *et al.,* 2002).

This study embarks on a comprehensive exploration of tigernut oil by addressing key aspects such as extraction methods, characterization techniques, and thermal stability analysis. In doing so, it aims to contribute to the expanding body of knowledge surrounding this distinctive vegetable oil, shedding light on its potential benefits, applications, and limitations. Employing a combination of analytical techniques and rigorous experimentation, this research endeavors to reveal the multifaceted nature of tigernut oil and its significance in contemporary food and industrial contexts.

1.1.2 STATEMENT OF PROBLEM

The exploration of tigernut oil, spanning its extraction, characterization, and thermal stability, is becoming increasingly significant due to the rising recognition of tigernuts as a nutritionally valuable source and the burgeoning interest in their oil's versatility within culinary and industrial domains. However, beneath the surface of this burgeoning interest,

several multifaceted challenges and noteworthy knowledge gaps persist, underscoring the need for comprehensive research and investigation in this domain.

Primarily, while tigernut oil is acclaimed for its distinctive fatty acid composition, the full spectrum of its chemical makeup and the intricate implications of this composition on its nutritional and functional attributes remain a subject that requires deeper scrutiny (Martinez-Ferez *et al.,* 2003). While extant literature has offered valuable insights, it has not yet provided a holistic understanding of the intricate interplay among the oil's constituents and their collective impact.

Secondly, the pivotal choice of extraction method carries far-reaching consequences for tigernut oil production, impacting not only the yield but also the quality and sustainability of the process. Cold pressing, characterized by its ability to maintain oil quality due to minimal heat exposure, is favored in this regard. However, it may not always be the most efficient in terms of yield. Conversely, solvent extraction methods promise higher yields but introduce potential risks to oil quality if not meticulously controlled (Oladejo *et al.,* 2018). Striking the delicate balance required for optimizing the extraction process continues to present a challenge. Furthermore, it is imperative to assess the thermal stability of tigernut oil to gauge its suitability for diverse culinary and industrial applications (Sado-Kamdem *et al.,* 2002). Nonetheless, there remains a palpable dearth of research exploring how tigernut oil performs under elevated temperature conditions and how its thermal stability compares with that of other commonly employed vegetable oils.

In light of these intricacies and the burgeoning enthusiasm surrounding tigernut oil, the present study endeavors to systematically address these knowledge gaps and significantly contribute to a more profound comprehension of tigernut oil extraction, comprehensive characterization, and thermal stability analysis. By doing so, this research aims to provide

valuable insights into the optimization of tigernut oil production processes, thereby enhancing its applicability across a spectrum of culinary and industrial contexts.

1.1.3 SIGNIFICANCE AND JUSTIFICATION OF THE WORK

The comprehensive exploration of tigernut oil, encompassing its extraction, characterization, and thermal stability, holds substantial significance in various aspects, ranging from health and nutrition to culinary arts and industrial applications. This section elucidates the profound importance of this research endeavor and justifies the allocation of resources and effort toward it, drawing upon relevant references.

Tigernuts are renowned for their nutritional richness, with potential health benefits attributed to their consumption. Tigernut oil, being a prominent constituent of tigernuts, is of particular interest due to its unique fatty acid composition. It is rich in oleic acid, a monounsaturated fatty acid with recognized cardiovascular benefits (Martinez-Ferez *et al.,* 2003). By comprehensively characterizing tigernut oil, this research can provide valuable insights into its nutritional attributes and potential health advantages, contributing to a better understanding of its role in human nutrition.

Understanding the thermal stability of tigernut oil is crucial for its effective utilization in culinary applications. The smoke point and oxidation stability of cooking oils determine their suitability for high-temperature cooking methods. By assessing the thermal stability of tigernut oil, chefs, and food processors can make informed decisions about its use in various cooking techniques, enhancing the quality and healthfulness of prepared dishes. Comparative studies with other commonly used oils can offer insights into its culinary advantages (Sado-Kamdem *et al.,* 2002).

Tigernut oil's unique properties make it a compelling candidate for various food processing applications. The food industry can benefit from the optimization of extraction methods to ensure efficient and sustainable tigernut oil production. Moreover, the oil's chemical characterization can aid in the development of innovative food products with improved nutritional profiles. For example, tigernut oil could be incorporated into functional foods with enhanced health benefits (Oladejo *et al.,* 2018).

Tigernuts are a valuable crop with the potential for economic growth in agriculture. Understanding the best practices for tigernut oil extraction can lead to increased yields and quality, benefiting farmers and agricultural industries. Moreover, the promotion of sustainable agricultural practices through tigernut cultivation can have positive environmental impacts by enhancing soil health and reducing the need for chemical inputs (Oladejo *et al.,* 2018). In the context of global food security and the increasing demand for alternative food sources, tigernuts have garnered attention as a drought-tolerant crop with the potential to thrive in diverse climates. Research on tigernut oil can contribute to the diversification of food sources and improve the adaptability of agriculture to changing environmental conditions, ultimately enhancing food security (Oladejo *et al.,* 2018).

1.1.4 SCOPE OF WORK

The scope of this research project encompasses a comprehensive exploration of tigernut oil, spanning its extraction methods, chemical characterization, and thermal stability assessment. It includes the optimization of extraction techniques and parameters, detailed chemical analysis, and functional group identification. The research also evaluates the thermal stability and culinary suitability of tigernut oil, comparing it with other vegetable oils, and explores its potential applications in nutrition, culinary arts, and various industries. Additionally, the study advocates for sustainable tigernut cultivation practices and assesses the economic and

ecological benefits. Overall, this research aims to provide valuable insights into tigernut oil, promoting its diverse uses while contributing to the broader understanding of its properties and applications.

1.1.5 AIM AND OBJECTIVES

AIM

The aim of this research is to extract, characterise and carry out thermal stability studies with tigernut oil.

OBJECTIVES

- To promote tiger nuts
- To Characterize Tigernut Oil
- To carry out thermal stability studies with the extracted oil

1.2 LITERATURE REVIEW

1.2.1 INTRODUCTION TO TIGERNUT AND TIGERNUT OIL

Tigernut, a member of the sedge family, is a versatile and perennial crop with a rich history. In Nigeria, it exists in three varieties—black, brown, and yellow—referred to as Aya in Hausa, Ofio in Yoruba, and Akiausa in Igbo. However, the yellow and brown cultivars are the most commonly available in the market (Oke *et al.,* 2017). These unassuming tubers have quietly held a treasure trove of nutritional benefits for centuries.

Tigernuts are a nutritional goldmine, containing high levels of soluble glucose and oleic acid, along with starch, fats, sugars, and proteins that are energy-rich. They are also abundant in essential micronutrients like phosphorus, potassium, calcium, and iron, crucial for bone

health, tissue repair, muscle function, and overall growth and development (Adejuyitan, 2011). Additionally, tigernuts boast high levels of vitamins C and E.

Tigernuts are available in various forms in Nigerian markets—fresh, semi-dried, and dried. They are consumed as snacks and used in culinary creations. However, their full nutritional potential and product diversity are often overlooked. Tigernuts can be transformed into various products, including tigernut flour, tigernut oil, and tigernut milk (Bamishaiye and Bamishaiye, 2011). As a snack, they can be roasted, dried, or baked. Additionally, tigernuts can be processed into a healthful beverage known as 'Kunnu Aya' in Northern Nigeria.

Tigernuts (Cyperus esculentus) are a versatile and globally significant crop cultivated in various countries (Sánchez-Zapata *et al.,* 2012). Despite their widespread presence, tigernuts have not received the attention they deserve in terms of research, which has limited their potential applications (Adejuyitan, 2011). A closer look at tigernuts reveals a treasure trove of nutrients and bioactive compounds, making them a promising subject for exploration.

Tigernuts exhibit a fascinating nutritional profile (Adel *et al.,* 2015). They typically contain 22.14–44.92% lipids, 3.28–8.45% proteins, 23.21–48.12% starch, 8.26–15.47% dietary fibers, and 1.60–2.60% ashes (Adel et al., 2015). These components contribute to their energy density and potential health benefits. Moreover, tigernuts are rich in bioactive compounds such as organic acids, alkaloids, and phenols (Nina *et al.,* 2019), further enhancing their nutritional value.

One of the most remarkable attributes of tigernuts is their high lipid content, primarily composed of monounsaturated fatty acids, which is reminiscent of the heart-healthy olive oil (Sánchez-Zapata *et al.,* 2012). This makes tigernut oil a valuable source of edible oils with potential applications in the food industry (Roselló-Soto *et al.,* 2018). Tigernuts are also abundant in starch, a renewable and cost-effective food ingredient (dos Santos Silveira Junior

and de Francisco, 2020). While their protein content is relatively modest, it is noteworthy for its suitability for individuals with diabetes or digestive issues and it's potential in preventing heart disease (Adejuyitan, 2011; Ogunlade *et al.,* 2015).

In addition to their macronutrient content, tigernuts are a rich source of dietary fiber, which has been proven effective in preventing colon cancer, combating obesity, and alleviating gastrointestinal disorders (Viuda-Martos *et al.,* 2010). Their antioxidant properties, attributed to flavonoids, position tigernuts as a natural source of antioxidants with potential health benefits (Jing *et al.,* 2015).

In summary, tigernuts are a nutritional powerhouse waiting to be fully explored. Their unique composition, including high lipids, starch, dietary fiber, and bioactive compounds, holds promise for various applications in the food industry and potential health benefits. Further research into tigernuts can unlock their full potential and pave the way for innovative culinary creations and enhanced well-being.

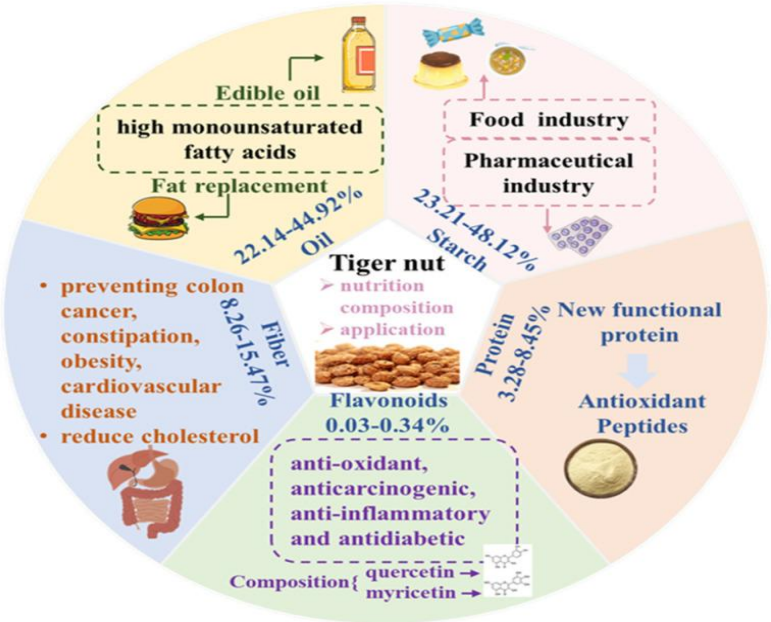


Figure 1: The main nutrition composition and nutrient-based applications of tiger nut (Yu *et al.*, 2022)

The nutritional richness of tigernuts and their derived products is a complex interplay influenced by numerous variables such as the type of tigernut variety, soil conditions, growth environment, farming methods, and how they are stored (Sánchez-Zapata *et al.,* 2012; *Nina et al.,* 2019; Roselló-Soto *et al.,* 2018).

The variety of tigernuts grown plays a pivotal role in shaping their nutritional content. A study conducted by Nina *et al.* (2019) compared black, brown, and yellow tigernut varieties, revealing distinct differences. For instance, the black tigernut had a higher level of crude fiber compared to the brown and yellow types, while the brown variety exhibited greater protein content compared to the black and yellow counterparts. These distinctions underscore the importance of selecting the most suitable tigernut variety based on specific nutritional objectives. Furthermore, the conditions in which tigernuts are cultivated, including environmental factors and agricultural practices, have a substantial impact on their growth and overall yield. Research by dos Santos Silveira Junior and de Francisco (2020) demonstrated that using a combination of wood biochar, cow dung biochar, and green manure can enhance soil fertility, resulting in improved tigernut growth and yield. This emphasizes the role of sustainable farming methods in enhancing the nutritional quality of tigernuts.

The way tigernuts are stored also plays a crucial role in determining their nutritional attributes. Refrigeration, for example, was found to increase the activity of α-amylase and lipase in tigernuts, whereas storing them under ambient or elevated conditions led to higher sugar content (Roselló-Soto *et al.,* 2018). These findings underscore the significance of appropriate storage practices to maintain the desired nutritional qualities of tigernuts.

Despite tigernuts having a long history as a crop, research into their nutritional composition, physicochemical properties, and product development lags behind that of other nut oil crops

like soybeans and peanuts. To address this disparity, it is imperative to stimulate further research in this field. The primary goal of this article is to ignite the curiosity of scientists by providing an overview of the current state of tigernut research and production.

While existing reviews predominantly concentrate on tigernut oil and milk processing, this article prioritizes the analysis of nutrients—a pivotal aspect for fully exploiting the potential of tigernuts. By summarizing the existing knowledge regarding the key nutrients found in tigernuts, their processing techniques, physicochemical attributes, functional properties, and industrial applications, this review aims to inspire comprehensive exploration and innovation in tigernut research.

In conclusion, tigernuts represent a versatile and nutritionally rich crop with untapped potential. Understanding the multitude of factors influencing their nutritional composition and applying this knowledge to research and product development can open up new horizons for harnessing the advantages of tigernuts across various industries.

1.2.2 TIGERNUT OIL

Tigernuts are a remarkable source of lipids, boasting a lipid content ranging from 22.14% to 44.92%. Interestingly, the lipid profile of tigernuts closely resembles that of olive oil, renowned for its health benefits (Ezeh *et al.,* 2014). This similarity underscores the potential of tigernut oil as a healthy dietary fat. In addition to lipids, tigernut oil contains essential vitamins like vitamin C and vitamin E, as well as an array of minerals such as potassium, calcium, and magnesium. Furthermore, phenolic compounds contribute to its antioxidant properties, giving tigernut oil exceptional resistance to oxidation compared to other vegetable oils (Nina *et al.,* 2020).

Beyond its nutritional value, tigernut oil is rich in bioactive compounds. Notably, it contains alkaloids, saponins, and tannins, which confer antibacterial and anti-inflammatory properties. These attributes position tigernut oil not only as a source of healthy fats but also as a potential natural remedy with therapeutic qualities (Vega-Moralesa *et al.,* 2019).

The sensory attributes of tigernut oil are equally intriguing. Recent research by Ola *et al*. (2012) identified a total of 75 odor-active compounds in roasted tigernut oil, including compounds like 5-hydroxymethylfurfural, ethyl hexadecanoate, and n-propyl-9,12-octadecadienoate. These compounds contribute to the delightful flavor profile of tigernut oil, making it a flavorful and aromatic addition to culinary creations (Lasekan, 2012).

In essence, tigernut oil is a nutritional powerhouse with a lipid profile akin to olive oil, enriched with vitamins, minerals, and potent antioxidants. Its bioactive components imbue it with potential therapeutic benefits, while its sensory qualities make it a delectable choice for diverse culinary applications. Unveiling the multifaceted attributes of tigernut oil not only paves the way for culinary innovation but also holds promise for potential health advantages.

1.2.3 METHOD OF EXTRACTION OF TIGERNUT OIL

Commonly, the tiger nut oil is produced by the conventional extraction method including Soxhlet extraction (SE) and mechanical expressing (ME). At present, some innovative techniques have been established for the sustainable and viable recovery of the oil, such as gas-assisted mechanical expression (GAME), mechanical compressing with enzyme assistance (MEEA), microwave-assisted extraction (MAE), microwave-ultrasonic assisted aqueous enzymatic extraction (MUAAEE), supercritical carbon dioxide fluid extraction (SC-CO_2) and subcritical *n*-butane extraction (SBE).

Tigernut oil extraction involves several methods, each with its advantages and limitations:

- Soxhlet Extraction (SE) is a conventional method known for its high oil yield (29.85 g/100 g dried powder). However, it uses organic solvents, raising safety and environmental concerns.
- Mechanical Expression (ME) is a safer method but offers a relatively lower oil yield (19.94 g/100 g). Cold pressing and hot pressing are subcategories, with the latter yielding more oil but consuming more electricity. Cold-pressed oil tends to have better quality, such as lower acid value and better color, but with a lower yield.
- Enzyme-Assisted Mechanical Extraction (MEEA) combines enzymes with ME to break down cell walls and enhance oil release. This method has shown promise, achieving an oil recovery rate of 20.79 g/100 g, surpassing ME alone.
- Microwave-Assisted Extraction (MAE) is quicker and uses less solvent compared to SE. It offers a shorter extraction time and lower solvent consumption, making it an efficient alternative.
- Microwave-Ultrasonic Assisted Aqueous Enzymatic Extraction (MUAAEE) combines microwave, ultrasonic, and enzymatic techniques. This method significantly shortens extraction time, resulting in an oil recovery of 25.44 g/100 g. However, it requires complex equipment.
- Supercritical Carbon Dioxide Fluid Extraction (SC-CO_2) is an eco-friendly technique that reduces the need for toxic solvents. It yields 27.79 g/100 g of tigernut oil but requires a longer extraction time and complex equipment.
- Gas Assisted Mechanical Expression (GAME) combines ME with SC-CO_2 to improve oil recovery. GAME has shown an oil recovery rate above 80%, and it enhances the oil's phenolic compound content, which contributes to better oxidation resistance.

- Subcritical n-Butane Extraction (SBE) is a method for extracting fat-soluble components under low pressure and temperature. While it offers some advantages over SC-CO_2, it still consumes a considerable amount of solvent.

In summary, conventional SE and ME methods are efficient for tigernut oil extraction but have drawbacks such as solvent use or lower yield. Unconventional methods like GAME and MUAAEE offer enhanced yields but may require complex equipment. Combining techniques is a promising direction for future industrial production. GAME, for instance, achieves a higher oil yield (28.48 g/100 g) than ME (19.94 g/100 g) and SC-CO_2 (27.79 g/100 g) alone.

Method	**Condition**	**Oil Yield**	**Reference**
SE	Powder: n-hexane = 1:10(*w*/*v*), extraction temperature 80 °C, 6 h	29.85 g/100 g	(Hu *et al., 2020)*
MAE	petroleum ether and acetone (2:1, *v*/*v*), microwave power 420 W, liquid to solid ratio 7.0 mL/g, 75 °C and 55 min	24.12 g/100 g	(Koubaa *et al.,* 2015)
ME	Pressing temperature 40 °C, 120 min, 30 MPa, speed 0.1 mm/s	19.94 g/100 g	(Ezeh and Gordon, 2016)
GAME	Temperature 40 °C, CO_2 pressure 20 MPa, CO_2 flow 8.5 kg/h, pump pressure 30 MPa, 120 min	28.48 g/100 g	(Ezeh and Gordon, 2016)
MEEA	Protease, α-amylase and Viscozyme L(1/1/1, *w*/*w*/*w*), mixed enzyme addition 1%, pH 8, 40 °C	20.79 g/100 g	(Ezeh *et al., 2016*)
MUAAEE	Cellulase, pectinase and hemicellulase (1/1/1, *w*/*w*/*w*), 40 °C, pH 3.5, ultrasonic power 300 w, microwave power 300 w, radiation time 30 min, enzyme concentration 2%, liquid to solid ratio 10 mL/g	25.44 g/100 g	(Ezeh and Gordon, 2016)

Method	Condition	Oil Yield	Reference
	and enzymolysis time 180 min		
$SC\text{-}CO_2$	Extraction temperature 60 °C, pressure 28 MPa, 90 min	27.79 g/100 g	(Ezeh *et al., 2016*)
SBE	Extraction temperature 40 °C, extraction time 50 min	26.03 g/100 g	(Hu *et al., 2018*)

Table 1: Oil yield of tigernut carried out by different extraction methods under separate conditions (Yu *et al.,* 2022)

- **Cold Pressing as an Extraction Method**

Cold pressing, also known as cold expeller pressing, is an oil extraction method that involves mechanically pressing oilseeds or nuts at temperatures below 122°F (50°C). Unlike traditional methods that use heat and chemicals, cold pressing relies solely on mechanical pressure to extract oil from the raw material. The process typically consists of crushing the oil-bearing seeds or nuts in a hydraulic press to release the oil. This method is commonly used for extracting high-quality, unrefined oils from various sources, including tigernuts.

Advantages and Disadvantages

Advantages

- Preservation of Nutrient Content: Cold pressing preserves the nutritional integrity of the oil, as it does not expose the oil to high temperatures that can degrade sensitive compounds like vitamins and antioxidants (Ezeh *et al.,* 2016).

- High-Quality Oil: Cold-pressed oils are often considered superior in flavor, aroma, and color compared to oils extracted using heat or chemicals. They maintain a more natural and mild taste profile (Ezeh *et al.,* 2016).

- No Chemical Residues: Since cold pressing avoids the use of solvents or chemicals, the resulting oil is free from chemical residues and is considered a more natural and healthier option (Ezeh *et al.,* 2016).

Disadvantages

- Lower Oil Yield: Cold pressing typically yields less oil compared to some other extraction methods like solvent extraction or mechanical expression (Ezeh *et al.,* 2016).

- Longer Processing Time: The cold pressing process can be slower and less efficient than methods involving heat. This may not be ideal for large-scale industrial production (Ezeh *et al.,* 2016).

Preservation of Oil Quality

Cold pressing is renowned for its ability to preserve the quality of the oil. The low-temperature extraction method minimizes oxidative damage and retains the oil's natural flavour, colour, and aroma. It also helps maintain the oil's content of sensitive bioactive compounds, such as vitamins and antioxidants, which can be compromised by high-temperature extraction methods (Ezeh *et al.,* 2016).

1.2.4 COMPOSITION AND CHARACTERIZATION OF TIGERNUT OIL

Tigernut oil, derived from the small tuberous rhizomes of the tigernut plant (Cyperus esculentus), is gaining attention due to its unique nutritional profile and potential health benefits. The chemical composition of tigernut oil is a critical aspect that determines its

quality, taste, and potential applications. Below is an in-depth exploration of the chemical composition of tigernut oil, including its fatty acid profile, vitamins, minerals, and bioactive compounds.

Fatty Acid Profile

Tigernut oil is predominantly composed of various fatty acids, making it a valuable source of dietary fats. The fatty acid composition may vary depending on factors such as the tigernut variety, cultivation conditions, and extraction methods (Ezeh *et al.,* 2016).

- **Monounsaturated Fatty Acids (MUFAs):** Tigernut oil is particularly rich in MUFAs, especially oleic acid (C18:1). Oleic acid is a monounsaturated omega-9 fatty acid, and its presence in tigernut oil is similar to that in olive oil. MUFAs are considered heart-healthy fats and are associated with various health benefits (Ezeh *et al.,* 2016).
- **Polyunsaturated Fatty Acids (PUFAs):** Tigernut oil contains polyunsaturated fatty acids, including linoleic acid (C18:2) and linolenic acid (C18:3). Linoleic acid is an omega-6 fatty acid, while linolenic acid is an omega-3 fatty acid. PUFAs are essential for the body and play a crucial role in various physiological processes (Ezeh *et al.,* 2016).
- **Saturated Fatty Acids (SFAs):** While tigernut oil is relatively low in SFAs, it still contains some saturated fats, such as palmitic acid (C16:0) and stearic acid (C18:0). These are generally considered less desirable than unsaturated fats due to their potential impact on cholesterol levels (Ezeh *et al.,* 2016).

Vitamins

Tigernut oil contains various vitamins, which contribute to its nutritional value:

- **Vitamin E:** Vitamin E, specifically α-tocopherol, is present in tigernut oil and acts as a potent antioxidant. It helps protect the oil from oxidation, extending its shelf life and maintaining its quality (Ezeh *et al.,* 2016).

Minerals

Tigernut oil also contains essential minerals:

- **Potassium:** Tigernuts are known for their high potassium content, and this mineral is partially retained in the oil. Potassium plays a vital role in regulating blood pressure and maintaining cardiovascular health (González-Martín *et al.,* 2012).
- **Calcium and Magnesium:** Tigernut oil contains trace amounts of calcium and magnesium, which are essential for bone health and various metabolic processes (González-Martín *et al.,* 2012).

Bioactive Compounds

Tigernut oil is not only a source of macronutrients but also contains bioactive compounds with potential health benefits:

- **Phenolic Compounds:** Phenolic compounds, including phenolic acids and flavonoids, are found in tigernut oil. These compounds possess antioxidant properties, which can help protect cells from oxidative stress and inflammation (González-Martín *et al.,* 2012).
- **Alkaloids and Saponins:** Tigernuts and their oil may contain alkaloids and saponins, which have demonstrated antibacterial and anti-inflammatory effects in some studies (Viuda-Martos *et al.,* 2012).

Understanding the chemical composition of tigernut oil is essential for its potential applications in the food industry and its recognition as a functional food with health-promoting properties.

1.2.5 NUTRITIONAL RELEVANCE OF TIGERNUT OIL COMPOSITION

Tigernut oil, derived from the tuberous rhizomes of the tigernut plant (Cyperus esculentus), offers a unique composition that presents a wide range of implications for both human health and its application in the food industry. This comprehensive note delves into the significant aspects of tigernut oil composition, emphasizing its health benefits, nutritional properties, and functional attributes in various food applications.

Health Benefits and Nutritional Properties:

- **Monounsaturated Fatty Acids (MUFAs):** Tigernut oil is notably rich in monounsaturated fatty acids (MUFAs), with oleic acid (C18:1) being a predominant component. Oleic acid, a monounsaturated fat, has been associated with several health benefits. Research suggests that diets rich in MUFAs, such as oleic acid, may help reduce the risk of cardiovascular diseases by improving lipid profiles and lowering LDL cholesterol levels (López-Marcos *et al.,* 2015).
- **Essential Fatty Acids**: Tigernut oil also contains essential polyunsaturated fatty acids (PUFAs) like linoleic acid (C18:2) and linolenic acid (C18:3). These omega-6 and omega-3 fatty acids play critical roles in various physiological functions, including brain health, immune system support, and inflammation regulation (López-Marcos *et al.,* 2015).
- **Antioxidants:** Phenolic compounds and antioxidants present in tigernut oil contribute to its ability to combat oxidative stress and protect cells from damage. These

antioxidants, which include vitamin E, may help reduce the risk of chronic diseases and support overall well-being (Ezeh *et al.,* 2014).

Functional Properties of Tigernut oil and its applications in food

Tigernut oil's composition also makes it a valuable ingredient in food applications due to its functional properties:

- **Flavor Enhancement:** Tigernut oil possesses a pleasant and mild nutty flavor, making it an excellent choice for enhancing the taste of various dishes. Its subtle nuttiness adds a unique dimension to culinary creations, from salad dressings to baked goods (Ezeh *et al.,* 2014).
- **Texture and Mouthfeel:** In food formulations, tigernut oil can contribute to desirable textures and mouthfeel due to its lipid content. It can be used to improve the richness and creaminess of sauces, spreads, and dairy alternatives (Roselló-Soto *et al.,* 2018).
- **Natural Antioxidant:** The natural antioxidants found in tigernut oil, including phenols, can help extend the shelf life of food products by reducing oxidative deterioration. Incorporating tigernut oil into food formulations can enhance product stability (Ezeh *et al.,* 2014).
- **Gluten-Free and Allergen-Friendly:** Tigernut oil is a suitable choice for gluten-free and allergen-friendly food products. It can be used as an alternative to oils that may trigger allergies or intolerances, broadening the range of accessible food options (Viuda-Martos *et al.,* 2012).

In conclusion, tigernut oil's composition, characterized by its MUFAs, essential fatty acids, and antioxidants, positions it as a healthful and versatile ingredient in various food applications. Its potential to enhance flavors, textures, and shelf life, while accommodating dietary restrictions, underscores its importance in the culinary and food manufacturing realms.

Further research and exploration of tigernut oil's functional attributes hold promise for future innovations in the food industry.

1.2.6 THERMAL STABILITY OF TIGERNUT OIL

The thermal stability of tigernut oil, like any other edible oil, is an important factor that affects its quality and suitability for various culinary and industrial applications. The thermal stability of an oil refers to its ability to withstand high temperatures without undergoing significant chemical changes, such as oxidation, that can lead to the degradation of the oil's nutritional value, flavor, and overall quality.

Here's a detailed exploration of the thermal stability of tigernut oil:

1. Oxidative Stability

- Tigernut oil, like many other vegetable oils, contains unsaturated fatty acids, particularly oleic acid and linoleic acid. These unsaturated fatty acids are susceptible to oxidation when exposed to heat, light, and oxygen.

- Oxidation of tigernut oil can lead to the formation of undesirable compounds, such as peroxides, aldehydes, and ketones, which can negatively impact the oil's flavor, aroma, and nutritional properties.

- The oxidative stability of tigernut oil can be influenced by factors such as the presence of antioxidants (natural or added), storage conditions, and the oil's initial quality.

2. Smoke Point

- The smoke point of an oil is the temperature at which it begins to produce visible smoke and break down into volatile compounds. For tigernut oil, the smoke point is typically higher

than that of some other edible oils like olive oil, making it suitable for high-heat cooking methods such as frying and sautéing.

- The smoke point of tigernut oil can vary depending on factors such as oil purity and processing methods.

3. Nutritional Quality

- High-temperature processing and prolonged heating can result in the loss of some of the oil's heat-sensitive nutrients, such as certain vitamins (e.g., vitamin E) and phytochemicals. Therefore, minimizing heat exposure during oil processing and cooking can help preserve its nutritional value.

4. Industrial Applications

- Tigernut oil is not only used in cooking but also finds applications in the cosmetic and pharmaceutical industries. In these applications, the thermal stability of the oil is crucial to maintain product quality and shelf life.

5. Improving Thermal Stability

- To enhance the thermal stability of tigernut oil, manufacturers may employ various strategies, including refining processes to remove impurities and antioxidants (natural or synthetic) to inhibit oxidation.

- Proper storage in cool, dark conditions and protection from oxygen exposure are essential to maintain the oil's quality.

1.2.7 USE OF TIGERNUT OIL IN CULINARY ARTS

Cooking Techniques (Frying, Sautéing, Baking):

- **Frying**: Tigernut oil, with its high smoke point and excellent thermal stability, is a great choice for deep frying. Its ability to withstand high temperatures without breaking down or producing harmful compounds makes it suitable for frying foods like French fries, chicken, and doughnuts. It imparts a pleasant, nutty flavor to fried dishes.

- **Sautéing**: Tigernut oil's medium-high smoke point makes it ideal for sautéing vegetables, meats, and seafood. Its subtle nutty taste complements a wide range of ingredients and enhances the overall flavor of dishes.

- **Baking**: Tigernut oil can be used in baking applications, where its mild flavor and moisture-retaining properties can contribute to moist and flavorful baked goods. It can replace other vegetable oils or butter in recipes for cakes, muffins, and bread.

Flavor and Sensory Attributes

- **Nutty Flavor**: Tigernut oil is known for its distinctive nutty flavor, which can add depth and richness to dishes. This unique taste is often appreciated in both savory and sweet culinary creations.

- **Mild Aroma**: The oil has a mild, pleasant aroma that doesn't overpower the other ingredients in a dish, allowing the natural flavors of the food to shine through.

- **Sensory Appeal**: Chefs and home cooks value tigernut oil for its sensory attributes. Its subtle flavor and aroma can enhance the overall dining experience, making it a versatile choice in various cuisines.

1.2.8 INDUSTRIAL APPLICATIONS OF TIGERNUT OIL

Tigernut oil isn't limited to culinary applications; it also finds use in various industrial sectors:

- **Cosmetic Industry**: Tigernut oil is used in cosmetic products, including skincare formulations and hair care products, due to its moisturizing properties. It is rich in oleic acid and vitamin E, making it beneficial for skin and hair health.
- **Pharmaceutical Industry**: Some pharmaceutical formulations incorporate tigernut oil as a carrier oil for certain medications. Its hypoallergenic and stable nature makes it a suitable choice for drug delivery systems.
- **Lubricants**: In some industrial applications, tigernut oil is used as a bio-based lubricant due to its lubricating properties and biodegradability.
- **Biofuels**: Researchers are exploring the potential of tigernut oil as a biofuel source. Its fatty acid composition and oil content make it a candidate for biodiesel production, contributing to sustainable energy solutions.

CHAPTER TWO

METHODS AND MATERIALS

2.1 SOURCING OF MATERIALS

The studied material- Tiger nut was bought from an open market in Ugbowo, Benin-city, Edo state.

2.2 MATERIAL PREPARATION

The tigernuts were examined to remove bad ones and the healthy ones were subsequently used. The tigernuts were wrapped aluminium foil paper and oven dried at a temperature of 105^0C. After drying, the dried tigernuts were allowed to cool before grinding. The ground material was thereafter used for the extraction process.

Figure 2.1 Picture of tiger nuts

2.3. EXTRACTION AND RECOVERY OF OIL FROM TIGERNUTS

Materials

- Soxhlet extractor
- Thimble
- Condenser
- 1L Round bottom/distillation flask
- n-hexane
- Tiger nut sample
- Anti-bumping stones
- Rotary evaporator

Method and Procedure

Solvent extraction of the oil was carried out using a soxhlet apparatus.

The apparatus was initially washed and then dried. After which 90g of the already crushed sample of tigernut was weighed and packed into the thimble, covered with a cotton ball (to avoid the sample directly falling into the extractor) which was then placed inside the soxhlet apparatus.

Six hundred millimeters of n-hexane was poured into a 1L round bottom flask. The flask which was fixed to a retort stand was set up on a heating mantle, the soxhlet extractor was connected to the flask and above the extractor, the condenser was attached and then tightly fixed to the retort stand with the aid of another clamp. The inlet of the condenser was connected to a running tap and the outlet was connected to the basin. The reflux was carried out for 6 to 8 hours.

The bottom flask is heated to boil the solvent, the vapor rises through the branch pipe of the extractor, is condensed and drops into the extractor, and the solvent is contacted with the solid for extraction. When the solvent surface exceeds the highest point of the siphon, the

solvent containing the extract is siphoned back into the flask. The solvent and the extracted material (oil) is concentrated in the flask.

After the reflux has been completed, the non-soluble portion of the extracted solid remaining in the thimble was discarded and the content in the round bottom flask (the solvent and oil) was then taken to the rotary evaporator to separate the oil from the solvent by distillation under vacuum. The round bottom flask was connected to the vapour duct with the aid of a clip (to prevent it from slipping off), the receiver flask was connected to the condenser and then the rotary evaporator flask was submerged in a water bath. The temperature of the water bath of the rotary evaporator was set to 68.7^0C which is the boiling point of the solvent (n-hexane).

The source of water supply to the condenser and the pump vacuum systems were then turned on. The motor was also turned on for the flask to start rotating, the rotating round bottom flask increases the liquid's surface area and thus the rate of evaporation. The solvent vapor travels into the cooler water condenser, where it condenses and drips into a separate receiving flask. The solvent is removed by this process, thereby leaving the oil in the original round bottom flask.

This whole process was repeated until a satisfactorily amount of the oil needed was gotten.

Precautions

1. Continuous water supply to the condenser was ensured to avoid over-heating.
2. Constant heat supply was ensured throughout the reflux process.
3. The thimble containing the sample was well covered with cotton balls to prevent the sample from falling directly into the extractor.
4. It was ensured that the flasks were properly fixed to the rotary evaporator.

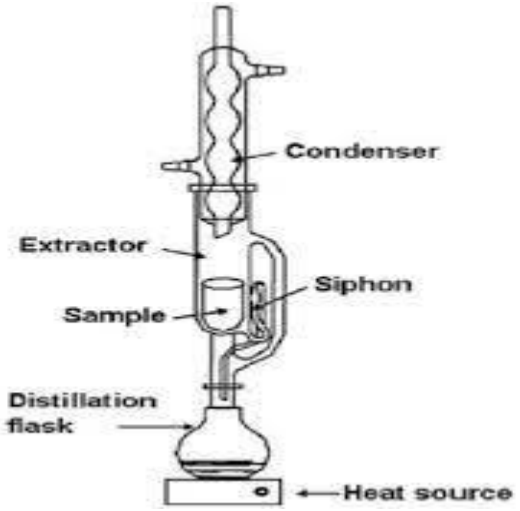


Figure 2.2 Schematic diagram of a soxhlet extractor setup

Figure 2.3 Picture of oil extracted in sample bottle

2.4. ANALYSIS CARRIED OUT ON EXTRACTED OIL

2.4.1. FREE FATTY ACID DETERMINATION

Free fatty acid content of the oil was determined in accordance with AOAC 940.28 method as prescribed by (Dr. Latimer, 2023).

Materials

- Ethanol
- Phenolphthalein
- Distilled water
- Weighing balance
- 50ml burette
- Oil sample
- 50ml Measuring cylinder
- Water bath
- 0.1M Sodium hydroxide solution (NaOH)
- Funnel
- 250ml Conical flask

PREPARATION OF REAGENTS

- **0.1M NaOH Solution (250ml)**

One gram of NaOH pellets was weighed and dissolved in a little amount of distilled water, the solution was then transferred into a 250ml volumetric flask and distilled water was added to make the solution up to the mark.

- **1% phenolphthalein indicator**

One gram of phenolphthalein powder was weighed and dissolved in 95% ethanol with continuous stirring until it dissolved.

Procedure

Ten grams of the oil sample was weighed into a 250ml conical flask, 50ml of ethanol was added to it, the solution was covered and boiled in a water bath for 5-15 minutes. The burette was first rinsed with distilled water and afterwards with the sodium hydroxide solution then the burette was filled with the 0.1M Sodium hydroxide solution. After 5 minutes had elapsed, the conical flask was removed from the water bath and 2-3 drops of phenolphthalein indicator was added. The solution was then titrated against the NaOH solution in the burette with continuous shaking until a pink colour which persisted for 30 seconds was observed, indicating the end-point of the titration. The volume of titrant used was recorded and thereafter the %free fatty acid was calculated from the formula;

$$\% \text{ FFA} = \frac{V \times M \times 28.2}{W}$$

Where V= Volume of NaOH used in ml

M= Molarity of NaOH

W= Weight of sample (oil) in g

2.5. THERMAL STABILITY TEST

A small amount of the oil sample was heated at 85^0C in a water bath for 1hour 30 minutes. From the heated oil, 5g of the oil was weighed and used for peroxide value analysis. A small quantity was also transferred into sample test bottles for UV analysis and refractive index test.

2.5.1. DETERMINATION OF PEROXIDE VALUE

The peroxide value of the oil was determined in accordance with AOAC 965.33 method as prescribed by (Dr. Latimer, 2023).

Materials

- Glacial acetic acid - chloroform (3:2)
- Saturated potassium iodide (KI)
- Distilled water
- 0.01M Sodium thiosulfate solution
- Starch indicator
- Filter paper

Preparation of reagents

- Starch indicator (1%)

One gram of starch soluble was weighed and poured into 100ml of boiling water, the solution was stirred until it dissolved completely. The solution was then brought down and thereafter filtered using a filter paper. The filtrate was kept in an indicator bottle as the starch indicator.

- **0.01M Sodium thiosulfate solution (250ml)**

Sodium thiosulfate crystal weighing 0.625g was dissolved in 250ml of distilled water, the solution was stirred till it completely dissolved and then transferred into a reagent bottle to be used for the analysis.

- **Saturated Potassium Iodide (KI) solution**

One hundred gram of potassium iodide salt was weighed and dissolved in 70ml of distilled water. The solution which had undissolved salts settled at d bottom was kept in the dark.

Procedure

Five gram of the oil sample was weighed into a 250ml conical flask then 18ml of glacial acetic acid was added followed by addition of 12ml of chloroform. 1ml of saturated KI solution was added, the solution was agitated for one minute after which 30ml of distilled water was added and further agitation for another one minutes was done. The burette was first rinsed with water followed by the titrant and was then filled with the titrant- 0.01M Sodium thiosulfate solution. The initial reading was recorded. 0.5ml starch solution was added to the mixture in the conical flask which gave a dark color, the mixture was then titrated against the sodium thiosulfate solution in the burette. Titration was carried out with vigorous agitation of the flask until the dark color changes to white. The end point is then reached and the final reading was recorded. This process was repeated for the heated oil sample.

$$\text{Peroxide value} = \frac{V \times M \times 1000}{W}$$

Where V = Volume of Sodium thiosulfate solution used in ml

M = Molarity of sodium thiosulfate solution

W = Weight of oil sample

2.5.2. UV ANALYSIS

Materials

- Heated oil sample and sample at room temperature
- UV spectrometer
- Reference or blank sample (n-hexane)

Principle

The amount of discrete wavelengths of UV or visible light that are absorbed by or transmitted through a sample in contrast to a reference or blank sample is measured by the analytical technique known as UV-Vis spectroscopy. The sample composition has an impact on this feature, potentially revealing what is in the sample and at what concentration (Justin, 2021).

Procedure

The sample at room temperature and heated samples were both taken to the spectrometer. The reference sample was first used to zero the spectrometer. Then the oil was placed into the cuvette and photometric scanning was done by exposing the sample to the UV/VIS wavelength and obtaining the wavelength at which the sample has maximum absorption. The wavelength of the sample was then obtained and the absorbance was determined at that wavelength. The wavelength was also set at 234nm to determine the absorption at that wavelength and the absorbance at both wavelengths were compared.

2.5.3 REFRACTIVE INDEX TEST

Materials

- Refractometer
- Oil samples
- Tissue paper

Principle

Instruments called refractometers are used to measure compounds that are dissolved in water and some oils for example, sugar. Utilizing the idea of light refraction via liquids, the refractometer operates. Light slows down as it enters a liquid from the air. This effect is what gives things that are partially submerged in water a "bent" appearance. Simply put, the slower light moves through water and the more pronounced the "bending" effect on light, the more

dispersed materials it contains. By shining light through a sample and displaying the refracted angle on a scale, refractometers employ this principle to calculate the concentration of dissolved solids in liquids. The Brix scale is the most frequently employed scale. The amount of pure cane sugar that dissolves in 100 grams of pure water is known as the Brix scale (grams sugar/100 grams H20).

Procedure

The prism was first cleaned with a tissue then little drop of the unheated oil was poured on the prism of the refractometer, the cover plate was closed and the sample was allowed to spread over the prism surface. The prism was pointed toward the source of light (in this case daylight). Through the eyepiece (lens), an internal scale was seen and the point in the scale where the boundary line intercepts i.e the point where the dim light and blue light met was read, that point was recorded as the %brix of the oil. The same process was repeated for the heated oil.

2.6. SPECTROSCOPIC STUDIES

2.6.1. FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FTIR)

Principle

Covalent bonds in molecules will selectively absorb light of particular wavelengths, changing the vibrational energy in the bond. The atoms in the link determine the sort of vibration (stretching or bending) caused by the infrared radiation. The transmittance pattern varies for different molecules because different bonds and functional groups absorb various frequencies. (The opposite of absorption is transmittance.) Wavenumber (cm-1) is plotted on the X-axis

and transmittance is plotted on the Y-axis to represent the spectrum. (Wavenumber is 1/wavelength and represents the energy of the molecular bonds' vibrational motion.)

Procedure

A small amount of the oil sample was measured into a test sample bottle and it was sent out to be analyzed. The functional groups present in the oil were identified by the different peaks seen in the signal sent as the result. The model of FT-IR machine used for the analysis is Cary 630 by Agilent Technologies Inc USA.

CHAPTER THREE

RESULTS AND DISCUSSIONS

3.1 RESULTS FOR PHYSIO-CHEMICAL ANALYSIS

The table below shows the result of FFA, Peroxide value, refractive index and UV absorbance tests carried out on the raw and treated Tigernut oil.

Temperature(^{0}C)	**RT**		**85(^{0}C)**	
% Free fatty acid (FFA)	0.48		_	
Peroxide value(meq/kg)	1.96		7.80	
Refractive index (Brix %)	69		69.5	
UV test				
Wavelength(nm)	270nm	234nm	270nm	234nm
Absorbance	1.009	1.014	0.004	0.002

Table 3.1 Selected physio-chemical properties of raw and treated oil

For assessing the quality of the raw material and its degradation during storage, as well as during the shelf life of different vegetable oils, the determination of free fatty acid (FFA) content is a crucial analysis (Medeiros *et al*., 2021). As seen from the value of the %FFA gotten from the analysis which is 0.48%, this value falls within the range of 0.20% to 0.87% according to Friday *et al*., 2021. High amounts of FFA make fats more vulnerable to oxidative aging and make go rancid more quickly.

The amount of oxidation reactions that takes place in an oil or fat is its peroxide value. Peroxide value PV is a measurement of the hydroperoxides present in the oil as a byproduct of primary oil oxidation. It is frequently given as milliequivalents of hydroperoxide (ROOH) per kilogram of oil (Saad *et al.*, 2006). This could result in the formation of bad aromas and odors. Upon heating of the oil, it can be seen from table (3.1) that the peroxide value

increased from 1.96meq/kg to 7.80meq/kg indicating that the oil was tending towards rancidity although the value obtained didn't exceed the limit of 10meq/kg set by the American Oil Chemists' Society (AOCS). High peroxide readings are a solid indicator of rancid fat and oil (Kamsiah and Yusof, 2012).

At greater temperatures, molecular motion (vibration and rotation) occurs; more energy is required at higher temperatures, increasing the absorbance (Saad *et al*., 2006). With each increase in the number of heating elements, the peak wavelength swings towards longer wavelengths as the oil temperature rises and the heating duration increases. It has been demonstrated that the absorbance rises as oil is heated to a greater temperature (Nawras and Chiad, 2021). In this study however, it is seen that the oil heated at 85^0C showed a decrease in absorption at both wavelengths (234 and 270nm) compared to that of the unheated tiger nut oil although greater absorption was seen at 234nm for the oil at RT while for the heated oil, greater absorption was observed at 270nm).This could be due to the change in molecular structure of the oil causing a decrease in the intensity amplitudes of absorption thereby leading to decrease in the absorbance of the oil i.e decrease in concentration of the oil (Mohammad, 2018).

From the result gotten, it was seen that the refractive index of the oil heated at 85^0C increased by 0.5% compared to the one at room temperature.

3.2 RESULT FOR FTIR ANALYSIS

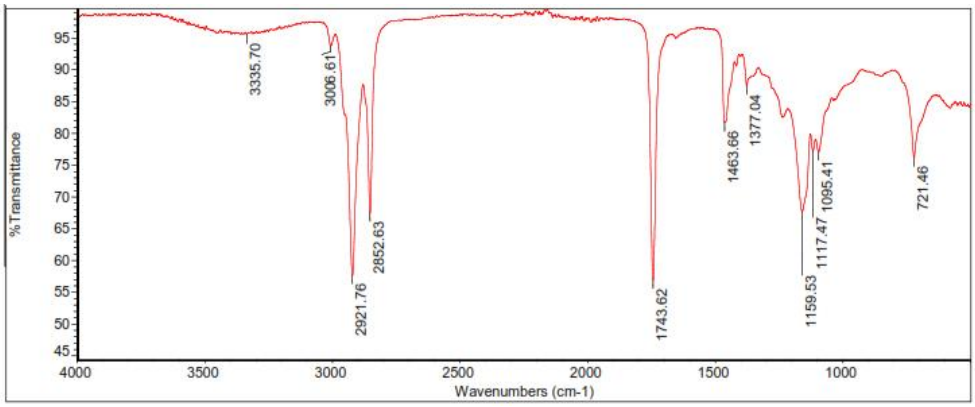


Figure 3. FTIR spectrum for tiger nut oil

The FTIR machine with model number Cary 630 set at a revolution of 4.000 with number of sample scans and background scans of 16, optical velocity of 0.4747 and aperture of 100 presented the spectrum above. The peaks in the spectrum can therefore be interpreted thus;

Wavenumber (cm^{-1})	**Absorption band range (cm^{-1})**	**Responsible functional group**	**Appearance**
3335.70	3550-2500	**O−H** stretch of alcohols (R−OH)	Broad
3006.61	3100-3000	**C−H** stretch of alkenes (=C−H)	Medium & sharp
2921.76	3300-2500	**O−H** stretch of carboxylic acids	Strong and broad
2852.63	3000-2840	**C−H** stretch of alkanes (−C−H)	Medium &sharp
1743.62	1750-1735	**C−O** stretch of esters (−C=O)	Strong
1463.66	1465-1415	**C−H** bend of alkanes (−C=H)	Medium
1377.04	1380-1370	**C−H** bend of alkanes (−C=H)	Medium
1159.53	1200-1070	**C−O** stretch of ethers (−R−O−R)	Strong
1117.47	1124-1087	**C−O** stretch of 2^0 alcohols (C−OH)	
1095.41	1124-1087	**C−O** stretch of 2^0 alcohols (C−OH)	
721.46	730-665	**C=C** bend of alkenes (−C=C−)	Strong

Table 3.2 Result for interpretation of FTIR spectrum

The primary fatty acids present in tiger nut oil are oleic acid ($C_{18}H_{34}O_2$), linoleic acid ($C_{18}H_{32}O_2$), palmitic acid ($C_{16}H_{32}O_2$) and also stearic acid ($C_{18}H_{36}O_2$). However, the most

prevalent fatty acid in the oil is oleic acid (Yu *et al*., 2022). From the interpretation in the table above, it can be seen that the tiger nut oil comprises of the alkane, alkene, hydroxyl, carboxylic and ester functional groups all combining to give the fatty acid composition and triglycerides structure in the oil. Oleic acid, the predominant fatty acid which can be used as the reference has a carboxylic functional group corresponding to the peak 2921.7 cm^{-1}, the broad peak at 3335.70 cm^{-1} corresponds to the stretching bond of alcohols from the carboxylic acid structure, the medium peak at 3006.61cm^{-1} corresponds to the stretching bond of alkene (double bond), the alkane bonds in the structure corresponds to the medium peaks at 1463.66cm^{-1} and 1377.04 cm^{-1} respectively. The peaks at 1117.47cm^{-1} and 1095.41cm^{-1} which corresponds to the C-O stretch of secondary alcohols and the peak at 1159.53cm^{-1} of the C-O stretch of ethers could be from other bioactive compounds present in the tigernut oil for example phenolic acids, flavonoids, alkaloids and saponins.

CONCLUSION

This study shows that tigernut oil can be used as an alternative source of fat and oils as seen from the free fatty acid content and peroxide value as it would not be easily susceptible to oxidation due to its thermal stability. These characteristics of the oil indicates that the oil is suitable for culinary and some industrial purposes.

The UV test emphasizes the importance of exercising caution while exposing tigernut oil to high temperatures. The variations in absorbance at particular wavelengths are signs of chemical changes that might impact the oil's sensory properties, nutritional value, and shelf life. Additional investigation could focus on the precise chemical alterations and their effects on a variety of purposes, such as tigernut oil's usefulness in industry and food.

The FTIR spectrum supports the tigernut oil's known composition by confirming the presence of both saturated and unsaturated components. It offers useful details on tigernut oil, which are crucial for comprehending its characteristics and prospective uses. The origin of particular functional groups may be made clearer with additional research.

REFERENCES

Adejuyitan, J. A. (2011). Tigernut Processing: Its Food uses and Health Benefits. ***American Journal of Food Technology***, *6*(3), 197–201. https://doi.org/10.3923/ajft.2011.197.201

Adel, A. A. M., Awad, A. M., Mohamed, H. H., & Iryna, S. (2015). Chemical composition, physicochemical properties and fatty acid profile of Tiger Nut (Cyperus esculentus L) seed oil as affected by different preparation methods. ***International Food Research Journal***, *22*(5).

Akubugwo, I. E., Obasi, N. A., Chinyere, G. C., and Ugbogu, A. E. (2007). Nutritional potential of the oils from some unconventional oilseeds. ***Plant Foods for Human Nutrition***, 62(4), 183-188.

Bamishaiye, E. I., & Bamishaiye, O. M. (2011). Tiger nut: as a plant, its derivatives and benefits. ***African Journal of Food, Agriculture, Nutrition and Development***, *11*(5). https://doi.org/10.4314/ajfand.v11i5.70443

dos Santos Silveira Junior, W., and de Francisco, A. (2020). Unconventional food plants as an alternative in starch production. (2020). ***Cereal Foods World***, *65*(2). https://doi.org/10.1094/cfw-65-2-0018

Dr. Latimer, George W, Jr. (2023). *Official Methods of Analysis of AOAC INTERNATIONAL*, 22 (online edn), Oxford Academic. https://doi.org/10.1093/9780197610145.001.0001

Ezeh, O., Gordon, M. H., & Niranjan, K. (2014). Tiger nut oil (*Cyperus esculentus* L.): A review of its composition and physico-chemical properties. ***European Journal of Lipid Science and Technology***, *116*(7), 783–794. https://doi.org/10.1002/ejlt.201300446

Ezeh, O., Niranjan, K., & Gordon, M. H. (2016). Effect of enzyme pre-treatments on bioactive compounds in extracted tiger nut oil and sugars in residual meals. ***Journal of the American Oil Chemists' Society***, *93*(11), 1541–1549. https://doi.org/10.1007/s11746-016-2883-9

Friday, O. A., Nina, G. C., & Ukeyima, M. T. (2021a). Quality characteristics of stored varieties of Tiger nut oil. ***Food and Life (Online)***. https://doi.org/10.5851/fl.2021.e3

Jing, S., Wang, S., Li, Q., Zheng, L., Li, Y., Fan, S., & Tao, G. (2016). Dynamic high pressure microfluidization-assisted extraction and bioactivities of Cyperus esculentus (C. esculentus L.) leaves flavonoids. ***Food Chemistry***, *192*, 319–327. https://doi.org/10.1016/j.foodchem.2015.06.097

Kamsiah, J., & Yusof, K. (2012). Repeatedly heated vegetable oils and lipid peroxidation. In ***InTech eBooks***. https://doi.org/10.5772/46076

Lasekan, O. (2012). Volatile constituents of roasted tigernut oil (*Cyperus esculentus*L.). ***Journal of the Science of Food and Agriculture***, *93*(5), 1055–1061. https://doi.org/10.1002/jsfa.5846

Lozano-Milo, E., Lopez-Gomez, A., Montano-Machado, V., Cucalo-Padial, J., & Lardos, A. (2017). Determination of fatty acid composition of edible oil by gas chromatography-

mass spectrometry (GC-MS). IntechOpen. ***In Gas Chromatography-Mass Spectrometry (GC-MS)*** (pp. 115-135).

López-Marcos, M. C., Bailina, C., Viuda-Martos, M., Pérez-Álvarez, J. Á., & Fernández-López, J. (2015). Properties of Dietary Fibers from Agroindustrial Coproducts as Source for Fiber-Enriched Foods. ***Food and Bioprocess Technology***, *8*(12), 2400–2408. https://doi.org/10.1007/s11947-015-1591-z

Martinez-Ferez, A., Salvador, M. D., and Aranda, F. (2003). Fatty acid composition and drying characteristics of chufa (Cyperus esculentus L.) tubers. ***Journal of the American Oil Chemists' Society***, 80(6), 575-578.

Medeiros, V. C., Rodolfo, R. P, Bernardo, G .C., Lopes, D. A. (2021). Determination of free fatty acids in crude vegetable oil samples obtained by high-pressure processes. ***Food Chem X***.;12:100166. doi: 10.1016/j.fochx.2021.100166. PMID: 34825173; PMCID: PMC8604745.

Mohammad, E. K. (2018). Effect of Temperature on Optical Properties of Vegetable Oils Using UV-Vis and Laser Fluorescence Spectroscopy. ***Optics and Photonics Journal***, Vol.8 No.7.

Nawras, K., & Chiad, B. T. (2021c). Spectroscopic study for effect of heating temperatures on the black seed oil. ***Journal of Physics***, *1999*(1), 012071. https://doi.org/10.1088/1742-6596/1999/1/012071

Nina, G. C., Friday, O. A., Ukeyima, M. T., Hleba, L., Císarová, M., Okuskhanova, E., Власов, С. А., Batishcheva, N., Goncharov, A. I., & Shariati, M. A. (2019). Proximate, mineral and functional properties of tiger nut flour extracted from different tiger nuts cultivars. ***The Journal of Microbiology, Biotechnology and Food Sciences***, *9*(3), 653–656. https://doi.org/10.15414/jmbfs.2019/20.9.3.653-656

Nina, G. C., Ukeyima, M. T., Friday, O. A., Hleba, L., Hlebová, M., Glinushkin, A. P., Laishevtcev, A., Derkanosova, A., Igor, P., Plygun, S., & Shariati, M. A. (2021). Investigation of physiochemical and storage conditions on the properties of extracted tiger nut oil from different cultivars. ***The Journal of Microbiology, Biotechnology and Food Sciences***, *9*(5), 988–993. https://doi.org/10.15414/jmbfs.2020.9.5.988-993

Ogunlade, I., Adeyemi, B. A., Aluko, O. G. (2015). Chemical compositions, antioxidant capacity of Tigernut (*Cyperus esculentus*) and potential health benefits. ***European Scientific Journal*** (Special edition), 217-224.

Oladejo, A. O., Okoh, O. O., and Adebayo, A. E. (2018). A comprehensive review on the extraction, characterization, and utilization of tigernut oil. ***Journal of Food Processing and Preservation***, 42(1), e13357.

Oke, E. K., Idowu, M. A., Sobukola, O. P., & Bakare, H. (2017). Quality Attributes and Storage Stability of Bread from Wheat–Tigernut Composite Flour. ***Journal of Culinary Science & Technology***, *17*(1), 75–88. https://doi.org/10.1080/15428052.2017.1404537

Roselló-Soto, E., Poojary, M. M., Barba, F. J., Lorenzo, J. M., Mañes, J., & Moltó, J. (2018). Tiger nut and its by-products valorization: From extraction of oil and valuable

compounds to development of new healthy products. ***Innovative Food Science and Emerging Technologies***, *45*, 306–312. https://doi.org/10.1016/j.ifset.2017.11.016

Saad, B., Wai, W. T., Lim, B. P., & Saleh, M. I. (2006). Flow injection determination of peroxide value in edible oils using triiodide detector. ***Analytica Chimica Acta***, *565*(2), 261–270.

Sado-Kamdem S. L., and Fachuang, L. (2002). Thermal degradation of oils during deep-fat frying. ***Journal of Food Engineering***, 54(3), 211-216.

Sánchez-Zapata, E., Fernández-López, J., & Pérez-Alvarez, J. (2012). Tiger Nut (Cyperus esculentus) Commercialization: Health Aspects, Composition, Properties, and Food Applications. ***Comprehensive Reviews in Food Science and Food Safety***, *11*(4), 366–377. https://doi.org/10.1111/j.1541-4337.2012.00190.x

Vega-Morales, T., Mateos-Díaz, C., Pérez-Machín, R., Wiebe, J. C., Gericke, N., Alarcón, C., & López-Romero, J. M. (2019). Chemical composition of industrially and laboratory processed Cyperus esculentus rhizomes. ***Food Chemistry***, *297*, 124896. https://doi.org/10.1016/j.foodchem.2019.05.170

Viuda-Martos, M., López-Marcos, M. C., Fernández-López, J., Sendra, E., & Vargas, J. H. L. (2010). Role of Fiber in Cardiovascular Diseases: a review. ***Comprehensive Reviews in Food Science and Food Safety***, *9*(2), 240–258. https://doi.org/10.1111/j.1541-4337.2009.00102.x

Yu, Y., Lu, X., Zhang, T., Zhao, C., Guan, S., Pu, Y., & Feng, G. (2022). Tiger Nut (Cyperus esculentus L.): Nutrition, Processing, Function and Applications. ***Foods***, *11*(4), 601. https://doi.org/10.3390/foods11040601

.

APPENDIX

CALCULATIONS FOR FFA ANALYSIS

Burette readings(ml)	1st titration(ml)	2nd titration(ml)
Final readings	1.50	3.40
Initial readings	0.00	1.50
Volume of NaOH used	1.50	1.90

Average volume of NaOH used (V) = $\frac{1.50+1.90}{2}$ = **1.70ml**

Molarity of NaOH (M) = 0.1M; Weight of oil (W)= 10g

%FFA = $\frac{V \times M \times 28.2}{W}$

= $\frac{1.70 \times 0.1 \times 28.2}{10}$

= **0.48%**

CALCULATIONS FOR PV ANALYSIS

Temperature(℃)	RT	85^0C
Weight of oil(g)	5.09	5.01
Final burette reading(ml)	1.00	4.90
Initial burette reading(ml)	0.00	1.00
Volume of $Na_2S_2O_3$ used(ml)	1.00	3.90

Peroxide value = $\frac{V \times M \times 1000}{W}$

- **For oil at RT**

Volume of $Na_2S_2O_3$ (V) = 1ml; Molarity of $Na_2S_2O_3$ = 0.01M; Weight of oil = 5.09g

PV = $\frac{1\times0.01\times1000}{5.09}$

= **1.96meq/kg**

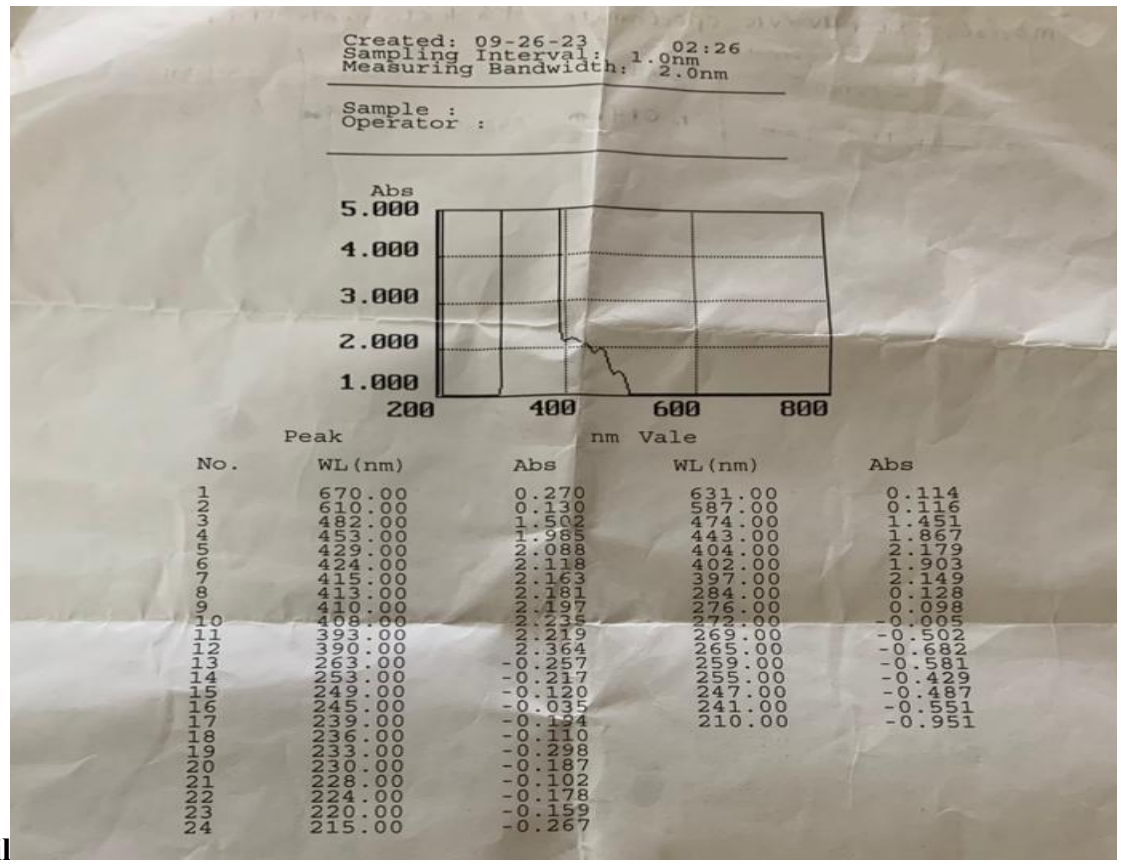
- **For oil heated at 85^{0}C**

Volume of $Na_2S_2O_3$ (V) = 3.90ml; Molarity of $Na_2S_2O_3$ = 0.01N; Weight of oil = 5.01g

PV = $\frac{3.90\times0.01\times1000}{5.01}$

= **7.78meq/kg**

UV Spectrum scanning for wavelength of Tigernut oil



Peak No.	WL(nm)	Abs	Vale WL(nm)	Abs
1	670.00	0.270	631.00	0.114
2	610.00	0.130	587.00	0.116
3	482.00	1.502	474.00	1.451
4	453.00	1.985	443.00	1.867
5	429.00	2.088	404.00	2.179
6	424.00	2.118	402.00	1.903
7	415.00	2.163	397.00	2.149
8	413.00	2.181	284.00	0.128
9	410.00	2.197	276.00	0.098
10	408.00	2.235	272.00	-0.005
11	393.00	2.219	269.00	-0.502
12	390.00	2.364	265.00	-0.682
13	263.00	-0.257	259.00	-0.581
14	253.00	-0.217	255.00	-0.429
15	249.00	-0.120	247.00	-0.487
16	245.00	-0.035	241.00	-0.551
17	239.00	-0.154	210.00	-0.951
18	236.00	-0.110		
19	233.00	-0.298		
20	230.00	-0.187		
21	228.00	-0.102		
22	224.00	-0.178		
23	220.00	-0.159		
24	215.00	-0.267		

SPECTRA SHEET FOR FTIR RESULT INTERPRETATION

The infrared spectroscopy absorptions by frequency regions (cm^{-1})

3700-3584	medium	sharp	O-H	stretching	alcohol
3550-3200	strong	broad	O-H	stretching	alcohol
3500 3400	medium		N-H	stretching	primary amine
3400-3300 3330-3250	medium		N-H	stretching	aliphatic primary amine
3350-3310	medium		N-H	stretching	secondary amine
3300-2500	strong	broad	O-H	stretching	carboxylic acid
3200-2700	weak	broad	O-H	stretching	alcohol
3000-2800	strong	broad	N-H	stretching	amine salt
3333-3267	strong	sharp	C-H	stretching	alkyne
3100-3000	medium		C-H	stretching	alkene
3000-2840	medium		C-H	stretching	alkane
2830-2695	medium		C-H	stretching	aldehyde
2600-2550	weak		S-H	stretching	thiol
2349	strong		O=C=O	stretching	carbon dioxide
2275-2250	strong	broad	N=C=O	stretching	isocyanate
2260-2222	weak		CΞN	stretching	nitrile
2260-2190	weak		CΞC	stretching	alkyne
2175-2140	strong		S-CΞN	stretching	thiocyanate
2160-2120	strong		N=N=N	stretching	azide
2150			C=C=O	stretching	ketene
2145-2120	strong		N=C=N	stretching	carbodiimide
2140-2100	weak		CΞC	stretching	alkyne
2140-1990	strong		N=C=S	stretching	isothiocyanate
2000-1900	medium		C=C=C	stretching	allene
2000			C=C=N	stretching	ketenimine
2000-1650	weak		C-H	bending	aromatic compound
1818 1750	strong		C=O	stretching	anhydride
1815-1785	strong		C=O	stretching	acid halide
1800-1770	strong		C=O	stretching	conjugated acid halide
1775 1720	strong		C=O	stretching	conjugated anhydride
1770-1780	strong		C=O	stretching	vinyl / phenyl ester
1760	strong		C=O	stretching	carboxylic acid
1750-1735	strong		C=O	stretching	esters
1750-1735	strong		C=O	stretching	δ-lactone
1745	strong		C=O	stretching	cyclopentanone
1740-1720	strong		C=O	stretching	aldehyde
1730-1715	strong		C=O	stretching	α,β-unsaturated ester

1725-1705	strong	C=O	stretching	aliphatic ketone
1720-1706	strong	C=O	stretching	carboxylic acid
1710-1680	strong	C=O	stretching	conjugated acid
1710-1685	strong	C=O	stretching	conjugated aldehyde
1690	strong	C=O	stretching	primary amide
1690-1640	medium	C=N	stretching	imine / oxime
1685-1666	strong	C=O	stretching	conjugated ketone
1680	strong	C=O	stretching	secondary amide
1680	strong	C=O	stretching	tertiary amide
1650	strong	C=O	stretching	δ-lactam
1678-1668	weak	C=C	stretching	alkene
1675-1665	weak	C=C	stretching	alkene
1675-1665	weak	C=C	stretching	alkene
1662-1626	medium	C=C	stretching	alkene
1658-1648	medium	C=C	stretching	alkene
1650-1600	medium	C=C	stretching	conjugated alkene
1650-1580	medium	N-H	bending	amine
1650-1566	medium	C=C	stretching	cyclic alkene
1648-1638	strong	C=C	stretching	alkene
1620-1610	strong	C=C	stretching	α,β-unsaturated ketone
1550-1500 1372-1290	strong	N-O	stretching	nitro compound
1465	medium	C-H	bending	alkane
1450 1375	medium	C-H	bending	alkane
1390-1380	medium	C-H	bending	aldehyde
1385-1380 1370-1365	medium	C-H	bending	alkane
1440-1395	medium	O-H	bending	carboxylic acid
1420-1330	medium	O-H	bending	alcohol
1415-1380 1200-1185	strong	S=O	stretching	sulfate
1410-1380 1204-1177	strong	S=O	stretching	sulfonyl chloride
1400-1000	strong	C-F	stretching	fluoro compound
1390-1310	medium	O-H	bending	phenol
1372-1335 1195-1168	strong	S=O	stretching	sulfonate
1370-1335 1170-1155	strong	S=O	stretching	sulfonamide
1350-1342 1165-1150	strong	S=O	stretching	sulfonic acid
1350-1300 1160-1120	strong	S=O	stretching	sulfone

1342-1266	strong		C-N	stretching	aromatic amine
1310-1250	strong		C-O	stretching	aromatic ester
1275-1200 1075-1020	strong		C-O	stretching	alkyl aryl ether
1250-1020	medium		C-N	stretching	amine
1225-1200 1075-1020	strong		C-O	stretching	vinyl ether
1210-1163	strong		C-O	stretching	ester
1205-1124	strong		C-O	stretching	tertiary alcohol
1150-1085	strong		C-O	stretching	aliphatic ether
1124-1087	strong		C-O	stretching	secondary alcohol
1085-1050	strong		C-O	stretching	primary alcohol
1070-1030	strong		S=O	stretching	sulfoxide
1050-1040	strong	broad	CO-O-CO	stretching	anhydride
995-985 915-905	strong		C=C	bending	alkene, monosubstituted
980-960	strong		C=C	bending	alkene, disubstituted (trans)
895-885	strong		C=C	bending	alkene, vinylidene
850-550	strong		C-Cl	stretching	halo compound
840-790	medium		C=C	bending	alkene, trisubstituted
730-665	strong		C=C	bending	alkene, disubstituted (cis)
690-515	strong		C-Br	stretching	halo compound
600-500	strong		C-I	stretching	halo compound
			Substituted benzene		
880 ± 20 810 ± 20	strong		C-H	bending	1,2,4-trisubstituted
880 ± 20 780 ± 20 (700 ± 20)	strong		C-H	bending	1,3-disubstituted
810 ± 20	strong		C-H	bending	1,4-disubstituted or 1,2,3,4-tetrasubstituted
780 ± 20 (700 ± 20)	strong		C-H	bending	1,2,3-trisubstituted
755 ± 20	strong		C-H	bending	1,2-disubstituted
750 ± 20 700 ± 20	strong		C-H	bending	monosubstituted benzene derivative