People's Democratic Republic of Algeria
Ministry of Higher Education and Scientific Research

**Amar Telidji University - Laghouat**

**FACULTY: SCIENCES**

**DEPARTMENT: AGRONOMIC SCIENCES**

# MASTER THESIS

**Presented by: DJERFAF Fatima Zahra and HADJADJ Abderrahmane**

**FIELD: NATURAL AND LIFE SCIENCES (SNV)**

**SECTOR: FOOD SCIENCE**

**OPTION: AGRI-FOOD AND QUALITY CONTROL**

***Theme***

# Study of some physicochemical and microbiological characteristics of some date-derived products

**Board of Examiners:**

| Full name | Grade | Quality |
|---|---|---|
| Mrs. ALLALI Khadidja | Associate Professor B | President |
| Mrs. LOUNICI Safia | Assistant Professor A | Examiner |
| Mrs. ZAAMOUM Miyada | Professor | Supervisor |
| Miss HOUICHER Aicha | Assistant Professor B | Assistant Supervisor |

Class: June 2023

**بسم الله الرحمن الرحيم**

«وَمِن ثَمَرَاتِ ٱلنَّخِيلِ وَٱلأَعْنَابِ

تَتَّخِذُونَ مِنْهُ سَكَراً وَرِزْقاً حَسَناً إِنَّ

فِي ذَٰلِكَ لآيَةً لِّقَوْمٍ يَعْقِلُونَ»

النحل الآية 67 ۩

# Acknowledgment

أول مشكور هو الله عز وجل على نعمة الصبر و القدرة على إنجاز هذا العمل، فالحمد لله على هذه النعم.

نشكر على وجه الخصوص الأستاذة الفاضلة **زعموم ميادة** حفظها الله ورزقها بركة في العمر لتكرمها بالنصح والإرشاد لإتمام هذا العمل، الشكر موصول إلى الأستاذة **هويشر عائشة** على مساعدتها لنا للمضي قدما و القيام بهذا العمل.

الشكر إلى أعضاء اللجنة المناقشة المؤقرة، الأستاذة **علالي خديجة** و الأستاذة **لونيسي صفية** .

جزيل الشكر و التقديرلجميع من وجهونا وأرشدونا في بحثنا ،لا سيّما الأستاذ **بن عمار إبراهيم** و الأستاذ **بن عجيلة عبدالرحمن**، و موجه كذلك الى كل **الأساتذة** الذين تتلمذنا على ايديهم و كان لهم فضل علينا و نخص بالذكر الأستاذ الفاضل **قوجال ياسين** ، نتقدم بكلمة الشكر **لمهندسي المخابر** بقسم علوم الزراعة على كرمهم وصبرهم الكبير.

الشكر إلى كل من ساهموا بشكل مباشر أو غير مباشر وساعدونا ولو باليسير لإنجاز هذا العمل، بارك الله فيهم وجزاهم كل خير.

# *Dedication*

أُهدَّي تخرُجَي وعَملَي و حَصّاد ما زرّعَتُه في سَبيل العلم إلى ذلك الإنسان العَظيم **أَبّي** غفر الله لك و رَحِمك و أَنس وحشَتك و جمَعنا بِك بِجنتِه.

لكِ يا سَّيّدة نساء الكون، إلى **أُمّي** الحَبِيبّة عَضُدَي و سَندَي حفِظَها الله و رزقّها بَرَكة في العمر والعمل.

إلى مَن إنتَظرُوا قِطَاف ثمّرة جُهدِي طَوِيلاً، فكَانوا شُركاء كل بَسمة وحَسرة; أحباب قلبي **إِخْوَتِي.**

إلى الزَّمِيلة المُحتَرمة **فاطمة الزهراء** التي كانت خيرَ مُعين.

إلى كل **أصدقَائِي ومعَارِفي** الذين تَطَيَّبَ الأوقات بصُحبتِهِم.

إلى كل **زُملائِي**، ولكل مَن عرفتُه أثناء دِراسْتي وفقكُم الله لما يُحِب ويرضّى.

***عبدالرحمٰن***

# *Dedication*

ولأن من لا يشكر الناس لا يشكر الله، فإن هذا العمل ما كـان ليـرى النور لولا فضل من الله و منة منه أولا و أخيرا وبعد.

أهدي هذا العمل المتواضع الذي يمثل عنوان كبريائي خلال مسيرتي الدراسية الى اللذان لم يبخلا بذرّة جهد وطاقة واحدة في سبيلي تقدّمي وزيادة تحصيلي العلمي **أمي و أبي** حفظه الله في غربته.

إلى **أخوتي** السند و العضد و اختي **سيرين** الذين شجعوني دائمًا لتقديم أفضل ما عندي.

الى **أجدادي** الأعزاء الاحياء منهم و الاموات الذين رافقوني بالدعاء وانتظروا هذا اليوم بصبر.

إلى زميل عملي المحترم **عبد الرحمن.**

الى السيد **بوعبيده.ع**. الذين كان مصدرًا للمعلومات والتحفيز بالنسبة لي خلال فترة التربص.

الى كل من ساعدني طوال الفترة الماضية من الأقارب.

الى كل **الأصدقاء** خير صحبة وخير زملاء الذين كانوا لنا الأنس خلال المسيرة الدراسية بارك لهم وبارك فيهم.

دفعة 2023/2022

***فاطمة الزهراء***

## List of Abbreviations

| | |
|---|---|
| **°A** | Acetic degree |
| **°B** | Brix degree |
| **°C** | Degree Celsius |
| **AFNOR** | French Association for Standardization |
| **ANOVA** | One way Analysis Of Variance |
| **APFA** | Accession to the Agricultural Land Ownership |
| **Cfu/ml** | Colony forming unit per milliliter |
| **E.C** | Electrical conductivity |
| **FAO** | Food and Agriculture Organization |
| **g** | Gram |
| **g/l** | Gram per liter |
| **ha** | Hectare |
| **IANOR** | Algerian Institute for Standardization |
| **ISO** | International organization for standardization |
| **JORA** | Official Journal of the Algerian Republic |
| **kg** | Kilogram |
| **Mg** | Milligram |
| **mS/cm** | Millisiemens per centimeter |
| **NaOH** | Sodium hydroxide |
| **NF** | French standard |
| **pH** | Potential of hydrogen |
| **PNDA** | National Program of Agriculture Development |
| **v/v** | Volume per volume |
| **VRBL** | Violet Red Bile Lactose Agar |
| **w/v** | Wight per volume |

## List of Figures

## List of Tables

**Full name:** DJERFAF Fatima Zahra **Directed by :** ZAAMOUM Miyada
HADJADJ Abderrahmane

**Theme:** Study of some physicochemical and microbiological characteristics of some date-derived products.

**Abstract**

Algeria is traditionally a significant producer of dates. This abundant production, dates have undergone essential technical processes to obtain new products that are easy to market. This work explores the physicochemical and microbiological characteristics of dates-derived products, namely vinegar, bioethanol, syrup, and liquid sugar. The study was carried out on 7 molasses samples (5 semi-industrial and 2 traditional) and 4 vinegar samples. The study examines two vinegar production methods, a semi industrial approach and a traditional approach. Moreover, the study examines the production of bioethanol and liquid sugar.

The physicochemical properties of bioethanol and liquid sugar, including pH, Brix degree (°B), and electrical conductivity, are examined to evaluate their quality and suitability for various applications.

The results showed that the qualities of traditional syrup are much greater than those of industrialized ones (90% of traditional and semi industrial samples comply with international standards). Also, the quality of semi industrial vinegar demonstrates acceptable performance compared to established standards, with an average similarity rate of 83%.

Microbiological analysis of the products indicated the presence of four different fungi species after 7 days, namely *Fusarium* sp., *Aspergillus niger*, *Aspergillus flavus*, and *Penicillium* sp. These fungi species were found in vinegar and syrup samples. These findings are crucial for understanding the microbial composition and potential risks of date-derived products.

**Keywords:** Date-derived products, vinegar, syrup, bioethanol, liquid sugar.

**الإسم و اللقب :** جرفاف فاطمة الزهراء **المؤطر(ة):** زعموم ميادة

حجاج عبدالرحمن

**العنوان :** دراسة بعض الخصائص الفيزيوكيميائية والميكروبيولوجية لبعض المنتجات المشتقة من التمور.

**ملخص**

تعتبر الجزائربلد منتجًا رئيسيًا للتمور ، بسبب هذا الإنتاج الوفير ، خضعت التمور لعمليات تقنية أساسية لتثمين و الحصول على منتجات جديدة يسهل تسويقها. يبحث هذا العمل في الخصائص الفيزيوكيميائية والميكروبيولوجية للمنتجات المشتقة من التمور ، وهي الخل، الإيثانول الحيوي، الدبس والسكر السائل. أجريت الدراسة على 7 عينات من دبس التمر (5 شبه صناعي و 2 تقليدي) و 4 عينات من الخل. تبحث الدراسة في طريقتين لإنتاج الخل ، ، طريقة شبه صناعية وطريقة تقليدية. بالإضافة إلى إنتاج الإيثانول الحيوي والسكر السائل..

يتم فحص الخصائص الفيزيوكيميائية للإيثانول الحيوي والسكر السائل ، بما في ذلك درجة الحموضة (pH) ودرجة البريكس (Brix) و الناقليّة النوعية (Electrical conductivity) ، لتقييم جودتها ومدى ملاءمتها للتطبيقات المختلفة.

أظهرت النتائج أن نوعية دبس التمر التقليدي أفضل من تلك الصناعية (90٪ من العينات التقليدية وشبه الصناعية تتوافق مع المعايير الدولية). أيضا، يظهر الخل شبه الصناعي جودة جيدة وفقًا للمعايير المتبعة بنسبة تشابه تعادل 83%.

تشير التحاليل الميكروبيولوجية للمنتجات إلى وجود أربعة أنواع مختلفة من الفطريات بعد 7 أيام وهي: *Fusarium* sp.، *Aspergillus niger* ، *Aspergillus flavus* و. *Penicillium* sp تم العثور على هذه الكائنات حية في عينات من الخل والدبس. هذه النتائج حاسمة لفهم التركيب الميكروبيولوجي والمخاطر المحتملة للمنتجات المشتقة من التمر.

**الكلمات المفتاحية:** منتجات التمر الثانوية ،الخل ، الدبس ، الإيثانول الحيوي ، السكر السائل.

**Nom et Prénom:** DJERFAF Fatima Zahra
HADJADJ Abderrahmane
**Encadrante :** ZAAMOUM Miyada

**Thème :** Etude de quelques caractéristiques physicochimiques et microbiologiques de quelques produits dérivés des dattes.

**Résumé**

L'Algérie est un grand pays producteur de dattes .En raison de cette production abondante, les dattes ont subi des processus techniques indispensables pour obtenir de nouveaux produits faciles à commercialiser. Ce travail examine les caractéristiques physicochimiques et microbiologiques des produits dérivés des dattes, à savoir le vinaigre, le bioéthanol, la mélasse et le sucre liquide. L'étude a été réalisée sur 7 échantillons de mélasses (5 semi-industriels et 2 traditionnelles) et 4 échantillons de vinaigre. L'étude examine deux méthodes de production de vinaigre, une approche semi-industrielle et une approche traditionnelle. De plus, l'étude examine la production de bioéthanol et le sucre liquide.

Les propriétés physicochimiques du bioéthanol et du sucre liquide, notamment le pH, le degré Brix (°B) et la conductivité électrique, sont examinées pour évaluer leur qualité et leur aptitude à diverses applications.

Les résultats ont montré que les qualités des mélasses traditionnelles sont beaucoup plus importantes que celles qui sont industrialisées (90% des échantillons traditionnelles et semi-industriels sont conformes aux normes internationales). Aussi, La qualité du vinaigre semi-industriel démontre des performances acceptables par rapport aux normes établies, avec un taux de similarité de 83 %.

L'analyse microbiologique des produits a indiqué la présence de quatre espèces des champignon différentes après 7 jours : *Fusarium* sp., *Aspergillus niger, Aspergillus flavus* et *Penicillium* sp. Ces espèces de levure ont été trouvées dans des échantillons de vinaigre et de mélasse. Ces découvertes sont cruciales pour comprendre la composition microbienne et les risques potentiels des produits dérivés des dattes.

**Mots clés :** Produits dérivés des dattes, vinaigre, mélasse, bioéthanol, sucre liquide.

## Table of Contents



### *Chapter II : Date's derived products*

***Chapter III : Materials and methods***

***Chapter IV : Resualts and discussion***

# *Introduction*

## Introduction

Palm date trees (*Phoenix dactylifera* L.) is an ancient fruit crop indigenous to the Arabian Peninsula, North Africa, and the Middle East **(Chao & Krueger, 2007).** It encompasses about 200 genera and more than 2,500 species **(Hadrami & Al-Khayri, 2012).** Date palm is a monocotyledon plant that passes through the stages of *hababauk, kimri, khalal, rutab,* and *tamer* during ripening. Date contains water, sugar, protein, fat, pectin, ash, crude fiber, and polyphenols **(Ashraf & Hamidi-Esfahani, 2011).**

Besides their fruit, date palms are highly valued as ornamental plants. As a result of its nutritional value, high yield, and long lifespan. The Date palm has been referred to as a "tree of life". Dates are a major source of income and a staple food for local populations in many countries. The economies, societies, and environments of those countries have benefited greatly from their presence **(Chao & Krueger, 2007).**

The date palm fruit consists of a single seed enclosed within a fibrous parchment, a fleshy mesocarp, and a skin (pericarp) covering the fruit. The pulp of dates contains easy-digestible sugars (70%), mainly glucose, sucrose, and fructose. Date pulp also contains dietary fiber, fewer proteins, and fewer fats **(Al-Farsi & Lee, 2008).**

Algeria is one of the most prominent world date producers, occupying fourth place with 12% of total world production. Algeria's date cultivars are numerous and estimated at more than 940 **(Hannachi *et al.*, 1998).** These resources could be better exploited. In light of this, finding more efficient methods to exploit and valorize dates and palm waste has become a priority.

However, the valorization of date derived products has become an important area of research, focusing on developing sustainable processes that can convert date waste into value-added products. Date palm products include semi-finished and ready-to-use products derived from the date, such as jam, juice, syrup, and fermented beverage.

Several reports show that many bioactive compounds can be extracted from these date-derived products, thereby adding industrial value, which could compensate for the economic loss from under-grading and/or deterioration dates.

In this context, this study takes place. We aim to investigate and analyze the physicochemical and microbiological characteristics of selected date-derived products such as vinegar, bioethanol, syrup, and liquid sugar.

We summarize the main contributions of this thesis as follow:

- The production of different secondary by-products from dates, namely vinegar, bioethanol, syrup, and liquid sugar that meets to microbiological and physicochemical quality standards.
- Evaluation the quality of different date's derived products produced in the laboratory.
- Isolation and identification of yeast strains from date's derived products.

Besides that, the findings of this thesis will contribute to the development of production processes for date-derived products, which can help to reduce waste and generate new economic opportunities.

# *Bibliographic Part*

# ***Chapter I***

*Date palm*

## 1.1 The Date Palm

The date palm (*Phoenix dactylifera* L.) tree belongs to the Arecaceae family (Angiosperms, monocotyledon), consisting of about 200 genera and more than 2,500 species **(Eoin, 2016).** Phoenix belongs to the tribe Phoeniceae, the subfamily Coryphoideae and the Arecaceae (Palmae) **(Jain *et al.*, 2011).** It is one of the oldest plants cultivated in the world. Its cultivation occurred 5000 years ago **(Chao & Krueger, 2007).**

The species' name was inspired by the finger-like shape of the fruit and the genus from the legendary bird of Ancient Greece. It is a long-lived monocotyledonous species and one of the tallest domesticated trees **(El Hadrami & Al-Khayri, 2012).** It comprises three parts: a thin-crusted flesh, a date pit, and a cap **(Ashraf & Hamidi-Esfahani, 2011) (Fig. 1).**

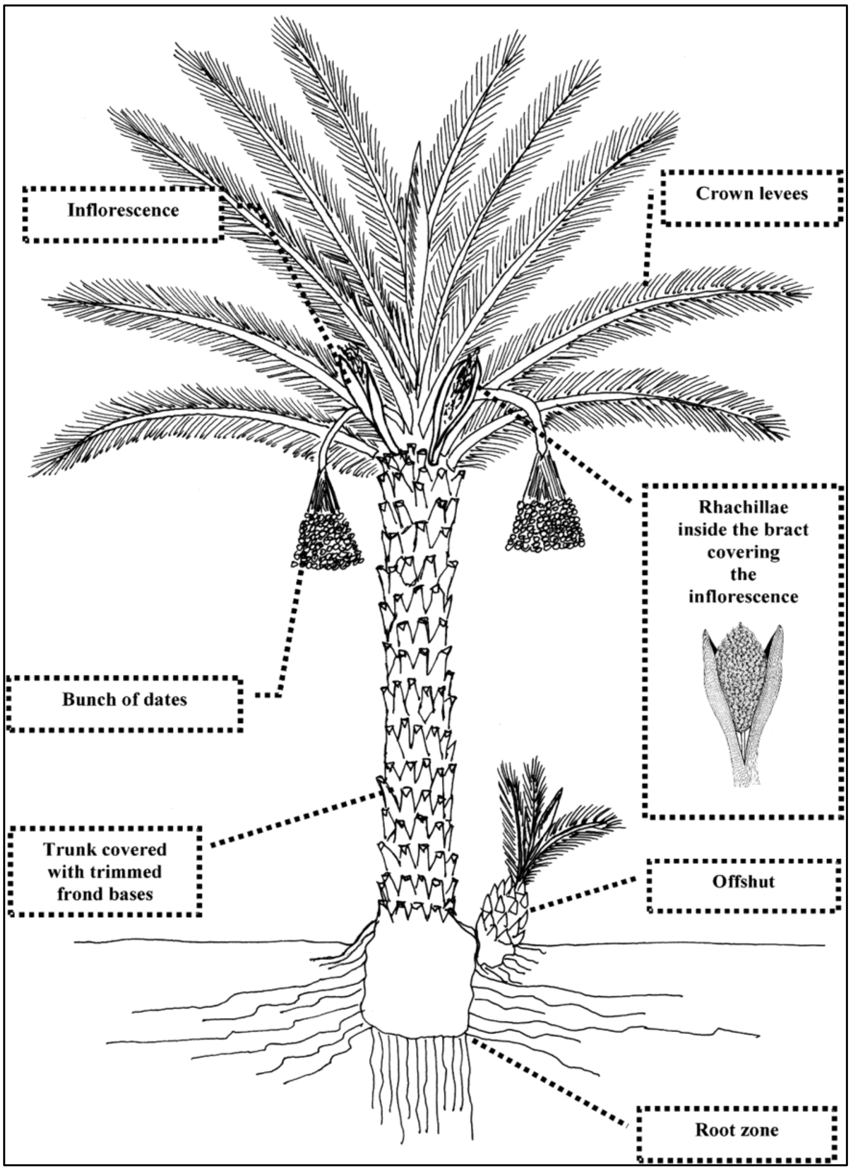



**Figure 1:** Diagrammatic construction of a date palm with its root system **(Paszke, 2019)**

Date palm has long been one of the most important fruit crops in the arid regions of the Arabian Peninsula, North Africa, and the Middle East. During the past three centuries **(Chao & Krueger, 2007).** Over 100 million trees are grown worldwide on an estimated 1 million ha **(El Hadrami & Al-Khayri, 2012).** Date palm is resistant to bad climatic conditions and can endure a wide range of temperatures (from –6 to 50°C). It is also resistant to the salinity of water and soil. The most appropriate area for this plant to grow is arid regions with hot and dry climates and limited rainfall **(Ashraf & Hamidi-Esfahani, 2011).**

Date palm fruit contains many carbohydrates, salts, vitamins, and minerals. The fats comprise 14 fatty acids, proteins composed of 23 different amino acids, and a considerable quantity of dietary fiber in the fruit **(Al-Shahib & Marshall, 2003).** The phytochemical investigation of this plant has shown that its fruits contain anthocyanins, procyanidins, phenolics, flavonoids, carotenoids, and sterols **(Ali *et al.*, 2020).**

The date palm is unquestionably the keystone tree species of agriculture in semiarid and arid lands of the Near East and North Africa. It is now successfully grown commercially in South Asia, Southern Africa, Iberia, the Americas, and Australia **(Krueger, 2021).** It is considered the main crop of traditional and modern Algerian Saharan agriculture. The economy of the southern provinces (Wilayates) is based primarily on date palm cultivation and the utilization of its fruit **(Bouguedoura *et al.*, 2015).**

The heavenly religions honored the date palm and showed concern for its cultivation and care. However, no other religion has stressed the regard for dates and the date palm as much as Islam. **The Holy Quran** mentions dates and the date palm in many suras (chapters) and verses. **Prophet Muhammad (peace be upon him)** is reported to have said that the best property is the date palm, that dates cure many disorders, and he urged Muslims to eat dates and tend the date palm **(Zaid & De J Arias-Jiménez, 1999).**

### 1.1.1 Date production

#### 1.1.1.1 Worldwide

The date palm can be cultivated in all five continents of the world. However, the central region of production is the Middle East and North Africa **(Ashraf & Hamidi-Esfahani, 2011).**

The available data on date production published by the **FAO (2022)** indicate that worldwide date production has significantly increased from 8,396,455 tons in 2017 to 9,656,377 tons in 2021. According to **FAO statistics**, Egypt, Saudi Arabia, Iran, Algeria, and Iraq were the top 5 producing countries of dates in 2021 **(FAO, 2022) (Table 1).**

**Table 1:** Production of Dates: Top 10 producers in 2021 **(FAOSTAT, 2022)**

| Country | Production in tons |
|---|---|
| Egypt | 1 747 714,68 |
| Saudi Arabia | 1 565 830,00 |
| Iran | 1 303 716,84 |
| Algeria | 1 188 803,00 |
| Iraq | 750 225,00 |
| Pakistan | 532 879,55 |
| Sudan | 460 096,55 |
| Oman | 374 200,00 |
| United Arab Emirates | 351 077,48 |
| Tunisia | 345 000,00 |

#### 1.1.1.2 In Algeria

A total of 1,188,803 t of date was produced by Algeria in 2021 **(FAO, 2022).** The curve shows the production per thousand quintals in **(Fig. 2).** Statistics show that date production in Algeria is mainly concentrated in the country's southeastern part, which is responsible for 76 % of national production. The province of Biskra ranks first with nearly 31 %, followed by El Oued (27 %) and Ouargla with 18 % **(Bouguedoura *et al.*, 2015).**

According to **Bouguedoura** et **al. (2015),** "the increase in date production is due to new plantations established within the framework of the Accession to the Agricultural Land Ownership (APFA) and the National Program of Agriculture Development (PNDA), as well as to the strong recent interest given to this crop."

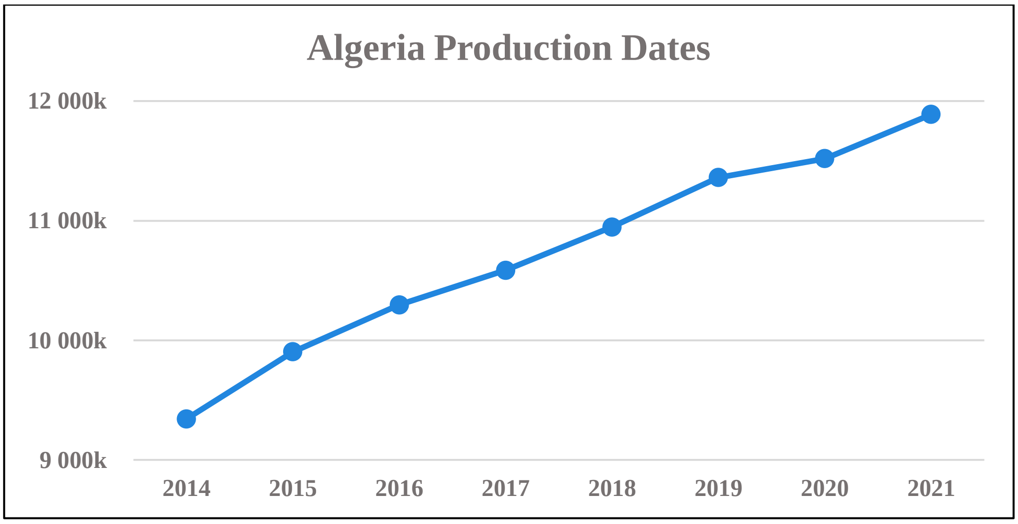



**Figure 2:** Annual average of production of date in Algeria **(FAO, 2022)**

## 1.2 Dates

### 1.2.1 Description of the date

Date palm fruits are berries containing a single seed enclosed by a fibrous parchment-like endocarp, fleshy mesocarp, and the fruit skin (pericarp). Different regions give different dates, which vary in shape, size, and weight. Also, they can vary in their organoleptic, physical, and chemical characteristics **(Al-Alawi *et al.*, 2017) (Fig. 3).**

The mesocarp, representing most fruit pulp, consists of enlarged parenchymatous cells and is divided into outer-mesocarp and inner mesocarp intermediated by 3–10 layers of stanniferous cells **(Shomer et al., 1998).**

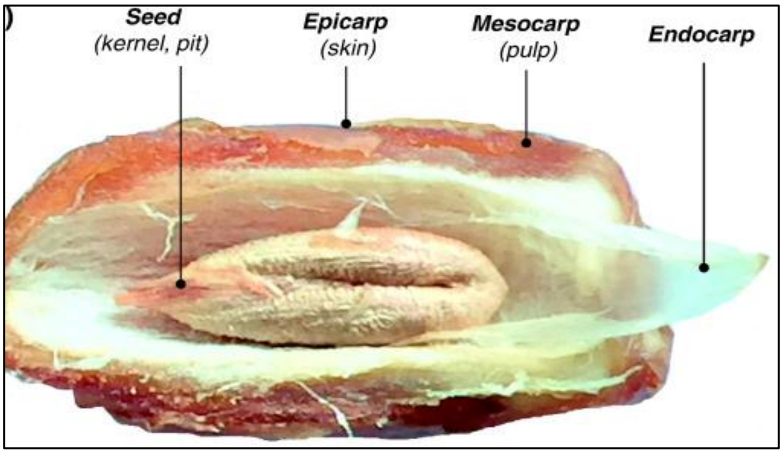



**Figure 3:** The anatomy of the date fruit at *Tamar* stage **(Ghnimi et al., 2017)**

### 1.2.2 Stages of evolution

The growth rate and development of seeded fruit follow a sigmoid growth curve. The date fruit goes through four distinct ripening stages. These four stages are usually referred to in terms derived from Iraqi Arabic as ‘‘*Kimri*,’’ ‘‘*Khalal*’’ (sometimes referred to as ‘‘*Bisr*’’), ‘‘*Rutab*," and *"Tamar"* to represent the immature green, the mature full colored, the soft brown, and the complex raisin like stages respectively **(Reuveni, 1986).**

According to **Ashraf & Hamidi-Esfahani, (2011),** the stage of development are **(Fig. 4):**

***Hababuk:*** This stage appears after pollination;

***Kimiri:*** The fruit enlarges and elongates (increases in weight and volume), is associated with high humidity levels, accumulations of reducing sugars, and higher high acids;

***Khalal***: It is characterized by a rapid increase in sugar, sucrose, and solid matter content, while the acidity and humidity levels decrease rapidly.

***Routab:*** The date becomes soft and loses its astringency (tannins under the skin precipitate in insoluble form);

***Tamr* or *Ripe:*** The date has almost wholly lost its water content.

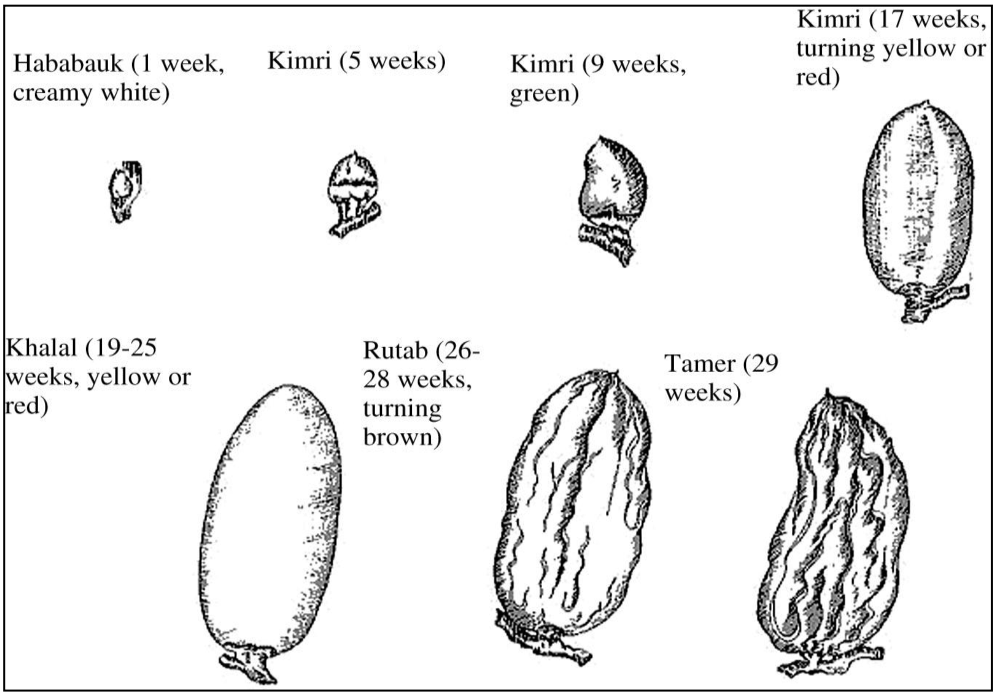



**Figure 4:** Forming and ripening of date **(Barreveld, 1993)**

### 1.2.3 Biochemical Composition of date

The date has much nutritive value and can play an influential role in providing the nutritional needs of humans. Each kilogram of fresh date contains approximately 1570 calories of energy, whereas dry date contains more than 3000 calories per kg **(Ashraf & Hamidi-Esfahani, 2011).** Date, which is very sweet, comprises about 50–88% of the total weight according to cultivar, stage of ripening, and water content, as shown in (**Table 2).**

**Table 2:** Main components of date **(Ashraf and Hamidi-Esfahani, 2011).**

| **Constituents** | **Quantity (%)** |
|---|---|
| Water | 5-20 |
| Sugar | 44-88 |
| Protein | 1-7 |
| Fat | 0,1-0,5 |
| Pectin | 1-4 |
| Ash | 1-2,5 |
| Crude fiber | 3-18 |
| Polyphenol | 3 |

#### A. Water content

The water content differs from class to class; according to **Munier (1973),** soft dates have a humidity level of more than 20%. On the other hand, the dry dates have humidity levels that are under 20%. The consistency of semi-dry dates has a humidity range of 20–30% **(Table 3).**

**Table 3:** Water content of some varieties of Algerian dates **(Belguedj, 2001).**

| Classes | Varieties | Levels of moisture (%) |
|---|---|---|
| Soft date | *Ghars* | 25.4 |
| Semi-dry date | *Deglet-Nour* | 22.6 |
| Dry date | *Mech-Degla* | 13.7 |

**B. Sugars**

Sugars comprise about two-thirds of date flesh, with water about one-fifth **(Ashraf & Hamidi-Esfahani, 2011).** Carbohydrates are the major chemical constituents of dates, including mainly reducing sugars such as glucose and fructose, non-reducing sugars such as sucrose, and small amounts of polysaccharides such as cellulose and starch **(Al-Shahib & Marshall, 2003).**

**C. Protein**

According to **Razi (1993),** "Dates are relatively low in protein. As a percentage of dry weight, it ranges from 1.5% to 2%". According to Ashraf, about 1–7% of protein can be found in date, but despite the low quantity, the protein contains essential amino acids the human body needs **(Ashraf & Hamidi-Esfahani, 2011).**

In **Al-Shahib and Marshall**'s study **(2003)**, date contains 23 different amino acids, including some not commonly found in oranges, apples, and bananas.

**D. Fat**

The date fruit contains little fat; their rates vary between 0.43 and 1.9% of the fresh weight **(Djouab, 2007).** It usually varies from 0.5% at the *khalal* stage to 0.1% at the *tamer* stage of maturity. Fat is usually concentrated in the crust, with date flesh having about 0.1–0.5% fat. Fat is more critical in protecting the fruit than in the nutritional value of the date flesh **(Ashraf & Hamidi-Esfahani, 2011).**

**E. Fiber**

Date fruit contains between 8.1 and 12.7% of its dry weight in fiber **(Al-Shahib & Marshall, 2003).** Several of these compounds, mainly cellulose, are insoluble **(Boukhiar, 2009).** They contain cellulose, hemicellulose, lignin, and pectin. Their percentages vary depending on the cultivars and the ecological conditions **(Aatef & Nadif, 1997).**

**F. Minerals and Vitamins**

The date palm contains a suitable concentration of calcium, potassium, phosphorus, and selenium which are very important for the human body and metabolic operations in the human cells **(Ashraf & Hamidi-Esfahani, 2011).**

The date palm fruit contains many kinds of vitamins like Vitamin A, B1, B2, and Nicotinic acid with concentrations of 0.04, 0.08, 0.05, and 2.20 mg/100g, respectively **(Hernández-Pérez *et al.*, 2005) (Table 4).**

**Table 4:** Minerals and vitamins per 100 g of pulp **(Benchelah & Maka, 2008).**

| Minerals | | Vitamins | |
|---|---|---|---|
| Potassium | 670 to 750 mg | B3 | 1,7 mg |
| Calcium | 62 to 65 mg | B5 | 0,8 mg |
| Magnesium | 58 to 68 mg | B2 | 0,1 mg |
| Iron | 3 mg | B6 | 1,15 mg |
| Phosphorus | 3 mg | Vitamin C | It is still present in small quantities on fresh dates, but almost all of it is absent from the dry dates. |
| Copper | 3 mg | | |
| Zinc | 3 mg | | |
| Manganese | 3 mg | | |
| Sodium | 1 to 3 mg | | |

**G. Other Components**

The necessary inherent enzymes of date are invertase, polygalacturonase, pectin methyl esterase, cellulase, and polyphenol oxidase. Polyphenols are also one of the constituents of date, making up 3% (dry basis) of flesh **(Ashraf & Hamidi-Esfahani, 2011).**

### 1.2.4 Classification of dates

Date fruits are generally classified into soft, semi-dry, and dry varieties. This classification depends mainly on the moisture and carbohydrate levels, including soluble sugars and fiber, which influence other quality characteristics, such as hardness, viscosity/stickiness, and textural parameters **(Kamal-Eldin & Ghnimi, 2018) (Table 5).**

**Table 5:** Classification of dates according to their consistency **(Espiard, 2002).**

| Consistency | Characteristic | Varieties and countries |
|---|---|---|
| Soft | -Moisture ≥ 30%.<br>-Rich with the inverted sugar (glucose and fructose). | *Ghars* (Algeria), *Ahmer* (Mauritania), *Kashram* and *Miskhrani* (Egypt and Saudi Arabia) |
| Semi-dry | -20% <H% < to 30%<br>-50% Saccharose and 50% glucose + fructose | *Deglet Nour* (Algeria), *Mahjoul* (Mauritania), *Sifri* and *Zahidi* (Saudi Arabia) |
| Dry | H% < 20%<br>-Rich with saccharose | *Degla-Beida* and *Mech Degla* (Tunisia and Algeria) and *Amsrie* (Mauritania) |

### 1.2.5 Nutritional value and therapeutic options

The earlier investigation reported that date fruits have the highest concentration of total polyphenols among the dried fruits due to the greater exposure to sunlight and extreme temperature for date fruits compared to other fruits **(Vinson *et al.*, 2005).**

In a different study, **Saafi *et al.* (2011)** looked at the effect of aqueous date fruit extract (*Deglet Nour* variety) on the protection against oxidative damage and hepatotoxicity brought on by subchronic exposure to dimethoate on rat liver, and the results revealed that the extract reversed the liver's degeneration.

Moreover, the health benefits of dates have been highlighted in Islam. In this regard, the use of *Ajwa* dates, a special kind of date homegrown in Arab countries, has been emphasized by **Prophet Mohammad** as he said, "If someone takes seven *Ajwa* dates in the morning, he would not be hurt by either magic or poison" **(Al-Bukhari, 1976).** In addition, **Prophet Muhammed (Peace Be upon Him)** said that the best asset is the date palm, as dates cure several disorders, and he suggested Muslims eat the date and have a tendency to use the date palm **(Zaid & De J Arias-Jiménez, 1999).**

## 1.3 Valorization of date

Date fruits provide an opportunity to improve human health and support economic development. However, this fruit needs to be more valued and requires intensive research to develop a sustainable date palm industry **(Sirisena *et al.*, 2015).** Date processing industries produce date products like date paste, date syrup, date dip, date honey, date jam, date vinegar, and so on **(Ahmed et al., 2005) (Fig. 5).**

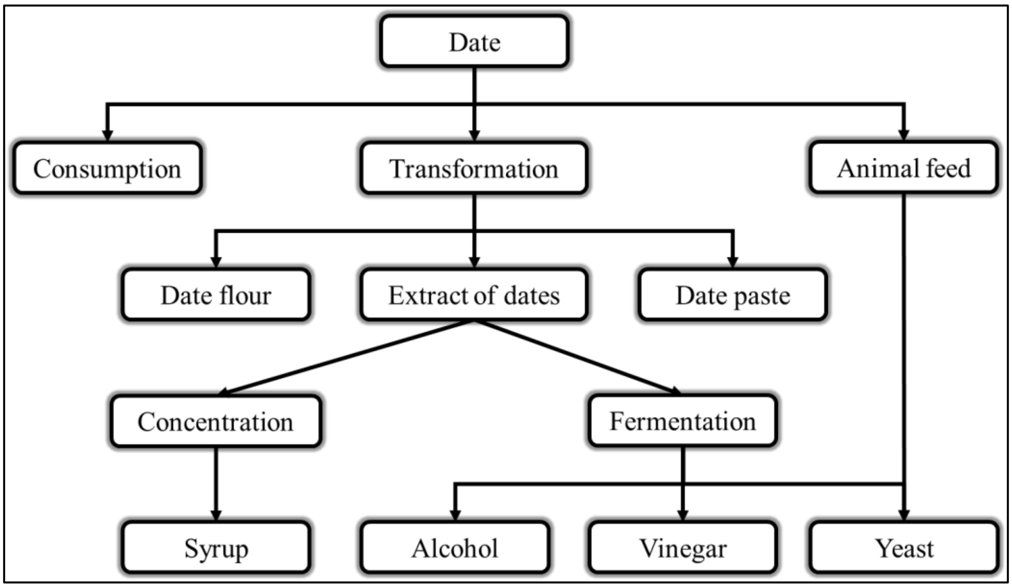



**Figure 5:** Dates processing industries **(Bouzaheur, 2016).**

### 1.3.1 Date paste

Date paste has been used as filler and a sugar substitute in many food formulations. The confectionery industries have been utilizing date paste as one of the significant ingredients for some time. The possibility of using date paste as a replacement for caramel or sugar paste in preparing candy bars was also studied in terms of processing conditions, nutritive value, and organoleptic properties of the prepared date bars, as well as their storability were evaluated **(Razavi & Karazhiyan, 2012).**

### 1.3.2 Date syrup

Date syrup has the same qualities as date juice but is more concentrated. Date syrup, which is also called "*Rub*" or "*Dibs*," is one of the most common date derivatives **(Ashraf & Hamidi-Esfahani, 2011).** It is created using healthy dates to prevent fermentation-related aftertaste **(Chibane, 2008).**

### 1.3.3 Date flour

The dates used in its preparation are either dry or have the potential to become so after drying. This flour, high in sugar, is used to make cookies, pastries, and child foods **(Aït Ameur, 2001).**

### 1.3.4 Date sugar

Date syrups are concentrated and dehydrated to create this product, which is a solid substance. Its color is roughly brown and has more sweetening capacity than glucose **(Chelghoum, 2012).**

### 1.3.5 Date vinegar

Vinegar can be made from dates. The last one was made by cultivating *Saccharomyces uvarum* yeast on date extract **(Ould El-Hadj *et al.*, 2001).** Various studies have demonstrated that date vinegar is good for health in terms of antioxidant properties, cholesterol-lowering, cancer, diabetes, and the prevention of cardiovascular diseases due to its phytochemical properties **(Akarca *et al.*, 2020).**

### 1.3.6 Alcohol

**Barneveld (1993)** claims alcohol is a fermentation by-product created from date sugar. It is possible to create a pure alcohol derivative that is useful for beverages. The chemical and pharmaceutical industries, cleaning goods, and alcoholic lights.

## 1.4 Microbiological aspect of dates and derivatives

### 1.4.1 Microbiology of dates

**a) Microorganisms involved in the deterioration of dates:**

Dates are notably vulnerable to rotting especially after heavy rains during the last stages of their ripening. Spoilage could even take place under poor stock management conditions.

**Ibiso (1988)** isolated 4 fungi from dates stored in bulk, *Aspergillus niger*, *Botryodiplodia theobromae*, and some species of *Rhizopus* and *Penicillium*.

He reported that *B. theobromae* caused a storage disease known as "Black Rot" in dates. The infected fruits are covered after two days of infection with a gray mycelium. The date turns black in the eighth day and as the infection advances, the fruits gradually lose their nutritional values, and in the 20th day the sugar level drops by 98.85%, leading to huge crop loss.

**b) Effect of the preparation on the number of microorganisms**

The rate of contamination depends on several critical factors such as growing weather conditions, size, stage of maturation, and post-harvest conditions such as hygienic practices during processing and handling, storage and transportation **(Sea, 2013).**

Fresh dates can carry non-pathogenic microorganisms, but they contain pathogens/microorganisms, including *Escherichia coli, Salmonella spp. Streptococcus spp., Staphylococcus aureus, Bacillus cereus, Aspergillus spp., Alternaria spp.,* and *Fusarium spp*. **(Hazzani *et al.*, 2014).**

Important cause that can affect the quality of date is the association of osmotolerant yeasts species in date flesh may occur even after packaging and refrigeration **(Risiquat, 2013).**

The effect of the preparation on the number of micro-organisms all operations of peeling, cleaning, disinfection, etc. participate in reducing the number of microorganisms. However, the steps can increase the microbial load or be a source of contamination by foodborne pathogens **(De Cruitis *et al.*, 2002).**

### 1.4.2 Factor influences the growth of date microorganisms

**A. Nutrient content**

Microorganisms require certain essential nutrients for growth and maintenance of metabolic functions. These nutrients include water, which is a source of energy **(Mossel & Thomas, 1988).** Most microorganisms in the food industry are heterotrophic but some can be autotrophic. For growth and nutrition of bacteria, the minimum nutritional requirements are water, a source of carbon, a source of nitrogen and some inorganic salts **(Guiraud, 2012).**

Molds also need water to grow, but when the ambient humidity is high, some molds can attack dry foods such as cold meats, powdered milk or even cereals.

**B. pH**

Most microorganisms, including most human pathogens, are neutrophils, which are organisms that prefer a neutral pH level .the pH notably influences cell permeability and the availability of substrates **(Guiraud, 2012).** Yeasts and molds that tolerate a very wide pH range for growth from (2 to 8.5) with an optimal pH between (4 and 6). When the pH is below 4.5, bacteria growth is inhibited, that's why acidic foods are best (such as lemon, orange, and tomato vinegar) and that's why vinegar is used to preserve certain foods **(Branger *et al.*, 2007).**

**C. Temperature**

In general, the higher the temperature, the easier it is for microorganisms to grow up to a certain point , very high and too low temperatures both obstruct the enzyme processes that microorganisms depend on to survive , most bacteria grow fast between 20 and 45 °c, we distinguish three different groups : Psychrophiles , mesophiles and thermophiles **(Guiraud, 2012).**

- **Psychrophiles:** prefer temperatures from 0°c to 5 degrees Celsius (they have a role in the degradation of dairy products and food stored in the cold).
- **Mesophiles:** like it in the middle, 20-45 degrees Celsius (these are pathogenic bacteria for humans).
- **Thermophiles:** like it hot, thriving in temperatures around or above 55 °C, refrigeration of food is effective in avoiding food poisoning because bacteria in the human body (Mesophiles) multiply slowly at temperatures below 15°C.

**D. Moisture**

All microorganisms need a certain level of water; but a few can survive low humidity conditions by conserving all the water they can find and remaining in a moisture-rich environment. As a general rule, though, the more moisture, the more microorganisms there will be found. Free flow of water is vital for microorganisms so that their cells can exchange substances and their metabolic processes.

**E. Oxygen influence**

Some bacteria grow only in the presence of oxygen, they are called strict aerobes, and others grow only in the absence of oxygen, strict anaerobes. And others that multiply in the absence or presence of oxygen are termed aeroanaerobes. Fungi are aerobic organisms but some can grow anaerobically and therefore deeper into food **(Branger *et al.*, 2007).**

**F. Influence of salt**

Since prehistoric times, salt has been used to preserve meat and fish because it reduces the multiplication of microorganisms. Indeed, salt has the ability to attract water and retain it. Therefore in the presence of salt the microorganisms are deprived of water and can no longer develop. Salt therefore has a bacteriostatic activity on most bacteria on the other hand certain bacteria called halophiles adapt to or require high concentrations of salt to live most molds support very high salt and sugar contents, which implies that charcuterie very salty jams and confectionery can be infected **(Leyral & Vierling, 2007).**

**G. Influence of nutrients**

The composition of food promotes the growth of different types of bacteria, for example, fruit juice that has been opened and left at room temperature promotes the growth of yeasts and will lead to alcoholic fermentation. Also for fresh milk there will be lactic fermentation by bacteria **(Guiraud, 2012).**

### 1.4.3 Microbiological controls and standards

The microbiological quality of our food has long been a major concern for the food industry as well as for control and hygiene services. Food quality and safety are now clearly required by consumers. It even has a regulatory connotation since the appearance of European regulation 178/200. In terms of legislation and standards **(ISO and Codex Alimentarius),** there is a set of laws, decrees and orders intended to Prevents the sale or possession of toxic or corrupt products and to promote quality. Moreover, there are sanitary regulations that set rules for food hygiene including cleanliness of personnel, equipment, places of manufacture, storage or sale, and conditions for food transportation **(Hatanaka *et al.*, 2005).**

- **Objectives of the microbiological analysis or control**

The microbiological control of food aims to control the less apparent but fundamental characteristics of a food product **(Faradji-Hamma, 2016).** It is about safety, that is to say the absence of toxic action, of pathogenic or toxigenic microorganisms as well as the level of populations of spoilage germs. In addition, in the case of preserves, he controls the stability of the products, that is to say the ability of the product not to deteriorate too quickly if the storage conditions are respected. Food products are an environment conducive to microbial growth. Harmful germs are those involved in the spoilage of food products. They affect the organoleptic and commercial hygienic quality of the product at the level of manufacture or conservation **(Andrews, 1996).**

These are the common germs of contamination that can cause serious problems in the industry. Common germs of contamination can have various actions that affect the food and commercial value of products ranging from a change in texture, modification of the coloring (appearance of a parasitic color), synthesis of toxic products or swelling of the containers following an intense release of gas then making the food product unfit for consumption or even dangerous under certain conditions **(Dziezak, 1987; Guiraud & Rosec, 2004).**

Common contamination germs only affect the food and have hygienic repercussions if there are a large number of them. Consequently, the control aims to detect the batches of food products whose level of populations of the flora of contamination exceeds the threshold tolerated by the standards in force.

Given that biological sterility is impossible at the risk of altering the nutritional values of the product, the quantitative factor intervenes at the level of the analysis of this flora.

Most often, qualitative knowledge of the composition of the flora is useless except in the manufacturing circuits to detect the agent responsible for the manufacturing accident **(Dziezak, 1987; Guiraud & Rosec, 2004).** The presence of pathogenic germs is totally undesirable in food products because of the health risk that this type of product poses for the consumer. In this case, it is the qualitative analysis that takes precedence and the detection of a single pathogen, makes the product unfit for consumption **(Bourgeois & Mafrt, 1991).**

- **Parameters to check**

In order to avoid the risk of manufacturing accidents and to locate the origin of the contamination, all the elements used in the manufacture of the product must be subjected to a microbiological analysis **(Ernoul, 2004).** In the food industry, this control concerns:

- The raw material before it enters the factory or even the origin of the raw material for example (dates before undergoing the transformation process).
- Water used for washing and transformation of products. In all cases, the water used in the food industry must be drinkable, including that used for washing premises, utensils and the production line.
- The surfaces of premises and utensils involved in storage, cutting or any other transformation process.
- Ambient air in processing workshops and hangars and storage.
- The staff of the unit who intervenes from the reception of the raw material until the storage of the finished product.
- Storage and packaging material **(Ernoul, 2004).**

### 1.4.4 Storage of dates

Generally speaking, the date packaging segment in the region, as in the other date-growing regions of the country, is mainly export-oriented because the Algerian market consumes little processed products, given that the purchasing power of consumers is low and those eating habits that favor the consumption of fresh produce.

Dates make it possible to obtain a certain number of by-products, which can sometimes be kept for a long time, and some of which are used in the preparation of traditional recipes.

Therefore, it is desirable to convert dates to obtain a certain number of by-products, which sometimes allow them to be preserved for a long time, and some of them are used in the preparation of traditional recipes.

# ***Chapter II***

## *Date's derived products*

## 2.1 Vinegar

### 2.1.1 Defintion

According to the **Codex Alimentarius Commission (1987)**, vinegar is a liquid that is fit for human consumption and produced exclusively from suitable products containing starch or sugars or starch and sugars by double fermentation processes, alcoholic and acetous. Vinegar shall not contain more than 0.5% alcohol, and a stabilizer is not permitted for use in fermented vinegar, according to European law. The vinegar shall not contain less than 50 grams per liter (w/v) of acetic acid.

Vinegar is a French word where “vin” means wine and “aigre" means sour, which is a liquid containing 5% acetic acid in water **(Singh, 2020).** Its color and aroma greatly depend on the material from which it is made **(Kehrer, 1921).**

### 2.1.2 Composition of vinegar

Acetic acid is vinegar's predominant flavoring and antimicrobial component **(Tan, 2005).** Which is found in concentrations higher than 50 g/l, usually expressed as a volume percentage (5% (v/v) of acetic acid or five acetic degrees) **(Mas *et al.*, 2016).** The secondary compounds, such as tartaric acid, succinic acid, and nitrogenous materials, result from the raw material, nutrients, and dilution water **(Follman, 1983).**

The following table **(Table 6)** shows the maximum concentrations of contaminants permitted in vinegar **(Official Journal of People's Democratic Republic of Algeria., 1998).**

**Table 6:** The maximum concentration of contaminants tolerated in the vinegar **(Official journal of People's Democratic Republic of Algeria., 1998).**

| Constituents | Quantity (%) |
|---|---|
| As | 1 |
| Pb | 1 |
| Cu + Zn | 10 |
| Fe | 10 |

### 2.1.3 Principe

Vinegar is produced by a two-stage fermentation process. The first one is the conversion of fermentable sugars to ethanol by yeasts, usually *Saccharomyces* species, and the second is the oxidation of ethanol by bacteria *Acetobacter* species **(Adams, 1998).** The reaction proceeds according to the following equation:

$$2C_2H_5OH \xrightarrow{\text{Anaerobic}} 2CH_3CHO \xrightarrow{\text{Aerobic}} 2CH_3CO_2H + 2H_2O$$

### 2.1.3.1 Alcoholic fermentation

Vinegar production typically involves a first fermentation, where simple sugars in raw material are converted to alcohol by yeasts **(Gullo & Giudici, 2008).** The first fermentation is an alcoholic fermentation that transforms sugars or processed starches into ethanol. This process is performed by yeast, mainly from *Saccharomyces cerevisiae*, although some other species can also perform alcoholic fermentation in part or total. The final result is considered the substrate of the second transformation, from ethanol to acetic acid **(Mas *et al.*, 2016).**

### 2.1.3.2 Acetous fermentation

According to **Mas *et al.* (2016)**, the second process is often called 'acetic' or 'oxidative' fermentation; it is not biochemically a fermentation but oxidation. The proper term is thus 'acetification.' This process involves two-step oxidation, from ethanol to acetaldehyde and further from acetaldehyde to acetic acid, which combines to produce the flavor and typical aroma; small quantities of volatile substances are formed (e.g., ethane, acetaldehyde, ethyl formate, ethyl acetate, isopentyl acetate, butanol, methyl butanol, 3-hydroxy-2 butanone or acetylmethylcarbinol). It vary from vinegar to vinegar depending on the starting material and which, because of their characteristics, produce vinegar with a variety of odor, taste, color, and other properties **(Plessi, 2003).** Whereby the end of this process requires an electron acceptor, with molecular oxygen being the most common. Acetic acid bacteria are the microorganisms responsible for this process **(Mas *et al.*, 2016) (Fig. 6).**

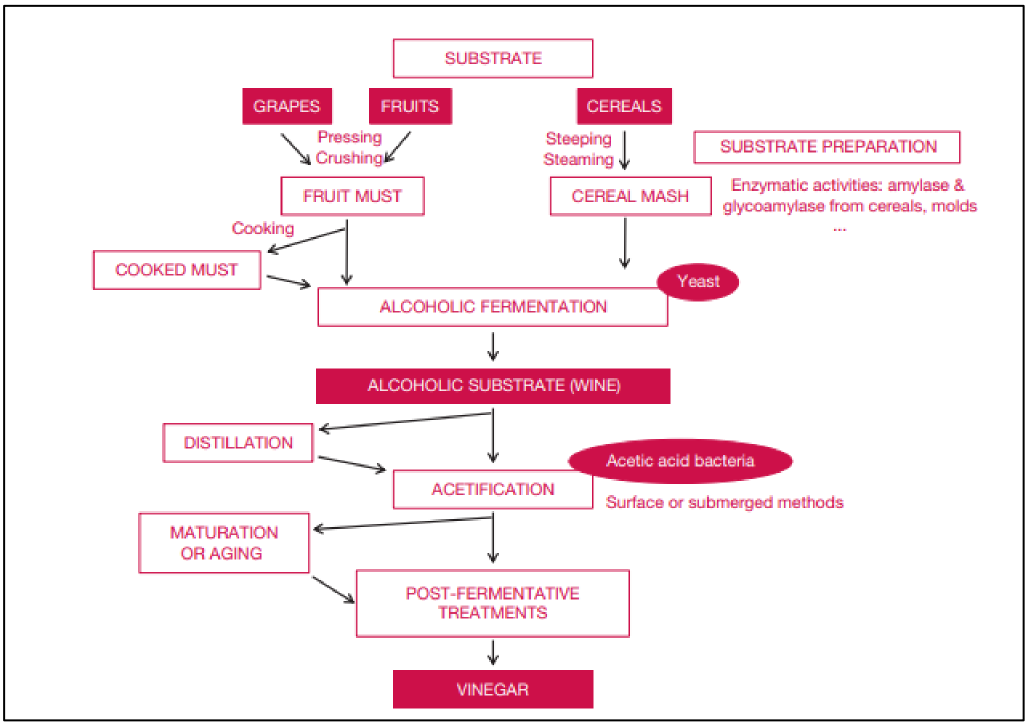



**Figure 6:** Process of vinegar production **(Mas *et al.*, 2016).**

### 2.1.4 Different type of vinegar

Vinegar can be made from various sources, including grains, alcohol, and animal products. According to **Mas *et al.* (2016),** vinegar can be produced from fruit vinegar (the most common type, especially from grapes, referred to as wine vinegar), grain vinegar (derived from a variety of cereals, such as malt or rice vinegar), and alcohol vinegar (produced mainly by distilling pure alcohol from different beverage industry by-products, like grape or another fruit pomace, corn) or even vinegar from products of animal origin (such as honey or milk serum).

### 2.1.5 Production of vinegar

Vinegar production ranges from traditional wood casks and surface culture methods to submerged acetate fermentation **(Morales *et al.,* 2001).**

#### 2.1.5.1 The Orleans process

Orleans process, which is called the slow method, was used in France in 1670, also called as French process **(Singh, 2020)**; it is still in use for the production of high-quality vinegar; the procedure has remained unchanged through the years but is very slow and requires a great deal of space. The starting liquor is placed in a large cask containing wood shavings or grape stalks, where the acetification process begins. After 8 days, the liquid is withdrawn and transferred into barrels, which are left only one-half or two-thirds full and where the liquid remains until acidity reaches its peak (about 3 months). From now on, two-thirds or three-quarters of the vinegar is withdrawn from the bottom of the barrel every week, and an equal volume of liquid from the generator cask is added from the top **(Plessi, 2003) (Fig. 7).**

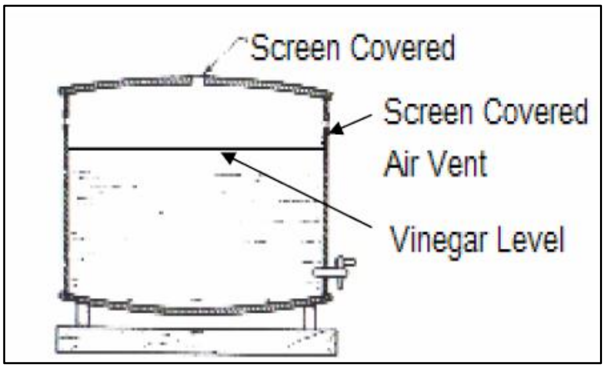



**Figure 7:** Orleans Process Barrel **(Tan, 2005).**

#### 2.1.5.2 Generator process

The generator tanks are generally made of wood or sometimes steel, are equipped with cooling coils, and are vented to allow the air to circulate; they have a perforated false bottom that supports the wood shavings (preferably beech) or grape stalks with which the tanks are filled. A spray mechanism distributes the alcoholic liquid over the surface of the packing **(Plessi, 2003).**

Re-circulated fermenting liquid or mash trickles over the packing material toward the bottom while air moves from the bottom through inlets toward the top. The acetification rate depends on oxygen concentration **(Cruess, 1958) (Fig. 8).**

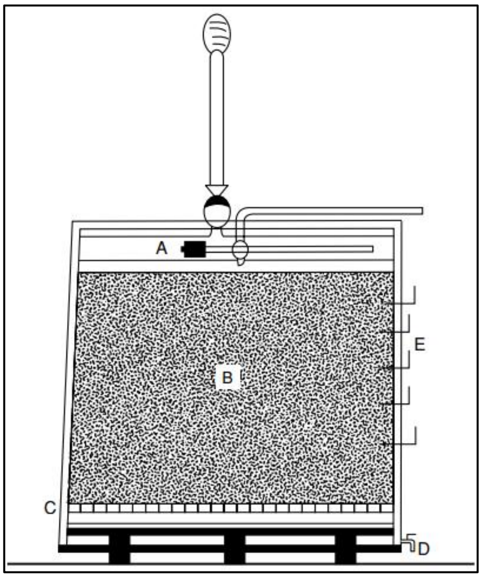



**Figure 8:** Circulating generator **(Plessi, 2003).**

### 2.1.5.3 Submerged Fermentation

Today, the most common production method is submerged culture, which improves general fermentation conditions like aeration, stirring, heating, etc. **(Tan, 2005).**

This occurs in fermenters with forced aeration systems (actuators, cavitation). Vinegar is manufactured using submerged culture fermentors at every stage **(Bouaziz, 2008) (Fig. 9).**

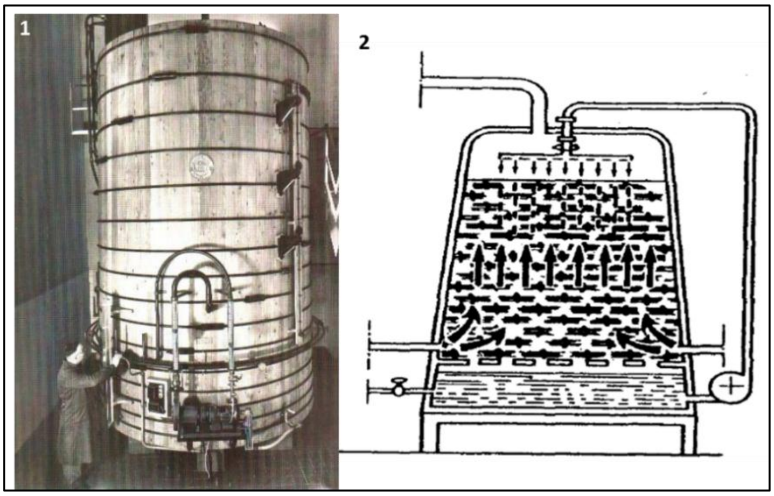
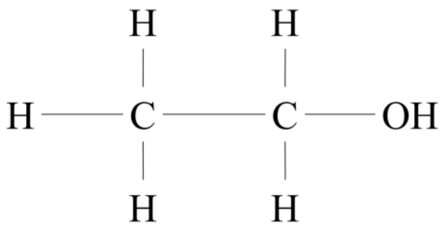



**Figure 9:** Submerged Process: **1)** Bioreactor for submerged fermentation **(Tan, 2005)**; **2)** Scheme of submerged process **(Bourgeois & Larpent, 1996).**

## 2.2 Bioethanol

### 2.2.1 Definition of ethanol

Ethyl alcohol, or $C_2H_5OH$ (EtOH) or bioethanol, is an alcohol produced by fermentation. The liquid is flammable, colorless, and has a pleasant smell. It is synthesized from microbial fermentation rather than synthetically from petroleum **(Walker, 2010).** The structural formula of the ethanol is shown below:




Bioethanol is produced by fermenting food-competing crops, lignocellulosic materials, and algae into ethanol by microbial degradation. Various other substrates, including rice, sweet potato, cassava, oats, and natural products, are also utilized for ethanol production. Producing ethanol from sugar or starch is relatively more straightforward than cellulosic biomass **(Nurhayati *et al.*, 2023).**

### 2.2.2 Physical and chemical properties of ethanol

The **table 7** provides a summary of the physico-chemical characteristics of ethanol.

**Table 7:** Physico-chemical properties of ethanol **(Ethyl Alcohol, 2017).**

| | |
|---|---|
| Molecular formula | $C_2H_5OH$ |
| Molecular mass | 46.07 g/mol |
| Appearance | Colorless liquid |
| Boiling temp. | 78 °C |
| Freezing point | -117 °C |
| Density | 0.81 kg/L |
| Aqueous solubility | Miscible |

### 2.2.3 Bioethanol feedstocks

According to **Walker (2010)**, Bioethanol can be extracted from every carbohydrate material with the typical formula $(C_2HO)_N$. It can be separated into three generations based on raw materials **(Table 8).**

**Table 8:** Major resources for bioethanol production **(Walker, 2010).**

| Sugary materials | Starchy materials | Cellulosic materials |
|---|---|---|
| • Sugarcane<br>• Sugar beet<br>• Sweet sorghum<br>• Fruits (surplus)<br>• Confectionery industrial waste | • Grains (maize, wheat,)<br>• Root corps (potato, cassava)<br>• Inulin (polyfructan) root corps (chicory, artichoke) | • Wood<br>• Agricultural residues (straws, Stover)<br>• Municipal solid waste<br>• Waste paper, paper pulp. |

#### 2.2.3.1 First generation feedstocks

The first generation of bioethanol is made from sugar- or lignocellulosic biomass, which is limited and used as a food source. Plant materials with cellulose, hemicellulose, and lignin as their main constituents are called lignocellulosic biomass **(Fan, 2014).** Namely, sugar molecules and cereal starches **(Walker, 2010).**

#### 2.2.3.2 Second generation feedstocks

Second-generation bioethanol, also known as "advanced biofuel", It is common practice to extract bioethanol from non-food biomass sources. Primarily lignocellulosic biomass **(Walker, 2010).**

#### 2.2.3.3 Third generation feedstocks

Marine microalgae (seaweeds) represent an exciting biomass resource for bioethanol and may represent an example of third-generation feedstocks for bioethanol production. They contain high amounts of carbohydrates such as alginic acid (structural) and laminarin, and mannitol (storage) that can potentially be fermented to ethanol; this biomass can be converted directly into energy **(Walker, 2010).** In general, using this raw material for bioethanol manufacturing depends on elements like technology and the marine environment **(Akbi, 2013).**

### 2.2.4 Production processes

The principle stages in ethanol production processes are summarized in **Figure 10.**

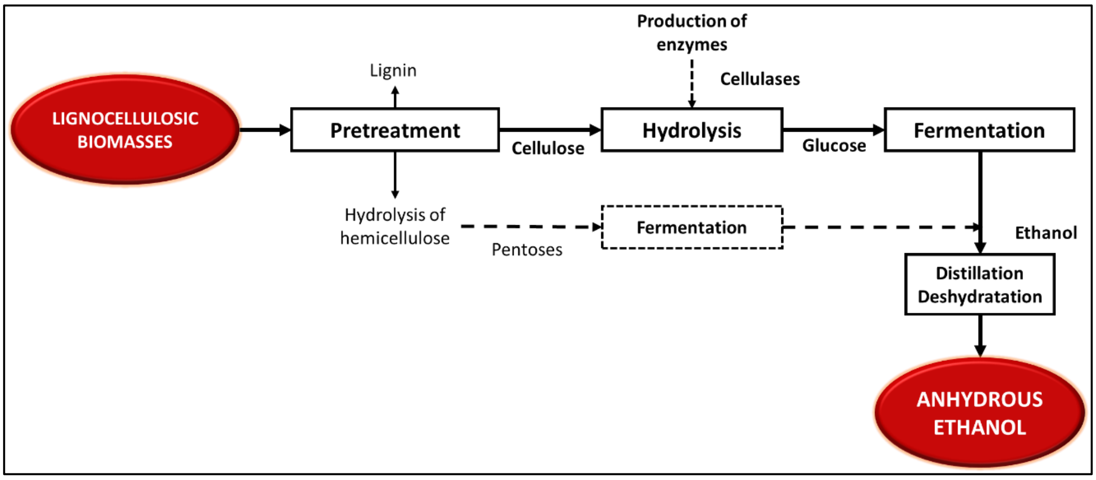



**Figure 10:** Process for producing ethanol from biomass **(Vaitilingom et al., 2021).**

### 2.2.4.1 Pretreatment of lignocellulosic biomasses

The pretreatment process' goal is to get the biomass ready for efficient fermentation and bioethanol production from cellulose and hemicellulose. There are four main pretreatment methods: physical, chemical, physicochemical, and biological **(Wyman, 2018; McMillan, 1994).**

Any pretreatment should aim to reduce the degree of lignocellulosic chain polymerization, preserve the pentose (hemicellulose) fractions, and restrict the creation of degradation products that hinder the growth of the fermentative microbe, minimize energy requirements, and keep costs low **(Biobased Industrial Products, 2000).**

### 2.2.4.2 Hydrolysis

According to **Dalena *et al.*, (2019),** a hydrolysis reaction breaks down cellulose and hemicellulose into monomers (simple sugars). As a result of enzyme hydrolysis, starch releases simple sugars, mainly glucose **(Walker, 2010).**

### 2.2.4.3 Fermentation process

In the third phase, bacteria or yeast convert carbohydrates into alcohol. Production of wood cellulose ethanol from biomasses involves the metabolization of the mentioned six-carbon (hexoses) and five-carbon (pentoses) sugars by specialized fermentation microorganisms **(Dalena *et al.*, 2019).**

### 2.2.4.4 Distillation

The ethanol is then recovered in a distillation system **(Walker, 2010).** The Distillates obtained by the distillation process consist of higher quantities of water, secondary products, and undesirable ions than required by the regulations; therefore, the alcohol must be purified **(Wyman, 2001).**

## 2.3 Date Syrup "*Robb*"

### 2.3.1 General about date syrup

Date syrup (locally called *Robb* or *dibis*) is possibly the most popular date product. It is produced in two different ways: either at the Artisanal transformation of date level by extracting and boiling the juice, or on a semi-industrial scale. The principle of the process based on the extraction and purification of date juice then concentrated the extract by cooking over low heat until a thick dark brown liquid is obtained. The sugars presented in date syrup are Glucose and fructose and the total sugar content reaches 88%. Date syrup contains elements that can play an important role in considering date syrup as a rich food **(Alkhateeb, 2008; Al-Hooti *et al.*, 2002).**

### 2.3.2 Nutritional value

From a nutritional point of view, molasses remains a product with a strong sweetening power and rich in carbohydrates that must be consumed in moderation. It also contains an interesting quantity of magnesium: the magnesium content which will help strengthen the immune defenses and promote the proper functioning of the muscles and the nervous system **(Gheraissa & Hamidani, 2018)**. Potassium: the transmission of nerve impulses is ensured by the supply of potassium in sufficient quantity, Iron: the iron supply of molasses is very important in order to fight against anemia and guarantee the good transport of oxygen in the body, calcium : is essential in maintaining good bone health but also to promote good heart and nervous system rhythm, and some antioxidant substances recent studies have shown that molasses is as rich in antioxidants as honey and agave syrup Regular consumption of molasses will therefore allow you to slow down cellular aging.

There are 3 types of molasses:

a) **Clear molasses:** it is a syrup which has been extracted following the first boiling of the sugar, it is rather used in baking;

b) **Black molasses:** this is the syrup obtained after the 2nd boiling; it is syrup a little less sweet than light molasses with a more pronounced taste;

c) **Green molasses:** this is the syrup obtained after the 3rd boiling, it is the most bitter and darkest syrup but also the most concentrated in minerals.

### 2.3.3 Uses of date syrup and modes of consumption

Modern dietary institutes around the world recommend the regular use of dates and its by-products for their effects on the body **(Abbes *et al.*, 2011).**

Date syrup is rich in certain nutrients (sugars, phenolic compounds, etc.). Due to its high sugar content, it provides a good source of quick energy. In effect; a high sugar content should justify their use as a source of liquid sugar. Research has been done to use date syrup in the preparation of cakes, cookies and sweet breads and for direct use on pancakes. Also, date syrup can be used to replace caramel in candy bars **(Barreveld, 1993).**

The robb can replace any source of fat, date or even honey and sugar. Indeed, according to Boussaid et al. (2020), date moelass is rich in nutrients with relatively high carbohydrate content ranges between 69.7-72%. There are many modes of consumption:

- It can be eaten with pancakes or bread, especially for breakfast instead of jam and butter.
- It can be added to "mesfouf" (fine couscous) and consumed especially during Ramadhan (Shour) instead of honey, fat and grapes.
- It can be eaten with many other traditional dishes.

### 2.3.4 Date Syrup Preparation Methods

#### 2.3.4.1 Process by pressing (traditional method)

The principle of this process is based on the packaging method. Usually placed in a canvas bag, which is a soft date storage medium. After the cleaning and hydration of the dates, the honey attracts under the effect of the temperature, the humidity and the weight of the fruits; the yield of the latter is very low, varying between 10 to 15% of the weight of the dates **(Mimouni, 2015).** The honey obtained (traditional honey) is a very concentrated natural product (about 82%), with an odor, taste and color of the date used **(Atef & Mohamed, 1998).**

#### 2.3.4.2 Soaking process in water at low temperature

Dates are soaked in water neither hot nor cold for about 8 hours and more. The extract obtained, after filtration and removal of fibers and nuclei, is heated again over low heat to remove excess water and condense it .The disadvantage of this process is the syrup which does not always have the same texture and concentration. In addition there is a risk of fermentation **(El-Ogaidi, 2000).**

#### 2.3.4.3 Process by soaking in water at high temearture

This process is frequently used. It consists of putting the dates in water at high temperature (about 80°C), using directly or indirectly the steam, the heating allows a very intense extraction; after filtration of the extract, the juice obtained contains impurities which are removed from the sugar solution by carbonation **(Mimouni, 2009).**

The honey obtained is dark brown in color, with a taste and smell of burnt sugar due to the use of high temperature **(Hassan, 2000).**

### 2.3.4.4 Extraction with enzymes (cellulase and pectinase)

It consists of soaking a paste of dates in water and then bringing it to a boil. After filtration, the solution undergoes an enzymatic treatment (cellulase and pectinase) **(Chikhrouhou *et al.*, 2006; AL-Sharnoubi *et al.*, 2014).**

### 2.3.4.5 Extraction by diffusion

This method is based on the maceration of dates in water maintained at 80°C for 24 hours. The principle is based on the passage, according to the laws of diffusion by passive transport, the juice is then obtained after decantation and passage through a gauze. To obtain concentrated honey, it is necessary to condense the juice recovered at a temperature of 60°C. **(Mimouni & Siboukeur, 2011).**

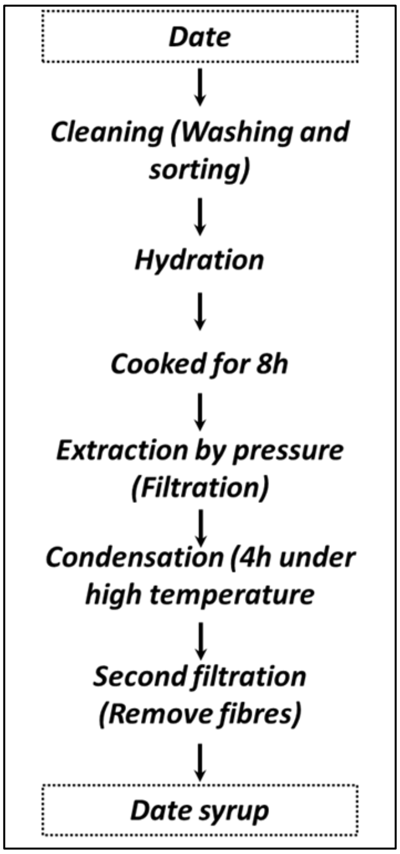



**Figure 11:** Process for producing date syrup **(Benmbarek & Deboub, 2015)**

## 2.4 Liquid sugar

Obtained by concentration of syrup, it comes in an amorphous state, its color is more or less brown. According to **Harrak** and **Boujnah (2012),** the sugar is obtained by grinding dates followed by kneading them in hot water. It is preferable to use a diffusion process which makes it possible to recover most of the sugars while limiting the diffusion of the sugars in the juice. The syrup is concentrated at 30 to 35 degrees Brix at low temperature (40 to 45°C) and under vacuum. A light brown to bright yellow concentrate is obtained depending on whether it is discolored or not. This concentrate represents a sweet product of easy use. If it has been purified, it does not bring color or astringency to diluted drinks, which makes it possible to use it directly in tea or coffee. Its sweetening power, like that of syrup, depends on the sugars that compose it.

The high amount of simple sugars (sucrose, fructose, and glucose) and other phytochemicals that naturally occur in date fruits make them an ideal nutritional source for the production of natural sugar. Therefore, the highly nutritious soluble date sugar extracted from date fruit would be a promising and novel food product for various applications. Moreover, it would be a suitable and superior alternative to commercially available refined sugar **(Dhenge *et al.*, 2022; Pingret *et al.*, 2013) (Table 9).**

**Table 9 :** Biochemical composition of date juice

| Composition (%) | Al-Eid (2016) | Al-Khateeb (2008) | Mimouni & Siboukeur (2011) |
|---|---|---|---|
| Water content | 13.5 | 16 | 13.7 |
| Soluble solids | 86.5 | 84 | 86.3 |
| Total sugar | 81 | 79.45 | 80.73 |
| Reducing sugars | 80 | 74.83 | 79.96 |
| Saccharose | 1 | 4.87 | 0.77 |
| Protiens | 2.2 | 0.84 | 1.15 |
| Pectines | 1.8 | 1.46 | 3.86 |

### 2.4.1 Industrialization process of date sugar

#### 2.4.1.1 Diffusion

In this operation, the sugar is extracted from the cossettes by diffusion using hot water (solvent) while limiting the transfer of impurities into the juice. The diffusion phenomenon is based on the movement of molecules from a region of high concentration (sugar stored in the cell tissue of the date) to another of low concentration (hot water) **(Arzate, 2005).**

### 2.4.1.2 Purification

This operation is done in two steps: The main purpose of purification is to separate the non-sweetened components of the Juice of diffusion (sweetened juice) by precipitation, then by sedimentation or filtration. Thus, it is usual to purify the juice by liming (adding lime – Cao) and successive carbonation (adding carbon dioxide – CO2) **(Arzate, 2005).**

**I. Liming**

Liming is done by adding lime in the form of calcium oxide (CaO) to the sugar solution, which leads to the elevation of the pH of the date juice. To avoid the hydrolysis of sugars by the acids existing in the date juice **(El-Ogaidi, 2000).** Recommended an amount of 1% calcium oxide based on the total weight of the dates. The addition of lime also makes it possible to precipitate the impurities which can easily be removed by filtration.

**II. Carbonation**

It is done by adding activated carbon to the limed juice, to precipitate the lime in the form of calcium carbonate (CaCO3) according to the following reaction:

$$CaO + CO_2 = CaCO_3$$

The purpose of turbid juice filtration is to separate the calcium carbonate from the manufactured product **(Arzate, 2005).**

### 2.4.1.3 Evaporation

The main purpose of evaporation is to concentrate the purified juice (10%–15% sucrose) by boiling down to the syrup at a concentration close to Saturation (68.5g dry matter/100g syrup) **(Arzate, 2005).**

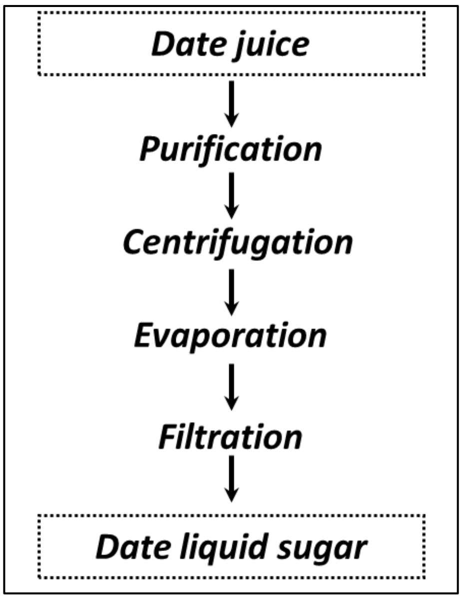



**Figure 12:** Process for producing date liquid sugar **(Arzate, 2005)**

# *Experimental Part*

# *Chapter III*

## *Materials and Methods*

## 3.1 Materiel

### 3.1.1 Plant materials

The varieties of dates retained in this present study are: *Balha*, *Ghars*, *Degla-Beida* and *Tamjouhert* from Laghouat region **(Fig. 13).** These varieties were selected because they contained sugar; and it is characterized by the low market value of some varieties.


**Figure 13:** Dates samples: **a)** *Balha* **(Azouz & Oubacha, 2022); b)** *Ghars* ; **c)** *Tamjouhert*; **d)** *Degla-Beida* **(Original photography, 2023)**

### 3.1.2 Biological material

Dry baker's yeast, *Saccharomyces cerevisiae*, is employed. Our inoculum will be used for alcoholic fermentation. **(Nguyen, 2016; Moussaoui & Necib, 2019).**

### 3.1.3 Equipment

#### 3.1.3.1 Fermentation device

An approach proposed by **Boukhiar (2009)** was employed in this work, performed with two devices. The first is for alcoholic fermentation, and the second is for acetic fermentation.

The alcoholic fermentation is carried out in bottles equipped with lids with a $CO_2$ evacuation system oriented in a small bottle full of water as an indicator of $CO_2$ release, as shown in **(Fig. 14).**

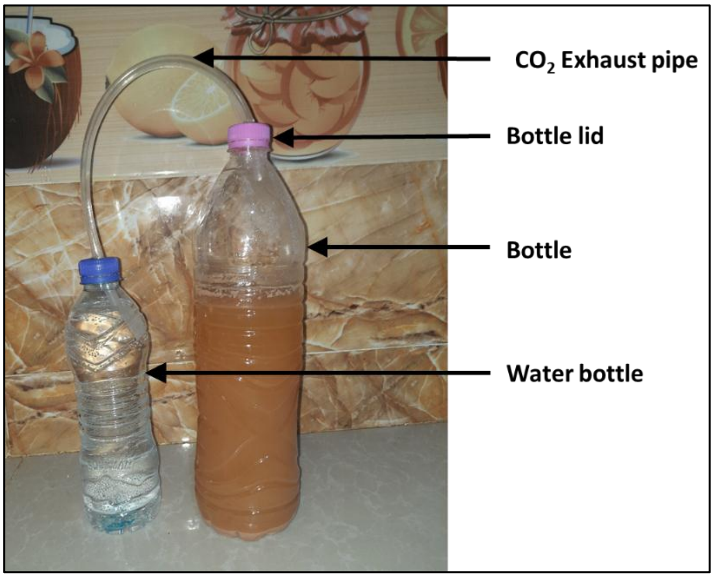



**Figure 14:** Alcoholic fermentation device **(Original photography, 2023)**

The acetic fermentation device has been designed to increase the contact surface between the fermented juice and oxygen. It was generated using a large plastic container. A textile fabric cover protects the container against insects while allowing air flow **(Fig. 15).**

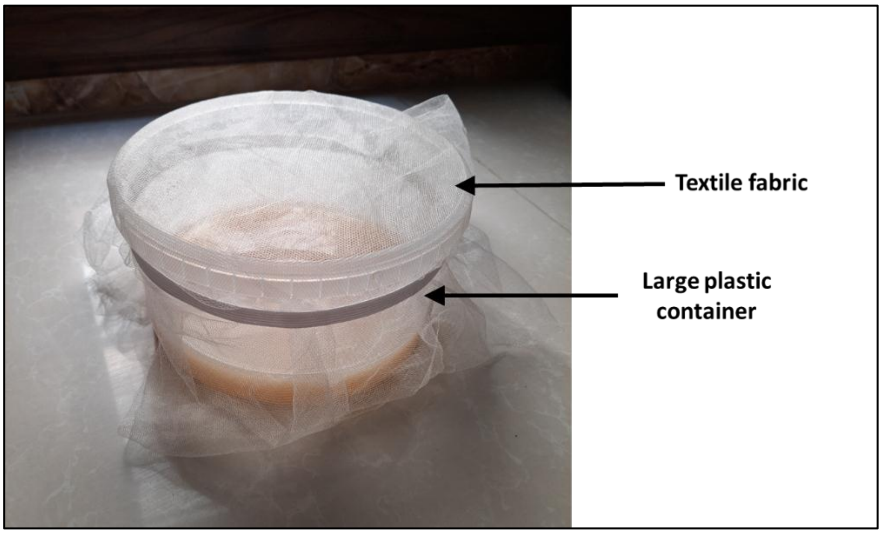



**Figure 15:** Acetic fermentation device **(Original photography, 2023)**

## 3.2 Methods

### 3.2.1 Date juice extraction

The extraction of date juice contained several stages according to **Hamden *et al.* 2022**. The dates are first sorted, washed, and drained in this experiment. Followed by heating (90 mins at 80°C) and digestion with water (at a ratio of 1:3); Dates are sliced into tiny fragments as a sample (to increase the contact surface with water and to extract the maximum amount of sugar). Finally, a filtration step is performed to separate the liquid phase (juice) from the solid phase (pulp). A textile fabric carries out the filtration **(Fig. 16) (Appendix 1).**

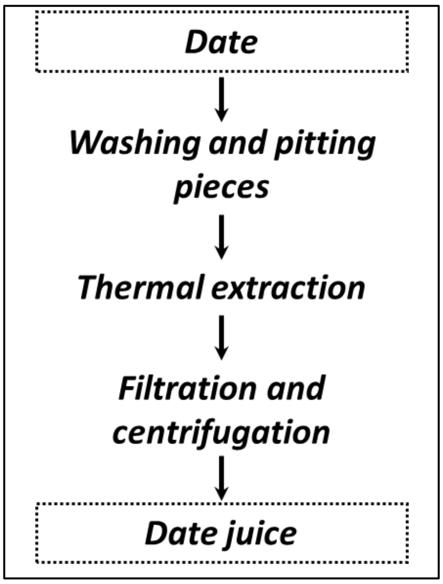



**Figure 16:** Diagram of date juice extraction

### 3.2.2 Date vinegar

#### 3.2.2.1 Production of semi industrial vinegar

The date juice is inoculated with active *Saccharomyces cerevisiae* yeast to implement alcoholic fermentation ( by dissolving 8 grams of yeast in 1 liter of date juice, homogenizing well, and letting the mixture sit at 25°C for 15 minutes) **(Azhar *et al.*, 2017).**

The alcoholic fermentation is carried out in bottles filled to 3/4 of its capacity under anaerobic conditions, and the temperature is fixed approximately at 30°C for 7 days. The fermentation broth was filtered to remove the solid biomass substrate from the liquid.

The acetic fermentation is carried out in a large plastic container under aerobic conditions, which consists of the oxidation of the alcohol into acetic acid, providing it with the characteristic vinegar taste. We use the mother of vinegar as acetic bacteria (**Hamden *et al.*, 2022)**. A textile fabric cover protects the container against insects while allowing airflow.

The following flowchart **(Fig. 17)** illustrates the production of semi-industrial vinegar process.

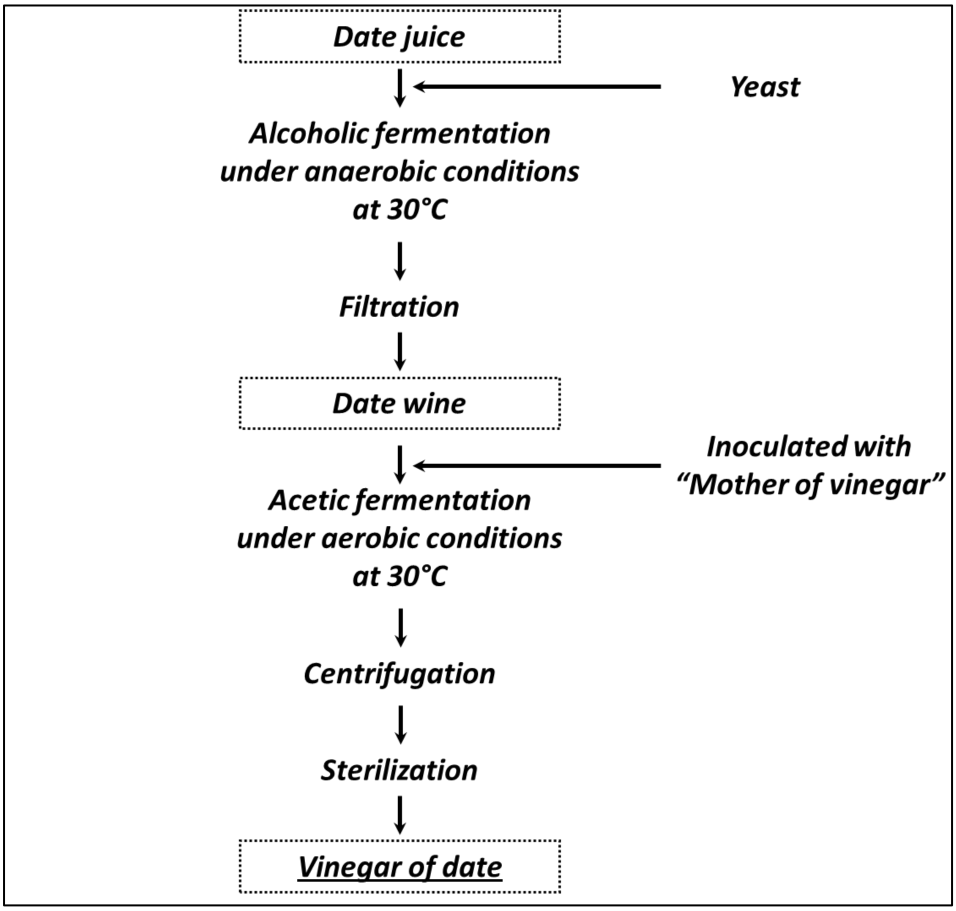



**Figure 17:** Semi industrial vinegar production diagram

The physicochemical parameters determined during this process are titratable acidity, pH, Brix degree and conductivity.

### 3.2.2.2 Production of traditional vinegar

*Degla-Beida* dates are used in this part to make traditional vinegar. After sorting and washing the dates, we add 3 measures of spring water to them. All items are placed in empty plastic bottles. The bottles are capped tightly and left at room temperature for 40 days. The diagram below represents the traditional vinegar production protocol **(Fig. 18).**

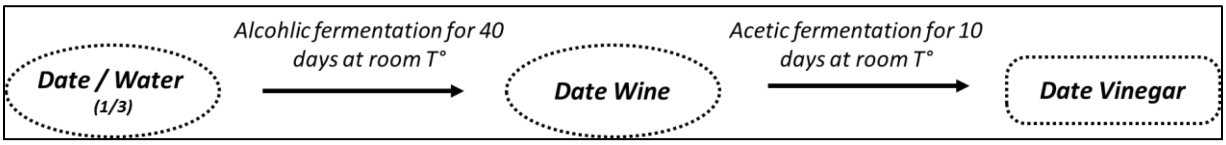



**Figure 18:** Traditional vinegar manufacturing procedure diagram

**Figure 19:** Production of traditional vinegar **(Original photography, 2023)**

After 40 days, the lid is removed, and filtration is performed to remove date waste. The entire thing is kept at the same temperature for 10 days for the acetic fermentation. After that the product is filtered. It boils for 30 minutes before being stored in small tightly closed bottles.

Titratable acidity, pH, Brix level, and conductivity are the physicochemical parameters that are determined throughout this process every 7 days.

A microbiological tests (enumeration and identification of yeast and mold) was performed for the samples.

### 3.2.3 Bioethanol

Stages of bioethanol production are: juice extraction, alcoholic fermentation, and distillation **(Hamden *et al.*, 2022) (Fig. 20).**

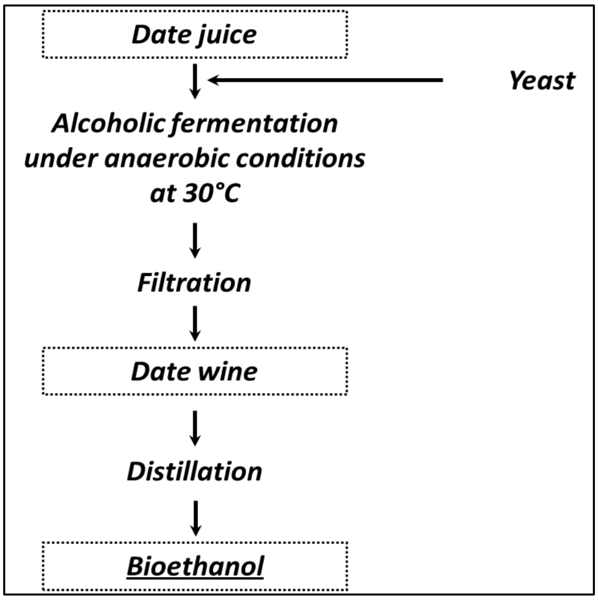



**Figure 20:** Bioethanol production diagram

The alcoholic fermentation was carried out with the same stages and the exact condition of the alcoholic fermentation of vinegar. After that, distillation at 78 °C was done to get the bioethanol samples using a distillation assembly **(Fig. 21).**

**Figure 21:** Distillation assembly for bioethanol production **(Original photography, 2023)**

The produced bioethanol was further characterized to measure its Titratable acidity, pH, Brix level, and electrical conductivity.

### 3.2.4 Date syrup "*Robb*"

We have adopted the traditional method for making date syrup **(Benahmed *et al.*, 2006) (Fig. 22) (Appendix 2).**

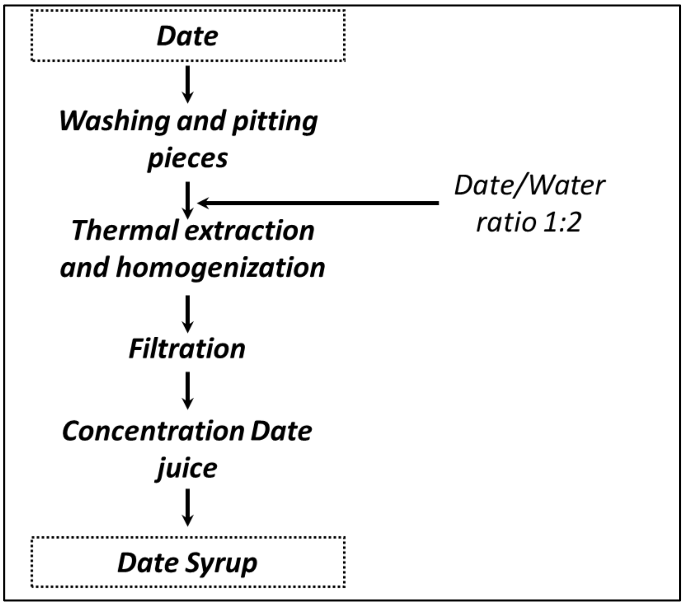



**Figure 22:** Syrup production diagram

After the concentration of the syrups produced during well-defined concentration times, we obtained syrups colored brown-red, viscous and syrupy **(Fig. 23).**

**Figure 23:** Syrups final products **(Original photography, 2023)**

### 3.2.5 Liquide sugar

The date juice will be used to produce liquid sugar. We obtain raw sugar juice that is liquid and contains as much sugar as possible. A large quantity of water must be evaporated to obtain sugar syrup, from which 90% or more of sugar and other dissolved products will be present **(Fig.24).**


**Figure 24:** Steam rotators for liquid sugar production **(Original photography, 2023)**

### 3.3 Analyzes

#### 3.3.1 Physicochemical analyzes

##### 3.3.1.1 Preparation of Samples

The date vinegar samples used for analysis was from:

- **Sample n°1:** Semi industrial vinegar obtained in the laboratory.
- **Sample n°2:** Traditionally (double fermentation) vinegar obtained in the laboratory.
- **Sample n°3:** Semi industrial vinegar from the Biskra region.
- **Sample n°4:** Semi industrial vinegar from the El Oued region.
- **Sample n°5:** Traditionally vinegar from the El Oued region **(Fig. 25).**


**Figure 25:** Date vinegar samples **(Original photography, 2023)**

For the date syrup samples used was from:

- **Sample n°1:** Semi industrial syrup from the Biskra region.
- **Sample n°2:** Semi industrial syrup from the Ouargla region.
- **Sample n°3:** Semi industrial syrup from the Djelfa region.
- **Sample n°4:** Semi industrial syrup from the Blida region.
- **Sample n°5:** Traditionally syrup obtained in the laboratory **(Fig. 26).**
- **Sample n°6:** Traditionally *Ghars* syrup obtained in the laboratory
- **Sample n°7:** Traditionally *Balha* syrup obtained in the laboratory **(Fig. 23).**


**Figure 26:** Date syrup samples **(Original photography, 2023)**

#### 3.3.1.2 The pH determination (NF T90-014)

The pH values are measured directly for all prepared solutions, including Robb, date juice, wine, vinegar and bioethanol at 20°C using a combined pH electrode. Electrodes are immersed in beakers containing the product to be analyzed according to the AFNOR standard. Once the pH value has stabilized, it is read. For each pH measurement, remove the electrode, rinse with distilled water, and dry **(Fig. 27).**

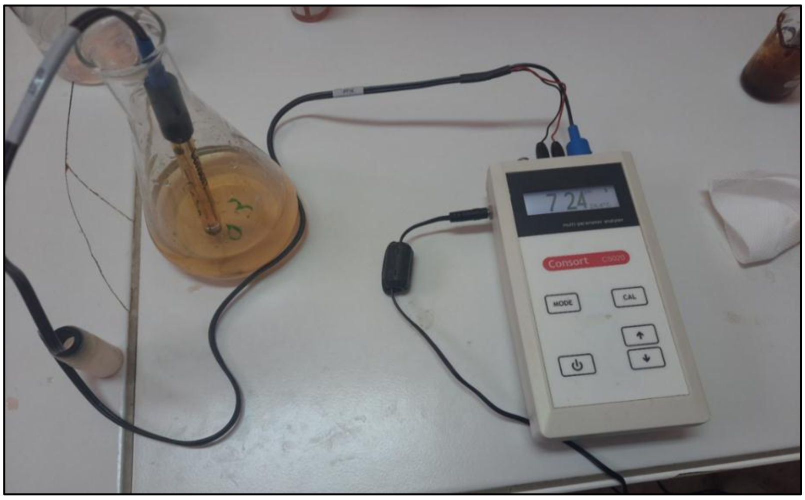



**Figure 27:** pH meter **(Original photography, 2023)**

### 3.3.1.3 Total soluble solids determination (Brix degree) (NF V05-109)

The Brix scale is used to determine the fraction of sucrose in a liquid, i.e. the percentage of soluble dry matter, in degrees Brix (°B). A higher Brix degree means a sweeter sample. **(Metelf *et al.*, 2022).**

The different measurement of Brix degree for all the product was obtained using an ATAGO Master-α "Alpha" series refractometers **(Fig. 28).**


**Figure 28:** ATAGO refractometer **(Original photography, 2023)**

### 3.3.1.4 Total acidity determination (ISO 6091:2010)

The total acidity was determined by potentiometric titration. According to the method described by **Hafzan *et al.* (2017),** a total of 80 mL of diluted of product samples were added three drops of phenolphthalein indicator then was titrated with 0.1 M NaOH solution **(Fig. 29).**

A sample's total acidity, expressed as grams of acid per liter, it’s given by the following formula **(Clavet, 1992):**

$$C\left(\frac{g}{l}\right) = \frac{F.V}{10} \times 60$$

**C:** Concentration of acetic acid in g/l.
**V:** Volume of NaOH added in ml.
**F:** Factor corresponding to the normality of NaOH 0.1N.
**60:** The molar mass of acetic /citric acid.

A vinegar's acidity is considered to be one of the most critical factors, it should be between 5 and 6 degrees **(Ousaaid et al., 2021).** Vinegar's acidity is measured by the following formula:

$$D° = \frac{m_{acid}}{m_{solution}} \times 100$$

$m_{acid}$**:** Mass of acetic acid.
$m_{solution}$**:** Solution density

**Figure 29:** Total acidity titration **(Original photography, 2023)**

### 3.3.1.5 Electrical conductivity (NF T90-111)

The method used is to rinse the electrode several times and measure it first with distilled water, then immerse it in a container containing the samples to be tested. The results are expressed in (mS/cm) **(Fig. 30).**

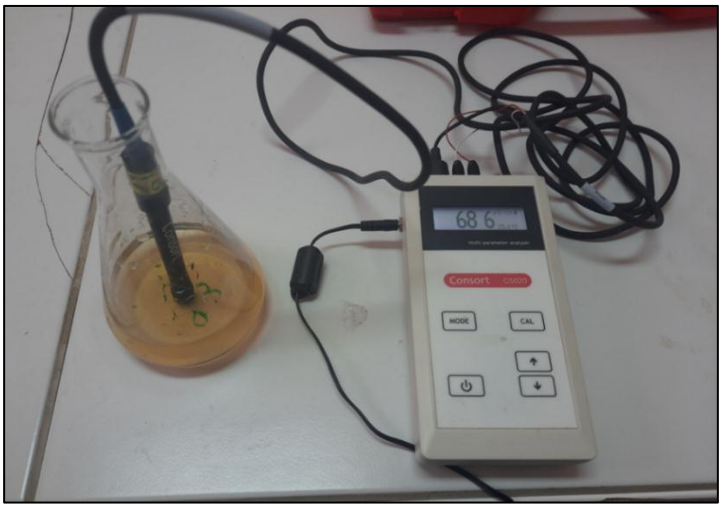



**Figure 30:** Electrical conductivity measurement **(Original photography, 2023)**

### 3.3.2 Microbiological analyzes

Food products are evaluated based on their microbial flora for hygienic quality. This assessment remains the most effective in assessing food quality **(Petransxiène & Lapied, 1981).**

The microbiological analysis was performed on: ***(i)*** Semi industrial vinegar obtained in the laboratory; ***(ii)*** Traditionally (double fermentation) vinegar obtained in the laboratory; ***(iii)*** Semi industrial syrup from Biskra; ***(iv)*** Semi industrial syrup from Blida; ***(v)*** Syrup from *Balha* variety obtained in the laboratory and ***(vi)*** Syrup from *Ghars* variety obtained in the laboratory.

#### 3.3.2.1 Preparation of dilutions (ISO 6887-1:2017)

In our study, we used the serial dilution method. Using a sterile pipette, add 1 ml of the sample to 9 ml of sterile physiological water. Agitate this primary dilution to achieve dilution $10^{-1}$. From the latter solution, a series of decimal dilutions are carried out up to $10^{-7}$.

#### 3.3.2.2 Determination of total and fecal coliforms (AFNOR 3M 01/02-09/89 C)

Coliform bacteria are considered indicator organisms because their presence in foods indicates that the conditions are suitable for enteric pathogens. They may signify insufficient sanitary conditions **(Halkman & Halkman, 2014).**

The search for coliforms in our samples is made in a solid medium which is Violet Red Bile Lactose Agar (VRBL) **(Appendix 3).** It is a selective medium for coliform isolation and enumeration in vinegar. 1 ml of the dilution is inoculated on the surface of $10^{-1}$, $10^{-3}$, $10^{-5}$, and $10^{-7}$ dilutions. The dishes are incubated at 30°C for total coliforms; and 44°C for fecal coliforms for 72h.

#### 3.3.2.3 Determination of yeast and molds (NF V08-059)

The Sabouraud agar medium **(Appendix 3)** is commonly used for the growth and the isolation of yeasts and molds. The fungal flora is counted by putting a 0.1 ml inoculum of stock solution or its dilutions over the surface of Sabouraud agar medium. **(Goudjal, 2022).**

A volume of 0.1 ml is inoculated on the surface of Petri plates for the $10^{-1}$, $10^{-3}$, $10^{-5}$, and $10^{-7}$ dilutions, the petri dishes were incubated at 25°C for 72h **(Fig. 31).**

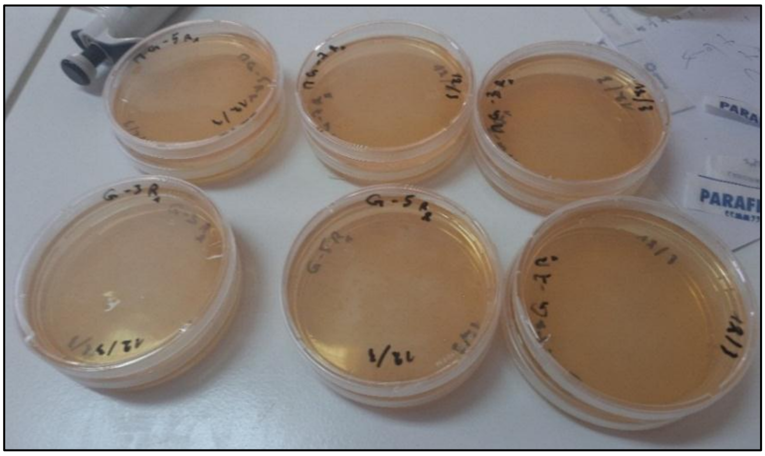


**Figure 31:** Isolation of fungal flora from different samples **(Original photography, 2023)**

### 3.3.2.4 Isolation and purification of strains

Once a strain has been incubated, it is subcultured several times on Sabouraud medium until it becomes pure **(Boukhelkhal, 2021).** A sterile loop can be used to remove spores or mycelial fragments from the margin of each colony to purify the strains. Inoculate fresh medium with the inoculum. The incubation takes place at 28°C for 24 hours **(Guiraud, 1998) (Fig. 32).**

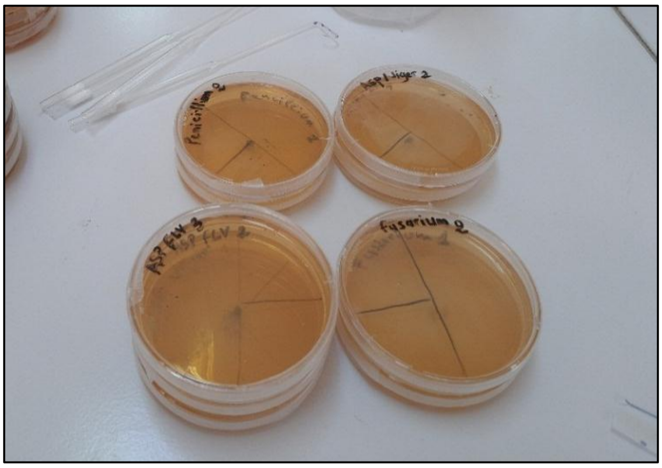


**Figure 32:** Isolation and purification of strains **(Original photography, 2023)**

Following is a formula for counting microorganisms from different dilutions, the results are expressed by (CFU/ml):

$$X = \frac{x_1 + x_2 + x_3}{3}$$

### 3.3.3 Statistical analyzes

The results are given as means ± standard deviation of at least three independent determinations. One way Analysis Of Variance **(ANOVA)** was used to compare the means and then were separated by Duncan's multiple range test. All statistical analysis was performed at $p < 0.05$ using the **XLSTAT 2023. 1. 3. 1407.**

# ***Chapter IV***

## *Results and discussion*

## 4.1 Physicochemical analysis results

### 4.1.1 Date vinegar

#### A. pH

The variation in pH during the fermentation of the traditional vinegar is illustrated by the curve in **(Fig. 33).**

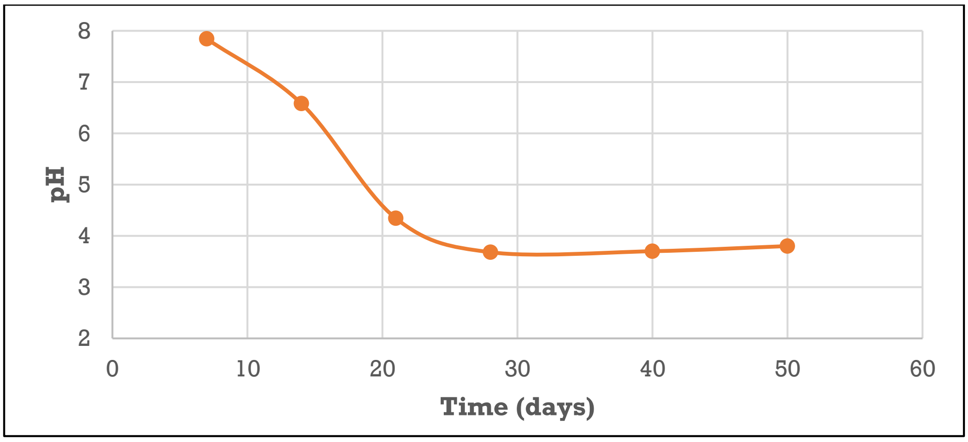



**Figure 33:** pH kinetics during the traditional fermentation of vinegar

The results in **(Fig.33)** indicate that the pH value decreases during fermentation to reach 3.68 on the 28th day, and it stays at this value throughout the process. After 50 days of fermentation, the pH measurement was 3.8. The pH lowered reflected yeast and mold activity in the medium fermentation. The reaction medium is favorable for acidophil development **(Ould El-Hadj *et al.*, 2001).**

The results obtained by measuring the pH of different vinegar samples are represented in the following figure (**Fig.34)**. The statistical analyzes has shown that the results are not statistically significant ($\rho$<0.001) **(Appendix 4).**

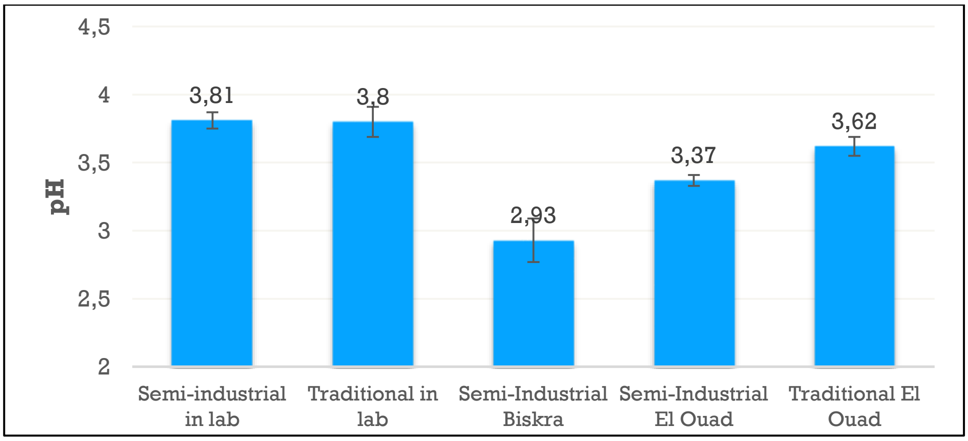



**Figure 34:** pH of different samples of vinegar.

All date vinegar samples have similar pH values with slight variations. The results are: 3.81±0.06; 3.8±0.11; 2.93±0.16; 3.37±0.04 and 3.62±0.07 respectively for Semi industrial in lab, traditional in lab, semi industrial of Biskra, and semi industrial of El Ouad and traditional of El Ouad. The samples have values between 2.93 and 3.81. Overall these findings are in accordance with results reported by **Ould El-Hadj *et al.* (2001).** They reported a pH of 3.12; 3.25 and 3.65 for their 3 samples of date vinegars, and also with the results reported by **Hamden *et al.* (2022),** they indicate pH values is 3.6 for *Khalas* vinegar.

This pH lowering is due initially to the diffusion of the acids contained in the vinegar. This is also caused by the acids metabolized by the microorganisms (mainly yeasts, etc.). The pH is also lowered by the formation of carbonic acid (a weak acid) that dissolves in water (**Hamden *et al.*, 2022).**

**B. Total soluble solids (Brix degree)**

The variation of brix degree during the fermentation of the traditional vinegar is represented in **Figure 35.**

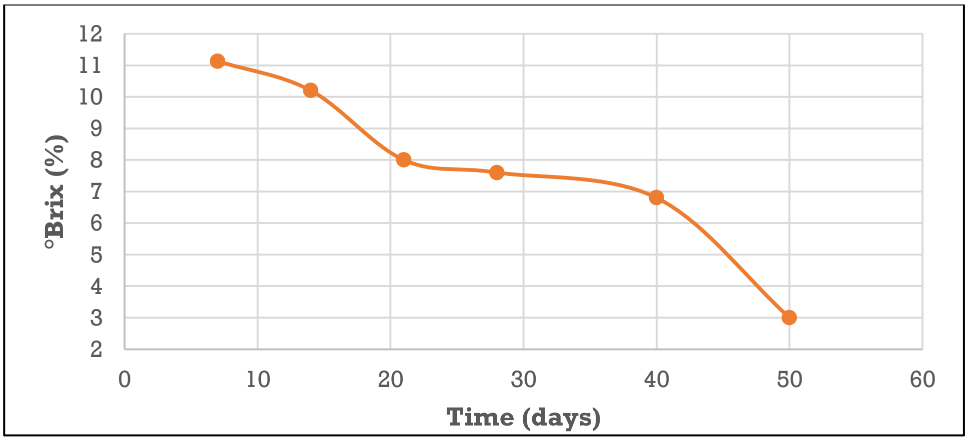



**Figure 35:**°Brix kinetics during the traditional fermentation of vinegar.

In the fermentation curve, we observe a significant decrease in the value of total sugars in the middle during the days of fermentation. At the end of the acetic fermentation, the value of the Total soluble solid value is 3.5%. The TSS rates slowed down. This was due to the acidification of the medium and the decrease in pH. The growth of yeast is stopped by ethanol, it has been proven that ethanol destroys the DNA of yeast cells and inactivates many enzymes as an inhibitor **(Tesfaw & Assefa, 2014).**

The variations in brix degree during the fermentation of the different samples studied are represented in the following **Figure 36.** The statistical analyzes has shown that the results are not statistically significant ($\rho$<0.001) **(Appendix 4).**

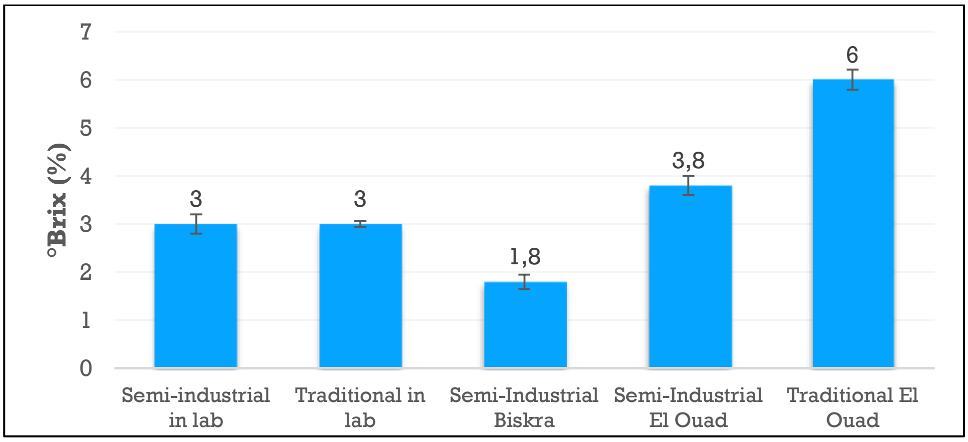



**Figure 36:** Total soluble solids (°Brix) of the different vinegar samples

Results show that the semi industrial Biskra has the lowest TSS rate with 1.8%. The semi industrial and traditional laboratory vinegar in laboratory has both 3%. The results were directly compared with the previously reported findings by **Dahmani and Rebbouh (2009),** who noted 3.25% for apple vinegar; and also comparable with the results from **Hamden *et al.* (2022)** studies. They report °Brix values of 5% for Khalas vinegar. The traditional El Oued vinegar has the highest TSS rate of 6%.

However, fermentation cannot reach a lower brix level despite the availability of sugars. A number of factors influence this:

- The varieties of dates
- The dissolved CO3
- The alcohol content
- The temperature
- The osmotic pressure
- Other limiting factors...etc. **(Boukhiar, 2009).**

There seems to be a close correlation between density and soluble solids content. Vinegars with the highest density indeed have the highest TSS **(Ould El Hadj *et al.*, 2001).**

### C. Electrical Conductivity

The curve giving the variation of the electrical conductivity (µS/cm) during the fermentation is given in the **(Fig. 37).**

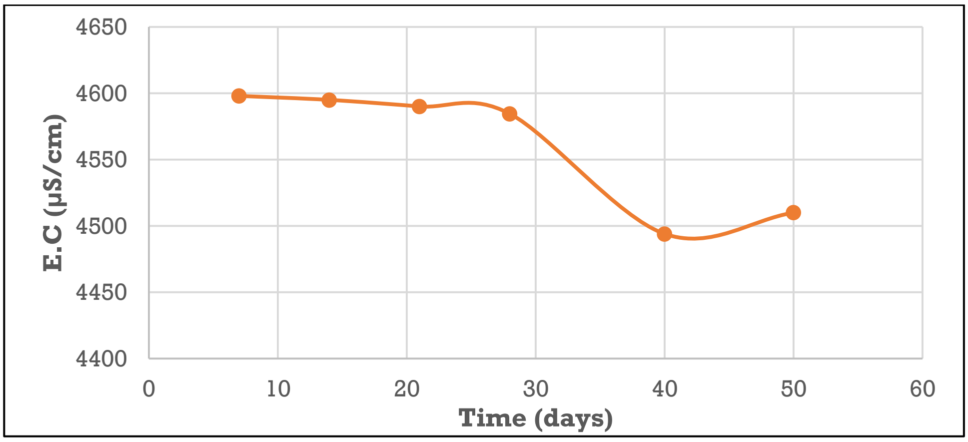



**Figure 37:** Conductivity kinetics during the traditional fermentation of vinegar.

According to the results shown on the curve, it reaches a maximum value of 4598 µS on the 7th day, then it decreases slightly until the end of fermentation to reaches 4510µS. The electrical conductivity increases due to minerals being diffused in water **(Boukhiar, 2009),** besides that, the using of mineral water in vinegar production.

The variations in electrical conductivity of the different samples studied are represented in the following figure **(Fig. 38)**. The statistical analyzes has shown that the results are not statistically significant ($\rho$<0.001) **(Appendix 4).**

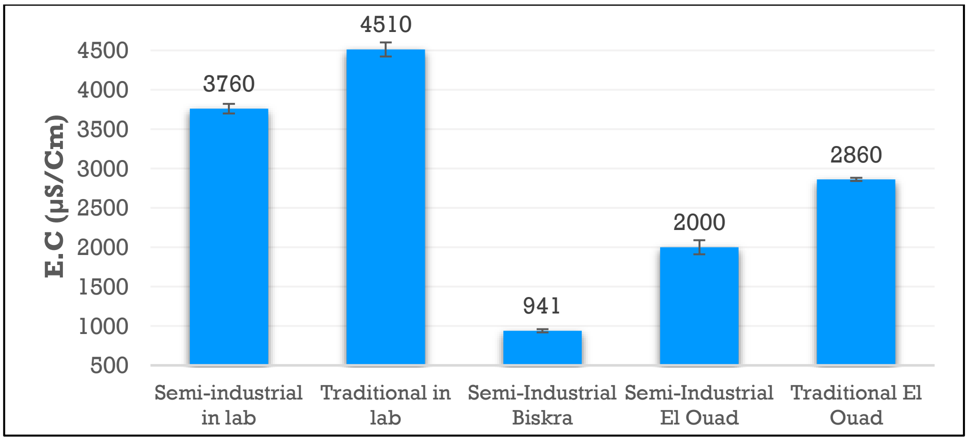



**Figure 38:** Electrical conductivity of different vinegar samples.

According to **Figure 38** results**,** the semi-industrial and traditional laboratory vinegar characterized by a 3760 ± 60 and 4510 ± 90 µS/cm values respectively. These values agree with those found by **Siddeeg *et al.* (2019)** and **Akarca *et al.* (2020)** with electrical values around 3100 and 4900 S/cm respectively. On the other hand **Hamden *et al.* (2022)**, noted a content of 5650 µS/cm which is higher than those obtained in our laboratory. This may be due to the raw materials used in vinegar production, the fermentation process and the storage time (**Hamden *et al.*, 2022)**.

Compared to the prepared samples, the other samples (commercial) have a lower electrical conductivity. It is likely that the low electrical conductivity is caused by the mineral-poor distilled water used in the process.

**D. Acetic Acid (Total acidity)**

The results in **figure 39** shows the dosage of acetic acid obtained values of different samples used. The statistical analyzes has shown that the results are not statistically significant ($\rho$<0.001) **(Appendix 4).**

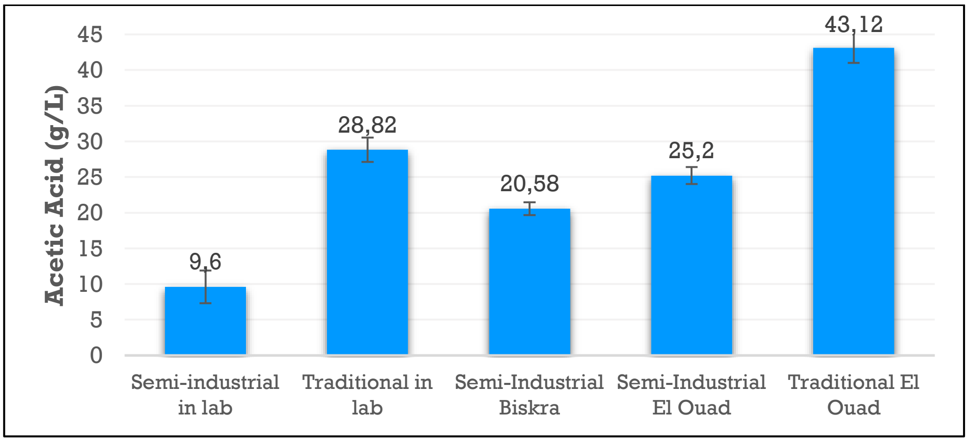



**Figure 39:** Acetic acid content of different samples of vinegar.

For traditional vinegar from the laboratory, the mean acetic acid value was about 28.82±1.7 g/l, close to those found by **Ould El-Hadj *et al.* (2001),** they reported acetic acid contents of 25.94 and 30.38g/l for vinegar made from the *H'Chef Deglet Nour* and *Harchaya* date varieties respectively; also it's close to that of **Elberkennou and Hafidi (2015),** who noted 23.41 and 27.62 g/l for *Hmira* and *Tinaceur* vinegar respectively.

The minimum levels are observed in the semi industrial laboratory vinegar with 9,6±2.3 g/l. This value is comparable to that found by **Hamden *et al.* (2022)** namely 4 g/l for the *Khalas* vinegar. Values that are confirmed by the North American legislation and set in more than 80% of the samples **(JUSTIA US Law, n.d.).** The maximum levels are observed in the traditional vinegar of El Ouad with a value of 43.12±2.12 g/l.

The degree of acidity for the semi industrial laboratory vinegar was about 3.81% which is represents 3.81°. These results are below the **Codex Alimentarius** vinegar values (at least 5% acetic acid). On the other hand, the result of traditional vinegar obtained in laboratory is 11.41%, which is 11.41°. These findings are in contrast with the values recommended by **the**

**Codex Alimentarius.** This non-conformity of a traditional product can be explained by the high oxygenation in the acetification process, where bacterial cells develop.

A Comparisons between different samples of date vinegar are shown in the following chart **(Fig. 40).**

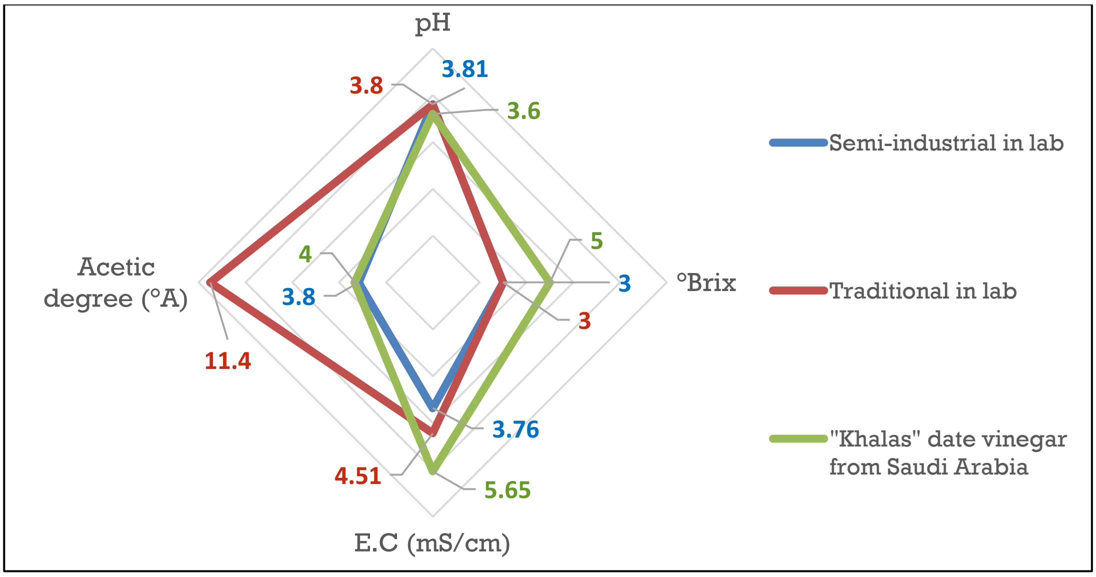



**Figure 40 :** Comparisons between different samples of date vinegar

There is an excellent similarity between *Khalas* vinegar and previous research since it is considered to be pure and high in quality according to standards. Based on a comparative analysis of *Khalas* vinegar and our product, there is a significant similarity specially the semi industrial one.

### 4.1.2 Bioethanol

The physicochemical analyzes of bioethanol are summarized in the following table **(Table 10).**

**Table 10:** Physicochemical analyzes of bioethanol.

| **Samples** | **Analyzes** | | |
|---|---|---|---|
| | **pH** | **°Brix** | **E.C (µS/Cm)** |
| Bioethanol | 3.27 | 3 | 20 |

The pH value was 3.27 close to those found by **Guetni-Larras, (2014),** there values was about 3; 3.1 for *Mech-Degla* and *Takerbucht* date varieties respectively. Yeast metabolism leads to a decrease in pH, which is directly connected to nitrogenous sources' assimilation. Additionally, carbon dioxide generated during alcoholic fermentation can dissolve in musts and further contribute to pH reduction **(Boulbaba *et al.*, 2013).**

The brix degree of bioethanol was 3%. This value are slightly lower than to results found by **Guetni-Larras, (2014),** they found 7.2; 6 for *Mech-Degla* and *Takerbucht* date varieties respectively. This lower value results from the transformation of sugars (sucrose) into ethanol by *Saccharomyces cerevisiae.* Additionally, it is due to the different sugars contents between the varieties **(Guetni-Larras, 2014).**

***Weight yield of bioethanol***

Weight yield is the ratio between bioethanol produced and fermented juice consumed, according to **Vogel** et **al. (1996),** is expressed as:

$$Yield\% = \left({}^{Volume\ of\ bioethanol}/_{Volume\ of\ fermented\ juice}\right) \times 100$$

**Table 11:** Biomass bioethanol weight yield.

| | |
|---|---|
| Volume of bioethanol in mL | 28 |
| Volume of fermented juice in mL | 400 |
| Yield of bioethanol in % | 7 % |

### 4.1.3 Date Syrup "*Robb*"

#### A. pH

The results in **figure 41** shows the pH obtained values of different syrup samples. The statistical analyzes has shown that the results are not statistically significant ($\rho$<0.05) **(Appendix 4).**

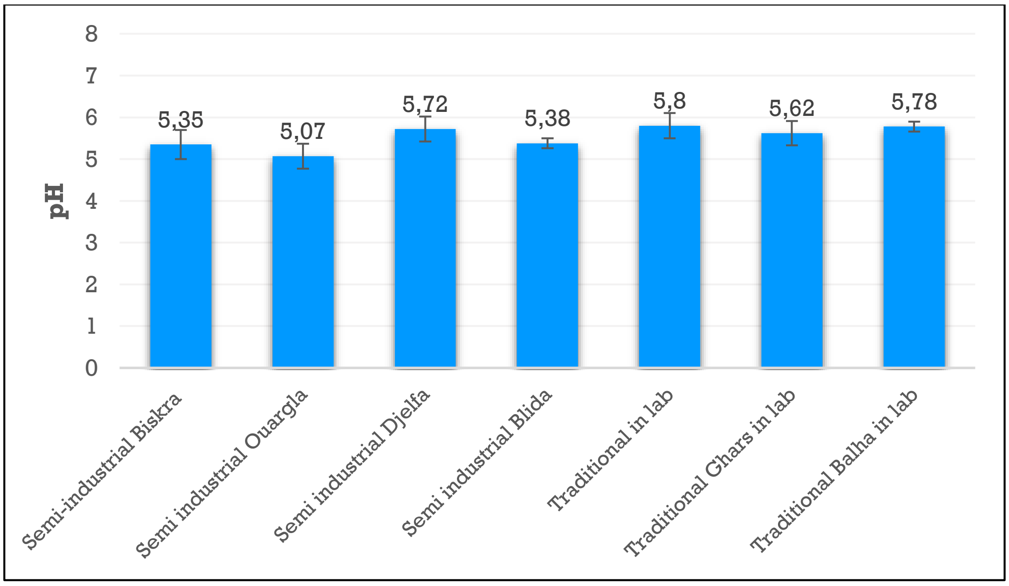



**Figure 41:** pH values of date syrup samples

The average of the pH of the date syrup samples vary between 5.35±0.35; 5.07±0.3; 5.72±0.3; 5.38±0.12; 5.8±0.3; 5.62±0.29; and 5.78±0.12, respectively for Semi-industrial in lab, traditional in lab, with an average of 4.70 for all the samples, the values are getting closer to each other. These values are associated with good quality dates **(Ould El-Hadj & Sayah, 2009)**, Ensuring a tart taste authenticated by the Codex Alimentarius standards **(Codex Alimentarius, 2001).** These pH values have advantages for the conservation of certain B vitamins such as vitamins B1, B2, B5, B9, and B12 predominant in dates **(Bourgeois, 2003).**

The pH determination informs us of the freshness of the sample and the nature of the raw material. Overall, the pH of the syrups is slightly acidic; it is in the range of 5. These values are comparable to those obtained by **Benahmed (2012)** and **Siboukeur (1997).**

**B. Total soluble solids (Brix degree)**

The results of the measurements of the total sugar content of syrup are shown in the graph below **(Fig. 42).** The statistical analyzes has shown that the results are not statistically significant ($\rho$<0.001) **(Appendix 4).**

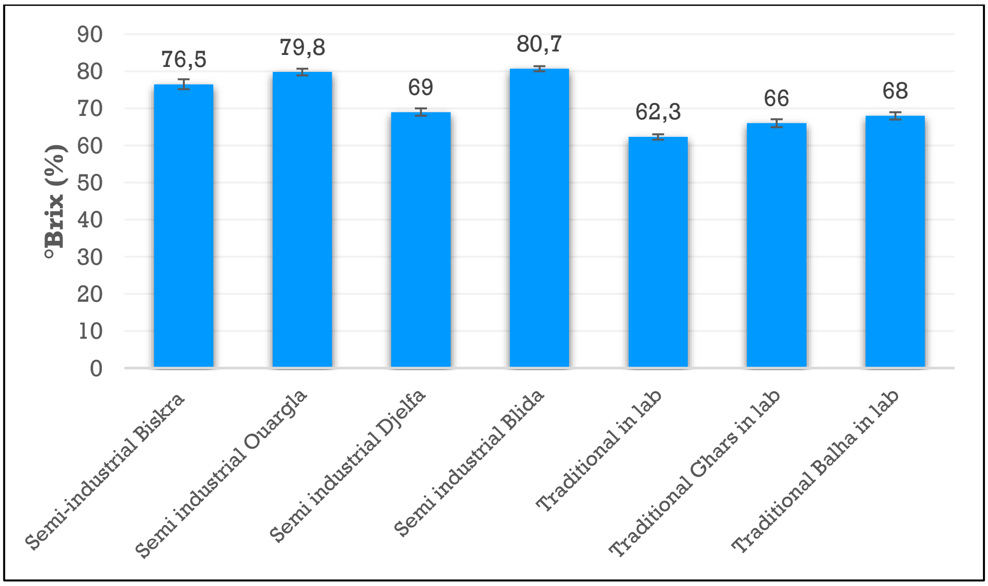



**Figure 42:** Brix degree of date syrup samples

The results obtained during the present study show that the TSS oscillates between 62.3 and 80.7 %. These values are not included in the interval reported by **(Alokaïdi, 1987; Abdelfatah, 1990; Ibrahim & Khalil1997).** Our results show that samples of the syrup traditional in laboratory has the lowest TSS rate with 62% and 66% .The results were directly compared with the previously reported findings by **Belguedj (2015)** who report °Brix values of 75% and 72.66% for “syrup” of *Ghars* dates respectively.

The concentration time has an effect on increasing the dissolved solids of date syrup. The longer the concentration time, the higher the consistency of the syrup, which is related to the evaporation of water (due to intense heating), varies from brand to brand depending on the dates used in the preparation and depends on the extraction technique used **(Benahmed, 2012).**

The heat treatment time has a great effect on the TSS of date syrup, the more the treatment time increases the more the TSS increases. This is explained by the release of soluble materials (sugars) from dates in the water **(Ould El-Hadj & Sayah, 2009).**

### C. Electrical Conductivity

The variations in electrical conductivity of the different samples studied are shown in the following graph **(Fig. 43).** The statistical analyzes has shown that the results are not statistically significant ($\rho$<0.001) **(Appendix 4).**

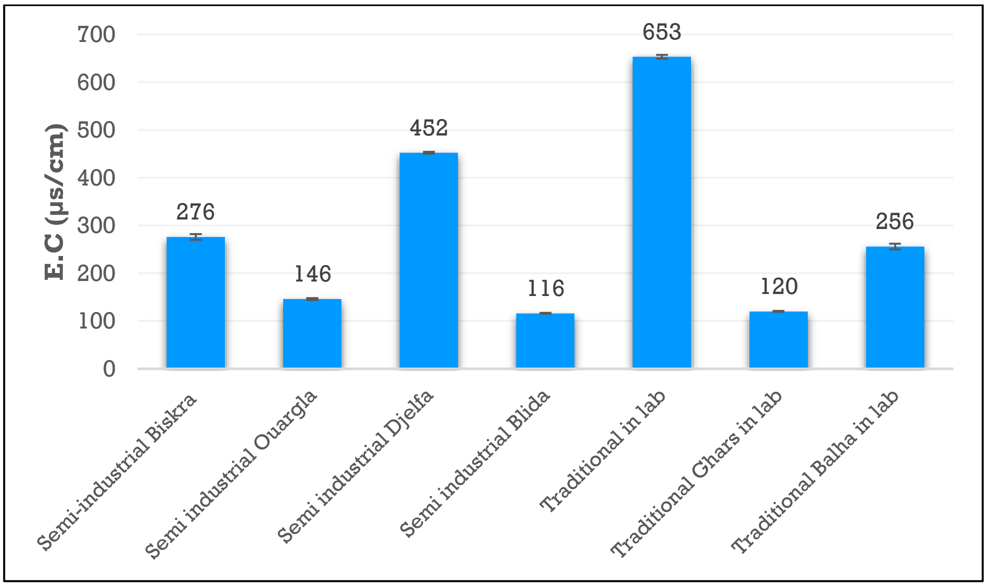



**Figure 43:** Electrical conductivity of date syrup samples

According to the **figure 43**, semi-industrial Djelfa and traditional in lab syrup characterized by a high value of electrical conductivity by a 452±2 and 653±4 µS/cm values respectively; compared to other samples which have almost similar values. The possibility that the reason for the decrease in the electrical conductivity in the other samples is due to the boiling of the condensate (concentration) , which led to the precipitation of salts and minerals, which led to a decrease in the electrical conductivity **(Mimouni, 2015).**.

### D. Total acidity

The results obtained by measuring the dosage of total acid of different syrup samples are represented in the following figure **(Fig. 44).** The statistical analyzes has shown that the results are not statistically significant ($\rho$<0.001) **(Appendix 4).**

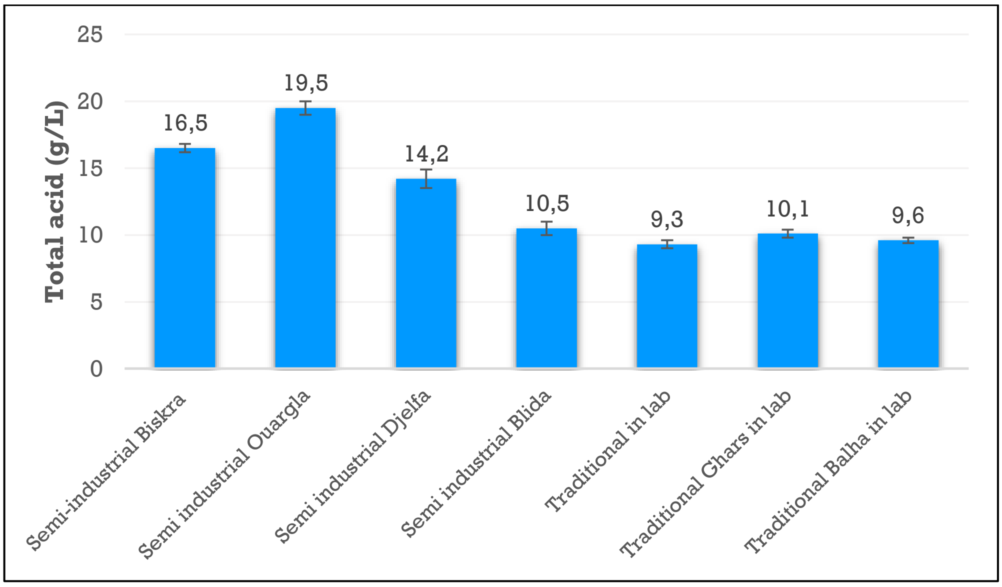



**Figure 44:** Total acid content in date syrup samples

According to the graph, the titratable acidity of the syrups studied varies between 9.3±0.3 g/L and 19.5±0.5 g/L; Ouragla's semi-industrial syrup has the highest value 19.5±0.5 g of citric acid per 100 g of syrup; the lowest value is recorded for the traditional syrup in lab 9.3±0.3 g/L. The values obtained are higher than those obtained by **Belguedj *et al.* (2015)** which vary between 3.20 g/L and 3.60 g/L. The variations observed may be due to two factors: the water content and the sugar nature of the raw material **(Mimouni, 2015).**

### 4.1.4 Date liquid sugar

The physicochemical analysis of liquid sugar are summarized in the following table **(Table 12).**

**Table 12:** Physicochemical analyzes of liquid sugar

| Samples | Analyzes | | |
|---|---|---|---|
| | **pH** | **°Brix** | **E.C (µS/Cm)** |
| liquid sugar: | 5.08 | 22 | 13.3 |

The pH is slightly acidic, it is close to the value of **Soltani (2007)** and **Benamara (2007)** with 5 and 6.14 respectively, but higher than the values of **Sibouker (1997)** with 4±0.005.

The content of soluble solid found does not conform to the results obtained by **Merabet (2014),** they report value of 62%, which means the lack of richness in sugar in soluble solid in this variety of dates.

The electrical conductivity is linked to the content of ionizable matter, of which the mineral matter constitutes the essential. It depends on the nature of the dissolved ions and their concentrations.

***Weight yield of liquid sugar***

**Table 13:** Biomass liquid sugar weight yield

| | |
|---|---|
| Volume of the liquid sugar in mL | 200 |
| Volume of date juice in mL | 400 |
| Yield of liquid sugar in % | 50 % |

The yield of the *Degla* variety in liquid sugar is 50 %. This high content in relation to the dry matter, provides an excellent advantage for replacing white sugar in food formulation.

## 4.2 Microbiological analyzes results

### 4.2.1 Date vinegar

After the incubation period is completed, the total coliform colonies can be observed clearly on the medium surface. Colonies tend to be pinkish or dark red in color **(Fig. 45).**

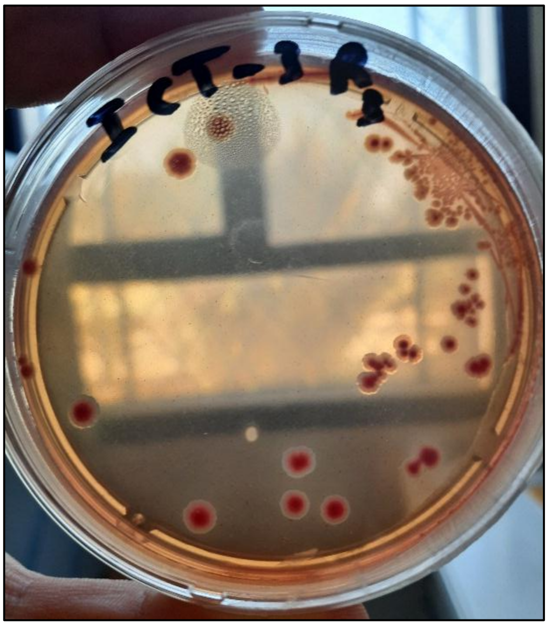


**Figure 45:** Total coliform on VRBL agar **(Original photography, 2023)**

The microbiological properties of the vinegar samples analyzed are listed in the next table **(Table 14).**

**Table 14:** Microbiological analyzes of vinegars samples after 72h.

| Samples | Microorganismes | | |
|---|---|---|---|
| | **Total coliforms(log 2 cfu/ml)** | **Fecal coliforms (log 2 cfu/ml)** | **Yeast and Molds (log 2 cfu/ml)** |
| Semi industrial in lab | 34 ±0.41 | Absence | 31±0.17 |
| Traditional in lab | 45.3±0.67 | 3.1±0.96 | 38±0.23 |
| Algerian Standards | $10^6$* | $10^3$* | <5000** |

* The decree of 23 July 1994 relating to the microbiological specifications of certain foodstuffs (Official Journal).
** NA 1207 /9 (IANOR).

It is known that the presence of coliforms in food indicates fecal contamination. Compared to semi industrial vinegar with 3400±41 germs/ml, traditional vinegar has a slightly higher result 4530±67 germs/ml. This may be explained by the lack of heat treatment for traditional vinegar, unlike semi-industrial vinegar which has undergone heat treatment (in the extraction of date juice). Or to the lack of hygiene practices throughout the production chain **(Boukhiar, 2009).**

In our study, there is a low level of mold and yeast in our vinegar samples comparing to the standards. The highest rate can be found in traditional vinegar, which has 3800±23 germs/ml of solution, while the semi-industrial vinegar has 3100±17 germs/ml. The mold causes fermentation to divert; this is evident by the bitter taste and unpleasant odor of our solutions **(Ould El Hadj *et al.*, 2001).** According to Algerian standards **(JORA, 1994),** the levels of yeasts and mold are below the maximum acceptable level. Due to this, the microbiological quality of the two varieties is satisfactory and complies with the standards.

### 4.2.2 Date Syrup "*Robb*"

The results of microbiological analysis of date syrup are summarized in the following table **(Table 15).**

**Table 15:** Microbiological analyzes of syrup samples after 72h.

| Samples | Microorganismes | | |
|---|---|---|---|
| | **Total coliforms(log 2 cfu/ml)** | **Fecal coliforms(log 2 cfu/ml)** | **Yeast and Molds (log 2 cfu/ml)** |
| Semi industrial Biskra | $2.03.10^1$ | Absence | Absence |
| Semi industrial Blida | $4.08.10^6$ | Absence | Absence |
| Traditional *Ghars* in lab | Absence | Absence | Absence |
| Traditional *Balha* in lab | Absence | Absence | Absence |
| Standards | $10^6$* | $10^3$* | $10^3$** |

* The decree of 2 July 2017 relating to the microbiological criteria of certain foodstuffs (Official Journal).
** NF ISO 4833-2008.

The value obtained for the count of total coliforms is 2030 germs/ml for semi industrial Biskra sample and $408.10^6$ germs/ml for the semi industrial Blida sample, according to the Algerian standard **(JORA, 2017),** the microbial load of the second sample is high in total coliforms than the standards.

The results obtained indicate a total absence of fecal coliforms from the analyzed syrup. The rate of conformity of the results is 100% for these absent germs. According to **Guiraud (1998),** This microbiological safety can be attributed to the quality of the raw materials, the efficiency of the heat treatment (concentrate) applied to the date molasses during its preparation, and the hygienic conditions under which the syrup was prepared The conformity analysis refers to the Algerian standards **(JORA, 2017)** relating to foodstuffs, these safety criteria must be imperatively respected.

The microbiological analysis of these samples showed a total absence of yeasts and molds. These results comply with the Algerian standard**.** The results obtained comply with international standards **(NF ISO 4833-2008)** where the acceptable limit value is $10^3$ CFU/ml. This is probably due to the good storage conditions. In addition, it can be proof that the products have undergone pasteurization.

### 4.2.3 Fungal flora

Four dominant fungi strains found were collected and purified from vinegar and syrup samples after 7days **(Fig. 46).**

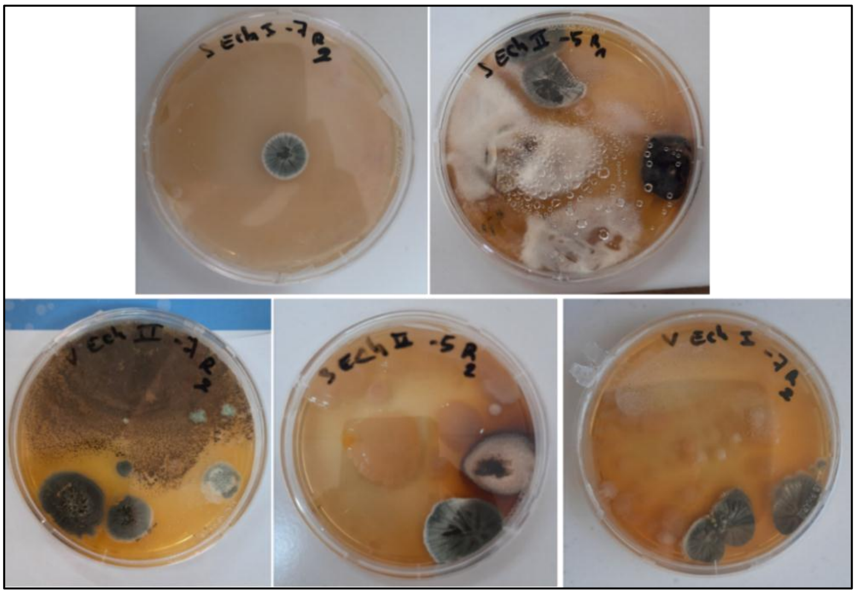


**Figure 46:** Fungi strains in vinegar and syrup on Sabouraud agar after 7days **(Original photography, 2023)**

Identification revealed that the 6 fungal isolates belonged to 3 genus: *Fusarium* sp., *Penicillium* sp. and *Aspergillus.*

*Aspergillus* includes two species:

- *Aspergillus flavus;*
- *Aspergillus niger.*

## I. Genus *Fusarium*

Based on identification keys from **Champion. (1997)**. Two isolates belong to the genus *Fusarium* sp. The colonies were circular to irregular shaped, white to cream color with aerial mycelium. *Fusarium* sp. are saprophytic fungi that produce macroconidia, microconidia, and chalmydospores as well as conidiasphores in large numbers **(Fig.47).**

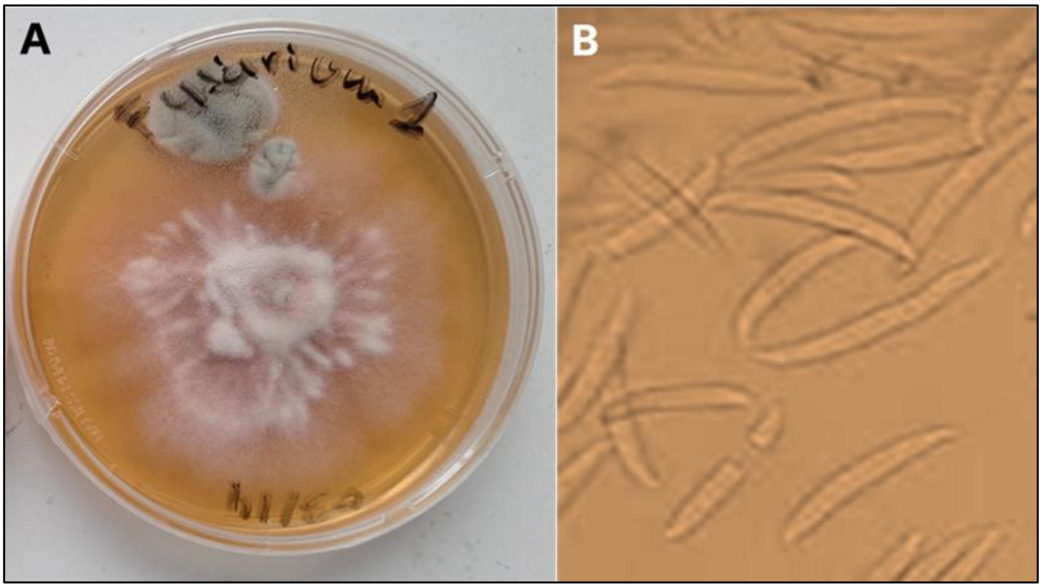



**Figure 47:** *Fusarium* sp.: **A)** Macroscopic observation; **B)** Microscopic observation (40x magnification) **(Original photography)**

## II. Genus *Penicillium*

According to macroscopic diagnosis, the color of the colonies appear blue-green in color with a yellowish surrounding. Microscopically it shows typical filamentous hyphae and conidia. The *Penicillium* sp. characterized with a colorless, branched, and septate hyphae with a long, cottony, or fluffy conidia **(Saif *et al.*, 2020) (Fig. 48).**

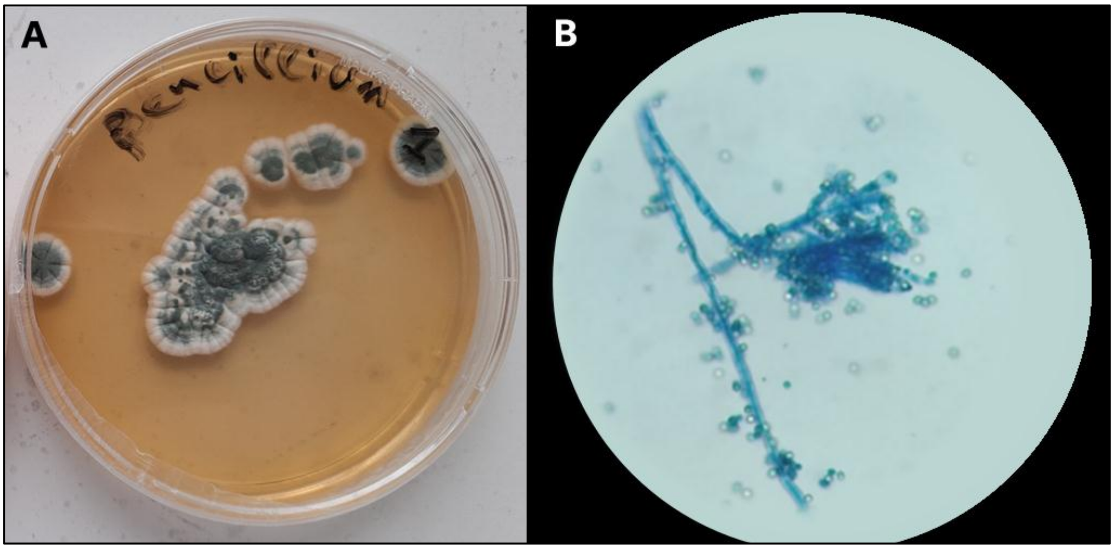



**Figure 48:** *Penicillium* sp.: **A)** Macroscopic observation; **B)** Microscopic observation (40x magnification) **(Original photography)**

### III. *Aspergillus flavus*

According to **Champion. (1997)**. One isolate was similar to *Aspergillus flavus*. The colonies appear powdery masses and yellowish-green color, they are characterized by hyphal growth that usually occurs by thread-like branching and produces mycelia as well as sexual spores (conidia) **(Fig.49).**

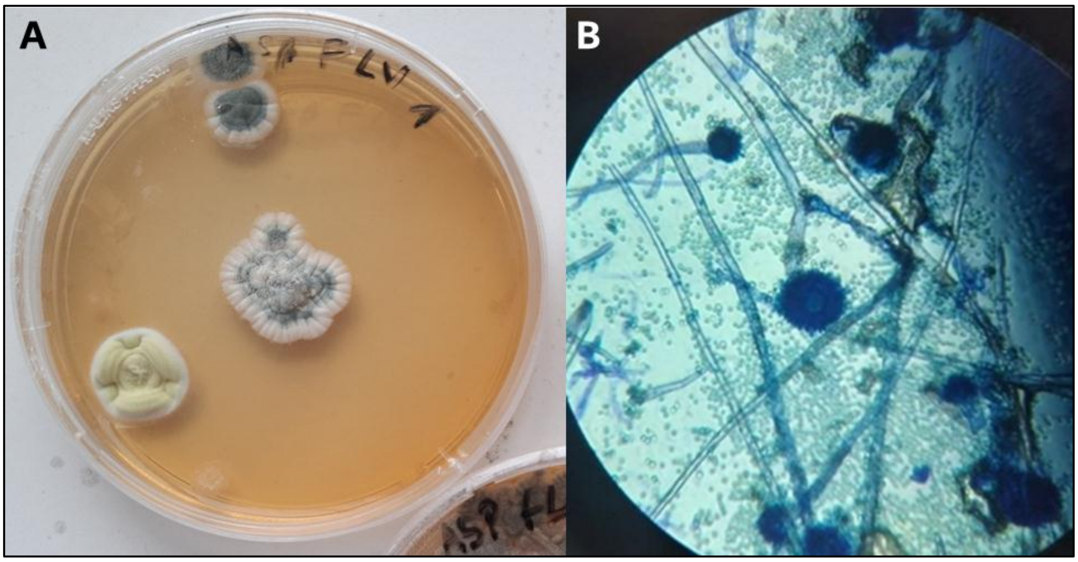



**Figure 49:** *Aspergillus flavus*: **A)** Macroscopic observation; **B)** Microscopic observation (40x magnification) **(Original photography)**

### IV. *Aspergillus niger*

In two isolates belonging to *Aspergillus niger* according to **Champion. (1997)**, the colony often invades the entire surface of the petri dish when grown on the Sabouraud medium.

This species grows rapidly. They have hyaline hyphae terminating in globose vesicles, and globose conidia with a rough texture and dark brown color **(Fig. 50).**

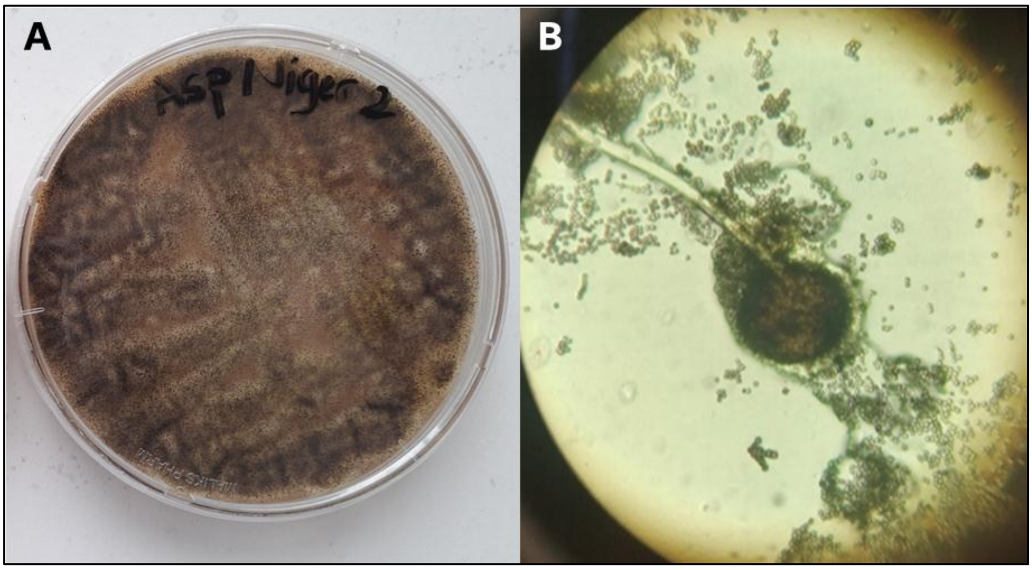



**Figure 50:** *Aspergillus niger:* **A)** Macroscopic observation; **B)** Microscopic observation (40x magnification) **(Original photography)**

### 4.2.4 Discussion

The presence of fungi strains in date-derived products can have significant implications for their quality and safety. Fungi usually grow on fruits with a high moisture content. By developing their mycelium outside the date, they can ferment date sugar **(Ghezzoul, 2010).**

The rate of the extreme deterioration can be attributed to storage genera' activity *Aspergillus* and *Penicillium*. Our identification of fungi strains is of 4 strains: *Fusarium* sp. with 33.33%, *Aspergillus niger* with 33.33%., *Penicillium* sp. with 16.66%, and *Aspergillus flavus* with 16.66%.

The appearance of this molds might be caused by:

• A wet cleaning of dates without dripping increases humidity and promoting fungus growth.

• For the *Ghars* variety, the vacuum at the "Betana" level increases respiration and humidity.

• Lack of humidity control and ventilation equipment in storage facilities.

• Mixing damaged dates with healthy ones.

• Lack of hygiene practices throughout the production chain.

• Lack or absence of adequate heat treatment for dates.

# ***Conclusion***

## Conclusion

The date is a fruit that constitutes the raw material for the elaboration of a good number of products such as vinegar, molasses, bioethanol, and liquid sugar. This thesis investigated and analyzed the physicochemical and microbiological characteristics of these products.

The varieties of dates used are semi-soft to dry in consistency: *Balha*, *Ghars*, *Degla-Beida*, and *Tamjouhert*.

The physicochemical analysis provided valuable insights into the composition, sensory attributes, and overall quality of the date-derived products. Parameters such as pH, TSS, electrical conductivity, and titratable acidity were thoroughly examined, allowing for a better understanding of the product characteristics and variations.

A high similarity rate was found between semi-industrial vinegar and standards compared to traditional vinegar in vinegar analyses. As a result, the semi-industrial approach is more reliable than the traditional approach.

As a result, traditional syrups were found to have superior quality across all the physicochemical and microbiological criteria studied. The results also revealed that traditional syrups were more nutritious and tasted more intensely than industrialized ones.

Microbiological evaluation of the products revealed that the total of traditional and semi-industrial derivatives samples meet international and national standards. Also, the presence of four different fungi species, namely *Fusarium* sp., *Aspergillus niger*, *Aspergillus flavus*, and *Penicillium* sp. These fungi species were found in vinegar and syrup samples after 7 days. Having this knowledge is critical to implementing quality control measures and ensuring product safety.

Furthermore, the investigation of different processing techniques unveiled the influence of various methods on the physicochemical and microbiological characteristics of date-derived products. The variations resulting from heat treatment, fermentation, and storage conditions were examined, providing valuable insights into the impact of processing on product quality.

Looking ahead, there are several prospectives areas for further research and improvement based on the findings of this thesis:

- Study of the organoleptic quality of vinegar, syrup and liquid sugar.
- Conduct further research on the therapeutic effects of this product.
- Look for different broad-spectrum antimicrobial activities and purify the bioactive molecules of date syrup and vinegar.
- Use the vinegar, syrup and liquid sugar as an ingredient in confectionery, baking, in the manufacture of beverages.

# ***References***

## References


1. **Aatef, M., & Nadif, M. (1995).** Le palmier dattier culture et entretient en payes arabes. *Ed Elmaaref. Egypte.*, 47-111.
2. **Adams, M. R. (1998).** Vinegar. *In: J. B. Wood (Ed.), Microbiology of Fermented Food*, 1-44.
3. **Ahmed, J., Ramaswamy, H. S., & Khan, A. (2005).** Effect of water activity on glass transitions of date pastes. *Journal of Food Engineering*, *66*(2), 253–258. https://doi.org/10.1016/j.jfoodeng.2004.03.015
4. **Aït Ameur, L. (2001).** *Analyse du processus de diffusion des sucres, des acides organiques et de l'acide ascorbique dans le système : Mech-Degla/Jus de citron.* [Master's thesis]. M'hamed Bougara University, Boumerdes.
5. **Akarca, G., Tomar, O., Caglar, A., & Istek, O. (2020).** Quality properties of date vinegar produced using conventional method. *Acad. J. Biotechnol. 8(8):*, 181–185. https://doi.org/10.15413/ajb.2020.0119
6. **Akbi, A. (2013).** *Les implications du développement des biocarburants.* [PhD's thesis]. Ecole doctorale 513 Droit Et Sciences Politiques, economiques et de Gestion Nice.
7. **Al-Alawi, R. A., Al-Mashiqri, J. H., Al-Nadabi, J. S. M., Al-Shihi, B., & Baqi, Y. (2017).** Date Palm Tree (Phoenix dactylifera L.): Natural Products and Therapeutic Options. *Frontiers in Plant Science*, *8*. https://doi.org/10.3389/fpls.2017.00845
8. **Al-Farsi, M. A., & Lee, C. (2008).** Nutritional and Functional Properties of Dates: A Review. *Critical Reviews in Food Science and Nutrition*, *48*(10), 877–887. https://doi.org/10.1080/10408390701724264
9. **Ali, Z., Li, J., Zhang, Y., Naeem, N., Younas, S., & Javeed, F. (2020).** Dates (*Phoenix Dactylifera*) and Date Vinegar: Preventive Role against Various Diseases and Related *in Vivo* Mechanisms. *Food Reviews International*, *38*(4), 480–507. https://doi.org/10.1080/87559129.2020.1735411
10. **Alkhateeb, A. (2008).** Enhancing the Growth of Date Palm (Phoenix Dactylifera) in Vitro Tissue by Adding Date Syrup to the Culture Medium. *Scientific Journal of King Faisal University (Basic and Applied Sciences), Vol. 9*(1).
11. **Al-Shahib, W., & Marshall, R. P. (2003).** The fruit of the date palm: its possible use as the best food for the future? *International Journal of Food Sciences and Nutrition*, *54*(4), 247–259. https://doi.org/10.1080/09637480120091982

12. **Andrews, W. H. (1996).** Validation of modern methods in food microbiology by AOAC INTERNATIONAL collaborative study. *Food Control*, *7*(1), 19–29. https://doi.org/10.1016/0956-7135(96)00007-2
13. **Arzate, A. (2005).** Extraction du Sucre de Betterave. *Center for Research, Development and Technology Transfer in Maple Syrup*, 13–27.
14. **Ashraf, Z., & Hamidi-Esfahani, Z. (2011).** Date and Date Processing: A Review. *Food Reviews International*, *27*(2), 101–133. https://doi.org/10.1080/87559129.2010.535231
15. **Azouz, C., & Oubacha, O. (2022).** *La valorisation des dattes Algériennes à travers la vente dans les boutiques spécialisées. Cas de la willaya de Tizi-Ouzou et Alger* [Master's thesis]. Mouloud Mammeri University, Tizi-Ouzou.
16. **Barreveld, W. H. (1993).** *Date palm products.* FAO Agricultural Services Bulletin No. 101. https://www.fao.org/3/t0681e/t0681e00.htm
17. **Belguedj, M. (2001).** Caractéristiques des cultivars de dattes dans les palmeraies du Sud-est. *Algérien, Ed. 3D. Alger*, 289.
18. **Benchelah, A., & Maka, M. (2008).** Les dattes: intérêt en nutrition. *Phytothérapie*. https://doi.org/10.1007/s10298-008-0296-0
19. **Benmbarek, S., & Deboub, I. (2015).** *Valorisation des sous-produits du palmier dattier et leurs utilisations* [Master's thesis]. Hamma Lakhdar University, El-Oued.
20. **Biobased Industrial Products. (2000).** *National Academies Press eBooks*. https://doi.org/10.17226/5295
21. **Booij, I., Piombo, G., Risterucci, A., Coupé, M., Thomas, D., & Ferry, M. (1992).** Etude de la composition chimique de dattes à différents stades de maturité pour la caractérisation variétale de divers cultivars de palmier dattier (Phoenix dactylifera L.). *Fruits*, *47*(6), 667–678. https://agritrop.cirad.fr/397808/
22. **Bouaziz, S. (2008).** *Caractérisation physicochimique et biochimique de quelques vinaigres traditionnels de dattes de la région d'Ouargla.* [Master's thesis]. Kasdi Merbah University, Ouargla.
23. **Bouguedoura, N., Bennaceur, M., Babahani, S., & Benziouche, S. E. (2015).** Date Palm Status and Perspective in Algeria. *Springer eBooks*, 125–168. https://doi.org/10.1007/978-94-017-9694-1_4

24. **Boukhelkhal, K. (2021).** *Caractéristiques physico-chimiques et microbiologiques de quelques échantillons du miel de la wilaya de Laghouat* [Master's thesis]. Amar Thelidji University, Laghouat.
25. **Boukhiar, A. (2009).** *Analyse du processus traditionnel d'obtention du vinaigre de dattes tel qu'appliqué au sud algérien : essai d'optimisation.* [Master's thesis]. M'Hamed Bougara University. Boumerdes.
26. **Boulbaba, L., Belgaib, J., Benamor, H., & Hajji, N. (2013).** Production of bio-ethanol from three varieties of dates. *Renewable Energy*, *51*, 170–174.
27. **Bourgeois, C. (2003).** *Les vitamines dans les industries agroalimentaires*. Tech & doc.
28. **Bourgeois, C. M., & Larpent, T. P. (1996).** *Microbiologie alimentaire. Aliment fermentés et fermentation alimentaires.* (2nd ed.). Tec & doc-Lavoisier.
29. **Bourgeois, C. M., & Mafrt, P. (1991).** Technologie globales d'évaluations de la microflore in : tehnique d'analyse et de contrôle dans les industries agro-alimentaire. *Ed. Tec & Doc Lavoisier APRIA*, *3*, 50-71.
30. **Bouzaheur, N. (2016).** *Etude comparative entre deux produits à base de datte : miel de datte traditionnel sirop de datte* [Engineering thesis]. University of Batna 1.
31. **Branger, A., Richer, M., & Roustel, S. (2007).** *Alimentation, sécurité et contrôles microbiologiques*. Educagri Editions.
32. **Bukhari, M., Zaibag, M., Al-Hinai, S., Al-Moslih, M. I., & Sahih, A. (1976).** The Collection of Authentic Sayings of Prophet Mohammad (Peace be Upon Him), Division 71 on medicine. *Division 71 on Medicine, Hilal Yayinlari, Ankara, Turkey, 2nd Edition*.
33. **Champion, R. (1997).** Identifier les champignons transmis par les semences. In *HAL (Le Centre pour la Communication Scientifique Directe)*. Le Centre pour la Communication Scientifique Directe.
34. **Chao, C. T., & Krueger, R. F. (2007).** The Date Palm (Phoenix dactylifera L.): Overview of Biology, Uses, and Cultivation. *Hortscience*, *42*(5), 1077–1082. https://doi.org/10.21273/hortsci.42.5.1077
35. **Chelghoum. (2012).** *Essai de production de biomasse "Saccharomyces cerevisiae" à partir des dattes "Ghars"* [Engineering thesis]. University of Batna 1.

36. **Chibane, H. (2008).** *Aptitude technologiques de quelques variétés communes des dattes : formulation d'un yaourt naturellement sucré et aromatisé.* [PhD thesis]. M'hamed Bougara University, Boumerdes.
37. **Clavet, L. (1992).** *Alcool méthylique, Vinaigre*. Ed, Béranger, Paris et liège.
38. **Codex Alimentarius. (2001).** Programme mixte FAO/OMS sur les normes alimentaires commission du Codex Alimentarius. *ALINORM 01/25*, Pp.1-31.
39. **Cruess, W. V. (1957).** *Commercial fruit and vegetable products: Chapter 21 – Vinegar manufacture.* (1st Ed.). McGraw-Hill Book Company, Inc.
40. **Dahmani, S., & Rebbouh, I. (2009).** *Etude comparative des caractéristiques physicochimiques de différents types de vinaigres : le vinaigre traditionnel de datte (Deglet Nour, Degla Beida, Tacherwit), vinaigre de pommes et vinaigre vendu en épicerie.* [Master's thesis]. University of Kasdi Merbeh, Ouargla.
41. **Dalena, F., Senatore, A., Iulianelli, A., Di Paola, L., Basile, M., & Basile, A. (2019).** Ethanol from Biomass. *Elsevier eBooks*, 25–59. https://doi.org/10.1016/b978-0-12-811458-2.00002-x
42. **De Cruitis, M. L., Franceschi, O., & De Castro, N. (2002).** Listeria monocytogenes in vegtables minimallt processed. *Archivos Latino Americanos De Nutricion (Venzuela)*, *52*(3), 282-288.
43. **Dhenge, R., Langialonga, P., Alinovi, M., Lolli, V., Aldini, A., & Rinaldi, M. (2022).** Evaluation of quality parameters of orange juice stabilized by two thermal treatments (helical heat exchanger and ohmic heating) and non-thermal (high-pressure processing). *Food Control*, *141*, 109150. https://doi.org/10.1016/j.foodcont.2022.109150
*44.* **Djouab, A. (2007).** Contribution à l'identification des constituants mineurs de la datte Mech-degla. *M'Hamed Bougara University. Boumerdes.*
45. **Dziezak, J. D. (1987).** *Rapid methods for microbiological analysis of food* (Vol. 41). Institute of Food Technologists, USA.
46. **Eid, S. A. (2006).** Chromatographic separation of fructose from date syrup. *International Journal of Food Sciences and Nutrition*, *57*(1–2), 83–96. https://doi.org/10.1080/09637480600658286
47. **Elberkennou, H., & Hafidi, M. (2015).** *Contribution à l'étude des caractéristiques physico-chimiques et biochimiques du vinaigre traditionnel de dattes ' Hmira, Tinaceur ' de la cuvette d'Adrar* [Master's thesis]. Ahmed Draia University, Adrar.

48. **El-Ogaidi, A. (2000).** *The date palm agronomic and industrial technological science.* Dar ezahran, Oman.
49. **Eoin, L. N. (2016).** Systematics: Blind dating. *Nature Plants*, *2*(5). https://doi.org/10.1038/nplants.2016.69
50. **Ernoul, R. (2004).** Gestion pratique des contrôles dans l'industrie. *MANAGEMENT DE LA QUALITÉ - VII-84*.
51. **Espiard, É. (2002).** *Introduction à la transformation industrielle des fruits*. Tec & Doc Lavoisier.
52. **Ethyl Alcohol. (2017).** *ACS Reagent Chemicals*. https://doi.org/10.1021/acsreagents.4135
53. **Fan, Z. (2014).** Consolidated Bioprocessing for Ethanol Production. *Elsevier eBooks*, 141–160. https://doi.org/10.1016/b978-0-444-59498-3.00007-5
54. ***FAOSTAT*. (n.d.).** FAO - Food and Agriculture Organization of the United Nations. https://www.fao.org/faostat/en/#home
55. **Faradji-Hamma, S. (2016).** Technique de contrôle microbiologique des aliments [Cours]. *Département De Microbiologie Techniques*.
56. **Follman., H. (1983).** Acetic acid. *In: H.J. Rehm and G.ReedEditors, Vol. 5, Chap. 3,* 388–407.
57. **Gheraissa, T., & Hamidani, I. (2018).** *Etude de quelques caractéristiques physico-chimiques du sirop traditionnel des dattes de deux variétés (Ghars et Tinissine)* [Master's thesis]. Hamma Lakhdar University.
58. **Ghezzoul, F. (2010).** *Les maladies fongiques des dattes en stockage du palmier dattier Phoenix dactylifera L dans la région de Ouargla* [Master's thesis]. Kasdi Merbah University, Ouargla.
59. **Ghnimi, S., Umer, S. M., Karim, A., & Kamal-Eldin, A. (2017).** Date fruit (Phoenix dactylifera L.): An underutilized food seeking industrial valorization. *NFS Journal*, *6*, 1–10. https://doi.org/10.1016/j.nfs.2016.12.001
60. **Goudjal, Y. (2022).** *Contrôle microbiologique des aliments* [Cours handout]. Amar Telidji University of Laghouat.
61. **Guetni-Larras, L. (2014).** *Production de bioéthanol à partir de dattes de faible valeur marchande* [Masters' thesis]. Saad Dahleb-Blida University.
62. **Guiraud, J. P. (2012).** *Microbiologie alimentaire*. Ed. Dunod.
63. **Guiraud, J., & Rosec, J. (2004).** *Pratique des normes en microbiologie alimentaire*.

64. **Gullo, M., & Giudici, P. (2008).** Acetic acid bacteria in traditional balsamic vinegar: Phenotypic traits relevant for starter cultures selection. *International Journal of Food Microbiology*, *125*(1), 46–53. https://doi.org/10.1016/j.ijfoodmicro.2007.11.076
65. **Hadrami, A. E., & Al-Khayri, J. M. (2012).** Socioeconomic and traditional importance of date palm. *Emirates Journal of Food and Agriculture*, *24*(5), 371–385. https://www.bibliomed.org/?mno=185750
66. **Hafzan, Y., Saw, J. W., & Fadzilah, I. (2017).** Physicochemical properties, total phenolic content, and antioxidant capacity of homemade and commercial date (Phoenix dactylifera L.) vinegar. *International Food Research Journal*, *24*, 2557–2562.
67. **Halkman, H. B. D., & Halkman, A. K. (2014).** Indicator Organisms. *Elsevier eBooks*, 358–363. https://doi.org/10.1016/b978-0-12-384730-0.00396-7
68. **Hamden, Z., El-Ghoul, Y., Alminderej, F. M., Saleh, S. M., & Flamini, G. (2022).** High-Quality Bioethanol and Vinegar Production from Saudi Arabia Dates: Characterization and Evaluation of Their Value and Antioxidant Efficiency. *Antioxidants*, *11*(6), 1155. https://doi.org/10.3390/antiox11061155
69. **Harrak, H., & Boujnah, M. M. (2012).** *Valorisation Technologique des Dattes*. INRA Edition, Maroc.
70. **Hatanaka, M., Bain, C., & Busch, L. (2005).** Third-party certification in the global agrifood system. *Food Policy*, *30*(3), 354–369. https://doi.org/10.1016/j.foodpol.2005.05.006
71. **Hazzani, A. a. A., Shehata, A. I., Rizwana, H., Moubayed, N. M. S., Alshatwi, A. A., Munshi, A., & El-Gaaly, G. A. (2014).** Postharvest fruit spoilage bacteria and fungi associated with date palm (Phoenix dactylifera L) from Saudi Arabia. *African Journal of Microbiology Research*, *8*(11), 1228–1236. https://doi.org/10.5897/ajmr2013.5378
72. **Hernández-Pérez, T., Carrillo-López, A., Guevara-Lara, F., Cruz-Hernández, A., & Paredes-López, O. (2005).** Biochemical and Nutritional Characterization of Three Prickly Pear Species with Different Ripening Behavior. *Plant Foods for Human Nutrition*, *60*(4), 195–200. https://doi.org/10.1007/s11130-005-8618-y
73. **Jain, S. M., Al-Khayri, J. M., & Johnson, D. V. (2011).** *Date Palm Biotechnology*. Springer Science & Business Media.

74. **JORA. (1997).** Arrêté sur le vinaigre. *Journal Officiel De La République Algérienne. Vol 18 N° 17.*
75. ***JUSTIA US Law*. (n.d.).** JUSTIA. Retrieved April 8, 2023, from https://law.justia.com/codes/rhode-island/2014/title-21/chapter-21-22/section-21-22-2/
76. **Kamal-Eldin, A., & Ghnimi, S. (2018).** Classification of date fruit (Phoenix dactylifera, L.) based on chemometric analysis with multivariate approach. *Journal of Food Measurement and Characterization*, *12*(2), 1020–1027. https://doi.org/10.1007/s11694-018-9717-4
77. **Kehrer, C. L. (1921).** The chemistry of vinegar. *Journal of Food Product and the American Vinegar Industry 1: 5-20.*
78. **Krueger, R. F. (2021).** Date Palm (Phoenix dactylifera L.) Biology and Utilization. *Compendium of Plant Genomes*, 3–28. https://doi.org/10.1007/978-3-030-73746-7_1
79. **Leyral, G., & Vierling, E. (2007).** *Microbiologie et toxicologie des aliments: Hygiène et sécurité alimentaires*. Canopé - CRDP de Bordeaux.
80. **Mas, A. J., Troncoso, A. M., García-Parrilla, M. C., & Torija, M. J. (2016).** Vinegar. *Elsevier eBooks*, 418–423. https://doi.org/10.1016/b978-0-12-384947-2.00726-1
81. **McMillan, J. D. (1994).** Pretreatment of Lignocellulosic Biomass. In *ACS symposium series* (pp. 292–324). American Chemical Society. https://doi.org/10.1021/bk-1994-0566.ch015
82. **Metelf, S., Zidane, A., & Gadouche, L. (2022).** Évaluation de la qualité physico-chimique d’un jus de fruit soumis à quelques traitements thermiques durant sa conservation. *Revue Nature Et Technologie, Hassiba Benbouali University of Chlef, Algeria.*, 34–41.
83. **Mimouni, Y. (2015).** *Développement de produits diététiques hypoglycémiants à base de dattes molles variété «Ghars», la plus répandue dans la cuvette de Ouargla* [PhD dissertation]. Kasdi Marbah, Ouargla University.
84. **Mimouni, Y., & Siboukeur, O. (2011).** Etude des propriétés nutritives et diététiques des sirops de dattes extraits par diffusion, en comparaison avec les sirops à haute teneur en fructose (isoglucoses), issus de l’industrie de l’amidon. *Annales Des Sciences Et Technologie*, *3*(1), 1–11.
85. **Morales, L., González, G. S., Casas, J. A., & Troncoso, A. M. (2001).** Multivariate analysis of commercial and laboratory produced Sherry wine vinegars: influence of

acetification and aging. *European Food Research and Technology*, *212*(6), 676–682. https://doi.org/10.1007/s002170100301

86. **Mossel, D. A. A., & Thomas, G. (1988).** Securite microbioligique des plats prepares refrigeres: recommendations en matiere d'analyse des risques, conception et surveillance du processus de fabrication. *Microbiol. Aliments Nutr.*, *6,* 289-309.
87. **Moussaoui, K., & Necib, S. (2019).** *Comparison des propriétés physico-chimique du vinaigre de pomme et de datte (Degla-Beida)* [Master's thesis]. Hamma Lakhdar university, El Oued.
88. **Munier, P. (1973).** *Le palmier-dattier.* Maisonneuve & Larose, Paris.
89. **Nguyen, T. D. (2016).** *Protection de la levure Saccharomyces cerevisiae par un système biopolymérique multicouche : effet sur son activité métabolique en réponse aux conditions de l'environnement.* [PhD dissertation]. University of Burgundy-Dijon, France.
90. **Nurhayati, N., Aisyah, I., Sundari, C. D. D., Sunarya, R. R., & Suryaningsih, S. (2023).** Optimization of bioethanol production from jackfruit straw waste through the addition of a starter and fermentation duration. *AIP Conference Proceedings*. https://doi.org/10.1063/5.0118625
91. ***Nutrition*. (n.d.).** PasseportSanté. https://www.passeportsante.net/portail/nutrition
92. **Ould El-Hadj, M., & Sayah, Z. (2009).** Etude comparative des caracteristiques physico-chimiques et biochimiques des dattes de la cuvette d'ouargla. *Annales Des Sciences Et Technologie*, *2*(1), 87–92.
93. **Ould El Hadj, M. D., Sebihi, A. H., & Siboukeur, O. (2001).** Qualité Hygiénique et Caractéristiques Physico-Chimiques du Vinaigre Traditionnel de Quelques Variétés de Dattes de la Cuvette d'Ouargla. *Revue Energie Renouvelable : Production Et valorisation-Biomasse*, 87-92.
94. **Ousaaid, D., Mechchate, H., Laaroussi, H., Hano, C., Bakour, M., Ghouizi, A. E., Conte, R., Lyoussi, B., & ElArabi, I. (2021).** Fruits Vinegar: Quality Characteristics, Phytochemistry, and Functionality. *Molecules*, *27*(1), 222. https://doi.org/10.3390/molecules27010222
95. **Petransxiène, D., & Lapied, P. (1981).** *Qualité Bactériologique du Lait et des Produits Laitiers, Analyses et Test*. Ed. Technique et Documentation Lavoisier, Paris.

96. **Pingret, D., Fabiano-Tixier, A., & Chemat, F. (2013).** Degradation during application of ultrasound in food processing: A review. *Food Control*, *31*(2), 593–606. https://doi.org/10.1016/j.foodcont.2012.11.039

97. **Plessi, M. (2003).** VINEGAR. *Elsevier eBooks*, 5996–6004. https://doi.org/10.1016/b0-12-227055-x/01251-7

98. **Razavi, S. M. A., & Karazhiyan, H. (2012).** Rheological and Textural Characteristics of Date Paste. *International Journal of Food Properties*, *15*(2), 281–291. https://doi.org/10.1080/10942912.2010.483615

99. **Razi, M. (1993).** *Contribution à l'étude de la valeur nutritive du jus de dattes de quatre variétés molles « Ghars, Litima, Tansilt et Takermoust »en comparaison avec le miel d'abeilles.* [Engineering Thesis]. I.T.D.A.S, Ouargela.

100. **Reuveni, O. (1986).** Date. *In: S.P. Monselise (Ed.). CRC Handbook of Fruit Set and Development.* 119–144.

101. **Risiquat, R. O. (2013).** Microbiological Assessment Of Date Fruits Purchased From Owode Market, In Offa, Kwara State Nigeria. *IOSR Journal of Environmental Science, Toxicology and Food Technology*, *4*(3), 23–26. https://doi.org/10.9790/2402-0432326

102. **Saafi, E. B., Louedi, M., Elfeki, A., Zakhama, A., Najjar, M. F., Hammami, M., & Achour, L. (2011).** Protective effect of date palm fruit extract (Phoenix dactylifera L.) on dimethoate induced-oxidative stress in rat liver. *Experimental and Toxicologic Pathology*, *63*(5), 433–441. https://doi.org/10.1016/j.etp.2010.03.002

103. **Saif, F. A., Yaseen, S. A., Alameen, A. S., Mane, S. B., & Undre, P. B. (2020).** Identification of Penicillium Species of Fruits Using Morphology and Spectroscopic Methods. *Journal of Physics*. https://doi.org/10.1088/1742-6596/1644/1/012019

104. **Sea, F. (2013).** Physicochemical-Microbiological Studies on Irradiated Date Fruits with Studying Migration Monomers of Packages Materials. *Journal of Microbial & Biochemical Technology*, *05*(01). https://doi.org/10.4172/1948-5948.1000091

105. **Shomer, I., Borochov-Neori, H., Luzki, B., & Merin, U. (1998).** Morphological, structural and membrane changes in frozen tissues of Madjhoul date

(Phoenix dactylifera L.) fruits. *Postharvest Biology and Technology*, *14*(2), 207–215. https://doi.org/10.1016/s0925-5214(98)00029-5

106. **Siddeeg, A., Zeng, X., Rahaman, A., Manzoor, M. F., Ahmed, Z., & Rahaman, A. (2019).** Quality characteristics of the processed dates vinegar under influence of ultrasound and pulsed electric field treatments. *Journal of Food Science and Technology*, *56*(9), 4380–4389. https://doi.org/10.1007/s13197-019-03906-3

107. **Singh, A. K. (2020).** Overview of vinegar production. *PalArch's Journal of Archaeology of Egypt / Egyptology, 17(6).* 4027–4037.

108. **Sirisena, S., Ng, K., & Ajlouni, S. (2015).** The Emerging Australian Date Palm Industry: Date Fruit Nutritional and Bioactive Compounds and Valuable Processing By-Products. *Comprehensive Reviews in Food Science and Food Safety*, *14*(6), 813–823. https://doi.org/10.1111/1541-4337.12162

109. **Tan, S. C. (2005).** *Vinegar fermentation* [Master's thesis]. Louisiana State University.

110. **Tesfaw, A., & Assefa, F. (2014).** Current Trends in Bioethanol Production by *Saccharomyces cerevisiae*: Substrate, Inhibitor Reduction, Growth Variables, Coculture, and Immobilization. *International Scholarly Research Notices*, *2014*, 1–11. https://doi.org/10.1155/2014/532852

111. **Vaitilingom, G., Mouloungui, Z., Benoist, A., Broust, F., Daho, T., & Piriou, B. (2021).** Vers une génération plus « verte » de biodiesels. *Oilseeds and Fats, Crops and Lipids*, *28*, 2. https://doi.org/10.1051/ocl/2020067

112. **Vinson, J. A., Zubik, L., Bose, P., Samman, N., & Proch, J. (2005).** Dried Fruits: Excellent*in Vitro*and*in Vivo*Antioxidants. *Journal of the American College of Nutrition*, *24*(1), 44–50. https://doi.org/10.1080/07315724.2005.10719442

113. **Vogel, A. I., Tatchell, A. R., Smith, P. W. G., Hannaford, A. J., & Furnis, B. S. (1996).** *Vogel's Textbook of Practical Organic Chemistry* (5th Ed.). Prentice Hall.

114. **Walker, G. M. (2010).** *Bioethanol: Science and technology of fuel alcohol*. Vents Publishing ApS.

115. **World Health Organization. & Food and Agriculture Organization. (1987).** Draft European Regional Standard for Vinegar. In *Codex Alimentarius Commission.*

116. **Wyman, C. E. (2001).** Twenty Years of Trials, Tribulations, and Research Progress in Bioethanol Technology: Selected Key Events Along the Way. *Applied Biochemistry and Biotechnology*, *91–93*(1–9), 5–22. https://doi.org/10.1385/abab:91-93:1-9:5

117. **Wyman, C. E. (2018).** Handbook on Bioethanol. In *Routledge eBooks*. https://doi.org/10.1201/9780203752456

118. **Zaid, A., & De J Arias-Jiménez, E. (1999).** Date palm cultivation. *Food and Agriculture Organization of the United Nations eBooks*, *156*. http://ci.nii.ac.jp/ncid/BA65262549

# *Appendix*

## Appendix

### Appendix 1: Date juice extraction


**Figure :** Date juice extraction **(Original photography, 2023)**

### Appendix 2: Syrup production


**Figure :** Syrup production **(Original photography, 2023)**

## Appendix 3: Growth medium

### Violet Red Bile Agar with Lactose

| | |
|---|---|
| Peptone | 7g |
| Sodium Chloride | 5g |
| Yeast extract | 3g |
| Neutral Red | 0.03g |
| Bile salts | 1.5g |
| Violet Crystal | 0.002g |
| Lactose | 10g |
| Agar | 15g |
| Distilled water | 1000 ml |

pH = 7.5

### Sabouraud Dextrose Agar (SDA)

| | |
|---|---|
| Glucose | 40g |
| Peptic digestion of animal tissues | 10g |
| Agar | 15g |
| Distilled water | 1000 ml |

pH = 5.6

## Appendix 4: One way Analysis Of Variance (ANOVA) statistical analyzes tables

### 1) Vinegar

**pH**

| Source | DDL | Somme des carrés | Moyenne des carrés | F | Pr > F | Codes de signification des p-valeurs |
|---|---|---|---|---|---|---|
| Modèle | 4,000 | 1,382 | 0,346 | 35,616 | **<0,0001** | *** |
| Erreur | 9,000 | 0,087 | 0,010 | | | |
| Total corrigé | 13,000 | 1,469 | | | | |

*Calculé contre le modèle Y=Moyenne(Y)*

*Codes de signification : 0 < *** < 0.001 < ** < 0.01 < * < 0.05 < . < 0.1 < ° < 1*

**Brix degree**

| Source | DDL | Somme des carrés | Moyenne des carrés | F | Pr > F | Codes de signification des p-valeurs |
|---|---|---|---|---|---|---|
| Modèle | 4,000 | 28,381 | 7,095 | 268,253 | **<0,0001** | *** |
| Erreur | 9,000 | 0,238 | 0,026 | | | |
| Total corrigé | 13,000 | 28,619 | | | | |

*Calculé contre le modèle Y=Moyenne(Y)*

*Codes de signification : 0 < *** < 0.001 < ** < 0.01 < * < 0.05 < . < 0.1 < ° < 1*

**Electrical conductivity**

| Source | DDL | Somme des carrés | Moyenne des carrés | F | Pr > F | Codes de signification des p-valeurs |
|---|---|---|---|---|---|---|
| Modèle | 4,000 | 771,124 | 192,781 | 46892,669 | **<0,0001** | *** |
| Erreur | 9,000 | 0,037 | 0,004 | | | |
| Total corrigé | 13,000 | 771,161 | | | | |

*Calculé contre le modèle Y=Moyenne(Y)*
Codes de signification : 0 < *** < 0.001 < ** < 0.01 < * < 0.05 < . < 0.1 < ° < 1

**Acetic acid**

| Source | DDL | Somme des carrés | Moyenne des carrés | F | Pr > F | Codes de signification des p-valeurs |
|---|---|---|---|---|---|---|
| Modèle | 4,000 | 1795,760 | 448,940 | 287,414 | **<0,0001** | *** |
| Erreur | 10,000 | 15,620 | 1,562 | | | |
| Total corrigé | 14,000 | 1811,380 | | | | |

*Calculé contre le modèle Y=Moyenne(Y)*
Codes de signification : 0 < *** < 0.001 < ** < 0.01 < * < 0.05 < . < 0.1 < ° < 1

**Physicochemical vinegar samples resualts**

| **Samples** | **Analyzes** | | | | |
|---|---|---|---|---|---|
| | **pH** | **°Brix** | **E.C (mS/cm)** | **Acetic Acid (g/L)** | **Acetic degree (°A)** |
| Semi-industrial in lab | 3,81±0.06$^{a}$ | 3±0.2$^{c}$ | 3,76±0.06$^{b}$ | 9,6±2.3$^{d}$ | 3.81±0.0 |
| Traditional in lab | 3,8±0.11$^{a}$ | 3±0.06$^{c}$ | 4,51±0.09$^{a}$ | 28,82±1.7$^{b}$ | 11.41±0.0 |
| Semi-Industrial Biskra | 2,93±0.16$^{d}$ | 1,8±0.15$^{d}$ | 9.41±0.02$^{e}$ | 20,58±0.9$^{c}$ | 8.16±0.0 |
| Semi-Industrial El Ouad | 3,37±0.04$^{c}$ | 3,8±0.2$^{b}$ | 2±0.09$^{d}$ | 25,2±1.2$^{b}$ | 10±0.0 |
| Traditional El Ouad | 3,62±0.07$^{b}$ | 6±0.21$^{a}$ | 2.86±0.02$^{c}$ | 43,12±2.1$^{a}$ | 17.09±0.0 |

**Data presented as mean ± SD of three replicates. Mean values followed by different superscripted alphabets in a column are significantly different at p < 0.05.*

**2) Syrup**

**pH**

| Source | DDL | Somme des carrés | Moyenne des carrés | F | Pr > F | Codes de signification des p-valeurs |
|---|---|---|---|---|---|---|
| Modèle | 6,000 | 1,338 | 0,223 | 3,089 | **0,038** | * |
| Erreur | 14,000 | 1,011 | 0,072 | | | |
| Total corrigé | 20,000 | 2,349 | | | | |

*Calculé contre le modèle Y=Moyenne(Y)*
Codes de signification : 0 < *** < 0.001 < ** < 0.01 < * < 0.05 < . < 0.1 < ° < 1

**Brix degree**

| Source | DDL | Somme des carrés | Moyenne des carrés | F | Pr > F | Codes de signification des p-valeurs |
|---|---|---|---|---|---|---|
| Modèle | 6,000 | 910,886 | 151,814 | 158,849 | **<0,0001** | *** |
| Erreur | 14,000 | 13,380 | 0,956 | | | |
| Total corrigé | 20,000 | 924,266 | | | | |

*Calculé contre le modèle Y=Moyenne(Y)*
*Codes de signification : 0 < *** < 0.001 < ** < 0.01 < * < 0.05 < . < 0.1 < ° < 1*

**Electrical conductivity**

| Source | DDL | Somme des carrés | Moyenne des carrés | F | Pr > F | Codes de signification des p-valeurs |
|---|---|---|---|---|---|---|
| Modèle | 6,000 | 717779,143 | 119629,857 | 8544,990 | **<0,0001** | *** |
| Erreur | 14,000 | 196,000 | 14,000 | | | |
| Total corrigé | 20,000 | 717975,143 | | | | |

*Calculé contre le modèle Y=Moyenne(Y)*
*Codes de signification : 0 < *** < 0.001 < ** < 0.01 < * < 0.05 < . < 0.1 < ° < 1*

**Total acid**

| Source | DDL | Somme des carrés | Moyenne des carrés | F | Pr > F | Codes de signification des p-valeurs |
|---|---|---|---|---|---|---|
| Modèle | 6,000 | 286,826 | 47,804 | 257,408 | **<0,0001** | *** |
| Erreur | 14,000 | 2,600 | 0,186 | | | |
| Total corrigé | 20,000 | 289,426 | | | | |

*Calculé contre le modèle Y=Moyenne(Y)*
*Codes de signification : 0 < *** < 0.001 < ** < 0.01 < * < 0.05 < . < 0.1 < ° < 1*

**Physicochemical syrup samples resualts**

| **Samples** | **Analyzes** | | | |
|---|---|---|---|---|
| | **pH** | **°Brix** | **E.C (µS/cm)** | **Acetic Acid (g/L)** |
| Semi-industrial Biskra | $5.35\pm0.35^{d}$ | $76.5\pm1.3^{b}$ | $276\pm6^{c}$ | $16.5\pm0.3^{b}$ |
| Semi-industrial Biskra | $5.07\pm0.3^{e}$ | $79.8\pm0.9^{a}$ | $146\pm2^{d}$ | $19.5\pm0.5^{a}$ |
| Semi-Industrial Biskra | $5.72\pm0.3^{b}$ | $69\pm1.0^{c}$ | $452\pm2^{b}$ | $14.2\pm0.7^{c}$ |
| Semi-Industrial El Ouad | $5.38\pm0.12^{d}$ | $80.7\pm0.7^{a}$ | $116\pm1^{e}$ | $10.5\pm0.5^{d}$ |
| Traditional in lab | $5.8\pm0.3^{a}$ | $62.3\pm0.7^{e}$ | $653\pm4^{a}$ | $9.3\pm0.3^{e}$ |
| Traditional *Ghars* in lab | $5.62\pm0.29^{c}$ | $66\pm1.1^{d}$ | $120\pm1^{e}$ | $10.1\pm0.3^{de}$ |
| Traditional *Balha* in lab | $5.78\pm0.12^{ab}$ | $68\pm1^{c}$ | $256\pm6^{c}$ | $9.6\pm0.2^{e}$ |

**Data presented as mean ± SD of three replicates. Mean values followed by different superscripted alphabets in a column are significantly different at p < 0.05.*

**Appendix 5: Laboratory equipment**

The devices we used to accomplish the analysis are:

- pH meter (Consort C5020 Multi-Parameter Analyzer).
- Conductivity meter (Consort C5020 Multi-Parameter Analyzer).
- Refractometer (ATAGO).
- Centrifuge (Sigma 4-15).
- Vortexer.
- Analytical Balance.
- Water Bath.
- Magnetic Stirrer.
- Various glassware.
- Reagents: Saline solution, Phenolphthalein, Ethanol and Sterile distilled water.