République Algérienne Démocratique et Populaire

Ministère de l'Enseignement Supérieur et de la Recherche Scientifique

Université A. MIRA - Béjaia

Faculté des Sciences de la Nature et de la Vie

Département de Sciences Alimentaires

Spécialité : Contrôle de Qualité et Analyses des Aliments

Réf :.....

Mémoire de Fin de Cycle

En vue de l'obtention du diplôme

MASTER

Theme

Impact of grape seed hydrophilic and lipophilic extracts on the oxidative stability of mayonnaise

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Defended: **03/07/2025**

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Année universitaire : 2024/2025

Acknowledgments

First and foremost i would like to express my deepest gratitude to my research supervisor, Sonia Medouni-Adrar, for her precious guidance, availability, and continuous support throughout this work. Her expertise and insightful advice have greatly enriched this thesis and my research skills, her insightful feedback and unwavering patience have been essential to the completion of this thesis.

I would also like to warmly thank the entire team at CEVITAL, where I had the privilege of completing my internship. My sincere appreciation goes to Mr. Tofik and ms.Linda, who supervised me during my time in the company, for their kindness, valuable advice, and availability. Their guidance throughout the experimental work has been of great help and made my experience both enriching and pleasant. I would also like to sincerely thank the Director of the Research and Development Laboratory mr .Hadjal Samir, my co-advisor . for his trust and for giving me the opportunity to work within such a stimulating environment.

A special mention goes to Lamia Medouni for her valuable assistance and guidance, and to Sabine, whose support and kindness I will always remember and appreciate.

I would like to thank Ms.salima deeb, our laboratory engineer, for always being there with support, solutions, and kindness when I needed help the most. Your calm presence and technical expertise made the lab feel like a second home, this thanks goes to ms. Nigro Leila on the same field.

I would also like to express my sincere gratitude to all the teachers and professors of the Department of Food Science, Ms.Smail leila, Ms. Geurfi, Ms. mekhoukh. as well as to the professors from other departments who have guided me and offered help along the way Your dedication to teaching and your constant encouragement have been instrumental throughout my university years.

Finally, I would like to express my gratitude to all those who have supported me, encouraged me, and believed in me throughout this journey. Your presence has been a true source of motivation during this chapter of my life.

Dedication

I dedicate this work to the people who have shaped me, supported me, and stood beside me throughout this journey with unwavering love and strength.

To my beloved mother,
Your unconditional love, sacrifices, and endless support have been my guiding light. Everything I have achieved is because of your strength and belief in me. Even when I doubted myself, your faith carried me through. I owe you everything.

To my father,
Thank you for your silent strength, your steady presence, and your belief in my abilities. I will always carry your hopes with me.

To my family,
For your love, patience, and quiet encouragement in every step I've taken. Thank you for always reminding me that no challenge is too great when you're surrounded by family.

To my dear aunties and uncle,
Your kindness, your encouragement, and your steady belief in me have been a quiet strength in the background of all I've accomplished. Thank you for your endless love and support.

To my soul friends , dearest Melinda ,Linda,
No words will ever be enough to thank you for what you've done for me. You have been more than just friends you've been my sisters, my chosen family. From the first days of this journey to the very last, you stood by me through every high and every low. Your unwavering encouragement, your endless patience, endless love, and your strength shaped this achievement as much as any of my own efforts did. I am forever grateful that life gave me the privilege of walking this path with both of you by my side , i pray we always remain together.

To my dearest friend, Aya,
Even though distance kept us apart, your heart was always beside mine. You never let the miles dim the light of our friendship. Your faith in me never wavered, even when mine did. Thank you for your constant love, your understanding, and for reminding me that true friendship endures everything.

To my classmates, CQAA class
Thank you for the shared laughter, the long nights, and the quiet support. This journey wouldn't have been the same without you.

This work is not mine alone it belongs to all of you who carried me through with love, strength, friendship, and hope.

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List of abbreviation

AAE: Ascorbic Acid Equivalents

AO / AOX: Antioxidant

AOA: Antioxidant Activity

BHA: Butylated Hydroxyanisole

BHT: Butylated Hydroxytoluene

DG: Dodecyl Gallate

DPPH*: 2,2-Diphenyl-1-picrylhydrazyl

DPPH-RSA: DPPH Radical Scavenging Activity

EDTA: Ethylenediaminetetraacetic Acid

FAC: Fatty Acid Composition

FRAP: Ferric Reducing Antioxidant Power

GAE: Gallic Acid Equivalents

GPx: Glutathione Peroxidase

GSE: Grape Seed Extract

GSEM / ME-GSE: Mayonnaise Enriched with Grape Seed Extract

GSO: Grape Seed Oil

$\mathbf{M_0}$: Initial Moles or Baseline Quantity (context-dependent)

OG: Octyl Gallate

OX / Ox: Oxidation

PG: Propyl Gallate

PV: Peroxide Value

ROO•: Peroxyl Radical

ROOA: Peroxyl–Antioxidant Adduct

ROS: Reactive Oxygen Species

TPC: Total Phenolic Content

Introduction

Oxidation is a chemical reaction that alters the structure and reactivity of food components, often resulting in the formation of reactive compounds such as hydroperoxides and aldehydes. These oxidative products are responsible for significant quality deterioration and are among the main causes of spoilage in food systems during processing, storage, and distribution (Mcclements & Decker, 2000; Wsowicz et al., 2004). In lipid-rich emulsions like mayonnaise, oxidation is a major concern due to the product's high fat content and structural characteristics. Mayonnaise, one of the most widely consumed sauces , it is particularly prone to lipid oxidation, which compromises its sensory qualities, nutritional value, and shelf stability (Ghorbani Gorji et al., 2016).

To mitigate lipid oxidation in mayonnaise, the Codex standard authorizes the use of specific synthetic antioxidants at regulated levels (Gavahian, et al.;2013). Common compounds such as BHA, BHT, TBHQ, and EDTA are widely used in the food industry due to their phenolic or benzene ring structures, which enhance their effectiveness in delaying rancidity (Kishk & Elsheshetawy, 2013; Mirzanajafi-Zanjani et al., 2019). However, despite their efficacy, the safety of synthetic antioxidants like BHA and BHT has been increasingly questioned. Some studies have associated them with potential tumor-promoting effects, while others suggest they may have anti-carcinogenic properties due to their antioxidant mechanisms (Labrador et al. 2007;Thorat et al., 2013; Kornienko et al., 2019); Carsono et al., 2022)These conflicting findings, along with evolving consumer preferences, have led to growing interest in reducing or replacing synthetic antioxidants. In this context, plant-derived antioxidants are gaining popularity as natural, safer alternatives that also provide functional and nutritional benefits (Atta et al., 2017;Oliveira et al., 2018; Gonçalves-Filho & De Souza, 2022; Gavahian et al., 2013; Mirzanajafi-Zanjani et al., 2019).

Natural bioactive compounds, especially polyphenols, have attracted increasing interest due to their potent antioxidant properties and associated health benefits, positioning them as viable alternatives to synthetic antioxidants (Ghouila et al., 2018; Patra et al., 2022; Sorrenti et al., 2023). Grapes (*Vitis vinifera L.*), which accounted for 73 million tonnes globally in 2023 (FAOSTAT, 2025), are rich in polyphenols, particularly in by-products like skins and seeds (Yanishlieva.N & Marinova, 2001; Iglesias-Carres et al., 2018); Nallathambi et al., 2020). Grape seeds, by-products of juice and wine production exhibit antioxidant, anti-inflammatory, cardioprotective, and gut-health-enhancing effects (JOSÉ M. L .R & DANIEL. F. R, 2016; Jovanovic et al., 2017; Nallathambi et al., 2020). They also yield 8–20% oil depending on the extraction method, which is rich in lipophilic antioxidants like tocopherols and tocotrienols (Konuskan et al., 2019); Zhao Yagiz et al., 2015; Zhao, 2017), making grape seed oil a valuable natural antioxidant for food applications (Matthäus, 2008; Jovanovic et al., 2017).

Studies have further highlighted the efficacy of phenolic-rich plant extracts in improving oxidative stability and shelf life in foods such as pomegranate peel reducing lipid peroxidation by 65% (Kelawala & Ananthanarayan, 2004), rosemary and oregano for meat preservation (Velasco & Williams, 2011), polyphenol-rich seaweed (Corsetto et al., 2020), and oak extracts against rancidity (Othón-Díaz et al., 2023).

While grape seed extract (GSE) is well-known for its antioxidant potential, its application in mayonnaise particularly in lipophilic forms like macerates oil, remains underexplored. Studies combining hydrophilic and lipophilic GSE are even scarcer, and their impact on mayonnaise's sensory and physicochemical properties is not well documented. Nevertheless, this dual incorporation offers a promising natural alternative to synthetic antioxidants, aligning with current consumer demand for clean-label and health-conscious formulations

The main objective of this study was to evaluate the individual and combined effects of hydrophilic and hydrophilic grape seed extract and grape seed macerate (lipophilic extract) on the oxidative stability, physicochemical characteristics, and sensory qualities of mayonnaise. Specific objectives included assessing their impact on lipid oxidation during storage, analyzing dose-dependent effects on parameters such as pH, emulsion stability, and viscosity, and identifying optimal formulations for both stability and sensory acceptability.

To achieve these goals, the study is structured in two main parts:

- The first part presents a literature review, covering the grapevine and grape, the history of viticulture in Algeria, global and local production statistics, and the antioxidant composition and extraction techniques of phenolic compounds.

- The second part details the experimental work, divided into: *Materials and Methods*, describing the procedures, and *Results and Discussion*, organized into five chapters. Chapter one addresses the physicochemical properties of grapes; chapter two focuses on optimizing phenolic extraction; chapters three and four examine the bioactive composition and phenolic profiles; and chapter five evaluates antioxidant activity.

Literature review

I. Generalities on mayonnaise

I.1. Historical background and global consumption trends of mayonnaise

The growth of international food restaurant chains, such as fast food and Japanese and Middle Eastern eateries, which utilize mayonnaise as a flavor for many of their meals, has led to an increase in mayonnaise consumption in recent decades (Blejan & Nour, 2023).

Additionally, it is frequently used as a component in the making of a variety of baked goods, including sandwiches and filled breads. There are many different mayonnaise product formulas and flavors being produced at the moment (Surin et al., 2025).

Throughout the world, mayonnaise is one of the most popular sauces. It was initially commercially made in the early 1900s and has been around for centuries. It gained popularity in America from 1917 to 1927 (Harrison and Cunningham, 1985). and in Japan from 1987 to 1990, when sales rose by 21% (Brabant et al., 1992).

I.2. Definition of mayonnaise

By definition, mayonnaise is a semi-solid oil-in-water emulsion that requires a certain combination of ingredients and processing techniques. It is usually prepared by carefully combining oil (70–80%), vinegar, egg yolk, and spices. Because of the high oil percentage, it has a reputation for having a high fat and calorie content (Abedi-Firoozjah et al., 2025).

I.3. Mayonnaise sauce: commercial variants and broader use

While the traditional formulation of mayonnaise is well established, the term "mayonnaise sauce" is increasingly used in the food industry to describe a broader range of products with modified compositions. The word "mayonnaise sauce" can refer to a wider range of products, including variants of the classic mayonnaise recipe. It could be used, for example, to describe reduced-fat or low-fat mayonnaise that uses fat substitutes or other components to get a comparable texture and flavor (Mozafari et al., 2017; Abedi-Firoozjah et al., 2025).

In contrast to conventional mayonnaise, the word "sauce" may also suggest a slightly different consistency or other components. The terms are sometimes used interchangeably, particularly when discussing commercial goods. However, the word "mayonnaise sauce" may refer to a product that differs from the classic mayonnaise recipe in terms of ingredients or preparation technique, but nonetheless has a comparable look and is used in cooking.

I.4. Nutritional composition of mayonnaise

Mayonnaise is composed of several ingredients, each contributing to its physicochemical and sensory properties. This section details the main components and their functional roles.

I.4.1. General composition

Mayonnaise is an oil-in-water emulsion It is made from vegetable oil, egg yolks, and acidifying agents, mayonnaise is a food ingredient used in salads and sandwiches. Vegetable oil emulsion-based goods are highly well-liked in the community due to their use in a wide range of food items that are rich in health and cosmetic advantages. The addition of sugar, salt, and spices, together with an acidifying component like lemon, vinegar, or tamarind, can enhance the flavor of mayonnaise. Generally speaking, mayonnaise consists of three primary ingredients: emulsifier ingredients in the form of egg yolks, dispersing and dispersion materials in the form of vegetable oils and acidifying agents (Yildirim et al., 2016 ; Meidian Daoed et al., 2022) The formulation of conventional mayonnaise generally follows a well-established composition, as shown in Table I

Table I : Standard composition of mayonnaise ref

Component	Typical Content (%)
Oil	70% no less than 68%
yolk Egg	14%
Vinegar (Acetic acid)	3–5%
Water (Aqueous phase)	12%
Salt (NaCl)	0.3–1.5%
Sugar	0 –2%
Mustard	1- 2%
Stabilizers/Thickeners	0–0.5%

I.4.2. Functional role of main ingredients

Each ingredient in mayonnaise plays a distinct and essential role in ensuring the stability, texture, flavor, and overall quality of the final product.

- **Vinegar**

Vinegar's primary function is to Balance pH. The pH of the mayonnaise significantly affects the structure of the emulsion. The maximum stability and viscoelasticity of mayonnaise would occur when the pH level reached the isoelectric point of the proteins in the egg yolk, to the extent

that the surface charge of the proteins is reduced. It is impossible for highly charged proteins to flocculate (Mirzanajafi-Zanjani et al., 2019).

- **Salt**

Regarding the salt, there are three primary reasons why adding it can improve the qualities of mayonnaise. First, salt helps spread out the granules in the egg yolk and makes more surface-active substances available. Second, proteins can readily adsorb to the oil droplets' surface because salt neutralizes their charges. Third, it makes oil droplets closer to one another, which strengthens their interaction. However, because of the salting-out effect **(Appendix 1)** too much salt can cause the proteins in the egg yolk to aggregate in the aqueous phase (Depree & Savage, 2001; Mirzanajafi-Zanjani et al., 2019).

- **Egg yolk**

The emulsion stability of mayonnaise depends entirely on egg yolk according to Nikzade et al. (2012), The emulsion properties of egg yolk enable the formation of mayonnaise while preventing flocculation to achieve the correct texture (Depree & Savage, 2001). The emulsifying properties of egg yolk stem from its composition of LDL (low-density lipoprotein) and HDL (high-density lipoprotein) along with phospholipids and nonbonded proteins including phosvitin and livetin (Giacintucci et al., 2016).

- **Oil**

The oil droplets create a dense network structure which produces mayonnaise's thick consistency and its viscoelastic behavior. The stability of emulsions and mouthfeel depends heavily on oil droplet size and amount because excessive oil content can lead to instability in the product (Mirzanajafi-Zanjani et al., 2019).

- **Mustard**

The emulsifying properties of mayonnaise receive additional support from mustard which also enhances flavor. The emulsion stability improves through sulfur compounds that prevent oil droplets from aggregating while adding color and flavor to the mixture. Mustard also has antioxidant effects that help prolong shelf life (Mirzanajafi-Zanjani et al., 2019).

I.5. Technological process of mayonnaise preparation

The industrial production of mayonnaise involves a sequence of carefully controlled steps aimed at obtaining a stable, high-quality oil-in-water emulsion. Mayonnaise is typically produced by transforming two immiscible phases an oil phase and an aqueous phase into a semi-solid, stable emulsion through the gradual incorporation of oil into a water-based pre-mixture containing

emulsifying agents and acidifiers, under continuous mechanical agitation (Harrison & Cunningham,1985 ; Depree & Savage, 2001). The primary emulsifier is egg yolk, which contains lecithin, a phospholipid compound that stabilizes the emulsion by reducing interfacial tension. Mustard is often included to enhance both flavor and emulsification capacity. Other common ingredients include vinegar or lemon juice, salt, sugar or sweeteners, and additional optional flavorings and stabilizers.

To ensure emulsion stability, oil is slowly added to the pre-mix typically composed of egg yolk, vinegar, and mustard. Simultaneous mixing of both phases without controlled addition can lead to the formation of a water-in-oil emulsion rather than the desired oil-in-water structure (H. Liu et al., 2007). In industrial-scale manufacturing, high-shear mixers, colloid mills, or rotor–stator systems are employed to achieve uniform droplet dispersion and reduce droplet size, which is essential for long-term physical stability (Yildirim et al., 2016) . The typical fat content of mayonnaise ranges from 70% to 80%, which contributes to its characteristic texture and mouthfeel (Depree & Savage, 2001 ; Liu et al., 2007).

The ratios of ingredients and the incorporation of stabilizers such as xanthan gum or sodium caseinate play an important role in enhancing viscosity and preventing phase separation (Harrison & Cunningham,1985; Yildirim et al., 2016) . Furthermore, recent trends in formulation focus on the development of healthier alternatives. These include low-fat and vegan mayonnaise, often based on the use of fat replacers and plant-derived emulsifiers such as aquafaba, as well as double-emulsion techniques designed to reduce oil content without compromising texture or stability (Yildirim et al., 2016); (Saget et al., 2021)

In addition to functional ingredients, natural flavorings and colorants such as beetroot peel powder may be incorporated to improve antioxidant properties and visual appeal (Mistrianu et al., 2022). Once production is complete, the mayonnaise is subjected to rigorous quality control procedures, including stability testing, viscosity measurements, and sensory evaluation, to ensure it maintains the desired properties throughout its shelf life (Jacobsen et al., 1999)

II. Oxidation in mayonnaise and its prevention

II.1. Lipid Oxidation in high-fat foods

A chemical process known as oxidation occurs when a substance's electrons are transferred to an oxidizing agent. Free radicals can be produced by oxidation processes. These free radicals can then trigger a series of events that harm cells (Mishra & Singh Bisht, 2011)

In addition to being a significant part of food, lipids are crucial structural and functional components of biological cell membranes. However, lipids especially those rich in unsaturated fatty acids are susceptible to oxidation through complex mechanisms involving free radicals and reactive oxygen species(Choe & Min, 2006.; Frankel, 2014). Their oxidative stability is influenced by multiple internal and external factors, such as the degree of unsaturation, presence of minor constituents (e.g., tocopherols, sterols), environmental conditions, processing and storage methods, and the presence or absence of antioxidants (Kumar et al., 2005 ;Fereidoon & Ying, 2010; Shahidi & Ambigaipalan, 2015).

In addition to being a significant part of food, lipids are crucial components of biological systems' cells, both structurally and functionally. However, oxidation can occur through a variety of mechanisms in this broad set of chemicals. Numerous internal and external factors, such as the unsaturation of their fatty acids that are susceptible to oxidation through complex mechanisms involving free radicals and reactive oxygen species (Choe & Min, 2006.). their oxidative stability is influenced by multiple internal and external factors, such as the degree of unsaturation, presence of minor constituents, environmental conditions, processing and storage methods, and the presence or absence of antioxidants (Fereidoon & Ying, 2010; Luo et al., 2011; Shahidi & Ambigaipalan, 2015)

The development of fatty foods with desired nutritional and physical qualities, such as mayonnaise, depends on the availability of better techniques to regulate their oxidative stability, which in turn depends on a comprehensive comprehension of the mechanisms underlying lipid oxidation (Mcclements & Decker, 2000) .

Mayonnaise, due to its high lipid content (typically 70–80% vegetable oil), is particularly susceptible to lipid oxidation, especially during storage or thermal processing (Hsieh & Regenstein, 1992). This oxidative degradation not only affects the sensory qualities of the product, such as flavor, aroma, and color, but also reduces its nutritional value and shelf life. The continuous phase of mayonnaise (aqueous) can promote the activity of pro-oxidant metal ions, while the oil droplets in the dispersed phase provide a large interfacial area that facilitates oxidation reactions. Furthermore, the presence of unsaturated fatty acids, mainly linoleic and oleic acids, makes the emulsion more prone to peroxidation, especially in the absence of effective antioxidants (Ghorbani Gorji et al., 2019). As a result, controlling lipid oxidation is a major challenge in mayonnaise formulation, and necessitates the use of appropriate strategies, including the incorporation of natural or synthetic antioxidants.

II.2. Mechanistic pathways of lipid oxidation in high-fat foods

Lipides when exposed to catalytic systems like light, heat, enzymes, metals, metallo-proteins, and microorganisms, can undergo complex processes of oxidation, including autoxidation, photooxidation, thermal oxidation, and enzymatic oxidation. The majority of these processes involve free radicals and/or other reactive species as an intermediary (Fereidoon & Ying, 2010).

It has been recognized as three different mechanisms, yielding different oxidation products: a free radical mechanism known as autoxidation, photo-oxidation and process related to lipoxygenase activity (Wsowicz et al., 2004)

There are many catalytic systems that can oxidize lipids. Among these are light, temperature, enzymes, metals, metalloproteins and microorganisms. Most of these reactions involve some type of free radical and/or oxygen species (J. R. Vercellotti Allen et al., 1992).

II.2.1. Autoxidation

Autoxidation, the most prevalent process of all, is the spontaneous reaction of lipids and molecular oxygen that results in oxidative degradation. It works in three steps using a free radical chain mechanism (**Appendex 2)** (Fereidoon & Ying, 2010).

There are Three separate stages typically make up the autoxidation process leading to a series of complex chemical changes : the production of radicals during the initiation stage, the growth of reactive compounds during the propagation stage, and the degradation or reaction of reactive compounds to form non-reactive compounds during the termination stage (Wang et al., 2023) .

Initiation

In the first step of oxidation, which typically happens extremely slowly, unsaturated lipid molecules lose a hydrogen atom and create free radicals when exposed to initiators such heat, light, ionizing radiation, and metal ions or metalloproteins. Following their contact with oxygen, the lipid radicals produce peroxyl radicals, which attack a new lipid molecule and serve as the chain carriers of the quickly advancing process. During propagation, this reaction may occur thousands of times until there is no more hydrogen available or the chain is broken, for instance, by antioxidants (Fereidoon & Ying, 2010).

Propagation

Phase of propagation wherein peroxy radicals (ROO•) are formed and have the ability to

react with unsaturated fatty acids (Wsowicz et al., 2004). As it spreads, the conjugated diene steals a hydrogen atom from a nearby unsaturated fatty acid to create hydroperoxides and turns into a highly reactive lipid radical when O_2 is present; This propagation procedure will continue until the termination step is reached after it has begun (Abeyrathne et al., 2021)

Termination

The termination step occurs when free radicals collide and cause stable molecules to form, which causes the free radicals to vanish (Martínez-Yusta et al., 2014) .

During this phase, radicals neutralize one another by disproportioning or combining with one another to create stable non-radical products, such as a range of polymer compounds. (Fereidoon & Ying, 2010) namely secondary oxidation products like ketones, ethers, alkanes, aldehydes, etc

II.2.2. Photooxidation

The photo-oxidation process is an alternative route to the free radical mechanism that generates hydroperoxides. It entails a photosensitizer being excited and energy being transferred to oxygen or lipid molecules (Fereidoon & Ying, 2010)**,** Light combined with a sensitizer can stimulate oxygen or unsaturated fatty acids (Wsowicz et al., 2004).

II.2.3. Enzymatic oxidation

Refers to the oxidation reaction involving enzymes, and there are two kinds of enzymes involved in lipid oxidation, namely lipoxygenase and hydroperoxidase (Lampi et al., 2020 ; Wang et al., 2023) .

Lipoxygenase is an enzyme which is a very important source of hydroperoxides formed during oil extraction (Wsowicz et al., 2004).

II.3. Impact of oxidation on food quality

The most important quality factor in food, particularly fatty foods, is lipid oxidation, which can damage food's flavor, texture, appearance, and nutritional value. It can also shorten the shelf life of foods that contain lipids and result in significant financial losses (Barden & Decker, 2016).

Food quality and human health are negatively impacted by lipid oxidation, hence measures must be taken to reduce oxidation and increase the oxidative stability of lipid products. The food business has effectively used antioxidant strategies to preserve the quality of food products, and the pharmaceutical industry has used them to lower the risk of many diseases caused by oxidative

stress (Fereidoon & Ying, 2010).

II.4. Antioxidants roles in preventing lipid oxidation

II.4.1. Mechanisms of antioxidants activity

One of the main defenses against deteriorating food quality as it travels from the farm to the consumer via processing and distribution is the use of antioxidants.

According to Abeyrathne et al. (2021) antioxidants are a class of substances that can prevent oxidation by functioning as reducing agents, free radical scavengers, radical species quenchers, or inactivators of prooxidants like metals (Abeyrathne et al., 2021). It is any material or nutrient that significantly slows down or prevents the oxidation of an oxidizable substrate when present in lower concentrations than that of the substrate is considered an antioxidant (S. Kumar et al., 2017).

Antioxidants can be classified according to the mechanism of action into two groups.

Primary antioxidants (chain-breaking antioxidants**)**

They are acceptors of free radicals. According to (Wsowicz et al., 2004), they can scavenge lipid radicals because they function as hydrogen donors:

$$ROO\bullet + AH = ROOH + A\bullet$$

By reacting with radicals, they can stop the radical chain process :

$$ROO\bullet + A\bullet = ROOA$$

The three main antioxidants that are most crucial are tocopherols, BHT, BHA, and PG.

Secondary antioxidants

When hydroperoxides and secondary or preventive antioxidants combine, non-reactive, non-radical compounds are produced. They do not break the chain of free radicals, but they can slow down or lessen lipid oxidation through a number of mechanisms, such as binding metal ions that can catalyze oxidative processes, chelating transition metal ions like citric acid, phosphoric acid, and EDTA, scavenging oxygen with ascorbic acid, ascorbyl palmitate, and sulfites, replenishing hydrogen to primary antioxidants, absorbing UV light and deactivating reactive species (Mishra & Singh Bisht, 2011) inhibiting enzymes, or breaking down hydroperoxides (Schwarz et al., 2001).

II.4.2. Antioxidants classes

Antioxidants can be classified into several categories based on their chemical structures and sources:

Polyphenols and phenolic compounds

The term "phenolic" or "polyphenol" can be chemically defined as a compound with an aromatic ring and one or more hydroxyl substituents, including functional derivatives such as esters, methyl ethers, glycosides, and so on. The majority of phenolics include two or more hydroxyl groups and are bioactive compounds found throughout plants (Mishra & Singh Bisht, 2011)

Many foods and food material contain phenolics, Most of them are found in cereals and legumes (barley, corn, nuts, oats, rice, sorghum, beans, and pulses), oil seeds (rapeseed, canola, flaxseed, and olive seeds), in fruits and vegetables, and beverages such as fruit juices, tea, coffee, cocoa, beer, and wine (Shi et al., 2003) , and consists of various classes with unique chemical structures and characteristics, including flavonoids, stilbenes, coumarins, lignans, tannins, curcuminoids, phenolic acid, etc., and can be divided into three categories: simple phenols and phenolic acids, hydroxycinnamic acid derivatives, and flavonoids. Additional classifications for the largest class, flavonoids, including anthocyanidins, anthocyanins, flavones, isoflavones, flavonols, flavanones, flavanonols, and flavanols (Overview & Ho, 1992).

Organosulfur compounds

Antioxidants of other significance include allicin from garlic and L-sulforaphane from broccoli (Kelsey et al., 2010) . These substances can either directly scavenge free radicals or indirectly boost the antioxidant defenses of the cells.

Non enzymatic antioxidants

Well-known exogenous antioxidants that can be found in a variety of fruits and vegetables include carotenoids, tocopherols (vitamin E), and ascorbic acid (vitamin C) glutathione and melatonin are also categorized as antioxidants. According to (Hamid et al., 2010) .

Enzymatic antioxidants

An essential class of endogenous antioxidants includes enzymes such as glutathione peroxidase, catalase, and superoxide dismutase (S. Kumar et al., 2017) . They have the ability to deactivate or stabilize free radicals before they harm cellular components. In order to make it stable, they either lower the energy of the free radicals or give up part of their electrons for its usage. To lessen the harm that free radicals inflict, they might also stop the oxidizing chain reaction (Atta et al., 2017).

Oligo-elements or trace elements

Elements like selenium, manganese, copper, zinc, and iron help neutralize harmful reactive oxygen species and protect cells from oxidative damage.They support antioxidant enzymes by functioning as their constituents or by directly neutralizing free radicals, which both strengthen antioxidant defenses (Ye et al., 2022). For example, selenium plays a crucial role in glutathione peroxidase (GPx), which neutralizes organic and hydrogen peroxides. Superoxide dismutase enzymes (MnSOD, Cu/ZnSOD) require manganese, copper, and zinc as essential components in order to catalyze the conversion of superoxide anions into oxygen and hydrogen peroxide (Wołonciej et al., 2016).

Synthetic antioxidants

Since they do not exist in nature, synthetic antioxidants are chemically created substances that are added to food as preservatives to help stop lipid oxidation. A number of synthetic antioxidants have been utilized to stabilize fats and oils because natural antioxidants are inherently unstable. The initial purpose of butylated hydroxytoluene (BHT) and butylated hydroxyanisole (BHA) was to shield petroleum from oxidative gumming (Tang et al., 2008) Another artificial antioxidant that is frequently utilized in the feed business is tert-butylhydroxyquinone, or TBHQ. TBHQ shares a phenol structure or benzene ring with BHT and BHA. Propyl gallate (PG), dodecyl gallate (DG), octylgallate (OG), and ethylene diaminetetraacetic acid (EDTA) are other examples of synthetic antioxidants (Atta et al., 2017).

III. Generalities about grapes and grape seeds

III.1. Global and national grape production

Before focusing on the bioactive compounds derived from grapes, it is important to highlight their global agricultural and economic importance. For thousands of years, grapes have been valued for their nutritional and therapeutic properties, making them one of the most popular fruits consumed worldwide (Arora et al., 2010; Balasubramani & Narendhirakannan, 2025)

III.1.1. Global grape production

Global grape production showed moderate fluctuations between 2015 and 2023 **(figure 2**), ranging from about 72 to 80 million tonnes. The peak occurred in 2018, while the lowest production was observed in 2023. Production remained relatively stable from 2019 to 2022. Despite minor variations, the trend highlights the strong and consistent importance of grape cultivation worldwide.

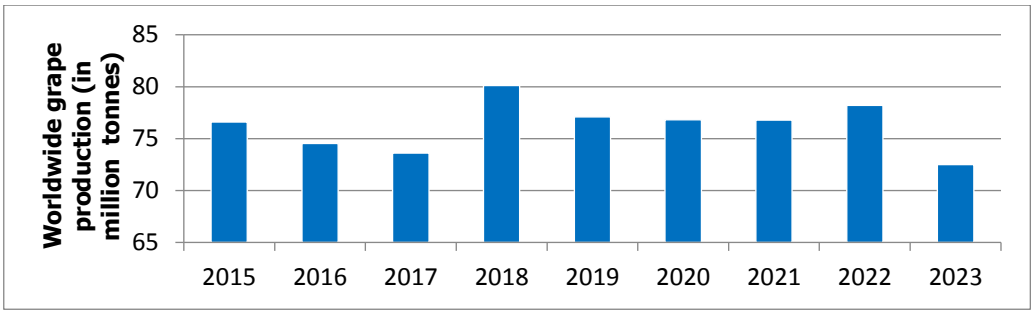

Figure 1: Global grape production (2015–2023) (FAOSTAT, 2025)

III.1.1. National grape production

Between 2015 and 2023 **(figure 2)** grape production in Algeria showed relatively stable trends, fluctuating between approximately 510,000 and 640,000 tonnes. The lowest production was recorded in 2018, while the highest was in 2021. From 2019 onward, a noticeable increase was observed, peaking in 2021, followed by a slight decrease but remaining high in 2022 and 2023. This overall stability and upward trend in recent years highlight the importance and resilience of grape cultivation in Algeria's agricultural sector.

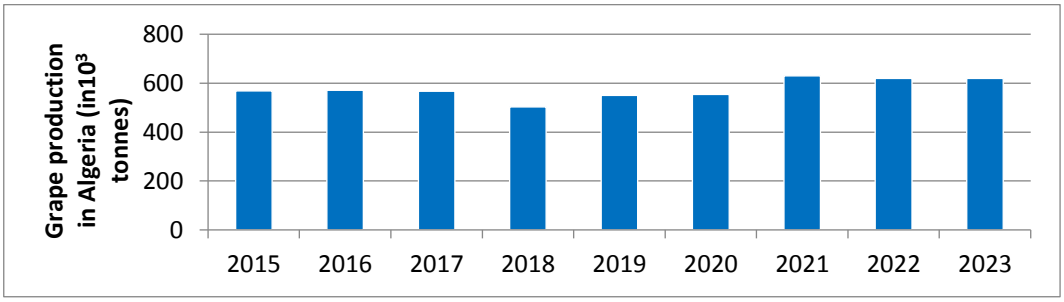

Figure 2 : National grape production (2015–2023) (FAOSTAT, 2025)

III.2. Grape seeds as a valuable by-product

Although most harvested fruit is turned into wine (used in viniculture), some varieties, such as table grapes, raisin grapes, and others, have edible seeds or are seedless. In addition to being eaten as fresh fruits, they can be used to make wine, raisins, juice, jam, jelly, vinegar, and grape seed oil. Numerous nutritional components, including carbohydrates, edible fibers, minerals, vitamins, and phytochemicals, are found in grapes, Phenolic compounds are the most important phytochemicals found in grapes (Thomas, 2016).

III.2.1. Chemical composition of grape seeds

Grape seeds constitute a relatively small portion of the total grape weight, typically accounting for about 5% (Garcia-Jares et al., 2015). However, they are a rich source of valuable bioactive compounds. Their composition varies depending on the grape variety but generally

includes.

Notably, grape seeds are estimated to contain 60–70% of the total polyphenolic content of the whole grape, contributing significantly to their antioxidant capacity (Garcia-Jares et al., 2015).

Table II: General composition of grape seeds

Component	**Approximate Content (%)**	**Function / Relevance**	**Refrencece**
Dietary Fiber	~40	Improves digestive health; contributes to texture in formulations	(Garcia-Jares et al., 2015)
Protein	~11	Source of amino acids	
Phenolic Compounds	5–8	Antioxidant activity	(Shi et al., 2003)
Lipids	10-20	Contains tocopherols and *β*-carotene	(Binzer et al., 2006 ; Tseng & Zhao, 2013),
Carbohydrates	Traces	Includes sugars	(Garcia-Jares et al., 2015)
Mineral Salts	Traces	Includes potassium, calcium, magnesium	

III. 2.2. Phenolic compounds in grape seeds

Grape seeds are a rich source of antioxidant compounds, with polyphenols being the most abundant and biologically significant class (Shi et al., 2003; Jebur et al., 2025). Among these, proanthocyanidins also known as condensed tannins represent the predominant group, accounting for approximately 92–95% of the total polyphenols in grape seed extract (Arora et al., 2010; S. X. Liu & White, 2012 ; Ghafoor et al., 2020). These compounds consist of oligomeric and polymeric flavan-3-ols, with monomeric units such as catechin, epicatechin, epigallocatechin, and epicatechin gallate, which serve as the fundamental building blocks of proanthocyanidins (Shi et al., 2003; ;(Yilmaz & Toledo, 2004)). Their structure ranges from simple dimers to highly polymerized forms, a complexity that contributes significantly to their biological activity (Leroy L. Creasy & Min T. Creasy, 2016; Thomas, 2016) . Notably, proanthocyanidins exhibit exceptional antioxidant capacity, reported to be up to 20 times greater than vitamin E and 50 times greater than vitamin C (Shi et al., 2003). Research has shown that these compounds may help protect cardiovascular cells, inhibit malignant cell proliferation, and preserve cellular integrity,

thus contributing to cancer prevention, cardiovascular protection, and anti-aging effects (Prior & Gu, 2005).

In addition to proanthocyanidins, grape seeds also contain phenolic acids, predominantly gallic acid, along with smaller quantities of other related acids (Yilmaz & Toledo, 2004) .Although resveratrol is found in lower concentrations compared to other polyphenols, it remains a compound of interest due to its potent antioxidant properties and its potential to modulate oxidative stress (Soleymani et al., 2019) .

Overall, the diversity, concentration, and bioactivity of phenolic compounds in grape seeds highlight their importance as a natural source of antioxidants with considerable health-promoting potential (Ghafoor et al., 2020)

III. 2.3. Grape seed oil

Grape seeds typically contain between 6% and 20% oil, and grape seed oil (GSO) is widely recognized for its versatility, particularly in cosmetic formulations (Lutterodt et al., 2011). In recent years, it has gained increasing popularity as an edible oil due to its pleasant sensory attributes and potential nutraceutical benefits (Dabetic et al., 2020) . The chemical composition of GSO can vary depending on factors such as grape variety, environmental conditions, and the degree of seed maturity, though it is generally classified as a linoleic-type oil. (Garavaglia et al., 2016).

Owing to its rich profile of bioactive compounds, GSO is associated with several health-promoting effects. It is especially rich in unsaturated fatty acids, notably polyunsaturated linoleic acid, which has been linked to cardiovascular health. Furthermore, GSO contains various antioxidant constituents including tocopherols, tocotrienols, phytosterols, flavonoids, phenolic acids, and carotenoids with its antioxidant activity largely attributed to its vitamin E content (Shinagawa et al., 2015; Gitea et al., 2023).

The unsaponifiable fraction of Grape seed oil, which comprises bioactive compounds such as tocopherols, phytosterols, and triterpenoids, has been shown to exhibit significant cytotoxic and anti-inflammatory properties, suggesting potential therapeutic applications in disease prevention and management (Soleymani et al., 2019) . In parallel, GSO demonstrates notable antimicrobial activity, with studies reporting inhibitory effects against common foodborne pathogens, including *Staphylococcus aureus* and *Escherichia coli*, highlighting its potential role as a natural preservative in food systems (Garavaglia et al., 2016).

Since its industrial production began in the 1930s particularly in European countries such as Germany, France, and Italy GSO has steadily gained popularity, primarily as a culinary oil due to its favorable sensory properties and health profile. Beyond its traditional uses, it is increasingly being investigated as a valuable source of specialty lipids for functional foods, nutraceuticals, cosmetics, and other industrial applications, driven by growing interest in natural, bioactive-rich ingredients (Camargo et al., 2010; Ghafoor et al., 2020)**.**

Materials and methods

The aim of this study was to evaluate the impact of enrichment with hydrophilic and lipophilic grape seed extracts on the oxidative stability and sensory quality of mayonnaise. The experimental work was conducted in collaboration with Cevital and the laboratories of the University of Béjaïa (3BS and Food Technology Laboratory).

Eight mayonnaise samples were prepared based on a formulation developed through a mixture design using JMP18 software. This formulation, selected as the reference recipe, was chosen for its optimal sensory characteristics as evaluated by an expert panel. The samples were enriched with hydrophilic grape seed extracts, lipophilic grape seed macerates, or both, in order to assess their effects on the oxidative stability and sensory quality of mayonnaise.

The grape seed extracts were characterized in the laboratory for their total polyphenol, proanthocyanidins and tocopherol content as well as their antioxidant activity. All mayonnaise samples were stored at 4 °C and 40 °C to assess their oxidative stability under both real and accelerated conditions. Peroxide value and acidity were regularly monitored over 5 days. Finally, the four most promising formulations were subjected to a descriptive sensory analysis to assess the impact of enrichment on organoleptic properties.

I. Preparation of lipophilic and hydrophilic grape seed extracts

I.1. Preparation of plant material

Grape berries (*Vitis vinifera* L.) were purchased from the local market. After manual sorting and washing, the berries were halved to collect the seeds. The seeds were then shade-dried at ambient temperature to preserve their bioactive content. Once dried, they were ground into a fine powder using a household coffee grinder. The powder was sieved (≤ 500 µm), then stored in airtight glass jars at 4 °C, protected from light and humidity until further use.

I.2. Extraction of hydrophilic compounds (hydrophilic extract)

Hydrophilic extraction was conducted using ethanol 70% (v/v), a commonly used food-grade solvent for polyphenols. This technique allows the recovery of phenolic compounds, flavan-3-ols, and other water-soluble antioxidants. The extraction procedure followed in this study is based on the optimized method established by (Medouni-Adrar et al., 2015) , which identified these optimal conditions for grape seed.

I.3. Preparation of lipophilic extract (oil macerate)

Oil maceration is a mild solid-liquid extraction technique used to extract lipophilic bioactives such as tocopherols, carotenoids, and certain non-polar polyphenols into a lipid medium. This method is particularly suitable for incorporation into emulsified food systems like mayonnaise (D'Eusanio et al., 2023).

A grape seed macerate was prepared by infusing grape seed powder in refined soybean oil under controlled conditions.

II. Optimization and selection of the reference mayonnaise formulation

II.1. Ingredients sourcing

The ingredients used for the preparation of the mayonnaise samples were sourced from both commercial and industrial suppliers. Fresh eggs, vinegar, salt, mustard, and sugar were all purchased from the local market. Tap water was first boiled to ensure microbial safety, then cooled to room temperature before use.

As for the oil, refined soybean oil was provided directly by the Cevital industrial complex (Béjaïa, Algeria). This oil was collected straight from the production line, before the addition of synthetic antioxidants, ensuring that it was free from any additives. This unfortified oil was particularly suitable for studying the effects of natural grape seed extracts on oxidative stability.

II.2. Formulation and preparation of base mayonnaise

A base mayonnaise formulation was developed using a mixture design approach (JMP18 software). The objective was to determine the optimal composition based on sensory quality criteria.

In this design, the proportions of three major ingredients, the oil phase, aqueous phase, and egg content, were varied within defined upper and lower limits, while other ingredients such as salt, mustard, sugar, and vinegar were kept constant. A total of 8 formulations were generated and prepared. The formulation that obtained the highest sensory scores was selected as the reference recipe.

III. Preparation of mayonnaise samples for the study

The selected reference formulation, identified through sensory optimization, was used as the base recipe for preparing 8 enriched mayonnaise samples. These samples were designed to

investigate the impact of grape seed extract enrichment, both hydrophilic and lipophilic, on the oxidative stability and sensory quality of mayonnaise.

Enriched mayonnaise samples were prepared using a standardized protocol. The hydrophilic grape seed extract (HGSE) was added to the aqueous phase at three increasing concentrations (E1 < E2 < E3) at the start of mixing, while the grape seed oil macerate, used at a single concentration, was incorporated directly into the oil phase. The 8 formulations differed by the type and concentration of grape seed extracts incorporated, as shown in Table III.

Table III: Formulations of the mayonnaise samples enriched with grape seed extracts

Code	Description
T	Control sample (no enrichissement)
Oi	Enriched with lipophilic extract only
EOi1	Enriched with both extracts (E1)
EOi2	Enriched with both extracts (E2)
EOi3	Enriched with both extracts (E3)
E1	Enriched with hydrophilic extract E1 only
E2	Enriched with hydrophilic extract E2 only
E3	Enriched with hydrophilic extract E3only

Hydrophilic extracts 1, 2, and 3 correspond to the different tested concentrations, with concentration 3 > 2 > 1.

IV. Experimental conditions

The study was conducted over a two-month period during which all mayonnaise samples were stored at a constant temperature of 40 °C to accelerate lipid oxidation and enable a faster evaluation of the effects of grape seed extract enrichment on oxidative stability. In parallel, the eight formulations were also stored at 4 °C to mimic standard refrigeration conditions and monitor their oxidative evolution under typical storage conditions.

V. Analytical methods

V.1. Physical chemical analysis

V.1.1. pH determination

The pH measurement was carried out using a pH meter at a temperature close to 20°C, following the procedure described by the AFNOR standard (1982). This method is based on the detection of free hydronium ions ($H_3\ O^+$) in solution (Meidian Daoed et al., 2022).

After calibration of the pH meter using two buffer solutions (pH 4 and pH 7), the probe was immersed into a beaker containing the mayonnaise sample. The pH value was recorded once the reading stabilized on the display screen of the pH meter.

V.1.2. Dry Extract

The dry extract of mayonnaise samples was determined in order to quantify the total solid content, which reflects the concentration of non-volatile substances such as proteins, carbohydrates, lipids, vitamins, and minerals. This parameter provides a more accurate assessment of nutritional and textural quality by eliminating the variability associated with water content.

2 g of mayonnaise were spread evenly on a pre-tared aluminum pan. The pan was then placed in a drying unit (desiccator or thermobalance), where the sample was subjected to controlled heating until complete evaporation of moisture. The dry extract value was automatically calculated and displayed as a percentage on the device screen, corresponding to the residual solid content that did not volatilize under the applied condition.

V.1.3. Salt content (NaCl)

The determination of salt content in the mayonnaise samples was performed using the Mohr titration method, which quantifies chloride ions (Cl^-) through an argentometric reaction. In this method, silver nitrate ($AgNO_3$) is used as the titrant and potassium chromate ($K_2\ CrO_4$) serves as the indicator (Mohr1856)

1 g of the mayonnaise sample was weighed into an Erlenmeyer flask, followed by the addition of 30 to 50 mL of distilled water heated to 55°C. The mixture was vigorously shaken until the sample was completely dissolved. Two drops of a 10% potassium chromate solution were then added as an indicator. The titration was carried out using a standard silver nitrate solution until a persistent brick-red coloration appeared, indicating the formation of silver chromate and the end point of the reaction (ISO 1989).

This method relies on the initial formation of a white silver chloride precipitate (AgCl) (recation1), and once all chloride ions are precipitated, excess silver ions react with chromate to form red silver chromate ($Ag_2\ CrO_4$) (reaction 2).

$$Ag^+ + Cl^- \rightarrow AgCl\,(s) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

$$2Ag^+ + CrO_4^{2-} \rightarrow Ag_2CrO_4\,(s) \ldots\ldots\ldots\ldots (2)$$

The salt concentration was calculated using the following formula:

$$\mathbf{NaCl\%} = \frac{\boldsymbol{V} \times \boldsymbol{N} \times \mathbf{58.5}}{\boldsymbol{w} * \mathbf{10}}$$

Where $\boldsymbol{V}$ is the volume of $AgNO_3$ used (in mL); $\boldsymbol{N}$ its normality, and $\boldsymbol{w}$ the weight of the sample grams).

V.1.4. Consistency

The consistency of the mayonnaise samples was evaluated using a Bostwick consistometer (Annexe 3), which measures the distance (in centimeters) traveled by the sample under gravity over a fixed period of 30 seconds. The instrument consists of a channel with a gate that retains the sample until released. To perform the test, the mayonnaise was loaded into the Bostwick consistometer up to the upper fill line. Upon releasing the gate, a stopwatch was started simultaneously. After exactly 30 seconds, the distance reached by the mayonnaise along the channel was recorded using the built-in scale. This distance reflects the product's flowability and is indicative of its consistency, with lower values generally corresponding to thicker or more stable formulations. The results are expressed in centimeters per 30 seconds (cm/30s) , ASTM International (2025).

V.1.5. Viscosity

The viscosity of the mayonnaise samples was evaluated to assess their flow behavior and consistency. As a non-Newtonian emulsion, mayonnaise exhibits shear-thinning properties, meaning its viscosity decreases with increasing shear rate. The analysis was performed at room temperature (approximately 25 °C) using a rotational viscometer (Katsaros et al., 2020a).

50 g of each sample were carefully placed into the measurement cup, ensuring no air bubbles were introduced. An appropriate spindle and rotation speed were selected for the emulsion type, and the torque required to rotate the spindle within the sample was measured to determine the viscosity, expressed in centipoise (cP).

V.2. Characterization of grape seed extracts

V.2.1. Preparation of extracts for analysis

- **Reconstitution of the dry hydrophilic extract**

A hydrophilic extract obtained by ethanolic extraction and rotary evaporation, then reconstituted in acidified ethanol with acetic acid to pH 3.5 (ethanol:water:acetic acid, 70:30 v/v) prior to analysis.

- **Extraction of phenolic compounds from grape seed oil macerate**

The extraction of total phenolic compounds from the grape seed oil macerate was performed following the method described by (Konuskan et al., 2019), with slight modifications. Briefly, 1 g of oil macerate was mixed with 3 mL of 80% methanol (v/v), vortexed for 2 minutes, and centrifuged at 3000 rpm for 5 minutes. The supernatant was collected. This extraction was repeated three times on the same sample to ensure the exhaustive recovery of phenolic compounds.

V.2.2. Quantification of total phenolic content (TPC)

The total phenolic content was determined according to the method of (Velioglu et al., 1998). Briefly, 200 µL of each extract was mixed with 1.5 mL of Folin–Ciocalteu reagent diluted 1:10 with distilled water. After three minutes, 1.5 mL of a 6% sodium carbonate solution was added. The mixture was then incubated for 60 minutes at room temperature, and the absorbance was measured at 760 nm. Results were expressed as milligrams of gallic acid equivalents per 100 gram of dry weight (mg GAE/100g DW), using a standard calibration curve.

V.2.3. Quantification of condende tannins (pronthocyanidines)

The quantification of condensed tannins, also known as proanthocyanidins, was performed using the acid butanol method described by (Lawrence J et al., 1986), with slight modifications. This colorimetric method relies on the transformation of proanthocyanidins into anthocyanidins, such as cyanidin, under high-temperature acidic conditions, which results in a measurable pink-red coloration. Ferric sulfate ($FeSO_4$) is included in the reaction mixture to catalyze the process and intensify the resulting color.

As it has been demonstrated by (Škerget et al., 2005); 0.25 mL of hydrophilic extract was mixed with 2 mL of a reagent composed of ferric sulfate and butanol–HCl in a 2:3 ratio. The tubes were then tightly closed and incubated in a water bath at 95°C for 50 minutes. After cooling, the absorbance of the solution was measured at 530 nm using a spectrophotometer. The concentration of proanthocyanidins was determined based on the absorbance measured at 530 nm and calculated

using the following equation. The results were expressed as milligrams of cyanidin-3-glucoside equivalents per 100 grams of dry weight (mg C3GE /100g DW).

$$C = \frac{A530 \times Pm \times Fd}{\varepsilon l}$$

Where: ***A530:*** *Absorbance at 530 nm;* ***Pm:*** *Molecular weight of cyanidin-3-glucoside (287.24g/mol);* ***ε:*** *Molar extinction coefficient (34700 l·mol⁻¹·cm⁻¹);* ***Fd:*** *Dilution factor;* ***L:*** *Path length of the cuvette.*

V.2.4. Quantification of tocopherols in grape seed oil macerate

The quantification of tocopherols was performed using a colorimetric redox method adapted from (Al-Anbakey et al., 2018). In this assay, tocopherols reduce potassium ferricyanide (Fe^{3+}) to ferrocyanide (Fe^{2+}), which then reacts with ammonium iron (III) sulfate to form a stable bluish-green complex. The absorbance of this complex was measured at 743 nm and is directly related to the tocopherol content.

100 µL of the grape seed oil macerate was first diluted in 10 mL of acetone. From this diluted solution, 1 mL was taken and mixed with 4 mL of potassium ferricyanide solution (0.01%) and 2 mL of ammonium iron (III) sulfate dodecahydrate solution (0.01%). The pH of the mixture was adjusted to 4 using 0.1 M HCl, and the final volume was brought to 10 mL with methanol. The mixture was left to react at room temperature for 10 minutes before the absorbance was read at 743 nm.

The tocopherol content was determined from the absorbance reading using a standard calibration curve. Results were expressed as milligrams of α-tocopherol equivalents per 100 grams of dry weight (mg α-TE/100 g DW).

V.2.4. Evaluation of antioxidant activity

- **DPPH* radical scavenging assay**

The antioxidant activity of grape seed extracts was assessed using the DPPH* radical scavenging assay according to the method of Milardović et al. (2006). A volume of 10 µL of each extract was mixed with 2.9 mL of a freshly prepared DPPH* solution (6×10^{-5} mM in methanol). The mixture was incubated in the dark for 30 minutes at room temperature. The decrease in absorbance, due to the reduction of DPPH* radicals by antioxidant compounds, was measured at 515 nm using a spectrophotometer. The antioxidant capacity was calculated based on a calibration curve and expressed as milligrams of ascorbic acid equivalents per gram of dry weight (mg AAE/100g DW).

- **Ferric reducing antioxidant power (FRAP) assay**

The reducing power of the extracts was evaluated by the FRAP assay following the method of Oyaizu (1986), as reported by (Kumar et al., 2005). In this procedure, 1 mL of extract was mixed with 2.5 mL of phosphate buffer (0.2 M, pH 6.6) and 2.5 mL of potassium ferricyanide solution (1%). The mixture was incubated at 50 °C for 20 minutes. After incubation, 2.5 mL of trichloroacetic acid (10%) was added, and the solution was centrifuged at 5000×g for 10 minutes. An aliquot of 2.5 mL of the resulting supernatant was mixed with 2.5 mL of distilled water and 0.5 mL of ferric chloride (0.1%). The absorbance of the green-colored complex formed was measured at 700 nm. The reducing antioxidant capacity was expressed as mg of ascorbic acid equivalents per gram of dry weight (mg AAE/100g DW).

V.3. Oxidative stability monitoring of mayonnaise

V.3.1. Peroxide value determination

The peroxide value was determined to assess the primary oxidation state of fats in the mayonnaise samples.

2 g of sample was weighed into an Erlenmeyer flask, to which 5 mL of a 3:2 (v/v) mixture of acetic acid and chloroform was added. A solution prepared from 0.5 g of saturated potassium iodide (KI) in 1 mL of distilled water was then added to the mixture. The flask was shaken and allowed to stand in the dark for 1 to 3 minutes. Next, 30 mL of distilled water and a few drops of starch solution were added as an indicator. The resulting mixture was titrated with 0.01 N sodium thiosulfate solution until the blue coloration disappeared, indicating the endpoint. The measurements were carried out regularly over a two-month period, with evaluations performed every 5 days. (ISO 3960:2007; Khiavi et al., 2025)

The results were expressed in milliequivalents of active oxygen per kilogram of sample (meq O_2 /kg of sample), and the peroxide value (PV) was calculated using the following formula:

$$\mathbf{IP} = \frac{\boldsymbol{V} \times \boldsymbol{N} \times \mathbf{1000}}{\boldsymbol{M0}}$$

***V**: Volume of sodium thiosulfate solution used in titration (in mL); **N** : Normality of the sodium thiosulfate solution (0.01 N); $\boldsymbol{M_0}$: Mass of the oil sample (in grams).*

The peroxide value (PV) was monitored over a period of 55 days, with measurements taken every 5 days. We determined the predicted oxidative stability of the mayonnaise formulations using Minitab modeling, based on the time required to reach a PV of 10 meq O_2 /kg, in order to compare the enriched samples to the non-enriched control.

V.3.2. Acidity determination

The acidity of the mayonnaise samples was assessed by acid-base titration using sodium hydroxide (NaOH), with phenolphthalein serving as the pH indicator. Approximately 1 g of mayonnaise was weighed into a clean Erlenmeyer flask, and 30 mL of distilled water preheated to 55 °C was added. The mixture was vigorously shaken to ensure full dispersion of the sample. Two drops of phenolphthalein were then added, and the solution was titrated with 0.1 N NaOH until a stable pale pink color appeared, marking the endpoint. The measurements were carried out regularly over a two-month period, with evaluations performed every 5 days (CODEX STAN 168-1989; ISO 3960:2007) .

The results were expressed as a percentage of acetic acid, and acidity was calculated using the following formula:

$$\textbf{Acidity} = \frac{\boldsymbol{V \times N \times Mm}}{\boldsymbol{M0}}$$

V: *Volume of NaOH solution used in titration (in mL);* ***N:*** *Normality of the NaOH solution (0.1 N);* ***Mm*** *: Molar mass (molecular weight) of acetic acid (60g/mol), used as the standard fatty acid;* $\boldsymbol{M_0}$ *: Mass of the oil sample (in grams).*

V.4. Sensory evaluation

Sensory analysis is a scientific method that uses human senses to evaluate a product's organoleptic characteristics, such as color, odor, taste, texture and sound, Is widely applied in quality control and product development (Piana et al., 2004; Van Trup' et al., 1995). The field is guided by two main approaches: a product-oriented perspective, which links sensory traits to physical properties, and a marketing-oriented perspective, which focuses on consumer preferences and market behavior (Van Trup' et al., 1995). Combining these approaches supports innovation and enhances product acceptance. Over time, sensory analysis has become more structured, incorporating trained panels, statistical tools, and advanced methods like fuzzy logic (Piana et al., 2004; Vivek et al., 2020). It is also increasingly integrated with instrumental techniques and emerging technologies such as virtual reality to improve accuracy and emotional insight(Segnini et al., 1999; Zulkarnain et al., 2024).

In this study, a descriptive sensory analysis was performed on three selected enriched mayonnaise samples, along with the non-enriched control, in order to investigate the effect of grape seed extract enrichment on the organoleptic quality of the products. This comparative evaluation aimed to assess possible sensory differences induced by the incorporation of grape seed extracts.

The assessment was conducted at the sensory analysis laboratory of the University by an expert panel composed of 13 trained members. The panelists evaluated key sensory attributes, including appearance, color, odor, texture, taste, and overall acceptability. In addition to the structured evaluation, they were encouraged to express their personal preferences, as indicated in the questionnaire provided in the appendix.

V.5. Statistical analysis

All experimental data were obtained from triplicate trials and expressed as mean values ± standard deviation using Microsoft Excel 2010. To compare the physicochemical parameters (salt content, pH, viscosity, consistency, dry extract), as well as oxidative stability indicators (lag time at 232 and 270 nm, and predicted time to PV = 10 meq O_2 /kg), a one-way ANOVA followed by the Least Significant Difference (LSD) test at a significance level of p= 0.05 was performed using STATISTICA 7.1.

Oxidative stability modeling was carried out in Minitab 18 using the stability analysis module to estimate the predicted time at which the peroxide value would reach the critical threshold (PV = 10 meq O_2 /kg). Formulation optimization was conducted using JMP 18 (SAS) based on a mixture design approach to determine the optimal ingredient combination. Sensory data were analyzed with XLSTAT software through descriptive analysis and Principal Component Analysis (PCA), allowing for comprehensive sample characterization and differentiation. Additional statistical analyses may be considered as needed.

Results and discussion

I. Selection of the optimal experimental mayonnaise formulation

An extreme vertices mixture design was employed to investigate the effect of three components: oil phase (%) egg yolk (%), and aqueous phase (%), on the sensory properties of mayonnaise. Eight formulations were automatically generated using JMP software, in accordance with the imposed proportional constraints on the ingredients.

I.1. Effect of formulation components on sensory criteria: ANOVA analysis

An analysis of variance (ANOVA), based on the LogWorth values, was conducted to evaluate the individual effect of each formulation component on the sensory response variables. The contribution of each factor to the overall sensory evaluation is illustrated in

All factors were found to be highly significant ($p < 0.001$), indicating that they have a strong influence on the sensory responses. Oil exhibited the most pronounced effect, having the greatest impact on sensory preferences and characteristics.

Statistical analysis reveals that the oil phase ($p = 0.00012$) exerts a highly significant influence on sensory responses. This strong effect can be attributed to the oil's central role in defining both flavor intensity and emulsion texture, which are key drivers of consumer acceptance. Indeed, oil content contributes directly to creaminess and mouthfeel while also serving as a medium for flavor compounds, thus enhancing palatability and preference (Upadhyay et al., 2020)

Similarly, the aqueous phase shows a significant impact, likely due to its role in modulating the viscosity and balance of the emulsion. An appropriate aqueous proportion helps control the overall consistency and fluidity of the product, ensuring that it remains smooth and homogeneous without being overly thick or runny. These aspects are crucial to the perceived quality and spreadability of mayonnaise, directly affecting consumer evaluations of consistency and overall enjoyment.

In contrast, the effect of egg yolk, while still statistically significant, is comparatively less pronounced. This suggests that within the tested proportions, egg yolk—despite being a natural emulsifier and contributor to richness—plays a more supporting role in shaping the final sensory experience (Chang et al., 2021). Its functional contribution to emulsion stability and slight flavor enhancement may be secondary to the dominant effects of oil and water phase ratios.

Overall, these findings demonstrate that optimizing mayonnaise formulation for sensory appeal requires a careful balance between the oil phase and aqueous phase, as they most strongly influence consumer perception of flavor and consistency. While egg yolk remains important for emulsion stability and texture, its impact appears more nuanced (Ali & Wu, 2021). These results

align with literature emphasizing the importance of emulsion structure, oil-water ratios, and fat perception in determining the sensory quality of mayonnaise and other emulsified food products.

I.2. Graphical interpretation of optimal regions (contour plots)

Figure 3 displays the mixture triangle diagram representing the distribution of the eight experimental mayonnaise formulations within the constrained design space. Each vertex corresponds to one of the three mixture components: oil phase (%), egg yolk (%), and aqueous phase (%).

All experimental points are located in the upper-central region of the triangle, indicating that the design space was limited to formulations containing: between 70% and 80% oil phase (%), approximately 5% to 12% egg yolk (%), and between 5% and 18% aqueous phase.

This localized region reflects the specific constraints defined during the mixture design process to identify the optimal formulation based on sensory acceptability criteria. The proximity of the points suggests a systematic variation of proportions in a narrow but strategically selected formulation zone, enabling a focused optimization of sensory responses.

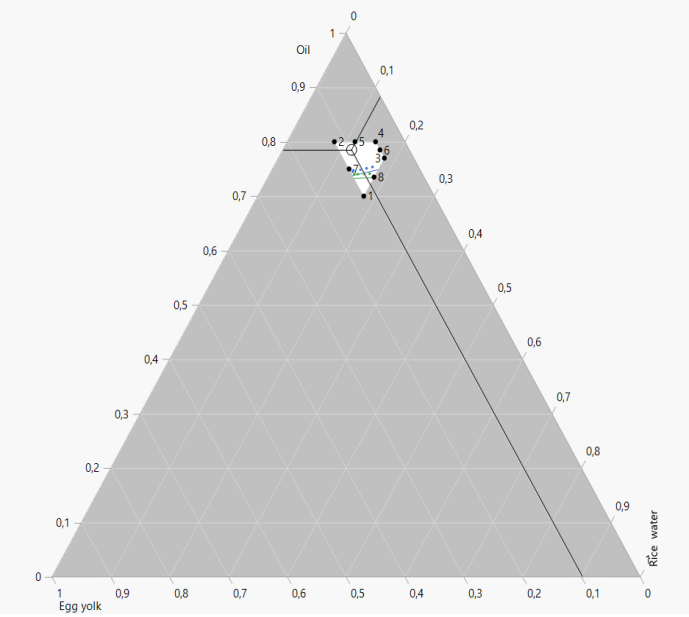

Figure 3: Mixture triangle showing the distribution of experimental formulations within the design space.

The contour plot (mixture design) highlights the effect of the proportions of oil phase (%), egg yolk (%), and aqueous phase (%) on the two evaluated sensory responses: overall preference (in green), and consistency (in blue). The areas enclosed by contour lines indicate the most

favorable combinations of proportions for each criterion. According to the model and the analysis of effects:

These visual trends from the diagram confirm that the interactions between the three components significantly influence the product's sensory perception. The diagram thus represents a valuable decision-making tool to guide formulation choices toward the optimal regions based on targeted sensory attributes.

I.3. Selection of the optimal formulation: synthesis through radar plot visualization

Following the modeling and validation of sensory responses, a radar plot was used to synthesize the results for the three main sensory attributes : flavor, consistency, and overall preference, across the eight experimental formulations. This type of graphical representation offers a comprehensive and intuitive overview of each formulation's performance, facilitating a direct comparison of their overall sensory profiles.

As shown in Figure 4, the formulations H (803), B(104) , and G(705) exhibit the most favorable balance between the three criteria. Among them, recipe G stands out by presenting a relatively extended and well-balanced radar shape, indicating strong performance across all sensory dimensions. More specifically: G exhibits the highest consistency score, outperforming all other samples on this criterion. It also achieves the highest overall preference score, suggesting strong consumer appeal. While its flavor score is slightly lower than some other formulations, it still ranks among the top-performing samples.

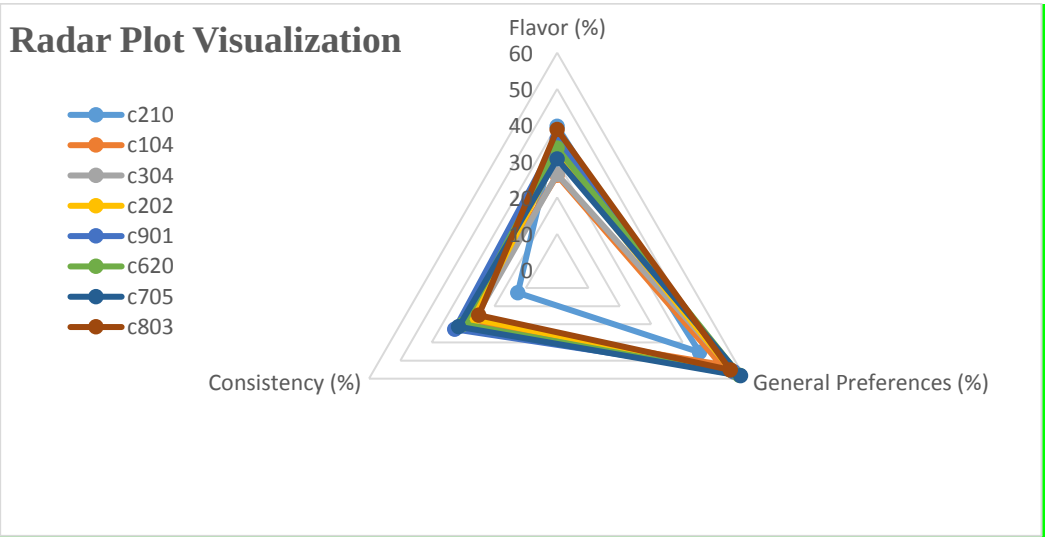

Figure 4: Radar chart of sensory evaluation results for different mayonnaise formulations.

These combined results make G the most promising formulation, offering the best compromise across the three key sensory attributes. Therefore, G is selected as the optimal formulation.

II. Bioactive composition and antioxidant potential of grape seed extract and macerated oil

II.1. Yield of Grape Seed Extracts

The yield of grape seed extract was determined gravimetrically. Given that 10 g of grape seed powder was used for extraction, the resulting yield was calculated to be 34.53%.

This yield of 34.53% obtained in this study is comparatively higher than those reported using conventional methods in the literature. For example, grape seed extract yields ranging from 4.96% to 22.24% were observed by (Samavardhana et al., 2015) using ethanol-based extraction. Similarly, supercritical CO_2 extraction achieved a yield of 14.49% under optimized conditions (Jokić et al., 2016). In contrast, ultrasound-assisted extraction under highly controlled parameters reached much higher yields, up to 82.9% (Böger et al., 2018) , while sonication prior to Soxhlet or Bligh-Dyer extraction enhanced yields by up to 32.10% (de Souza et al., 2020). These findings highlight how extraction yield is highly dependent on method type, solvent system, and pre-treatment steps, underlining the importance of optimizing conditions to maximize phenolic recovery.

II.2. Quantification of Bioactive Compounds and their antioxidant activity

The antioxidant potential and the bioactive compound profiles of both the grape seed dry extract and grape seed macerate were comprehensively evaluated, with the results summarized in **Table IV** The grape seed dry extract exhibited a higher total polyphenol content (1479.47 ± 8.61 mg EAG/100 mg DW) than the oil macerate (1016.15 ± 64.41 mg EAG/100 mg DW). While this difference may appear modest, it is important to note that polyphenols are predominantly hydrophilic compounds, and therefore are more efficiently extracted with polar solvents (e.g., ethanol–water) than with lipophilic media like vegetable oil. This limited solubility in oil likely explains the lower polyphenol concentration in the macerated oil, as many antioxidant molecules remain in the solid matrix during extraction. TPC values recorded between 1300 and 1600 mg GAE/100 g DW, depending on the solvent composition and grape variety (Felhi et al., 2016). On the other hand, significantly lower polyphenol levels, often below 100 mg GAE/100 g, have been reported in grape seed oil due to the poor solubility of polyphenols in lipophilic environments (Zhou et al., 2020), confirming the limited extraction efficiency of oil-based maceration.

Table IV: Bioactive Content and Antioxidant Properties of Grape Seed Extract and Oil maceration

Parameter	Grape seed dry extract	Grape seed oil maceration
Total Polyphenol content (mg EAG/100mg DW)	1479.47 ± 8.61	1016.15 ± 64.41
Proanthocyanidins (mg EC/100 g DW)	2095.62 ± 120.15	/
Tocopherols (mg α - tocopherol/mg oil)	/	139.85± 1.87
DPPH* (mg EAA/100g DW)	8157.46 ± 143.37	1074.94 ± 79.52
FRAP(mg EAA/100g DW)	3045.06 ± 21.96	855.07 ± 98.32

In terms of proanthocyanidins, which are powerful polyphenolic antioxidants, they were found only in the dry extract (2095.62 mg EC/100 g DW) and were absent in the oil macerate. This again highlights the inefficiency of oil-based maceration in extracting hydrophilic compounds, particularly large, polar flavonoids. reported proanthocyanidin contents in dry grape seed extracts between 18 and 22 mg/100 g, depending on extraction efficiency and grape variety .(shi and al 2003)

The grape seed oil macerate contained 139.85 mg of α-tocopherol / kg of oil, which represents its main antioxidant component. However, this level is noticeably lower than those reported in the literature for pure grape seed oil, where α-tocopherol concentrations typically range from 230 to 400 mg/kg, depending on the grape variety and extraction method (Göktürk Baydar et al., 2007); Sabir et al., 2012). More recent studies have also confirmed values between 250 and 350 mg/kg in cold-pressed grape seed oils (Banjanin et al., 2019) This reduced content in the macerate may be explained by the nature of the sample, which is not pure grape seed oil but rather a vegetable oil infused with grape solids. As a result, it may not fully capture the spectrum of naturally occurring lipophilic antioxidants present in grape seeds. Furthermore, the maceration process may not allow complete transfer of tocopherols and other lipid-soluble compounds from the grape matrix into the oil phase .This disparity is also reflected in the antioxidant activity assays.

The DPPH* radical scavenging activity of the dry extract (8157.46 mg EAA/100g DW) was substantially higher than that of the oil macerate (1074.94 mg EAA/100g DW), as was the ferric reducing antioxidant power (FRAP) (3045.06 vs. 855.07 mg EAA/100g DW, respectively). These results confirm that the dry extract, being richer in both total phenolics and proanthocyanidins, exhibits greater overall antioxidant capacity. In contrast, the oil macerate shows moderate activity, primarily due to its tocopherol content, but likely limited by both the extraction method and the

lower yield of transferred antioxidants.

The grape seed dry extract offers a more potent antioxidant profile due to its high polyphenol content and is especially effective in aqueous systems. The oil macerate, while less active overall, provides valuable lipophilic antioxidants like tocopherols. Their complementary nature makes them suitable for combined use in emulsified food systems such as mayonnaise, where both aqueous- and lipid-phase oxidative stability are desired.

III. Physicochemical analysis of mayonnaise samples

Eight mayonnaise formulations were analyzed to assess the impact of grape seed derivatives on their physicochemical properties. One sample was prepared with grape seed maceration oil only (Oi), three were enriched with hydrophilic grape seed extracts (E1, E2, E3), and three others combined both oil maceration and hydrophilic extract (EOi1, EOi2, EOi3). A control formulation (T) without any enrichment served as a reference.

The objective was to determine how these enrichments affect sodium chloride content, consistency, pH, dry extract content, and viscosity. The results are summarized in Table X.

III.1. NaCl content

Salt is a common ingredient in mayonnaise used to enhance flavor and extend shelf life. The salt content in mayonnaise is variable but typically falls within a range depending on the desired taste and preservation requirements of the product (Gaipova et al., 2021) Exact average values for salt content are not specified in the retrieved studies, but commercial mayonnaise usually contains about 1-2% salt by weight (Depree et al; 2001).

The statistical analysis of NaCl content revealed slight but significant differences between samples ($p < 0.05$), with values ranging from 1.04% to 1.12%. However, these variations remain minimal and do not reflect a direct impact of enrichment, as all formulations were prepared using the same base recipe. For instance, sample Oi (1.12 ± 0.02%) showed the highest salt content, while E1 had the lowest (1.04 ± 0.04%). Although statistically grouped differently, the variations are not systematically associated with the type or level of enrichment. These differences are more likely due to minor inconsistencies in the manufacturing process, such as weighing, homogenization, or mixing, rather than deliberate changes in salt content.

The NaCl content of all samples ranged from 1.04% to 1.12%, placing them within the typical formulation range (1.0–1.5%) found in commercial mayonnaise (Ares et al., 2021; de Souza et al., 2022). This ensures not only acceptable sensory saltiness but also contributes to

microbial and emulsion stability.

Table V : Physicochemical characteristics of mayonnaise samples

Sample	Nacl content (%)	Consistancy Cm/30s	Viscosity (mPa·s)	pH	Dry extract (%)
T	1.10 ± 0.024^{ab}	3.50 ± 0.10^{a}	38718.5 ± 415.5^{h}	4.48^{a}	80.05 ±0.36^{f}
Oi	1.12 ±0.020 ^{a}	1.05 ± 0.05^{c}	83519.5± 224.5^{d}	4.45 ^{b}	81.40 ±0.15^{b}
EOi1	1.06 ± 0.04^{b}	1.25 ± 0.05 ^{b}	72595.5± 136.5^{g}	4.5 ^{ab}	81.82 ±0.20^{a}
EOi2	1.1 ± 0.020 ^{ab}	1.25 ± 0.05 ^{b}	74105± 23.00^{f}	4.49 ^{a}	80.32 ±0.22^{ef}
EOi3	1.11 ± 0.01 ^{a}	1.25 ± 0.05 ^{b}	76154.5± 439.5^{e}	4.5^{ab}	80.66 ±0.25^{de}
E1	1.04 ± 0.04 ^{c}	0.95 ± 0.05 ^{cd}	94851± 1.00^{a}	4.45^{b}	80.8 ± 0.15^{c}
E2	1.07 ± 0.04 ^{abc}	0.95 ± 0.05 ^{cd}	92805± 503.00^{c}	4.5^{ab}	80.71 ± 0.31^{d}
E3	1.06 ± 0.02 ^{bc}	0.85 ± 0.05 ^{d}	93743.5± 268.5^{b}	4.48^{a}	80.28 ±0.07^{ef}

Values with different letters in the same column are significantly different (P < 0.05). Results are ranked in descending order.

III.2. Consistancy

The consistency of the mayonnaise samples was assessed using the Bostwick method, where higher flow values indicate a more fluid product and lower values reflect a thicker, more cohesive emulsion.

The statistical analysis of consistency measurements revealed highly significant differences between the mayonnaise samples ($p < 0.05$). The control sample (T) exhibited the highest Bostwick flow value (3.50 cm/30 s), indicating the lowest consistency (most fluid texture). In contrast, the enriched samples showed significantly lower values, reflecting thicker textures. All enriched samples exhibited significantly lower flow values ($p < 0.05$), meaning they were more consistent than the control. Among them, E3 showed the highest consistency (0.85 cm/30 s), followed by EOi1, EOi2, and EOi3, which did not differ significantly from each other (1.25 cm/30 s). The Oi, E1, and E2 samples showed intermediate values but remained significantly more consistent than the control.

These results suggest that enrichment with grape seed derivatives, especially in the aqueous phase, significantly enhances mayonnaise consistency, likely due to the presence of polyphenols and other compounds that influence emulsion structure and water-binding capacity. The observed

differences confirm the impact of the enrichment strategy on textural properties, with aqueous-phase enrichment leading to the most pronounced thickening effect.

When compared to values reported in the literature, the consistency results of the enriched mayonnaise samples in this study fall within or below the typical range observed for standard mayonnaise, which generally flows between 1.0 and 2.5 cm/30 s using the Bostwick consistometer (Depree & Savage, 2001; Kadian et al., 2021) . Notably, several enriched formulations, particularly E3, E1, and E2, exhibited values below 1.0 cm/30 s, indicating a markedly thicker and more stable emulsion. This enhanced consistency may be attributed to the structural effects of grape seed derivatives, especially polyphenols, which are known to strengthen emulsion systems and reduce flowability. These findings confirm that the enrichment strategy not only maintains but can even exceed the textural quality standards expected of commercial mayonnaise products.

III.3. Viscosity

The viscosity of the mayonnaise samples was measured to evaluate the impact of grape seed enrichment on emulsion thickness and texture. Statistical analysis revealed significant differences between samples ($p < 0.05$), indicating that the enrichment strategy had a marked effect on this parameter.

The control sample (T) showed the lowest viscosity (38718.5 mPa·s), reflecting a thinner and less structured emulsion. In contrast, all enriched samples exhibited significantly higher viscosity values ($p < 0.05$), confirming their enhanced thickness. The highest viscosities were recorded for samples E1 (94851 mPa·s), E3 (93743.5 ± 268.5 mPa·s), and E2 (92805 mPa·s), enriched with grape seed extract in the aqueous phase, either alone or in combination. These were significantly more viscous than all other samples.

Intermediate viscosities were observed in EOi3, EOi2, and EOi1 (76154.5, 74105, and 72595.5 mPa·s respectively), enriched with both grape seed oil and extract. The Oi sample (grape seed oil only) showed a moderate viscosity (83519.5 mPa·s), significantly higher than the control but lower than most aqueous-phase enriched samples.

These results suggest that enrichment with grape seed derivatives, especially in the aqueous phase, significantly enhances mayonnaise viscosity. This effect is likely due to the presence of polyphenols and other hydrophilic compounds that strengthen the emulsion network and improve water retention. Overall, the findings confirm the influence of enrichment strategy on the

rheological behavior of mayonnaise, with aqueous-phase formulations offering the most pronounced thickening effect.

Mayonnaise typically exhibits a semi-solid, oil-in-water emulsion structure, where consistency and viscosity are key quality parameters. These properties are influenced by formulation factors such as the type of oil, stabilizers (like egg yolk or xanthan gum), and added functional ingredients (Mozafari et al., 2017; Mirsadeghi Darabi et al., 2022). The enhanced consistency in enriched samples may be attributed to the presence of bioactive compounds or fiber-rich ingredients, which are known to increase structural firmness and improve emulsion stability (Mistrianu et al., 2022). This aligns with previous findings that highlight the role of formulation components in determining the texture of mayonnaise (Chetana et al., 2019; (Katsaros et al., 2020); Wiguna et al., 2023).

III.4. pH

Mayonnaise is typically a slightly acidic product, with a pH generally ranging from around 3.5 to 4.5. This acidity is primarily due to the presence of vinegar or lemon juice, which are common ingredients in mayonnaise formulations (Shen et al., 2022).

The pH values of the mayonnaise samples ranged from 4.45 to 4.50, indicating slightly acidic conditions typical of standard mayonnaise formulations. Statistical analysis showed no significant differences between most samples ($p > 0.05$), suggesting that the enrichment strategy did not markedly affect the acidity of the products.

The control sample (T) had a pH of 4.48, which was statistically similar to most enriched formulations, including E3 (4.48), EOi2 (4.49), and EOi1/EOi3/E2 (4.50). Slightly lower values were observed in samples Oi and E1 (4.45), but these remained within the expected range and did not represent meaningful deviations.

These results indicate that the incorporation of grape seed derivatives, whether hydrophilic or lipophilic, did not significantly influence the pH of the mayonnaise. This stability in acidity is crucial for both microbial safety and product quality, confirming that the enrichment process preserved the essential physicochemical balance of the emulsions.

III.5. Dry extract

The dry extract content of the mayonnaise samples ranged from 80.05% to 81.82%. Statistical analysis revealed highly significant differences between the samples ($p < 0.05$),

indicating that the enrichment strategy influenced the concentration of solids in the formulations.

The sample EOi1 exhibited the highest dry extract value (81.82%), followed closely by Oi (81.40%), confirming that oil-phase enrichment contributes to higher solid content, likely due to the concentration effect of oil incorporation. Sample E1 also showed a relatively high dry extract (80.80%), while E2 and EOi3 presented slightly lower but still elevated values (80.71% and 80.66%, respectively). In contrast, the control sample (T) recorded the lowest value (80.05%), which was significantly different from most enriched formulations.

Initially, the higher values observed in the enriched samples could be attributed to the addition of grape seed extract, which is known to contain considerable amounts of dietary fiber, proteins, polyphenols, and minerals components that contribute significantly to total dry weight .

Overall, these findings confirm that enrichment with grape seed derivatives, particularly in the oil or dual phases, leads to an increase in dry extract, reinforcing the functional contribution of these natural ingredients to mayonnaise formulations.

IV. Monitoring of oxidative stability in enriched mayonnaise samples

IV.1. Acidity test monitoring

The figure below illustrates the evolution of acidity in the control and antioxidant-enriched mayonnaise samples over a 53-day storage period. Acidity was monitored as an indicator of lipid hydrolysis and potential microbial activity.

The acidity of mayonnaise is important for both flavor and preservation. It is influenced by the type and concentration of acidulants like vinegar or citric acid. Studies have shown that altering the formulation can affect the acidity levels, but exact average values may vary based on specific recipes or production methods (Mirsadeghi Darabi et al., 2022; Mohammadi et al., 2024).

The acidity of all mayonnaise samples increased progressively over the 50-day storage period at 40 °C. As shown in Figure 5. The control sample (T) exhibited the highest acidity increase indicating significant lipid hydrolysis and microbial activity in the absence of protective agents.

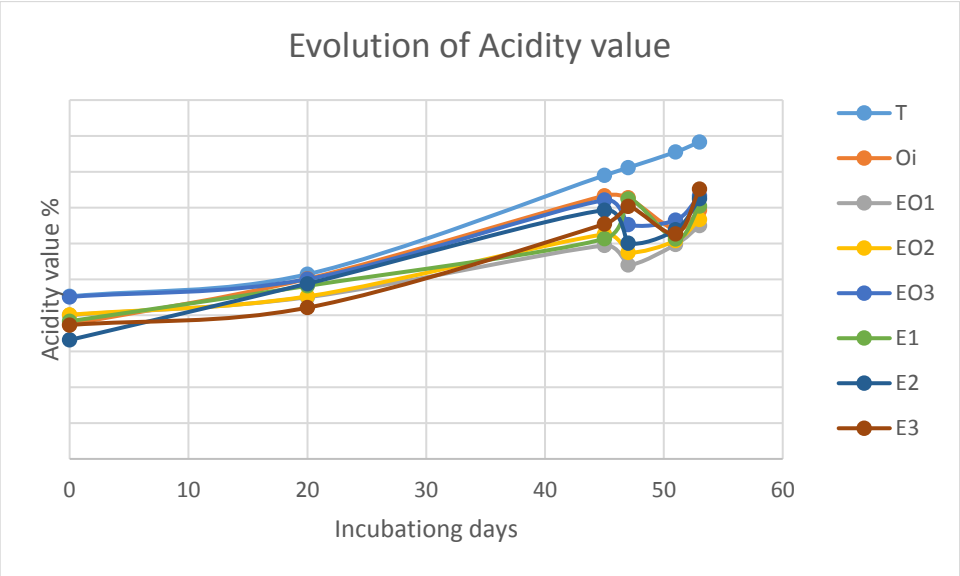

Figure 5: Evolution of acidity value (%) of mayonnaise samples over storage at high temperature.

In contrast, antioxidant-enriched samples (EOi1, EOi2, EOi3, E1, E2, and E3) demonstrated a slower rate of acidity development during the early stages. However, a notable rise in acidity was observed across all samples after approximately 40 days meaning after temperature acceleration, indicating a general decline in stability

Samples enriched with macerated oil of grape seed (EOi1–EOi2) demonstrated improved stability. EO1 showed the lowest increase, suggesting superior inhibition of acid-producing reactions, likely due to the antimicrobial and antioxidant properties of the essential macerated oil constituents.

The samples containing grape seed extract (E1–E3) also exhibited delayed acidity progression compared to the control, though E2 and E3 reached values slightly higher than their essential oil counterparts. This suggests that while grape seed extract can mitigate oxidation, its effect on acidity buildup may be less pronounced, possibly due to weaker antimicrobial effects. Overall, both enrichment strategies reduced acidity rise, with essential oils showing slightly better performance in controlling acid development during storage.

Among the enriched formulations, samples E2 and E3 appeared to offer the greatest protection against acidity development, suggesting a higher efficacy of their antioxidant systems compared to Oi and EOi1.

The effectiveness of antioxidants is also closely related to their partitioning behavior within the emulsion system. Lipophilic antioxidants like tocopherols tend to concentrate in the oil phase, while hydrophilic compounds may localize at the oil–water interface or in the aqueous phase. This

uneven distribution may leave parts of the emulsion insufficiently protected, allowing localized oxidation and acid formation to proceed (Jacobsen et al., 1999). Moreover, thermal storage conditions can degrade antioxidant compounds over time, reducing their capacity to prevent oxidative reactions (Shaygannia et al., 2021)

In addition to their chemical degradation, antioxidants may also interact with other components in the formulation, such as emulsifiers, proteins, or salts, in ways that limit their activity. Poor emulsifier performance or destabilization of the oil–water interface can further promote lipid oxidation and acid accumulation (Venkataraman et al., 2024). These physicochemical factors likely contributed to the increased acidity observed after day 40, even in samples enriched with antioxidants.

Although lipid oxidation appears to be the dominant mechanism behind acidity development, potential microbiological contributions should also be considered.

IV.2. Peroxide value monitoring

The peroxide index of the mayonnaise samples remained stable (0 meq O_2 /kg) during the first 40 days of storage, indicating no detectable lipid oxidation during this period. Based on preliminary observations, oxidation typically began after day 40 when the samples were incubated at 40 °C.

It is important to note that samples stored at 4 °C remained stable throughout the duration of the study.

The **figure 6** presents the evolution of peroxide values (PV) over time in the 8 different mayonnaise samples subjected to accelerated oxidation. Peroxide value is a primary indicator of lipid oxidation and reflects the formation of hydroperoxides in the early stages of fat degradation.

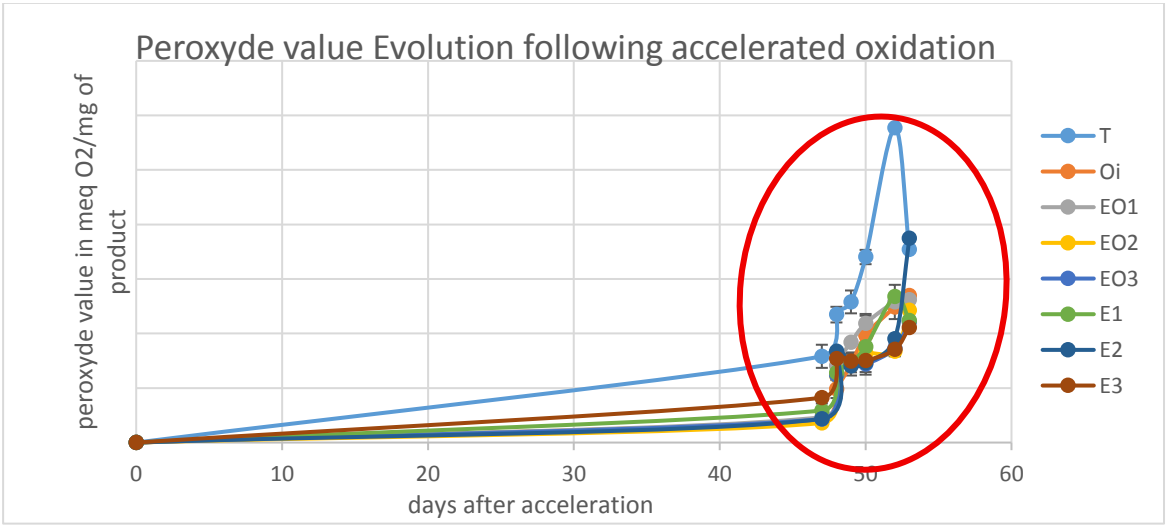

Figure 6: Peroxide Value Evolution Following Accelerated Oxidation During Storage at 60 °C.

Given the limited clarity observed in specific regions of the original peroxide index graph, particularly those marked in red these segments were extracted and magnified to generate a separate, detailed graph.

The evolution of peroxide values in the tested mayonnaise formulations under accelerated oxidation conditions reflects the efficiency of antioxidant enrichment strategies. The results show that both of them (oil vs. aqueous phase) and concentration of seeds extracts significantly influenced the oxidative stability of the emulsions.

The control sample (T), which lacked any antioxidant enrichment, exhibited a rapid and continuous increase in peroxide value (PV). Throughout storage. This trend indicates high susceptibility of emulsified lipids to oxidation in the absence of protective agents. Similar behavior has been reported in simple emulsified systems, where hydroperoxide formation progresses quickly when no antioxidants are present (Frankel, 1998). These results reaffirm that base formulations are inherently unstable without antioxidant intervention.

Sample Oi, enriched only in the oil phase, showed improved oxidative stability compared to the blank. Its lower PV trajectory suggests that lipophilic antioxidants, such as tocopherols, effectively scavenge lipid radicals, particularly within the lipid droplets and at the oil–water interface, where primary oxidation tends to occur (Liu et al., 2014; Ghorbani Gorji et al., 2019). However, this protection remained relatively limited. This may be due to the inability of oil-phase antioxidants to fully neutralize radicals generated in or near the interfacial region, where water-soluble pro-oxidants can also be active.

Formulations EOi1, EOi2, and EOi3, which were enriched in both the oil and aqueous phases, combined tocopherols with increasing concentrations of grape seed extract. These dual-phase systems showed substantially better oxidative stability, especially in EOi2 and EOi3, which demonstrated clear *latency phases*, during which PVs remained nearly constant. This indicates strong antioxidant buffering capacity and suppression of hydroperoxide formation. This phenomenon is well supported by (Mcclements & Decker, 2000) and Laguerre et al., 2007), who emphasized that antioxidant partitioning between the oil, water, and interfacial regions is critical in emulsified systems. These authors explained that antioxidants residing in both phases can act synergistically, scavenging free radicals before they initiate lipid oxidation.

The observed synergistic effect between lipophilic and hydrophilic antioxidants aligns with recent literature, which shows that antioxidants distributed across both emulsion phases can act

cooperatively to form a more effective barrier against lipid oxidation (Budilarto & Kamal-Eldin, 2015 ; Zhang et al., 2022). The best performance was observed in EOi3, with 150 ppm aqueous-phase enrichment, suggesting a concentration-dependent effect. The pronounced latency phase indicates improved radical scavenging and interfacial stabilization.

This behavior supports the supramolecular antioxidant model, which emphasizes that both chemical activity and spatial distribution of antioxidants are crucial for protecting emulsions against oxidation (Chaiyasit et al., 2005; Farooq et al., 2021) The enhanced oxidative resistance in dual-phase systems is consistent with recent findings that interface-localized antioxidant clusters can offer extended protection (Mitrus et al., 2019)

Samples E1, E2, and E3 also showed improved oxidative stability compared to the blank. Among them, E3 demonstrated the best performance, with lower PVs and delayed oxidation onset. The appearance of latency phases in E2 and E3, although shorter than those in dual-phase samples, confirms that aqueous antioxidants can migrate toward the oil–water interface and offer protection. However, their weaker performance may be due to lower interfacial affinity or shorter residence time Cliquez ou appuyez ici pour entrer du texte. ; Kiokias & Oreopoulou, 2022)

One notable trend in the graph is the drop in PV observed in several enriched samples after an initial increase. This behavior was especially clear in EOi2, EOi3, E2, and E3, all of which showed a decline in peroxide value after reaching a peak around 6–8 hours. In contrast, the control sample (T), which lacks antioxidant enrichment, showed a continuous PV increase with no such decline, indicating uncontrolled oxidation.

The PV drop in enriched samples is most likely due to the neutralizing action of antioxidant compounds, which scavenge lipid radicals and interrupt the oxidative chain reaction. Natural polyphenols and flavonoids are known to exert such effects by neutralizing reactive oxygen species and preventing further peroxide formation (Li et al., 2016; Shebis et al., 2013; Chen et al., 2022)). Their effectiveness here confirms the protective role of the added bioactives, improving oxidative stability under accelerated conditions.

Overall, these findings confirm that antioxidant partition governed by molecular polarity, structure, and emulsion architecture is a key factor influencing oxidative stability. The dual-phase enrichment strategy proved most effective, supporting current understanding that combining lipophilic and hydrophilic antioxidants offers superior protection in emulsified lipid systems (Ghorbani Gorji et al., 2019 ; Zhang et al., 2021).

IV.3. Predicted oxidative stability of mayonnaise formulations using Minitab modeling based on peroxide value (PV = 10 meq O_2 /kg)

To better compare the oxidative stability of the different mayonnaise formulations, statistical modeling was conducted using the Minitab stability module. The predicted time required to reach the oxidation threshold, defined as a peroxide value (PV) of 10 meq O_2 /kg, was estimated under accelerated storage conditions at 40 °C. Although these results do not reflect actual shelf-life, they provide a comparative index of antioxidant effectiveness across formulations.

The results presented in the following table summarize the predictive trends derived from the data, offering insight into the relative effectiveness of the antioxidant treatments in delaying oxidation.

The predicted time to reach the oxidation threshold, defined as a peroxide value (PV) of 10 meq O_2 /kg (Time to PV=10) allow for a clear comparison of the effectiveness of hydrophilic and lipophilic grape seed extracts across formulations **(Appendix 6).**

The oxidative stability of the different mayonnaise formulations was assessed using predictive modeling of peroxide value (PV) evolution under accelerated conditions. Statistical analysis revealed highly significant differences between samples for both the predicted oxidation time and the oxidation rate ($p < 0.05$), confirming that the enrichment strategy had a substantial impact on lipid peroxidation behavior.

The control sample (T) exhibited the shortest predicted time to reach the oxidation threshold indicating low oxidative stability and rapid degradation in the absence of antioxidants.

In contrast to the control, all enriched samples exhibited significantly enhanced oxidative stability, as indicated by longer times to reach the oxidation threshold (PV = 10 meq O_2 /kg) ($p < 0.05$). The most remarkable performance was recorded for EOi3, which combined the highest concentrations of both lipophilic (oil phase) and hydrophilic (aqueous phase) grape seed enrichments.

E3 and EOi2 also demonstrated excellent oxidative resistances almost double that of the control. These two samples belonged to statistically distinct groups (b and c) compared to most other formulations ($p < 0.05$), confirming the effectiveness of aqueous-phase enrichment, particularly at higher concentrations.

E1 and Oi were statistically less stable than E3 and EOi2 but significantly more stable than the control ($p<0.05$). EOi1 and E2 ranked lowest among the enriched formulations. Their oxidation

times and rates were statistically closer to each other and to the control (groups f and b), suggesting limited antioxidant efficiency, potentially due to lower concentrations or less effective dispersion.

Overall, these results confirm that the oxidative stability of mayonnaise can be significantly enhanced through enrichment with grape seed derivatives, particularly when both hydrophilic and lipophilic antioxidants are combined. This dual-phase approach not only delays the onset of oxidation but also slows its progression, offering a promising strategy to extend shelf life and improve the quality of lipid-based food products. These results confirm the effectiveness of using a combination of oil- and water-phase antioxidants as a targeted strategy to retard lipid oxidation in emulsified food systems (Ghorbani Gorji, et al. 2019).

V. Descriptive sensory evaluation

V.1. Model coefficients and attribute characterization

In this test, the coefficients of the selected model are presented for each descriptor and each product. The results are illustrated in **Figure 7.** The analysis of each graph allows for the characterization of individual products: descriptors with significantly positive coefficients are shown in blue, those with significantly negative coefficients in red, and non-significant coefficients in white.

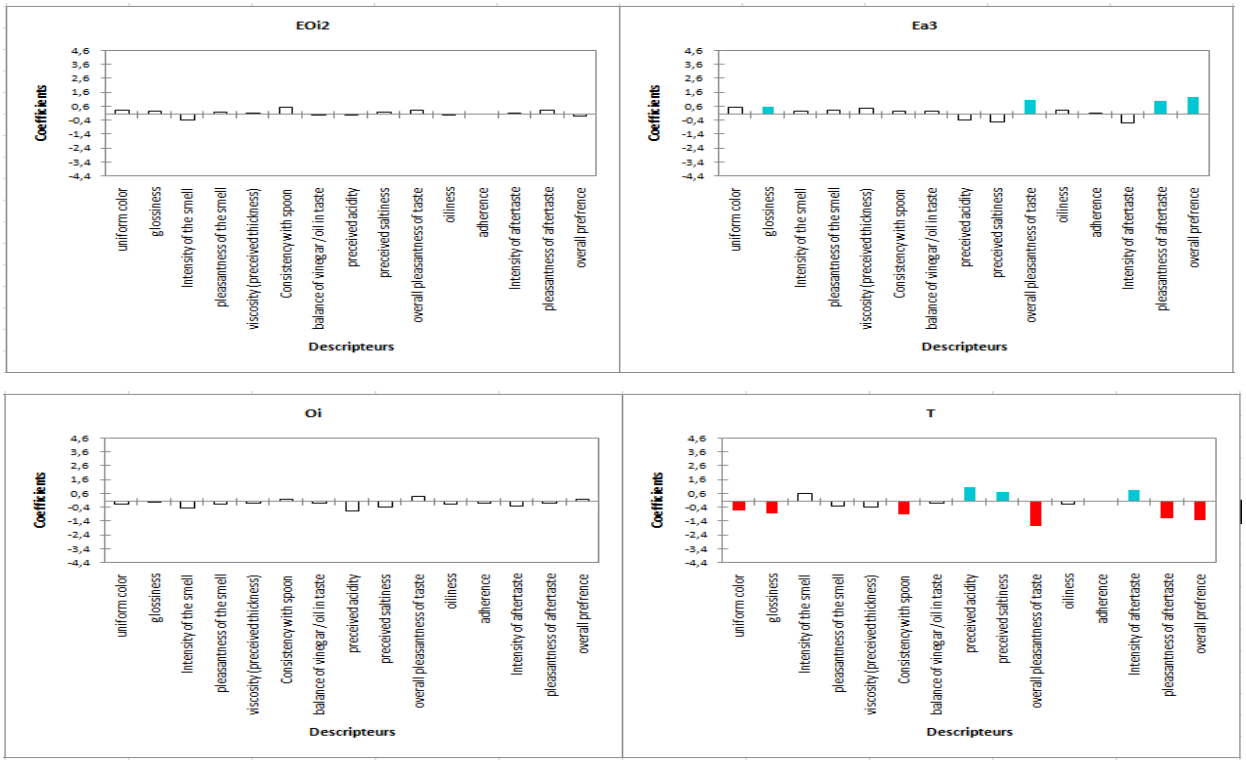

Figure 7: Regression coefficients for sensory descriptors of each sample

The model coefficients are presented in Figure 10, which also illustrates the sensory profiles of the

products.

In white, the characteristics with scores close to the judges' average ratings are shown, indicating no significant differences between the products for most attributes. However, some slight differences are observed: in blue, characteristics with significantly positive coefficients are shown, while in red, characteristics with scores below the average are displayed.

According to the results, there is no significant difference between the mayonnaise samples enriched in both phases and the one enriched in the oil phase only. Sample T is characterized by a slightly lighter color and a less glossy texture; however, its acidity, saltiness, and aftertaste are slightly more intense compared to the other samples. As for sample E3, it stands out due to its glossiness, taste, and aftertaste, which were more appreciated.

2. Discriminative power by descriptor

The current test ranks the descriptors based on their discriminative power, from highest to lowest, in differentiating between the products. The results for the discriminative power of each descriptor are illustrated in **Figure 8.**

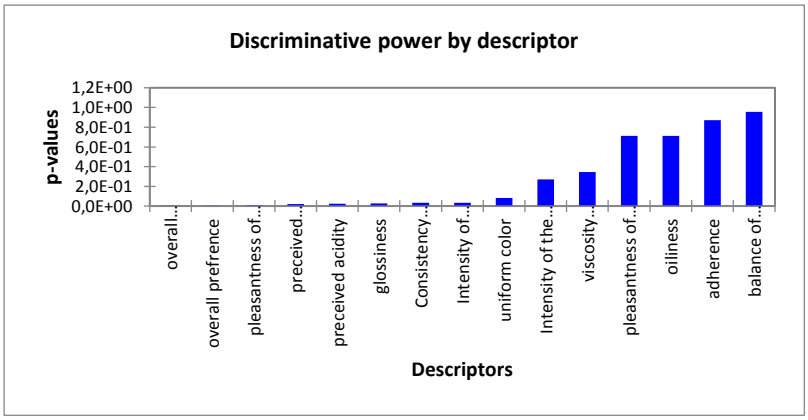

Figure 8: Results for the discriminative power of each descriptor

In Figure 11, he descriptors of the mayonnaise samples are presented in descending order of their discriminative power, from the most to the least significant. The blue bar representing 'firmness' shows a p-value significantly higher than those of the other descriptors, indicating that it lacks meaningful discriminative power.

A high *p*-value ($p > 0.05$) suggests that 'firmness' does not contribute significantly to distinguishing between the samples. In contrast, the other descriptors exhibit low *p*-values (close to zero), implying strong discriminative power and the ability to effectively differentiate the samples. Overall, these results indicate that the mayonnaise samples possess distinct sensory attributes that

differentiate them from one another except for 'firmness,' which appears to remain relatively consistent across all samples.

graph illustrates that overall preference, taste, perceived saltiness, aftertaste, acidity, brightness, and spoon consistency are the most discriminating descriptors. This indicates that these sensory attributes vary significantly between the mayonnaise samples. In contrast, other attributes, particularly the vinegar/oil balance, adhesiveness, odor preference, and creaminess, are less discriminating, suggesting minimal differences among the samples for these characteristics.

3. Preference Mapping

Preference mapping is a fundamental analytical approach in consumer research, as it enables the visual and statistical association between consumer preferences and specific product attributes. This technique is widely utilized in sensory evaluation, marketing, and product development to identify the key sensory drivers influencing consumer acceptance. Nonetheless, as noted by (Yenket et al., 2011) no single multivariate method consistently aligns with actual consumer preferences, and the reliability of the results may vary depending on the selected methodology and analytical software.

3.1. Principal Component Analysis (PCA)

Principal Component Analysis (PCA) is a fundamental statistical method used to reduce the dimensionality of complex datasets while retaining the maximum variance (Gewers et al., 2018). It transforms correlated variables into a set of uncorrelated components ranked by the variance they explain. Although PCA is simple and effective, it can be sensitive to outliers and computationally demanding in high-dimensional settings. Robust PCA methods have been developed to address these issues (Zou et al., 2006 ; Nie et al., 2011). Additionally, federated PCA enables secure, privacy-preserving analysis of distributed data using encryption techniques (Froelicher et al., 2023).

Figure 12 illustrate the PCA analysis. This PCA biplot represents the sensory descriptors of the samples projected onto the first two principal components (F1 and F2), which together explain 94.94% of the total variance a very high cumulative percentage, indicating that this 2D

representation captures almost all the variability in the data.

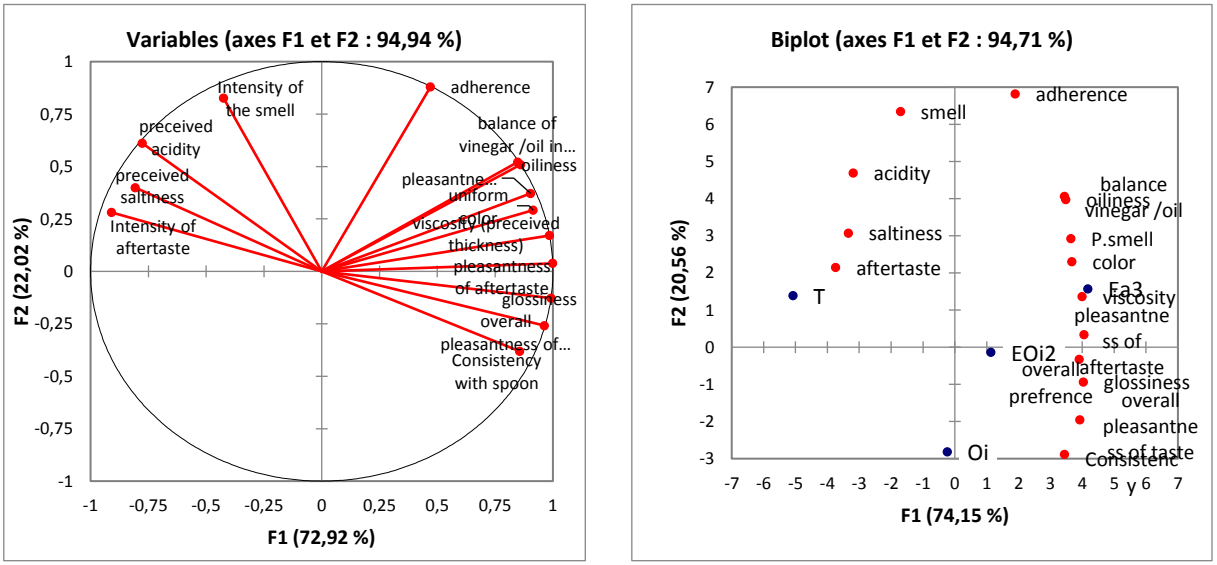

Figure 9: Correlation between variables and factors"

With F1 explaining 72.92% and F2 accounting for 22.02%. This high cumulative variance indicates that the two-dimensional representation captures the essential structure of the data.

The first component (F1) was primarily associated with positive sensory characteristics such as glossiness, overall pleasantness of taste, consistency with spoon, viscosity (perceived thickness), balance of vinegar/oil in taste, and oiliness. These variables, which are strongly and positively correlated, suggest that F1 reflects a dimension of overall sensory quality and consumer acceptability.

In contrast, the second component (F2) was mainly influenced by attributes related to flavor intensity, including perceived acidity, saltiness, intensity of aftertaste, and intensity of the smell. This axis appears to represent a dimension of sensory sharpness or pungency. Notably, attributes such as perceived acidity and saltiness were negatively associated with the pleasantness-related attributes on F1, indicating a potential trade-off between flavor intensity and overall sensory appeal. These findings provide a clear visualization of how the sensory characteristics are structured and interrelated, offering valuable insights for guiding product formulation and optimization.

It is possible that the samples enriched with both grape seed Macerat grape seed extract contribute positively to this axis, potentially due to improvements in texture, visual appearance, and flavor balance. Similarly, the sample enriched with lipophilic extract only , might also align with these attributes, possibly due to the oil's effect on viscosity and mouthfeel. On the other hand, the second component (F2) was more influenced by attributes such as perceived acidity, saltiness, smell

intensity, and aftertaste intensity.

The sample enriched with grape seed extract in the aqueous phase only, as well as the non-enriched control sample, appear to be more associated with these intensity-driven attributes, which may suggest a less favorable sensory profile from a consumer perspective. the sample enriched solely with lipophilic extract may be more closely associated with the second principal component (F2), which is driven by intensity-related attributes such as acidity, saltiness, and aftertaste. This could be due to the potentially bitter or disagreeable notes naturally present in grape seed oil, especially if used in higher concentrations c

In contrast, the sample enriched with grape seed extract in the aqueous phase may have had less impact on these intensity attributes and might instead align more with the first component (F1), which reflects pleasantness-related sensory qualities.

These observations suggest a potential trade-off between sensory intensity and overall pleasantness, where increased acidity or saltiness could reduce general acceptability. However, these interpretations should be considered indicative rather than conclusive, and further sensory or consumer testing would be needed to confirm the specific contributions of each enrichment type.

According to the previous figure, acidity, saltiness, aftertaste, and odor appear to be correlated and are characteristic of sample T. In contrast, the other sensory attributes are correlated with and define samples E3 and EOi2.

3.2. Hierarchical Cluster

The application of Hierarchical Cluster Analysis (HCA) produces several tables and graphs. One of the key outputs is the class profile plot, generated from preference data, which visually compares the average preferences across the different clusters formed. This profile plot is presented in **Figure 10**.

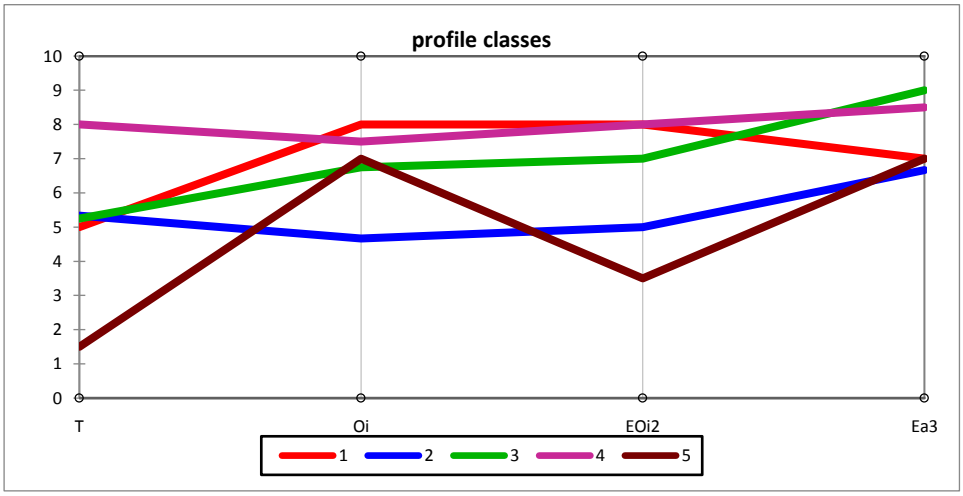

Figure 10: Profile of the different classes created

The hierarchical cluster analysis (CAH) performed on the sensory data grouped the descriptors into five distinct classes, each reflecting specific sensory trends across the four samples: T (control), Oi (oil macerate), EOi2 (enriched in oil phase with oil macerate and in the aquese phase 100ppm GSDE), and E3 (enriched with only Grape seed dry extract).

Class 1 (red) showed an increase in intensity from the control (T) to Oi and remained high in EOi2 before decreasing slightly in E3. This suggests that the descriptors grouped in this class—likely linked to pronounced sensory notes such as bitterness or acidity—were more expressed in the oil-based samples, particularly in Oi and EOi2.

Class 2 (blue) remained relatively stable across all samples, with low scores overall and a slight increase in E3. These descriptors may represent subtle or less perceivable attributes that were not strongly influenced by any specific formulation, although E3 may have slightly enhanced their perception.

Class 3 (green) displayed a progressive increase from T to E3, indicating that the descriptors within this class were positively impacted by enrichment, especially in the E3 sample. These may include favorable attributes such as pleasentense of smell, Acidity pleasentness, or balanced flavors.

Class 4 (pink) maintained consistently high intensity values across all samples, with a slight peak in E3. This class likely includes stable, dominant descriptors present regardless of formulation, which were further reinforced in the enriched sample.

Class 5 (brown) started at a very low intensity in the control (T), peaked strongly in Oi, decreased notably in EOi2, and then rose again in E3. This pattern suggests that descriptors in this class were strongly influenced by oil maceration but were somewhat suppressed by the enrichment process in EOi2. However, E3 appears to restore or enhance these attributes.

Overall, this CAH profile highlights that the E3 sample exhibited the most balanced and elevated sensory profile, particularly in Classes 3 and 4, which may correspond to desirable sensory characteristics. Conversely, the control sample (T) showed lower intensity in several descriptor groups, while oil-based formulations (Oi and EOi2) influenced specific traits differently depending on the class of descriptors.

4. Cartographie des préférences (PREFMAP)

Following Principal Component Analysis (PCA) and Hierarchical Cluster Analysis (HCA), a preference map (PREFMAP) was generated using XLSTAT software, as shown in Figure 14. In this study, the preference map was developed based exclusively on the expert panel's evaluations, as it was not possible to conduct a hedonic analysis with untrained consumers due to the limited quantity of available product.

The purpose of the preference map was to determine the panelists' overall preferences for the various samples. Based on the map, products E3 and EOI2 were the most appreciated, each receiving 100% of the preferences, which explains their positioning in the red zone of the map with their appreciation driven by attributes such as viscosity, color, odor, and aroma . They were followed by the OI sample, with a preference rate of 40%. Conversely, sample T was the least appreciated, with a preference score of 0%.

These results suggest that the enriched samples were generally more appreciated, especially those enriched in the aqueous phase or in both phases. This finding represents a significant outcome, as it supports one of the main objectives of this study: demonstrating that enrichment not only

enhances the antioxidant properties of mayonnaise but also improves its sensory appeal compared to the standard formulation.

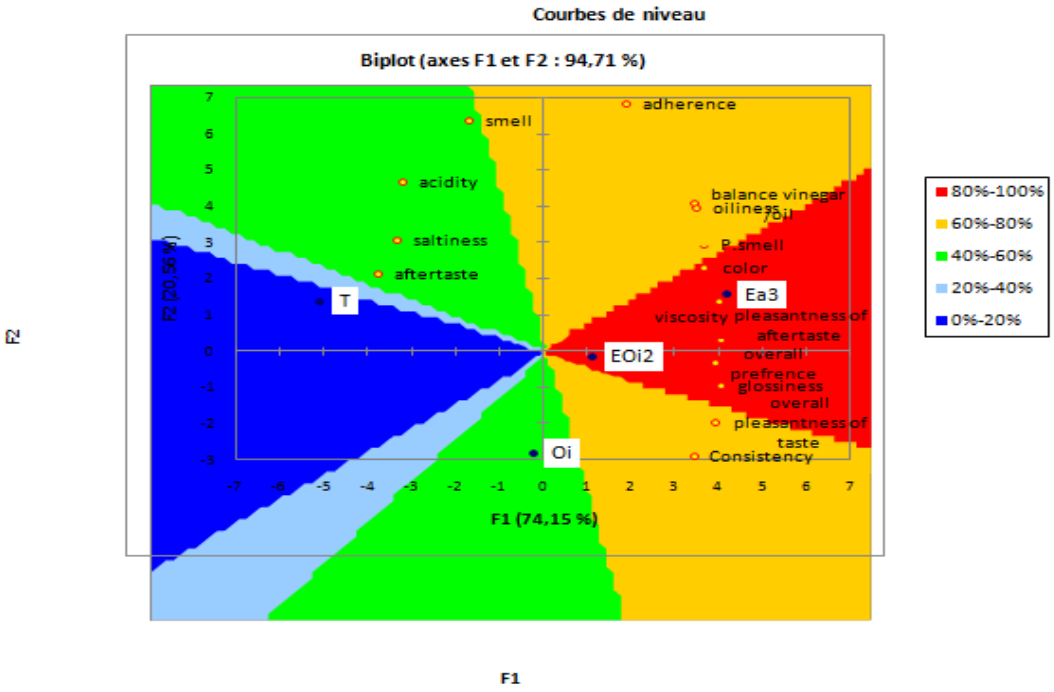

Figure 11: Preference mapping (PREFMAP) of mayonnaise samples

5. Product evaluation based on judges' average scores

In this study, four products (T, Oi, EOi2, E3) were evaluated by 13 judges, who assigned scores to each product based on their sensory perceptions. To present the results of this analysis in a clear and concise manner, the average scores for each product were calculated. These averages provide an overall view of how each product was perceived by the judges **(Figure12).**

The sensory profile radar chart provides a comparative overview of the four mayonnaise formulations evaluated by panelists across multiple sensory attributes. The sample **E3** (enriched with grape seed extract in the aqueous phase) consistently achieved the highest scores in several key sensory attributes, including overall preference, pleasantness of aftertaste, overall pleasantness of taste, uniform color, and glossiness. This suggests that aqueous-phase enrichment contributed positively to both the visual and organoleptic qualities of the product, likely improving consumer acceptance.

The **E3** sample, enriched with grape seed extract in the aqueous phase, was the most appreciated formulation in the sensory analysis. It consistently received the highest scores among the four samples, particularly in overall preference (score of 8), pleasantness of aftertaste (8), overall pleasantness of taste (7), uniform color (8), and glossiness (8). These results suggest that aqueous-

phase enrichment significantly enhanced both the visual and organoleptic properties of the mayonnaise, contributing to strong consumer acceptability.

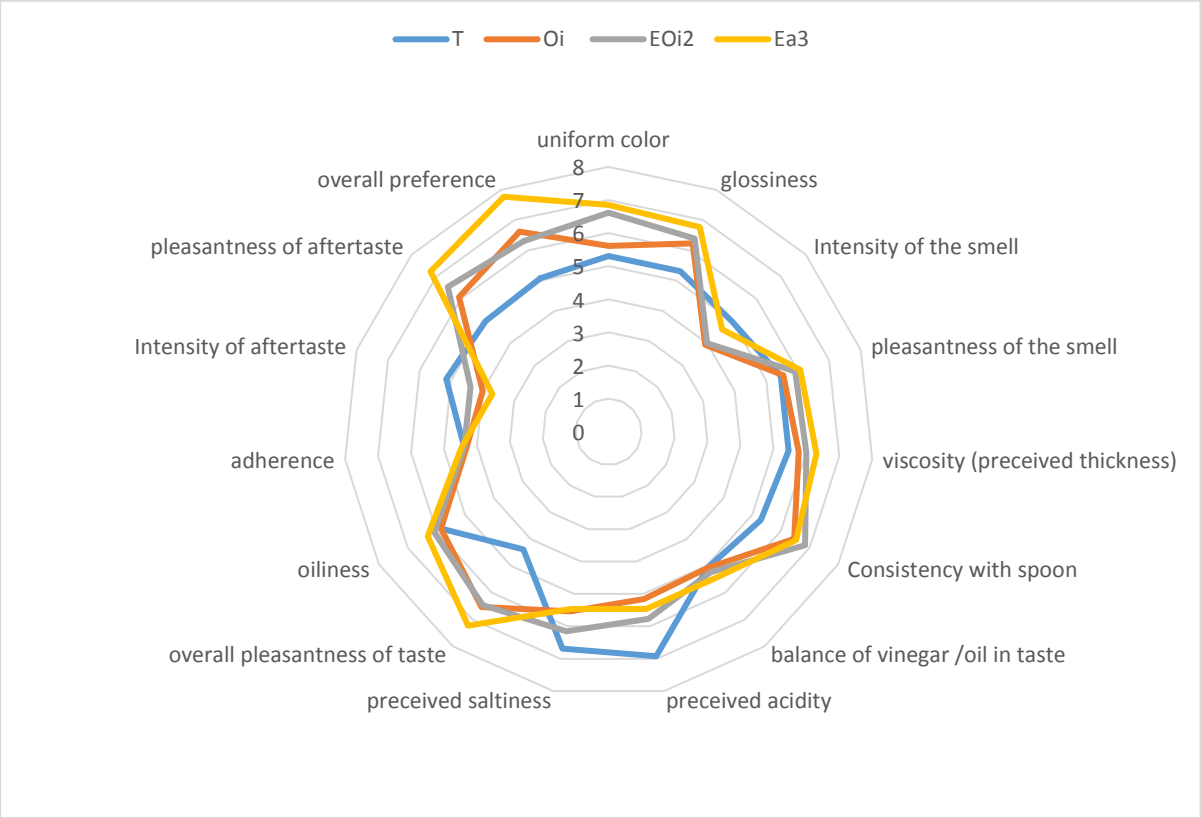

Figure 12: Radar chart showing sensory evaluation scores of control and enriched mayonnaise samples.

The **EOi2** sample, enriched in both oil and aqueous phases, closely followed E3 in several sensory attributes. It achieved high ratings in viscosity (perceived thickness) (7), consistency with spoon (7), pleasantness of smell (7), and overall preference (7). This indicates that dual-phase enrichment may produce a balanced and well-rounded product by improving both texture and flavor-related characteristics, likely benefiting from the combined effects of oil-based and water-soluble bioactives.

The **Oi** sample, enriched with grape seed macerat oil only, showed a more moderate profile. It reached a score of 6 in viscosity, glossiness, and overall preference, but had lower values in taste-related attributes such as overall pleasantness of taste (5) and pleasantness of aftertaste (5). This may be due to the presence of certain polyphenolic compounds in grape seed oil, which are sometimes associated with a slightly bitter or astringent taste, especially at higher concentrations.

The **T** sample, which served as the control (no enrichment), received the lowest scores in several

categories. It scored 5 in overall preference, 4 in oiliness, and 5 in pleasantness of aftertaste, indicating a relatively neutral or bland profile compared to enriched variants. Its slightly higher score in perceived saltiness (6) may reflect the absence of added phenolic compounds that could otherwise modulate flavor perception.

These findings align with earlier research on the incorporation of grape seed by-products in food systems. For example, when added to muffins and waffles, grape seed flour enhanced antioxidant profiles but affected sensory attributes such as texture and sweetness (Antonic et al., 2021; (Yalcin et al., 2022). Despite increased hardness in some baked goods, overall sensory scores remained within acceptable ranges. Similarly, the addition of grape seed extract in meat products like chicken nuggets improved oxidative stability and shelf life without compromising sensory quality (Kaur et al., 2015) These studies underscore the importance of optimizing concentration and application form to enhance functional properties without negatively impacting consumer perception.

The radar chart results confirm that aqueous-phase enrichment with grape seed extract (E3) leads to the most favorable sensory outcomes, while dual-phase enrichment (EOi2**)** provides a good balance of texture and flavor. Sample (Oi) with enrichment is oil phase only showed some limitations, and the non-enriched control (T) was the least appreciated. These results demonstrate that careful formulation using grape seed derivatives can yield products that are both functionally beneficial and sensorially acceptable and expected by consumers.

Conclusion

This study evaluated the impact of natural antioxidant enrichment, particularly grape seed extract and macerated grape seed oil, on the oxidative stability, physicochemical properties, and sensory characteristics of mayonnaise. By enriching formulations in both the aqueous and oil phases and subjecting them to accelerated storage conditions, the study aimed to identify the most effective strategies for inhibiting lipid oxidation in emulsified food systems.

A mixture design approach identified formulation G (75% oil, 12% egg yolk, 13% aqueous phase) as the most balanced recipe. This formulation achieved the best sensory scores in terms of consistency, flavor, and overall preference, confirming the value of the design model in optimizing mayonnaise composition.

The antioxidant properties of grape seed derivatives were first confirmed by in vitro assays. The extract exhibited high total phenolic content (1479.50 mgGAE/g DW) and proanthocyanidins (20955.62 mg/kg), while the macerated oil was rich in tocopherols (17.50 mg/g). Both showed strong radical scavenging activity (DPPH and FRAP), supporting their potential for oxidative stabilization.

Oxidative stability monitoring revealed that all enriched formulations outperformed the control in terms of peroxide value evolution. Dual-phase enrichments (especially EOi2 and EOi3) showed the best results, with significantly longer lag times, highlighting the synergistic effect of combining hydrophilic and lipophilic antioxidants.

Physicochemical analysis showed that antioxidant enrichment influenced parameters such as viscosity, pH, NaCl content, and consistency, though all values remained within acceptable ranges. Samples enriched in both phases displayed distinct rheological behavior, suggesting better antioxidant distribution and improved emulsion structure.

Sensory analysis further confirmed the success of the dual-phase strategy. EOi2, in particular, combined excellent oxidative resistance with high sensory acceptability. Similarly, sample E3 (aqueous enrichment at a high concentration) also showed good oxidative stability and was well appreciated by the panel. These results indicate that natural antioxidant strategies can enhance product quality without compromising consumer acceptance.

Overall, the findings validate the use of grape seed derivatives as efficient natural antioxidants in mayonnaise. The dual-phase enrichment approach appears especially effective in extending shelf

life, improving oxidative stability, and aligning with clean-label and sustainability trends in food innovation. However, the study also presents some limitations. Accelerated storage conditions, while useful for comparative purposes, may not fully replicate real-time oxidation behavior. Further research is needed to address these aspects and strengthen industrial relevance.

Building on these results, several future research directions are recommended:

- ❖ Real-time storage studies are needed to validate the oxidative stability of enriched formulations under typical market conditions.
- ❖ Since the macerated oil used in this study is relatively diluted, further investigations should assess the impact of pure grape seed oil to better understand its full antioxidant potential. Additional exploration of natural antioxidant sources may reveal synergistic or more effective combinations.
- ❖ Extended sensory analysis during storage, as well as application of this enrichment strategy to other emulsified products, will help generalize and expand its relevance across the food industry

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Appendixes

Appendixes

Appendix 1

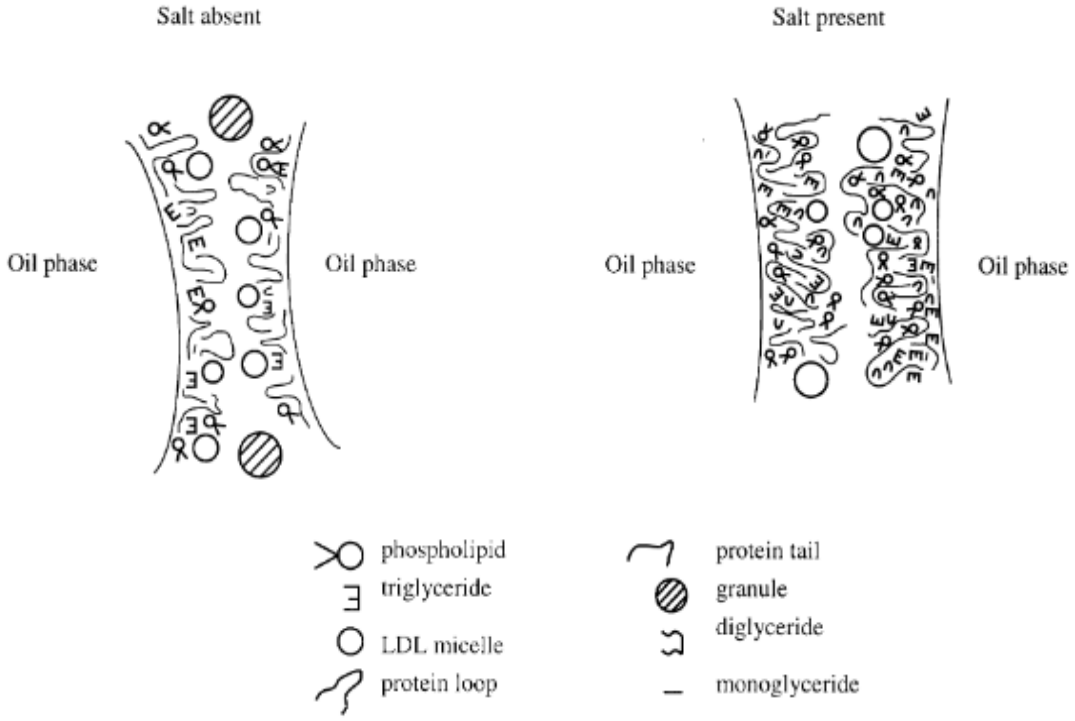


Fig. 1. Diagram of egg yolk components absorbed on adjacent oil drops (redrawn from Koisseoglou & Sherman, 1983).

Appendixes

Appendix 2

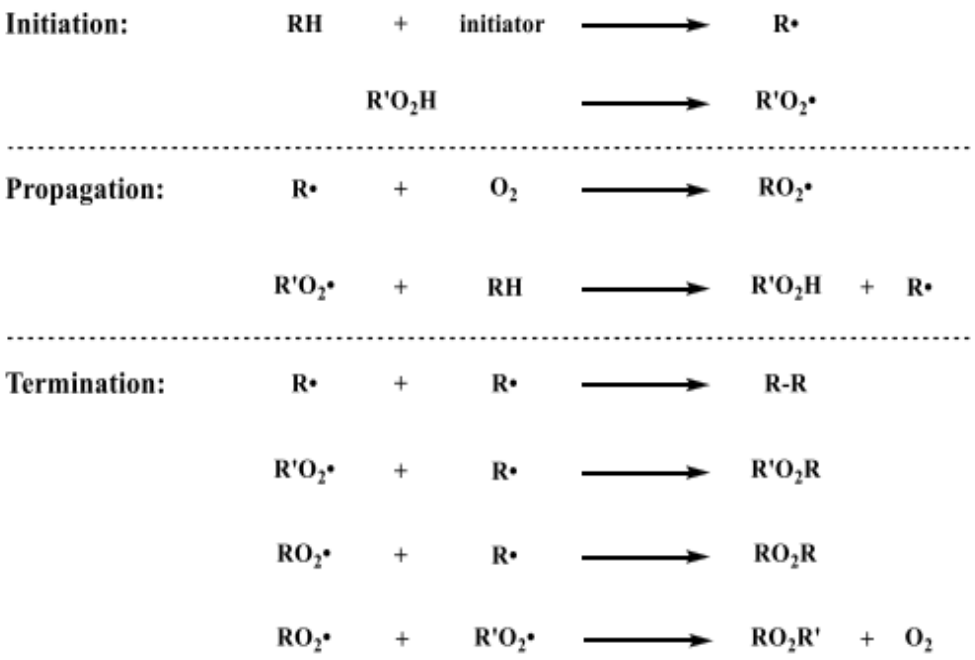
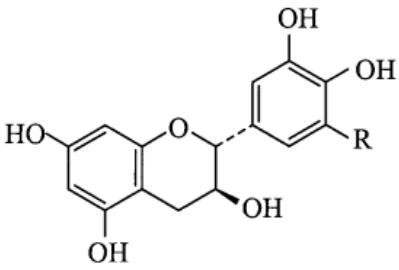


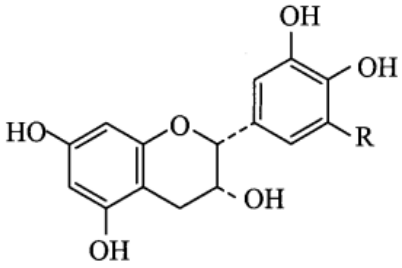
Figure 2: The autoxidation pathway of lipids

Appendixes

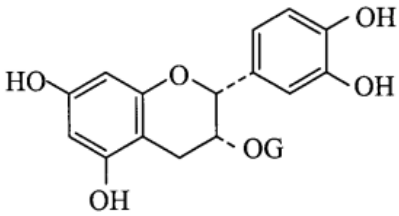
Appendix 3



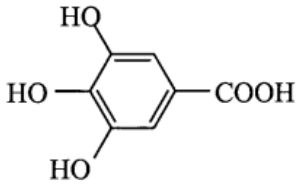
R = H: (+) -Catechin
R = OH: (+) -Gallocatechin



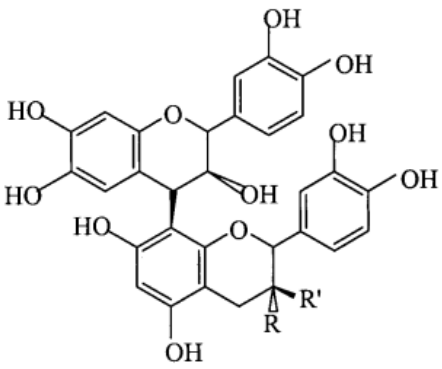
R = H: (-) -Epicatechin
R = OH: (-) -Epigallocatechin



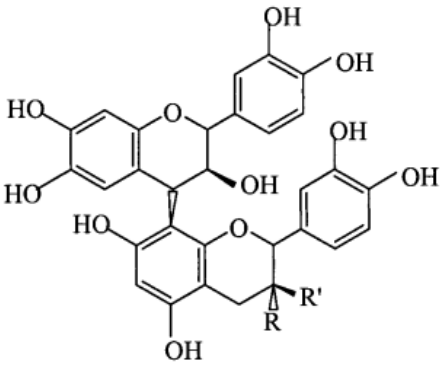
Epicatechin-3-gallate
G = Gallic acid



Gallic acid



Procyanidin B1: R' = OH, R = H
Procyanidin B2: R' = H, R = OH



Procyanidin B3: R' = OH, R = H
Procyanidin B4: R' = H, R = OH

Figure 3: most abundant antioxidants found in grape seeds

Appendixes

Appendix 4

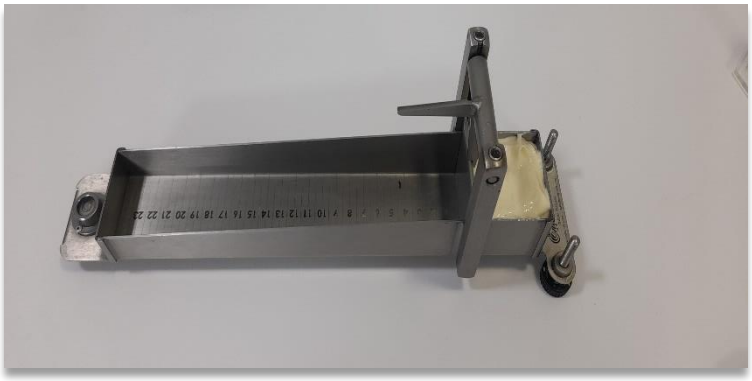
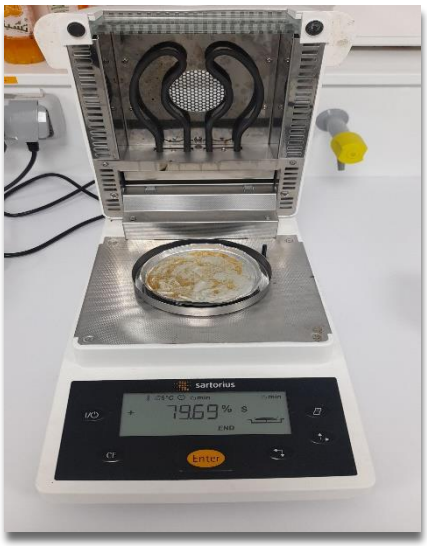


Figure 4: consistency test for mayonnaise samples using bostwick scale

appendix 5

Figure 5 : Infrared moisture analyzer used for measuring the dry matter content in mayonnaise.



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Appendix 6

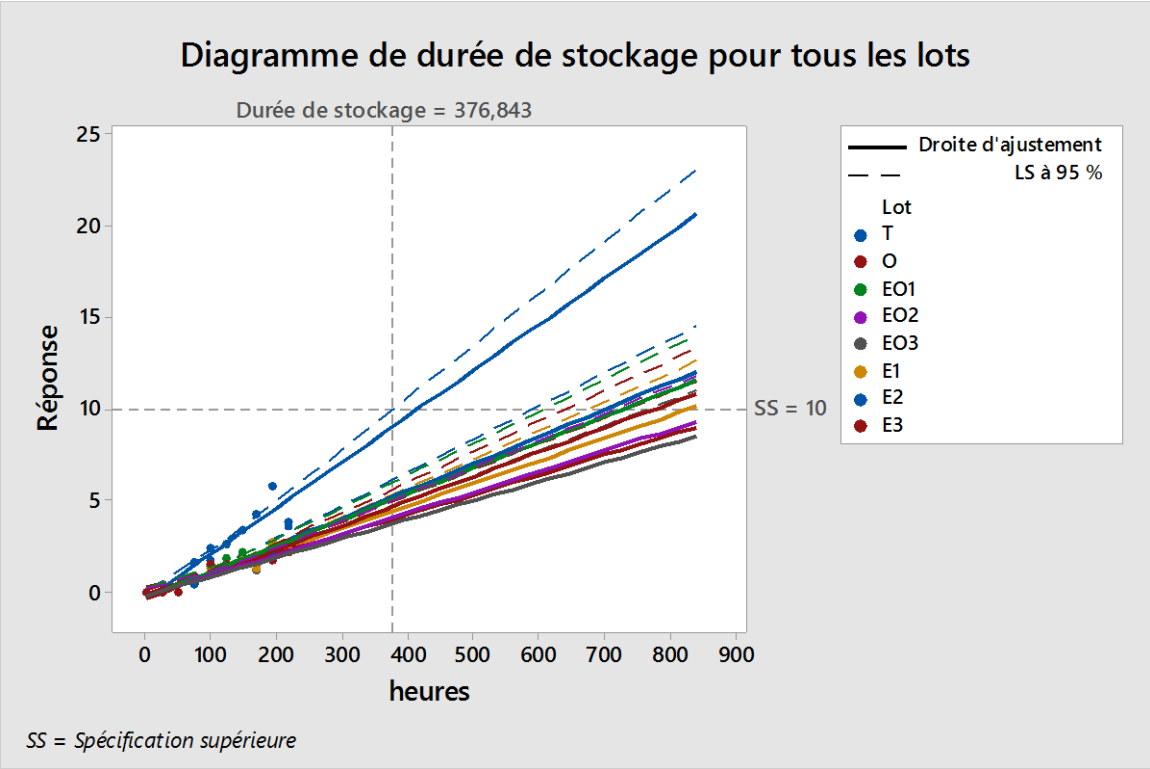

Figure 06: Minitab-Based Statistical Treatment of Peroxide Index

Appendix 7 Sensory Evaluation Questionnaire – Mayonnaise Expert Panel

Informations générales du participant

• Nom : _____

• Sexe : ☐ Homme ☐ Femme

• Date : ___ / ___ / _____:

Dans le cadre d'une **analyse sensorielle visant à évaluer des variantes de mayonnaise**, vous allez déguster **quatre échantillons codés de manière aléatoire** : **147**, **203** **319** , **650.** Cette évaluation a pour objectif de recueillir vos perceptions sensorielles (aspect, odeur, goût, texture, arrière-goût…etc.) de manière objective et indépendante.

- Merci de **rincer votre bouche entre chaque échantillon** et de remplir soigneusement le questionnaire en attribuant une note à chaque critère selon l'échelle fournie.

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Pour chaque critère, attribuez une note de **1 à 9** selon l'échelle suivante :

Note	Signification
1	Très faible / Pas du tout
2	Faible
3	Légèrement faible
4	Moyennement faible
5	Modéré / Moyen
6	Moyennement fort
7	Assez fort
8	Fort
9	Très fort / Extrêmement

1. Aspect visuel

• Couleur uniforme :

147	203	319	650

• Brillance :

147	203	319	650

2. Odeur

• Intensité :

147	203	319	650

• Agréabilité :

147	203	319	650

3.Viscosité et consistance

• Viscosité (épaisseur perçue) :

147	203	319	650

• Consistance à la cuillère:

147	203	319	650

4.Goût

• Équilibre vinaigre/huile :

147	203	319	650

• Acidité perçue :

147	203	319	650

• Salinité perçue :

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147	203	319	650

• Agréabilité globale du goût :

147	203	319	650

5. Texture en bouche

• Onctuosité :

147	203	319	650

• Adhérence (collante ou non) :

147	203	319	650

6. Arrière-goût

• Intensité :

147	203	319	650

• Agréabilité :

147	203	319	650

7. Appréciation globale

147	203	319	650

➢ Commentaires généraux :

• Avez-vous remarqué des différences particulières entre les échantillons ?

……
……..……

• Quel est votre échantillon préféré ? Pourquoi ?

……
……..……
……..……
……..

• Suggestions ou remarques :

……
……..……
……..……
……..……
……..

Merci pour votre participation

Résumé

La mayonnaise, en raison de sa forte teneur en lipides, est particulièrement sujette à l'oxydation. Cette étude a évalué l'efficacité antioxydante d'extraits de pépins de raisin sous formes hydrophile (extrait hydroalcoolique) et lipophile (huile macérée), en tant qu'alternatives naturelles aux antioxydants synthétiques comme l'EDTA. L'extrait hydrophile a montré un rendement en matière sèche de 34,53 %, une teneur élevée en composés phénoliques totaux (1479,47 mg EAG/100 g MS) et une activité antioxydante supérieure (DPPH : 8157,46 mg AAE/100 g ; FRAP : 3045,06 mg AAE/100 g) comparé à l'extrait lipophile. Les proanthocyanidines ont été identifiées dans l'extrait, tandis que de faibles quantités de tocophérols ont été relevées dans l'huile. L'enrichissement des échantillons de mayonnaise n'a pas altéré leurs propriétés physico-chimiques (pH, viscosité, matière sèche, sel), et a permis de stabiliser l'acidité dans le temps. Les valeurs de peroxyde ont montré une réduction marquée de l'oxydation lipidique, surtout dans les formulations double phase.

Mots-clés : Mayonnaise, antioxydant naturel, pépins de raisin, oxydation lipidique, polyphénols.

Abstract

Mayonnaise is highly prone to lipid oxidation due to its high fat content. This study evaluated the antioxidant efficiency of hydrophilic and lipophilic grape seed extracts as natural alternatives to synthetic antioxidants (e.g., EDTA). The hydroalcoholic extract showed a 34.53% dry matter yield, high phenolic content (1479.47 mg GAE/100 g DW), and superior antioxidant activity (DPPH: 8157.46 mg AAE/100 g; FRAP: 3045.06 mg AAE/100 g) compared to oil macerates. Proanthocyanidins were detected in the hydrophilic extract, while only trace tocopherols were found in the oil. Mayonnaise samples enriched with these extracts maintained stable physicochemical properties (pH, viscosity, dry matter, salt) and stable acidity over time. Peroxide values showed reduced lipid oxidation, especially in dual-phase.

Keywords: Mayonnaise, natural antioxidant, grape seed extract, lipid oxidation, phenolic compounds.