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INTEGRATED QUALITY-RISK MANAGEMENT IN MANUFACTURING SYSTEMS IN THE FERMENTATIVE FOOD INDUSTRY

SUMMARY

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CONTENT

Introduction

Fermented functional foods have emerged as a subsector of the functional food industry, which has been steadily growing in recent years.

Fermentation has many potential health benefits such as: the supply of beneficial lactic acid bacteria, helps metabolism, increased nutritional availability, improved mood and behavior, but also benefits for cardiovascular health.

Dairies and meat are the most popular fermented products widely consumed. On the other hand, alongside dairy and meat, fermented fruits and vegetables are well known for their worldwide consumption and health benefits.

The awareness of benefits of the alternative proteins has recently expanded the consumption of fermented products that have as basic ingredients insects and seaweed. Furthermore, to maximize the bioconversion efficiency of food waste, it is necessary to optimize food waste and the use of cutting-edge biotechnological methods.

Innovative techniques, such as precision fermentation, are suitable for producing desired proteins, lipids, and carbohydrates, as they allow generation of molecules with similar compositions with their homologues conventionally grown.

In the future fermentation industries will continuously operate, automation can be installed and this will lead to a reduction of human involvement and the prevention of errors. In future fermentation processes, instead of steel, it can be used low-cost materials such as plastic, cement or ceramics.

Hereafter, artificial Intelligence will be responsible for controlling fermentation, thus providing more efficient and sterile processes with reduced energy, water, and labor requirements.

Another direction of future research could be the use of innovative technologies, such as high hydrostatic pressure, ultrasound, microwaves, pulsed electric fields, etc., and their effects in the production of fruit vinegar of high-quality standards. Some of these technologies have been tested for the production of grape vinegar, and the results have been promising.

The aim of this research was to test ultrasound technology on different varieties of fruits, possibly grown in different regions, and to observe the influence of ultrasound on the extraction process of various compounds during the vinegar-making process.

In CHAPTER 1: **An overview of fermentation in food industry - looking back from a new perspective**, there were studied reports and scientific articles related to fermentation and its benefits.

Scientific research have demonstrated numerous health benefits associated with the consumption of fermented foods: lowering cholesterol levels; strengthening the immune system; protection against infections, cancer, osteoporosis, obesity, diabetes, allergies, and atherosclerosis; as well as reducing lactose sensitivity.

Non-communicable diseases, such as cardiovascular diseases, type 2 diabetes, cancer, and allergies, have become more prevalent. The consumption of fermented foods can reduce the risk of these diseases due to the beneficial properties of bioactive peptides produced during fermentation (Şanlier et al., 2019).

Fermentation requires less ground, generates fewer greenhouse gas emissions, and consumes less water than conventional agriculture. Additionally, fermentation technology exploits food waste to produce valuable by-products such as enzymes, pigments, and biofuels (Knorr and Augustin, 2022).

This chapter also provides an overview of the cultural significance and ecological importance of fermentation, of technological progresses in fermented foods and beverages, health benefits, nutritional value, and also microbiological information on various fermented foods.

There is also a global discussion about how the industrial revolution has affected the fermented food process and how fermentation is used to exploit food waste. The growth of global population is often correlated with an increased demand for food. Industrialization generates large amounts of food waste, and there is a noticeable lack of adequate waste management strategies.

Estimates suggest that by 2030, 2.1 billion tons of food waste will be generated annually. The agricultural and food processing industries face two major challenges: it is essential to limit their environmental impact to minimize the effects of climate change and to address the insufficiency of superior alternatives to health-promoting diets (Rastogi et al., 2022).

Food shortages and environmental consequences result from the loss and waste of agricultural production during processing and distribution. A large proportion of nutrients is found in plant-based food processing waste, including pulp, peels, and silage, but these materials are usually discarded in landfills or washed away, causing rapid depletion of dissolved oxygen.

Fermentation in food industry is an environmentally friendly alternative for enhancing the nutritional value of products. More and more products can be obtained through fermentation in a safe, environmentally friendly and sustainable process.

However, for this technology to become more competitive in the future, some issues need to be resolved. Some of these are freshwater scarcity, high energy consumption, microbial contamination, complexity of sterile operations, poor oxygen use in crops, food-related ingredients as substrate. For these reasons, future fermentation processes should be more efficient.

In CHAPTER 2: **Fermentative degradations. Types of vinegar**, a classification and description of fermentative degradations is provided, along with an overview of the main types of vinegar and their benefits. There are analyzed fermentations types, and also is presented the chemical composition of vinegar.

Also in this chapter, there are presented the types of fermentations encountered in food industry: lactic fermentation used in yogurts and cheese production, and also in other fermented dairy products; alcoholic fermentation used for the production of alcoholic beverages such as beer and wine; propionic fermentation used for the production of propionic acid, which is applied in food preservation; acetic fermentation used in the production of vinegar; malolactic fermentation used in winemaking in order to improve the taste of wine by reducing its acidity. There also described the most representative microorganisms involved in fermentation processes of food industry.

In CHAPTER 3: **Current state of vinegar production methods** presents a study of new techniques of vinegar production, maturation, post-maturation treatments, etc.,.

The essential principle of vinegar production is to maximize the contact surface between the raw material, acetic bacteria, and air in order to ensure a complete oxidation of the ethyl alcohol contained by the raw material. The various industrial techniques used in vinegar manufacture are distinguished by using different types of equipment.

Chapter 4: **The aim and objectives of the PhD thesis** presents the objectives of the thesis.

Chapter 5: **Bibliometric analysis in the field of vinegar production** analyzes the productivity of scientific research in the field of vinegar technology published in literature, as well as the most significant trends in the field.

The results obtained present the current state of literature, highlighting the most eloquent journals in the field, authors and trending keywords.

By examining the most recent scientific articles, there have been identified the emerging trends in vinegar research. It is believed that detailed analyses of omics data will create an emerging focus on microorganisms, while also serving as a means of determining the spatiotemporal description of microbial ecology in vinegar fermentation. This type of research will also include the identification of basic aromatic chemome of vinegar through the use of a uniform chemometric model in order to integrate data from multiple sources.

Although they are numerous scientific articles related to vinegar production, there are still required research to determine the ideal conditions for producing fruit vinegar and optimized biotechnological processes from which result a higher profitability of the product. Since the commercial initiators do not use acetic fermentation, this can lead to issues in vinegar production and to economic losses.

Another future research direction could be the use of innovative technologies such as high hydrostatic pressure, ultrasound, microwaves, pulsed electric fields, etc., and their effects on the production of high-quality fruit vinegar. Some of these technologies have been tested for grape vinegar production, and the results have been promising.

In Chapter 6: **Research on the use of ultrasound in vinegar production technology**, there are the new technologies applied to the vinegar production process. Numerous studies in the field confirm the current interest in a variety of emerging technologies capable of generating cost-effective and high-quality products. The studies conducted have demonstrated the advantages of these emerging technologies in the vinegar production process. Implementing these emerging technologies could offer consumers superior products with added value in terms of nutritional and sensory characteristics, and also ensuring a greater profitability for industry by reducing the process duration and the efficient use of natural resources, energy, and raw materials.

Chapter 7: Entitled: **Analysis of the current state of quality-risk management in vinegar manufacturing technology** is analyzed the quality management in vinegar industry.

In this chapter, quality management standards are analyzed so that companies which operate in the field of vinegar production to constantly recognize the challenges for effectiveness and efficiency in external and internal circumstances.

Current approaches to Management Systems for the food industry are also analyzed in this chapter. The Hazard Analysis and Critical Control Points (HACCP) system, based on ISO 9001, not only ensures the safety product, but also enables a better and more efficient implementation of the entire quality system. Food producers are legally required to apply HACCP, while other systems are applied voluntarily in food industry.

Chapter 8: **Experimental research on applying the ultrasound treatment in the laboratory process of vinegar production** there have been carried out research with the aim of:

- Intensifying the extraction of phenolic compounds during vinegar production using ultrasound treatment as an emerging technology.
- Developing and scaling up the ultrasonic process from the laboratory level to industrial scale.
- Assessing the effect of ultrasound parameters (amplitude, temperature, duration) on the total polyphenol content and antioxidant properties of fruit vinegars.
- Evaluating the impact of ultrasound treatments on microorganisms during vinegar production.
- Optimizing the antioxidant properties of blueberry, raspberry, and blackberry vinegars obtained from substrates treated with ultrasound.

The data obtained demonstrated that ultrasonically treated and optimized blueberry vinegar was of a higher quality as compared to the content of polyphenols and anthocyanins, while also having better antioxidant properties. For optimal values of these parameters, the best results for amplitude and ultrasound duration were found to be 78.50% and respectively of 3.95 minutes.

Chapter 9: **Implementation of food safety management system with ultrasound treatment as a processing stage in vinegar production** - contains all the steps taken for the implementation of HACCP plan in food safety management system for vinegar production, using ultrasonic treatment technology.

Chapter 10: **General conclusions and summary of contributions** presents a synthesis of the research undertaken and highlights the original contributions in the field of vinegar production technology and management, particularly focusing on the use of ultrasound treatment as a newly introduced operation, aimed to increase the extraction of biologically active compounds. In this context, a series of unresolved research problems have been identified, which could easily become future research directions.

PART I - CURRENT STATUS OF INTEGRATED QUALITY-RISK MANAGEMENT IN FERMENTED FOOD INDUSTRY SYSTEMS

CHAPTER 1: A comprehensive overview of fermentation in food industry - Looking back from a new perspective

1.1 Introduction

Benefits of fermentation: increased storage stability, fermented products are more resistant to degradation, having a longer life; reduced risk of food poisoning, fermentation reduces the amount of toxins and pathogens in food; improve digestion, fermentation decomposes food compounds into more digestible forms; high in probiotics, and the beneficial bacteria in fermented foods support digestion and nutrient absorption.

Scientific research has demonstrated numerous health benefits associated with the consumption of fermented foods, including: lowering cholesterol levels; strengthening the immune system; providing protection against infections, cancer, osteoporosis, obesity, diabetes, allergies, and atherosclerosis; and reducing lactose sensitivity (Tamang & Thapa, 2022; Şanlier et al., 2019).

In conclusion, fermentation is both a traditional and modern method for preserving and improving food, offering extensive health and environmental benefits. Advanced fermentation technologies provide sustainable solutions for food production and the optimal utilization of available resources.

1.2 Technological progresses in fermentation

The fermentation process includes the biochemical activity of organisms throughout their life cycle, from growth to death. Industrial-scale production of food, pharmaceuticals, and alcoholic beverages use fermentation technology supplied by these organisms. Industrial fermentation technology is based on the fundamental principle of cultivating organisms under optimal conditions.

Ultrasound systems are easy to be applied on an industrial scale as they do not require immersing the product in a liquid medium. This allows the hydrophilic nutrient compounds to be maintained, enabling the use of these systems on a large scale. In addition, the application of ultrasound has result the improvement of the physiological, phytochemical, biochemical and organoleptic characteristics of alcoholic beverages Celotti et al. (2020) investigated the impact of high-power ultrasound on anthocyanin and phenol levels in young red wines. After 15 and 30 days of storage, the tannin content of the wine treated with high-amplitude ultrasound (81%) decreased by 15%,and respectively by 40%.

1.3 Fermentation in food industry as an ecological alternative to improve nutritional value

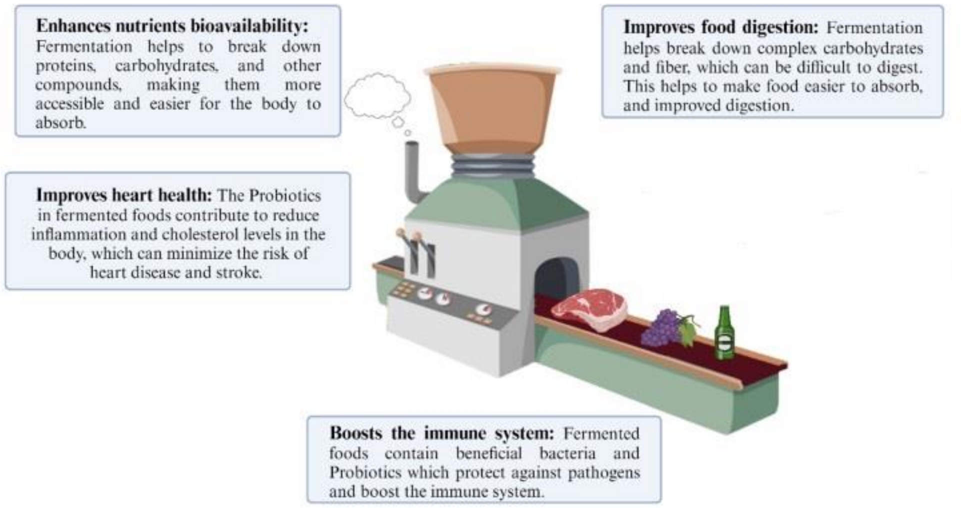


Figure 1.1 Fermentation's primary benefts (Shaida et al., 2023)

The global population growth is often correlated with an increased demand for food. Industrialization results in significant amounts of food waste and highlights the absence of effective waste management strategies (Ng et al., 2020).

Food fermentation technology offers a range of benefits, including environmental and health advantages (Paramithiotis et al., 2022; Rastogi et al., 2022).

In addition, fermentation can produce nutritious foods and sustainable food supplies. Compared to traditional chemical synthesis methods, fermentation is more flexible, cost-effective, and eco-friendly (Figure 1.1).

1.4 Fermented foods and non-communicable diseases

Dietary habits are significant in the hierarchy of essential factors that trigger non-communicable diseases (NCDs). High levels of saturated faty acids, sodium, a sedentary lifestyle, and a diet low in fruits and vegetables are some of the risk factors for developing NCDs (Angeles-Agdeppa et al., 2020).

The health benefits associated with fermented products are primarily attributed to metabolic activities of the fermenting microbial community or to their biologically active metabolites. For example, it has been found that various lactic acid bacteria (LAB) produce exopolysaccharides (EPS) during fermentation. EPS products have been linked to various health benefits, including antidiabetic, cholesterol-lowering, antioxidant, and immunomodulatory effects (Nampoothiri et al., 2017; Patel and Prajapat, 2013).

1.8 Food waste recovery through fermentation

Food waste (FW) includes complex carbohydrates, proteins, lipids, organic acids, enzymes, and nutraceuticals (Carmona-Cabello et al., 2018; O'Connor et al., 2021; Ravindran and Jaiswal, 2016).

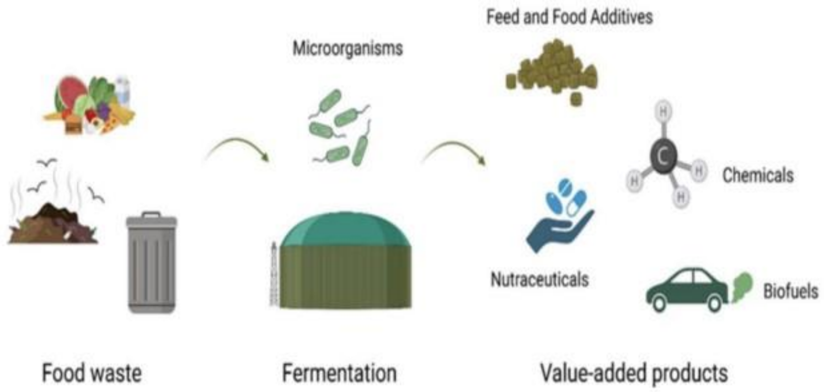


Figure 1.2 Recovery of Food Waste Through Fermentation (Created by BioRender.com)

Although the definition of waste has been widely debated, the Food and Agriculture Organization (FAO) defines it as "the total loss of food quality and quantity occurring during the supply chain process, which happens at various stages such as production, post-harvest, and processing." FW is generally considered non-hazardous, except for animal-origin waste strictly regulated by European Regulation (EC) No. 1069/2009. FW is becoming an increasing and crucial issue both locally and globally.

Aiming to valorize food waste, several technologies employ acidogenic fermentation (Figure 1.2) with anaerobic microbial communities, thereby generating various value-added products from food waste (Ortiz-Sanchez et al., 2023).

Chapter 2: Fermentative degradations. Types of vinegar

2.1 Introduction

The industrial exploitation of microorganisms largely constitutes the fermentation industry. The term "fermentation" is derived from the Latin verb "fervere," which means "to boil." Initially, the term was closely associated with alcoholic fermentation and to its early phase, where the release of carbon dioxide creates foam on the liquid surface, giving the impression of boiling. Over time, the meaning of the term expanded to include all oxidation processes by which organic substances, carbon sources, and energy sources are transformed into various fermentation products.

Fermentation plays a crucial role in the food, biochemical, and pharmaceutical industries due to its ability to transform organic substrates into high-value economic and nutritional products. The progresses in biotechnology and the use of starter cultures have enabled the production of safer, more uniform, and higher-quality products. Therefore, fermentation remains an essential technology for food production and industrial sustainability.

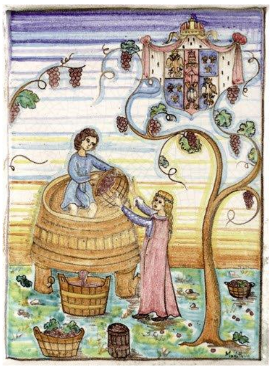
2.7 History of vinegar

The term "vinegar" is derived from Latin ("vinum acre"), which evolved into the French expression "vin-aigre," meaning "sour wine" in English. Today, the term is used to refer to all types of vinegar resulting from the acetic fermentation of ethanol, irrespective of their origin. The production of wine has been a concern for at least 10,000 years, and it is assumed that vinegar has been around for just as long (Duddington, 1962; Galoppini and Fiorentini, 1984). In antiquity, the factors that caused wine to turn into vinegar were not understood, although the phenomenon of vinegar formation was well-known.

Various types of vinegar are produced worldwide, depending on the raw materials and manufacturing methods used. Among the most common varieties are "apple cider vinegar," "balsamic vinegar," "wine vinegar," "rice vinegar," and "malt vinegar." These types are produced in different regions of the world and are known for their distinctive qualities.

Vinegar is the final product obtained through the acetic fermentation of alcoholic liquids in the presence of acetic bacteria (Figure 2.4). Acetic bacteria in contact with air develop rapidly on the surface of alcoholic liquids, forming a more or less compact film (Fumi et al.,1992) and converts ethanol from the proper medium into acetic acid.

The general chemical reactionof vinegar production is (Ebner, 1982):



$$\underset{\text{Ethyl Alcohol}}{CH_3 - CH_2 - OH} + O_2 \xrightarrow{\text{Acetic acid bacteria}} \underset{\text{Acetic Acid}}{CH_3 - COOH} + \underset{\text{Water}}{H_2O}$$

Figure 2.4: Conversion of Ethanol to Acetic Acid

CHAPTER 3. Current status of vinegar production methods

3.1 General aspects of vinegar production

The technological process for vinegar production does not vary significantly depending on the type of alcoholic substrate used: wine or an aqueous-alcoholic solution prepared from spirits.

The basic principle of vinegar production is illustrated in Figure 3.1.

The fundamental principle of vinegar production is to maximize the contact surface between the raw material, acetic bacteria and air, to ensure complete oxidation of the ethanol present in the raw material. The various industrial techniques used in vinegar manufacturing are distinguished by the use of different types of equipment.

Raw materials used in acetic fermentation include altered wines with low alcohol content, alcohols derived from fruit juices, cereals, beets or potatoes, whether refined or diluted, starchy materials, and others.

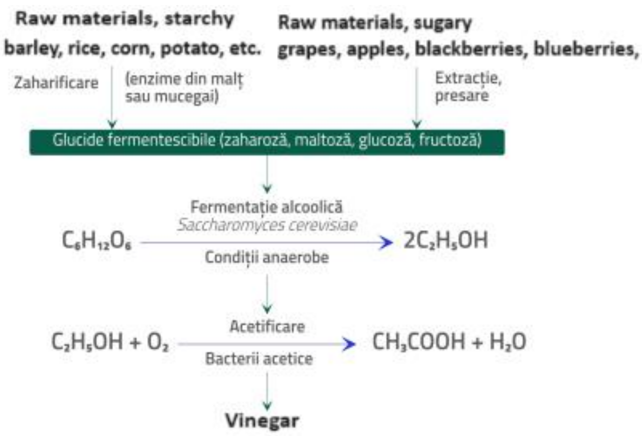


Figure 3.1 General technological process for vinegar manufacturing from starchy and sugary raw materials

Chapter 4: Aim and objectives of the PhD thesis

4.1 Aim of the PhD thesis

The research theme addressed in this PhD thesis is formulated in line with recent trends identified in specialized literature, as listed in the references at the end.

4.2 Objectives of the PhD thesis

The overall objective of the PhD thesis is: **To identify technological and managerial factors applicable to the use of ultrasonic treatment in vinegar production that lead to increased extraction of biologically active compounds and reduced processing times.**

The thesis aims to achieve the following operational objectives:

O.1. Analysis of the Current State of Achievements in Vinegar Production Technologies and Management

This objective focuses on assessing the current level of knowledge regarding conventional systems used in vinegar production, the chemical compounds of a phenolic nature found in fruits, common extraction methods, and new technologies used to enhance the extraction of phenolic compounds during vinegar production. Additionally, it includes the evaluation of the current status of food industry management systems and to identify the underexplored factors in literature.

O.2. Characterization of the Effects and Identification of Issues Related to Ultrasound Treatment in Vinegar Production

This objective aims to analyze and synthesize the existing literature on ultrasonic systems used for extraction, and the application of ultrasonic treatment during vinegar production, as well as the technological constraints that have an impact on the the industrial, to adopt and to commercial viability of ultrasonication systems.. Furthermore, it seeks to develop and expand the ultrasonic process from laboratory scale to industrial scale.

O.3. Investigation and Improvement of Results from Laboratory-Scale Ultrasound Treatment as an Aid in Extracting Biologically Active Compounds from Fruits and Its Impact on Vinegar Quality

This objective focuses on designing an experimental model to enhance the extraction of biologically active compounds from fruit juice used for vinegar production, testing and optimizing this model at the laboratory scale, and to determine the quality parameters of the final vinegar product.

O.4. Identification and Specification of Technical and Managerial Aspects of Implementing Ultrasound Treatment in a Vinegar Production Facility

This objective aims to conduct research on the implementation of food safety management systems concerning the use of ultrasound treatment as a processing step in vinegar production technology. In addition, the economic and financial aspects of applying ultrasound treatment within a vinegar production line are targeted.

The research approach is based on a well-structured algorithm, beginning with an overview of current management and technologies in vinegar production, identifying limitations of conventional technologies, exploring emerging technologies, and analyzing the effects of applying ultrasound treatment at various stages of vinegar production.

Reviewing the literature provides a snapshot of the current state of research on the application of ultrasound treatment in field. According to the studied literature, ultrasonic treatment is used to increase the extraction degree of phenolic compounds and reduce the maceration-fermentation duration of fruit juice, to control the alcoholic fermentation process.

Based on the literature studies and research conducted, a lack of standardized parameters for ultrasound treatment was identified. This has led to the need for theoretical and experimental research on ultrasound treatment management, aimed to increase the extraction of bioactive compounds from fruit juice and understanding the impact of ultrasound treatment on the maceration duration and, consequently, on the quality of the final vinegar product.

PART II – THEORETICAL CONTRIBUTIONS ON INTEGRATED QUALITY-RISK MANAGEMENT IN VINEGAR PRODUCTION SYSTEMS

Chapter 5: Bibliometric Analysis in Vinegar Production

5.1 General Aspects of Utilizing Surplus and By-Products from Fruits in Vinegar Production

According to the FAO (Food and Agriculture Organization, 2019), 21.6% of fruits produced worldwide are wasted from post-harvest stages to distribution.

Therefore, alternatives that can use this surplus and thus to reduce the impact generated on fruit industry are extremely valuable. Potential options related to the vinegar industry might include macerating fruits with vinegar, enriching vinegar with fruit fibers, or using fruits for vinegar production.

The acetic nature of fruit vinegars and the significant sensory impact that this acid has on the organoleptic properties of the product allow the use of almost any type of fruit in its production. Although Asian countries were the first to show interest in this type of product, more and more scientific research are carried out in other parts of the world on this matrix (Figure 5.1).

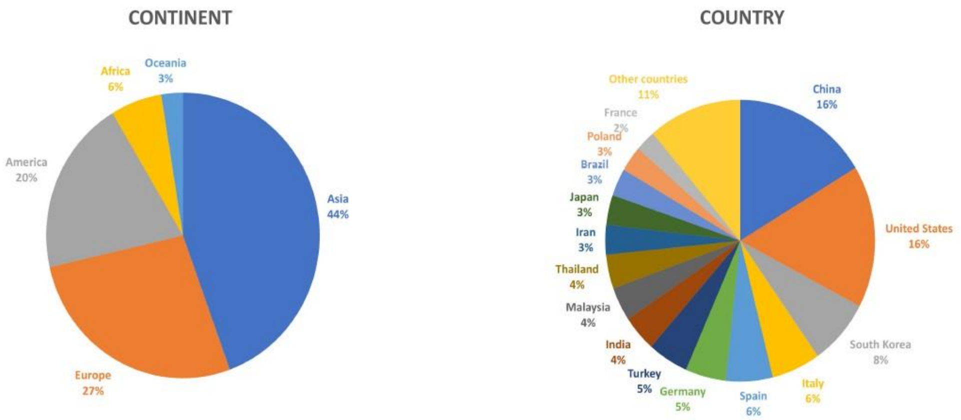


Figure 5.1 Percentage distribution of scientific articles on fruit vinegar published from 2015 to 2020 by research group origin (Continent/Country) (Source: Scopus)

Figure 5.2 illustrates the distribution of the number of scientific articles on fruit vinegar published between 1990 and 2020. As it can be seen, there has been an exponential increase in recent years, indicating a growing interest from the scientific community in this type of product.

Therefore, this bibliographic review will aproach the technological influence and biotechnological processes of producing fruit vinegar other than grapes. On the one hand, it will analyze the different procedures for preparing and extracting juice, such as crushing, grinding, or peeling fruits. It will also examine the various conditions and methods applied in both alcoholic and acetic fermentation, and study the microorganisms responsible for each process, along with the physical and chemical properties of the final products.

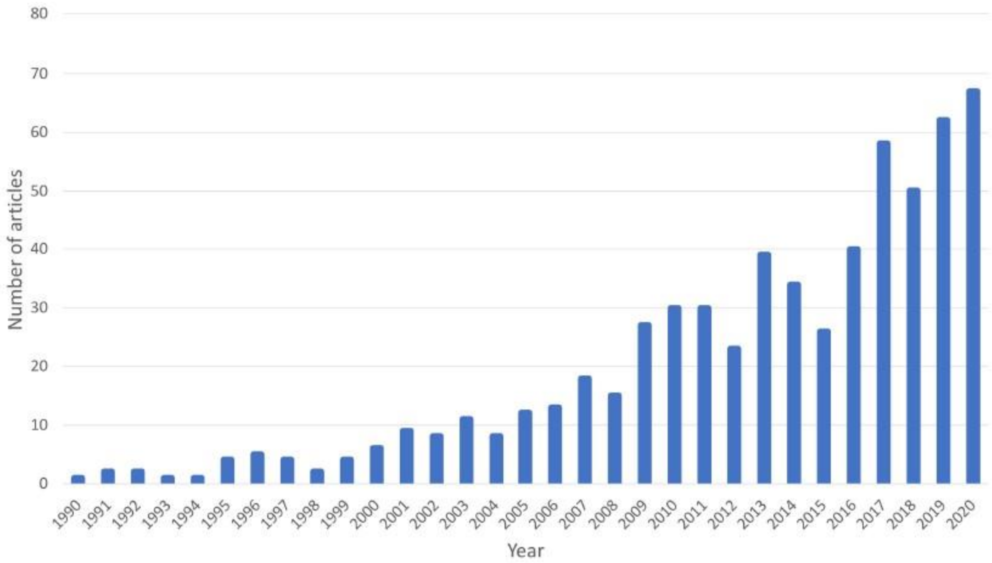


Figure 5.2 Number of scientific articles on fruit vinegar published annually (Source: Scopus)

5.2 Aspects of Vinegar Production and Marketing

The increasing interest in high-quality vinegar products was first observed fifteen years ago, and this trend has not lost interest in European countries, and now it can be obeserved an increase of production and development in this area

As shown in Figure 5.4, a logical model depicts the relationship between research topics, divided into main themes: substances, health functions, production technologies, adjunctive medicines, vinegar residues, and those currently being studied in the field of vinegar research.

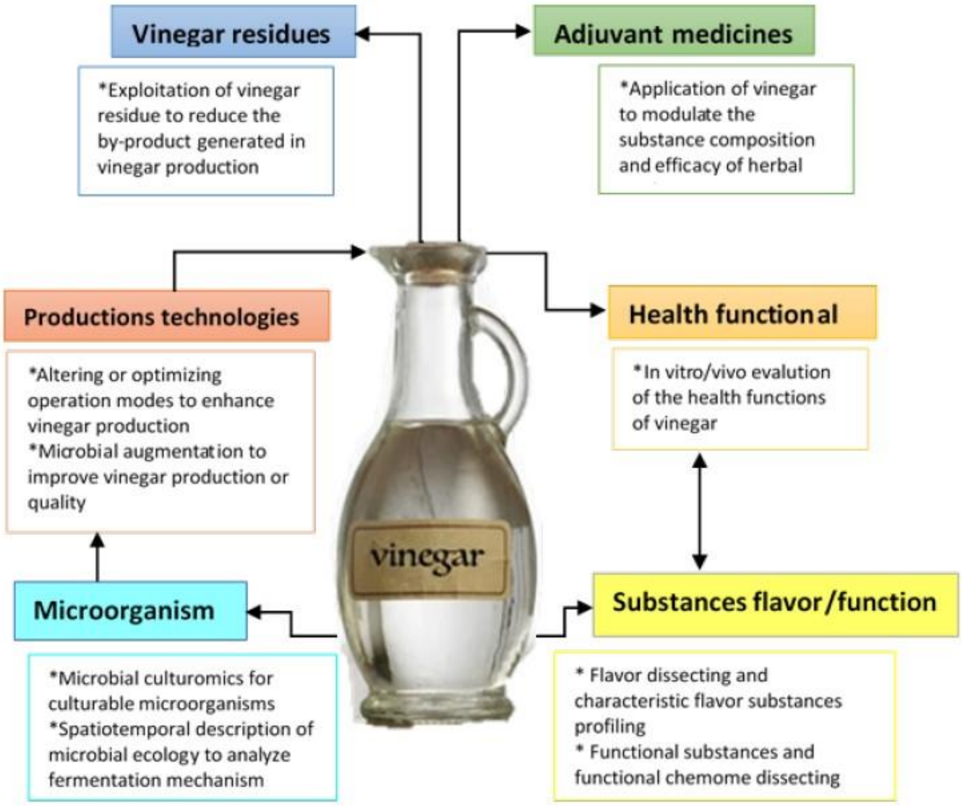


Figure 5.4 Research Themes Model in the Field of Vinegar Production (Boistean, 2021)

Chapter 6. Research on the Use of Ultrasound in Vinegar Production Technology

6.7 Use of ultrasound in food industry

The evolution of ultrasound used in processing technologies began before World War II, initially focusing on emulsions and surface cleaning. By the 1960s, power ultrasound had been widely accepted and used in industry, particularly for cleaning and welding plastics. The use of low-intensity ultrasound for food characterization was first studied 60 years ago. Recently, the full potential of this technology has been realized (Dolatowski, Stadnik, and Stasiak, 2007).

In the field of food technology, ultrasound finds application across a wide range of processes. Until recently, most applications of ultrasound in food technology focused on non-invasive analysis, with an emphasis on quality assessment. These applications use techniques similar to those in diagnostic medicine or non-destructive testing, involving high-frequency ultrasounds. Examples include detecting foreign bodies in food, analyzing droplet sizes in fat and oil emulsions, and determining the degree of crystallization in dispersed emulsions.

In addition to the cavitation phenomenon, ultrasound is able to weaken the physical structure of the material or medium, on the condition that the dimensions of the support are similar to those of the ultrasonic wavelength used. One application of power ultrasound in biochemistry is the breakdown of biological cell walls to release their contents. It has also been demonstrated that power ultrasound can be used to activate immobilized enzymes, enhancing substrate transport to the enzyme. Additionally, ultrasound can be used to inhibit enzymes. The use of power ultrasound significantly improves the extraction of organic compounds from plant materials and seeds. The mechanical effects of ultrasound ensure deeper solvent penetration into cellular materials and enhance mass transfer.

Other uses include improvements in flavor extraction, filtration, mixing and homogenization, as well as the precipitation of powders in air and the destruction of foams that can cause difficulties in process control, such as in fermentation.

6.9 Use of High-Frequency Ultrasound in Fermentation

Studies have highlighted that high-frequency ultrasound based on measurement systems are non-invasive, hygienic, precise, rapid, cost-effective and suitable for automation. These measurement methods can be used to monitor concentrations in solutions as well as to assess composition, structure, physical state, and molecular properties (McClements, 1991; Novoa-Díaz et al., 2014; Stillhart and Kuentz, 2012).

6.10 Effects of Ultrasound Treatment on Polyphenols and Their Bioactivity

In the extraction of biologically active compounds from biological matrices, such as polyphenol extraction from plants and food products, it is mainly used the ultrasonic power range. Most ultrasonication equipment operates at low ultrasonic frequencies, generally between 20 and 40 kHz. Although low-frequency ultrasound appears to be effective for generating faster fragmentation, the exact optimal frequency depends on the specific acoustic amplifier and system. Therefore, there is a need for the design of optimization tests and specific configurations for different types of extractions (Bhangu, Ashokkumar, and Lee, 2016; Wen et al., 2018).

Chapter 7. Quality-risk management in vinegar manufacturing technology

7.1 Quality management in vinegar production

In food industry, quality—understood as a level of excellence—can be defined as a product that achieves outstanding rewards and performance. A high-quality food product is defined as one that meets the requirements of both the consumer and the producer in terms of performance, quality standards, preferences, excellence, safety, and health. Standards include physical-chemical quality, organoleptic characteristics, high sensory properties, improved shelf life, absence of pathogenic microorganisms (product safety), and a reasonable price.

Quality management involves controlling all these factors that can adversely affect the quality of the final product. Management involvesobjectives and planning the control of these parameters without compromising the final product's quality. The beverage industry relies on quality parameters preferred by consumers, such as taste, texture, appearance, and microbiological safety, whereas the product must not contain any health risks.

7.2 ISO 9004: A quality management standard for the creative leaders of contemporary sustainable organizations

International management standards can only provide organizations challenging opportunities only if they understand the goals and characteristics of these standards and apply them creatively and integratively in their business management. Business leaders play a key role in making this happen.

The Quality Management Standard (QM ISO 9004) is one of the international management standards that can be applied to all types of organizations.

Many organizations, which have not recognized the differences and relationships between ISO 9004 and ISO 9001 standards, often apply ISO 9000 standards inappropriately and, therefore do not have the capacity to exploit the potential opportunities of standards.

7.9 Current approaches to management systems in food industry

Ensuring the safety of production and the supply of safe food products are the primary objectives of the food and beverage industry. These objectives can be achieved through the adoption of a systematic and organizational structure, controlling activities, processes, procedures, and resources according to standards that form the basis of quality and hygiene systems, including Hazard Analysis and Critical Control Points (HACCP) and the ISO 9000 series. Implementing a Quality Management System according to the ISO 9000 series in the food sector involves ensuring quality procedures for food companies and reinforcing legislative requirements (Aggelogiannopoulos, Drosinos, and Athanasopoulos, 2007).

HACCP, based on ISO 9001, as part of a quality system, not only succeeds in ensuring product safety but also provides better and more effective implementation of the entire quality system (Efstratiadis and Arvanitoyannis, 2000). Food manufacturers are legally required to apply HACCP, while other systems are applied voluntarily within the food industry.

PART III: EXPERIMENTAL STUDIES AND RESEARCH ON ULTRASOUND TREATMENT MANAGEMENT DURING VINEGAR PRODUCTION PROCESS. LABORATORY RESEARCH

CHAPTER 8: Experimental research on the application of ultrasound treatment during vinegar production in laboratory conditions

8.2 Experimental research plan

Being one of the most widely used acidic spices in the world, vinegar serves multiple physiological functions, including antibacterial, antioxidant, and weight-reducing effects. Due to their polyphenol content, blueberries (Vaccinium myrtillus L), raspberries (Rubus idaeus L), and blackberries (Rubus caesius L) are valuable sources of natural flavors and antioxidants for vinegar.

The use of ultrasound could significantly enhance the content of these valuable compounds. Thus, the purpose of this research was to evaluate the total polyphenol content (TPC), total anthocyanin content (TAC), and antioxidant capacity (using ABTS and DPPH free radicals) of experimental vinegar variants produced through the fermentation of juices from these berries, with the juices treated using ultrasound. The main stages of producing experimental vinegar variants are illustrated in Figure 8.1.

8.3 Raw materials used for experimental research

Experimental vinegar variants were prepared and monitored at the Oenology Laboratory of the Faculty of Food and Tourism at Transilvania University of Brașov. Preliminary tests and analytical and physico-chemical determinations of the samples were conducted also at the laboratory mentioned above and to the Biochemistry Laboratory at the National Research and Development Institute for Potato and Sugar Beet in Brașov, and at the Interdisciplinary Research Platform, USAMV, Bucharest.

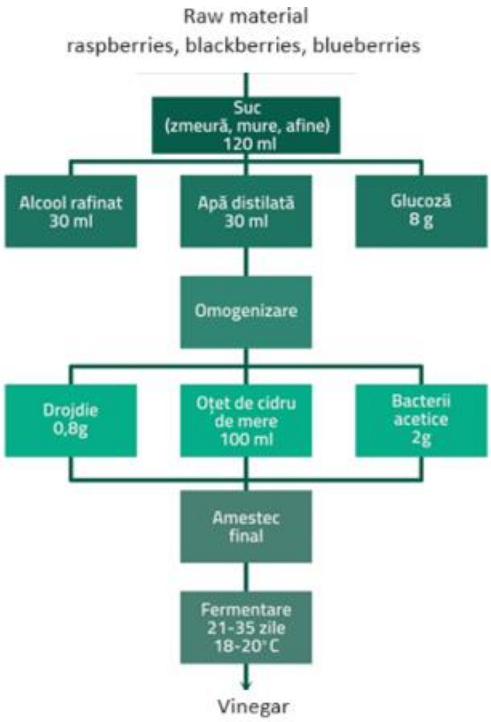


Figure 8.1 Main stages in the process of obtaining experimental variants of vinegar

8.4 Experimental research on the total polyphenol, anthocyanin content, and antioxidant properties of various vinegars obtained from berries

Raspberries, blueberries, and blackberries are valuable sources of natural flavor and antioxidants for vinegar. The aim of this research was to evaluate the total polyphenol content (TPC), total anthocyanin content (TAC), and antioxidant capacity (using ABTS and DPPH free radicals) of several experimental vinegar variants obtained through the acetic fermentation process, from various substrates containing different percentages of berry juice (raspberries, blueberries, blackberries) (40%; 60%) and acetic bacteria (0.5%; 1%). For the single-stage acetic fermentation process, a natural source of Acetobacter aceti in different concentrations was used.

8.4.1 Obtaining berry vinegar

Berry fruits from three species—raspberry (*Rubus idaeus* L.), blackberry (*Rubus fruticosus* L.), and blueberry (*Vaccinium myrtillus* L.)—were used as raw materials for the production of berry vinegar through a one-step (acetic) fermentation process. The berries were sorted, the defective ones were set aside and the healthy fruits were mechanically crushed. The juice was extracted using a manual press. The remaining fruit pulp was reprocessed through the press to extract as much juice as possible (Figure 8.2). During vinegar processing, the berry juices (Figure 8.3) were first mixed with water and apple cider vinegar in two different percentages: 60% juice: 20% water: 20% apple cider vinegar and 40% juice: 40% water: 20% apple cider vinegar, according to the ID of each sample presented in Table 8.1. These substrates were mixed with ethanol (7%) and glucose (4%). The inoculation was carried out with a Saccharomyces cerevisiae culture at a concentration of 0.4% (v/v) (2.12 x 10^9 cells/mL).

Preparation of yeast inoculum has been made by mixing 2 g of yeast with 60 mL of warm water. Subsequently, these substrates, having sufficient alcohol for acetic fermentation, were inoculated with a natural culture of acetic acid bacteria (Acetobacter aceti) to concentrations of 0.5% and 1% (v/v).

a. b.

Figure 8.2 Fruit processing used in experimental trials: a – fruit processing; b – blueberries, raspberries, and blackberries pulp.

Figure 8.3 Samples of blueberry, raspberry, and blackberry juice used in experimental research.

The acetic fermentation process was completed after periods between 21 to 35 days after inoculation. Sampling was carried out at certain periods of time to collect and test the berry vinegar. These experimental variants were stored at 4 °C in sterile 100 ml vessels until analysis (Figure 8.5).

The initial alcohol content of raw material was 7% (v/v). Taking into account that the surface crop fermentation method is not 100 % efficient, the acetic acid content values were within the prescribed range.

8.4.2 General physical and chemical analysis of fermentation substrates and vinegar samples

The pH, acidity (% acetic acid), and total soluble solids content were determined for substrates (the mixture before fermentation) and for the vinegar samples. pH was measured using a pH meter (Consort C5020T, Consort, Belgium). Acidity (as acetic acid equivalent) was determined by titration with a standard 1N NaOH solution using phenolphthalein as an indicator. The soluble dry matter content (°Brix) of the samples was measured using a refractometer (OE Germany/OE Swiss/MASS) at room temperature.

8.4.2.1 Total acidity determination in vinegar – Titrimetric method STAS 6182/14-72

Total acidity, also known as potential acidity, is determined by the total amount of hydrogen ions released by an acid during neutralization until complete dissociation occurs. Total acidity is expressed as the sum of volatile and fixed acidity Total acidity is the sum of titratable acids; it does not include carbonic acid and free and combined anhydra The method is based on titrating the product with a known concentration of sodium hydroxide solution, using phenolphthalein as an indicator.

8.4.2.2 Soluble dry matter content

The soluble dry matter content was evaluated by refractometry using an Abbe refractometer. The method involves determining the refractive index or percentage of sucrose mass (soluble dry matter) and estimating the sugar content expressed in g/L.

A few drops of berry vinegar were pipetted into the refractometer's prism chamber, and the refractive index and soluble dry matter content (sucrose percentage) were read from the visible scale in the eyepiece.

8.4.2.3 Total polyphenol content – Folin-Ciocalteu reagent

All phenolic compounds present in the samples (Figures 8.5 and 8.6), including berry wine and vinegar, are oxidized by the Folin-Ciocalteu reagent. This reagent is composed of a mixture of phosphotungstic acid ($H_3PW_{12}O_{40}$) and phosphomolybdic acid ($H_3PMo_{12}O_{40}$), which, after oxidizing the phenols, is reduced to a mixture of blue tungsten oxides (W_8O_{23}) and molybdenum oxides (Mo_8O_{23}). The blue color produced has a maximum absorption in the 750 nm region and is proportional to the total amount of phenolic compounds initially present.

Calibration curves were obtained using gallic acid as a standard (concentration range 5-250 μg/ml). The results of total polyphenol content were expressed in mg/L as gallic acid equivalent (GAE) (Figure 8.7). Calibration samples are shown in Figure 8.7. For each determination, three repetitions were performed.

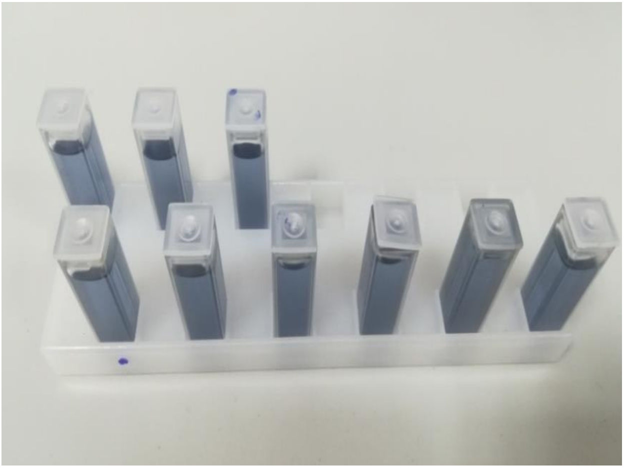


Figure 8.5 Preparation of Samples for Total Polyphenol Content Determination Using Folin-Ciocalteu Reagent

Figure 8.6 Preparation of Samples for Calibration Curve Procedure

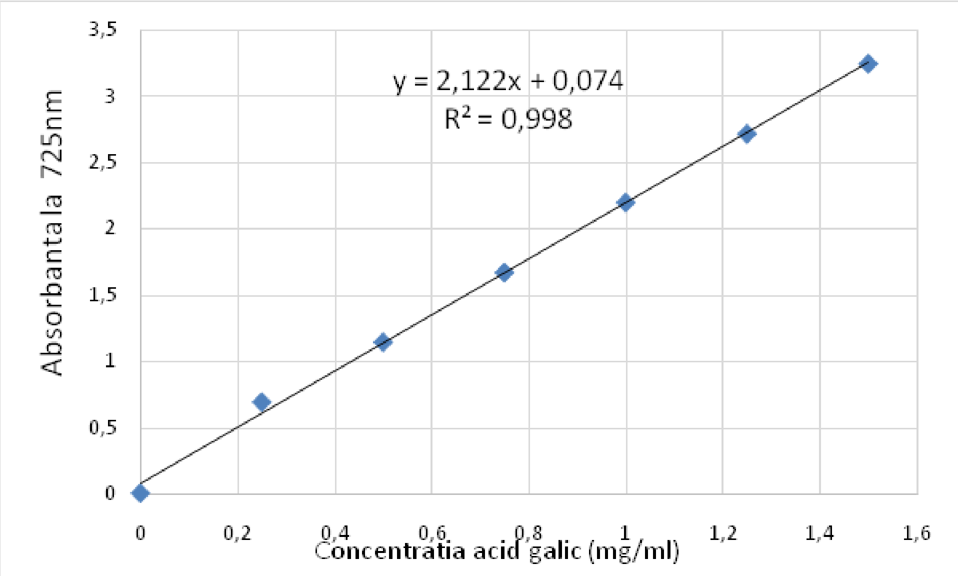


Figure 8.7 Calibration Curve: Absorbance vs. Concentration (mg GAE/mL)

8.4.2.4 Total monomeric anthocyanin content

The total content of anthocyanin pigments was determined using the differential pH method, adapted (ELISA plates with 96 wells, Nunk, Denmark). This method is based on the property of anthocyanin pigments to change color depending on pH (Figure 8.5).

Two dilutions were prepared: 1 ml of sample in 10 ml of solution: the first in potassium chloride (0.025 M, pH 1.0), and the second in sodium acetate (0.4 M, pH 4.5), with pH adjusted using 2N HCl. After equilibrating at room temperature (15 minutes), absorbance of the two solutions was read at wavelengths of 510 nm and 700 nm. The total content of anthocyanin pigments (mg equivalent cyanidin 3-O-glucoside/100 g fresh weight).

All determinations were made in three replicates, and results are expressed as mean ± standard deviation (Figure 8.8).

8.4.2.5. DPPH test

The DPPH test is based on the redox reaction with the 2,2-diphenyl-1-picrylhydrazyl radical. The DPPH radical has a violet color. As a result of the reaction with an antioxidant, DPPH-H (2,2-diphenyl-1-picrylhydrazine), a yellow-colored compound, is formed. The test relies on this color change. The antioxidant potential of the analyzed sample can be assessed by measuring the absorbance at 515 nm for the yellow-colored reduced compound using a spectrophotometer.

8.4.2.6. Statistical analysis

Data were processed using SPSS software (Statistical Package for the Social Sciences, version 21, IBM, New York, NY, USA) and analyzed by ANOVA at a significance level of 0.05, and Duncan's multiple range test (noted as significant if $p < 0.05$). The correlation between variables was analyzed using Pearson correlation coefficients. Analyses were performed in triplicate, and results were presented as mean ± standard deviation.

8.5. Results

8.5.1. Obtained vinegar variants and their physicochemical properties

In general, the acidity values were much lower than those obtained in a previous study concerning strawberry vinegar (5.5%) after 80 days of acetic fermentation (Hidalgo, 2013) or in other types of vinegar (Aguiar et al., 2005; Arvaniti et al., 2019), but similar to the data presented by Boonsupa et al. (2013) for raspberry and blueberry vinegars. The maximum acetic acid content (4.94 g/100 mL ± 0.12) and the minimum pH value (2.78 ± 0.01) were obtained for the Z3 variant (40% berry juice: 1% acetic bacteria).

For all berries used as substrates, in the 40% juice variants inoculated with 0.5% and 1% acetic bacteria, the acidity values were significantly higher compared to vinegars obtained from substrates containing 60% fruit juices and 0.5% acetic bacteria. These variants exhibited the greatest difference from the initial acidity of the substrates, with the increase in these acidity values being significantly higher compared to variants using 60% berry juice for substrates inoculated with 0.5% acetic bacteria (Figure 8.8).

As illustrated in Figure 8.8, all berry vinegars showed a significant increase in acetic acid content through fermentation, as the alcohol in the substrate was converted into acetic acid by the acetic bacteria.

8.5.2. Total polyphenol content (TPC) of vinegar variants

Significant differences were found among the analyzed variants, with variant B1 (60% juice : 0.5% Acetic Bacteria) having the highest TPC values (180.83 ± 1.96 mg GAE/L). The lowest TPC value was found in vinegar samples obtained using substrates with 40% blackberry and raspberry juices (inoculated with acetic bacteria at 0.5% and 1%) and with a high acidity level (48.10 ± 5.14 mg GAE/L for variant M3, 35.87 ± 5.89 mg GAE/L for variant R3). The composition of the substrates subjected to acetic fermentation had a major impact on TPC, particularly the acidity of the samples and the percentage of fruit juice used.

Vinegars obtained from fruit juices retain only a portion of these beneficial compounds for consumer health, as the methods commonly used during vinegar production affect the types and content of these biologically active substances (Seydi, 2019). Andlauer et al. (2000) reported that acetic fermentation could reduce the polyphenol content of vinegar obtained through traditional methods. In our study, TPC and TCC levels significantly decreased after fermentation, as observed in Figures 8.9 and 8.10.

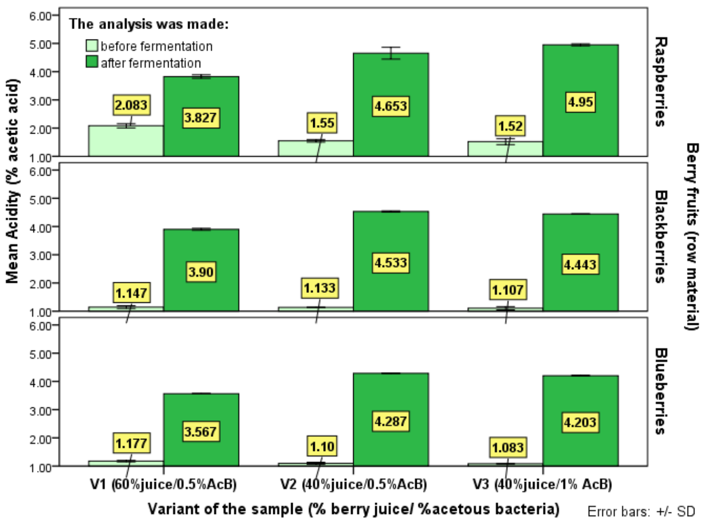


Figure 8.8 Changes in acetic acid content (%) in samples tested during the production of berry vinegar.

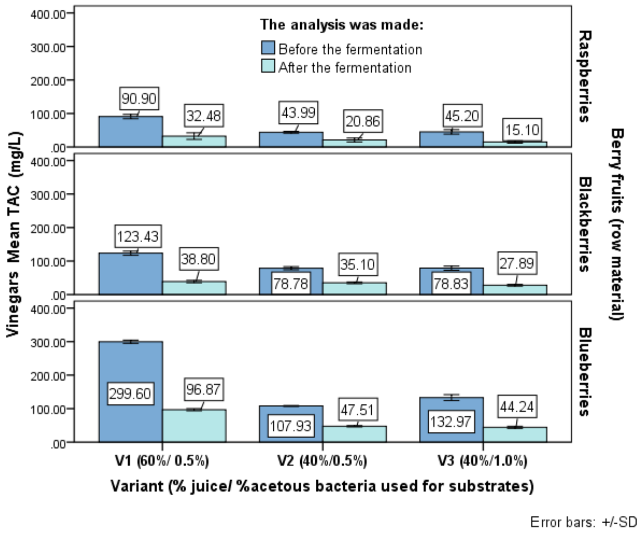


Figure 8.9 Changes in TAC values of berry vinegar samples depending on the percentage of berry juice and the acetic acid bacteria used

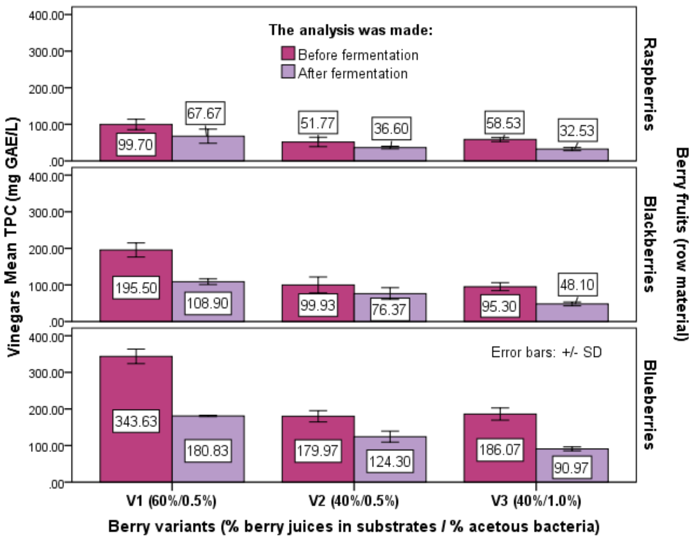


Figure 8.10 Changes in TPC values (total phenolic content values before and after substrate fermentation) for experimental berry vinegar samples depending on the percentage of berry juice in the substrate and the acetic acid bacteria used.

It is important to note that the same fermentation process was used for all variants; the only factors that could influence the TPC levels were the type of berries, the percentage of berry juice in the substrates, and the percentage of acetic bacteria used. Regarding the influence of the type of berries on the total polyphenol content (TPC) in vinegar, the use of blueberries resulted in significantly higher values for this indicator compared to the other berries used.

8.5.3. Total monomeric anthocyanin content (TAC) in vinegar variants

The use of 60% berry juice exhibited significantly higher total anthocyanin content compared to the other variants for all types of berries used in the study. The highest TAC value was obtained for variants B1 and B2 (values ranging from 96.87 ± 3.12 mg/L to 47.51 ± 2.10 mg/L). For all berry juices used, samples with high acidity (variants V2 and V3) had the lowest TAC values. The high acidity level might contribute to the degradation of anthocyanins and anthocyanidins.

8.5.4. Antioxidant capacity of vinegar

As regards the antioxidant capacity assessed by the two methods, significant differences were observed between variants. Blueberry juice-based vinegars (60%, 0.5% Acetic Bacteria) (B1) exhibited the highest antioxidant capacity, with free radical scavenging activities of 1.52 ± 0.07 mM TE/L for the ABTS test and 65.90 ± 2.12% for the DPPH test. The lowest antioxidant potential was found in vinegar made from raspberry juice (R1, R2), with values of 0.44 ± 0.02 mM TE/L for the ABTS test and 17.81 ± 1.91% for the DPPH test.

Evaluating the antioxidant activities of a selected antioxidant requires several test methods. The DPPH free radical is widely used for evaluating antioxidant activity in samples. In the DPPH test, the percentage of inhibition of the DPPH radical by antioxidants is attributed to their ability to donate hydrogen ions.

The results indicate that a good antioxidant capacity is best achieved using 60% juice in the substrates and 0.5% acetic bacteria inoculum, likely due to the high sensitivity of phenolic compounds and monomeric anthocyanins to environmental acidity. Research findings show that ABTS test values were well correlated with TPC and TAC (Figure 8.11). In all cases, the best correlations were identified with TPC compared to TAC. Some differences in the content of phenolic compounds might influence antioxidant capacity, as the samples might also contain additional non-oxidizable phenolic antioxidants that emerged due to fermentation.

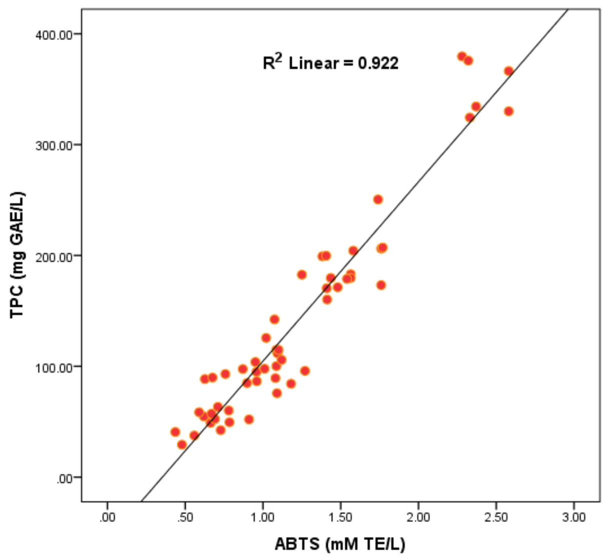


Figure 8.11 Correlations between TPC and antioxidant capacity determined by ABTS.

In our study, experiments were conducted without using an optimization program. Future research will aim to establish optimal conditions for producing the best berry fruit vinegar rich in biologically active compounds, especially antioxidants. Moreover, any study of the antioxidant potential and TPC of berry fruit vinegar should consider the structure of antioxidants, their intake, raw materials, technology, and the maturation process.

8.6. Influence of Ultrasound on Polyphenol Content in the Technological Process of Blueberry Vinegar Production

Further research was conducted to evaluate the total polyphenol content (TPC), total anthocyanin content (TAC), and antioxidant capacity (using ABTS and DPPH tests) of several samples obtained through the fermentation process of blueberry juice treated with ultrasound. The blueberry juice was treated with ultrasound (at a frequency of 20 kHz) at amplitudes of A40%, A60%, and A80% (A) for three, four, or five minutes. After optimization studies, the optimal values for the variables were found to be amplitude A78.50% and time 3.96 minutes, with the following determined values: TPC 628.01 mg GAE/L, TAC 22.79 mg C3G/L, ABTS 391.7 μmol/100 mL, and DPPH 229.17 μmol/100 mL. Ultrasound treatment of all blueberry vinegar samples had beneficial effects on polyphenol and anthocyanin enrichment, improving antioxidant activity and the quality of blueberry vinegar.

Therefore, the study aimed to optimize (using RSM) the extraction of polyphenols and anthocyanins and the antioxidant capacity of vinegar variants obtained from alcoholic substrates containing blueberry juice treated with ultrasound.

8.6.1 Production of blueberry vinegar variants

Fresh blueberries were sorted, mechanically crushed (using a mixer), and the blueberry juice was obtained under laboratory conditions using a Bosch MES3500 centrifugal juicer (700 W) (Bosch GmbH, Stuttgart, Germany), by following a procedure typically applied on a household scale. The clarified blueberry juice obtained was used for vinegar production according to the technological scheme presented in Figure 8.12.

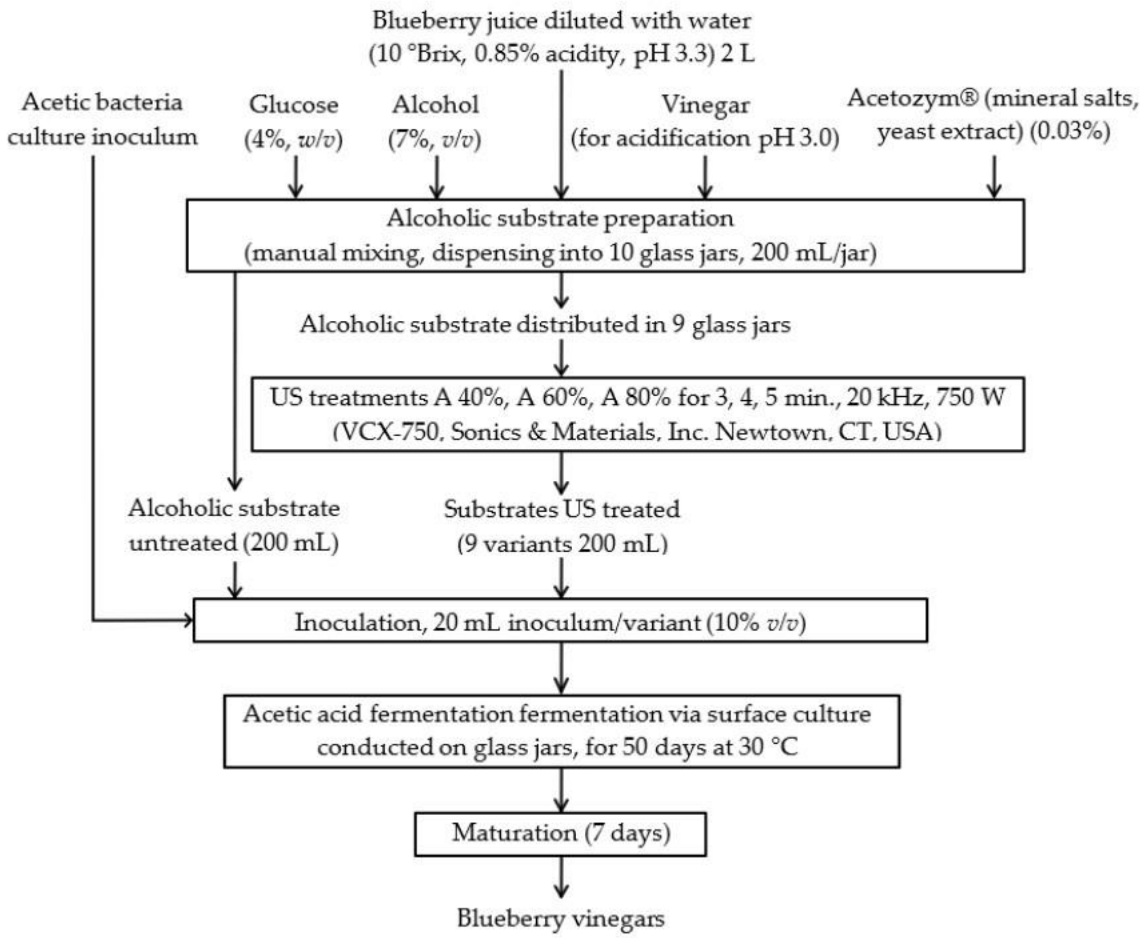


Figure 8.12 The method used to obtaining blueberry vinegar (US = ultrasound)

Acetic fermentation was carried out in 0.5 L glass jars containing 220 mL of substrates inoculated with acetic bacteria. The fermentation process was stopped when the acetic acid content became stable for 7 days and until the alcohol content was between 0.5% and 1%. The acetic fermentation experiments were completed after 50 days from inoculation. The control (Sample A) consisted of untreated blueberry vinegar. The vinegar samples were stored at 4°C.

8.6.2 Titratable acidity, pH, and total soluble substances

The values of pH, titratable acidity (% acetic acid), and total soluble substances were determined for substrates (before fermentation) and for the vinegar samples. The pH was measured using a pH meter (Consort C5020T, Consort, Belgium). The method for determining acidity (% acetic acid) involved titration with a 1N NaOH solution in the presence of phenolphthalein as an indicator, and the total soluble substances (TSS, °Brix) content was analyzed using a refractometer (OE Germany/OE Swiss/MASS) at room temperature.

8.6.3 Ultrasonic treatment

Before inoculation, the substrates were treated with ultrasound (VCX-750, Sonics & Materials, Inc. Newtown, CT, USA) using a continuous frequency of 20 kHz, an amplitude of A40%, A60%, and A80%, and a power of 750 W for periods of 3, 4, and 5 minutes for 19 mm and 500 ml samples as a mixture (Brezan et al., 2020).

8.6.5 Total phenolic content (TPC) and total monomeric anthocyanin content (TMA)

The total phenolic content (TPC) of the samples was analyzed using the Folin–Ciocâlteu method (Singleton et al., 1999) using a spectrophotometer with tiles (Tecan, SunRiseTM, MagellanTM software, Männedorf, Switzerland). TPC was indicated in milligrams of gallic acid equivalents (GAE) per liter of vinegar, and in milligrams of gallic acid equivalents per 100 g DW (dry weight).

The total monomeric anthocyanins were determined using the pH differential method, and absorbance was measured on a DR2800 spectrophotometer (Hach Lange, Loveland, USA) (Wang H et al., 2017). Results were reported as mg of cyanidin 3-O-glucoside (C3G) per liter of vinegar, and per 100 g DW.

8.6.6 ABTS method

The antioxidant potential of the vinegar variants in reaction with the ABTS radical (2,2′-azino-bis (3-ethylbenzothiazoline-6-sulfonic) (ABTS·+)) was determined according to the method described by Re et al. (Re R et al., 1999). Trolox standards were used to generate the calibration curve. Results were reported in µmol Trolox equivalents per liter of vinegar (µmol TE/L) and per gram of dry weight (µmol TE/g DW).

8.6.7 DPPH method

The DPPH (2,2-diphenyl-1-picrylhydrazyl) assay was performed according to a method described by Kumara et al. (Kumaran and Karunakaran, 2006). The absorbance was analyzed at 517 nm (DR2800 spectrophotometer, Hach Lange, Loveland, USA) with methanol as control sample. The antioxidant capacity of the samples against DPPH radicals was expressed as micromoles of Trolox equivalents per 100 mL of vinegar (µmol TE/100mL) and per gram of dry weight (µmol TE/g DW), using a calibration curve previously established.

8.6.8 HPLC analysis

The profile of polyphenolic compounds was determined by high-performance liquid chromatography (HPLC) according to the method described by Abdel-Hameed et al. (Abdel-Hameed et al., 2013). Separations were performed using an Agilent Technologies 1200 chromatograph equipped with a

UV-DAD detector, using a 250 mm x 4 mm Licrocart (Licrospher PR-18 5μm) column (Merck, Darmstadt, Germany) operated at 30 °C. All recorded data were processed using Agilent Chem Station B.04.03 software (Agilent, USA). The mobile phase consisted of water/acetic acid (97:3, v/v) (eluent A) and acetonitrile (eluent B) at a flow rate of 1 mL/min. The linear gradient profile was as follows: 97% A (0 min), 97–91% A (5 min), 91–84% A (5-15 min), 84–65% A (15-20.8 min), 65–64.5% A (20.8-36 min), 64.5–50% A (36-37 min), 50% A (37-38 min), 50–97% A (38-39 min) and 97% A (42 min). The injection volume was of 20 μL.

8.6.9 Sensory analysis

The general acceptability of vinegar samples was evaluated by considering the profiles of smell and taste (general impression, pungent sensation, aromatic intensity, taste, and smell of ethyl acetate). Twelve women and ten men participated to the evaluation of the vinegar samples. Each sample was dispensed in glass containers of 20 ± 1 g and kept at 4…6 °C until serving. A 9-point hedonic scale was used for the analysis. The scale scores were as follows: excellent, 9; very good, 8; good, 7; acceptable, 6; and poor, <6.

8.6.10 Statistical analysis

To optimize the antioxidant potential of the blueberry vinegar, RSM (Minitab 18.1, Minitab, Inc) was used. Three-dimensional graphs were obtained (Sigma Plot 12.0 Statistical Analysis Software, Systat Software, Inc.). The analysis was performed three times.

8.7 Results and Discussion

8.7.1 Analysis of raw material (Blueberries) and blueberry juices obtained

There were determined the titratable acidity (TA), pH and soluble dry matter (total soluble solids, TSS indicator expressed in °Brix) of raw material, and also of blueberry juice and of blueberry juice diluted with water. Each value represents the average of three determinations ± standard deviation (n = 3).

There were conducted reserach on the total phenolic content (TPC), total anthocyanin content (TAC) and on antioxidant potential, expressed as ABTS test and DPPH test, of blueberry fruits (raw material) and of blueberry juices used.

The average acidity values of the vinegars obtained in this study are presented in Figure 8.13. Generally, the acidity values were similar to the data presented by Fonseca et al. for blueberry vinegars (Da Silva Fonseca et al., 2018). The maximum acetic acid content (4.307%) was obtained for variant B805 (Figure 8.13).

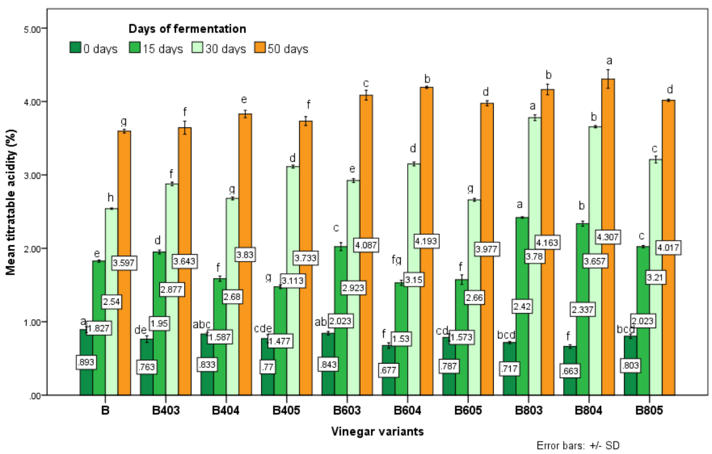


Figure 8.13 Average value of acidity (% acetic acid) for blueberry vinegar variants obtained by using substrates treated with ultrasound (graph of acetic acid formation at different stages of acetic fermentation). Data are expressed as mean values of 3 determinations ± standard deviation. Values with different letters are significantly different by ANOVA and Duncan's test ($p<0.05$)

8.7.3 Total phenolic content and total anthocyanin content of blueberry vinegars

There were determined the average values of the total polyphenol content, total anthocyanin content, and antioxidant potential, expressed as ABTS test and DPPH test, of the blueberry vinegars obtained by using the process described in the chapter the materials and methods. The data were expressed as the mean of 3 determinations ± standard deviation. Values with different letters differ significantly by ANOVA and Duncan's test ($p<0.05$).

To maintain the level, a quadratic model was identified ($R^2 = 0.9614$ for TPC and $R^2 = 0.9889$ for TMA). The effect of amplitude on TPC and TAC in the samples was statistically significant at $p < 0.001$.

In the case of samples initially treated with ultrasound at 80% amplitude for 4 minutes, the highest TPC values were observed (643.2 mg GAE/L). The lowest TPC value was found in samples treated with ultrasound at 40% amplitude for 3 minutes (441.4 mg GAE/L) (Figure 8.14).

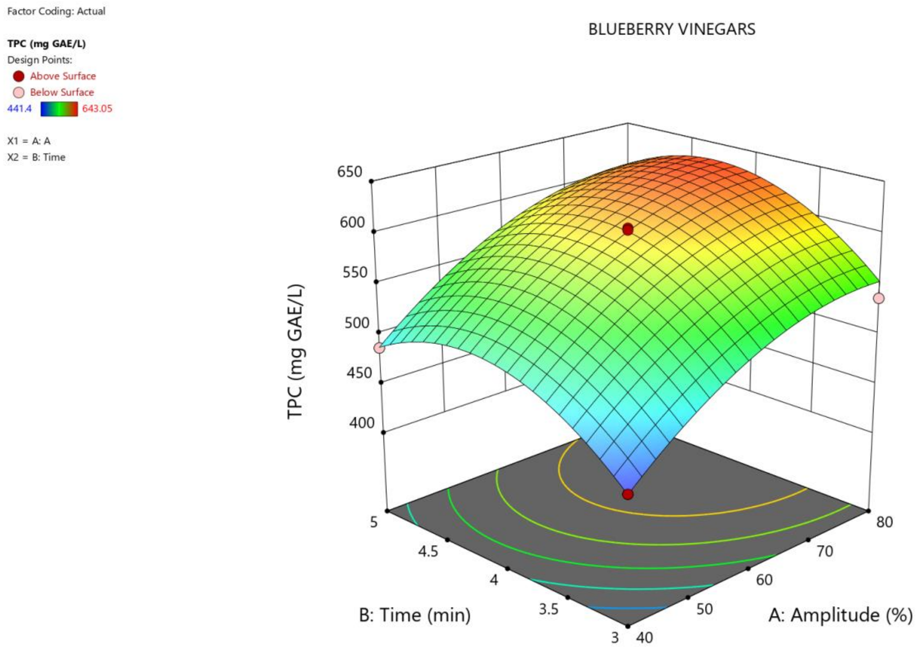


Figure 8.14 Response surface showing the effect of time (minutes) and amplitude (A%) of ultrasound treatments on the bioactive compounds of blueberry vinegars: CTP (mg GAE/L).

Changes in TPC and TAC values, based on ultrasound time and amplitude, are shown in Figures 8.14 and 8.15. In the optimization model, it was determined that the TPC was 628.02 mg GAE/L and the TMA was 22.79 mg C3G/L after 3.96 minutes and treatment with 78.50% amplitude, showing an increase of 45.6% in TPC compared to the untreated control, and an increase of 69.3% in TMA. Similar results (regarding the positive effects of ultrasound treatments on TPC in vinegars) were reported by Yikmiş et al. for apple and tomato vinegar, by Lieu et al. for grape juice, by Brezan et al. for apple cider, and by Bhat et al. for lime juice (Yıkmış, 2019; Yıkmış et al., 2021; Brezan, 2020; Bhat et al., 2011).

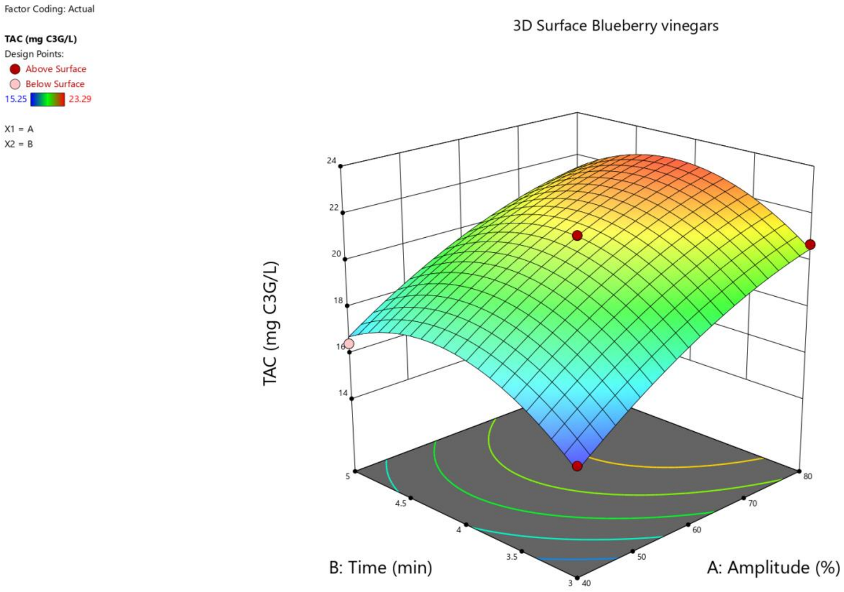


Figure 8.15 Response surface showing the effect of time (minutes) and amplitude (A%) of ultrasound treatments on the bioactive compounds of blueberry vinegars: CTA (mg C3G/L)

8.7.4 Antioxidant Capacity of Berry Vinegars

The variation of ABTS and DPPH values, depending on the ultrasound variables (amplitude and time), is shown in Figure 8.16.

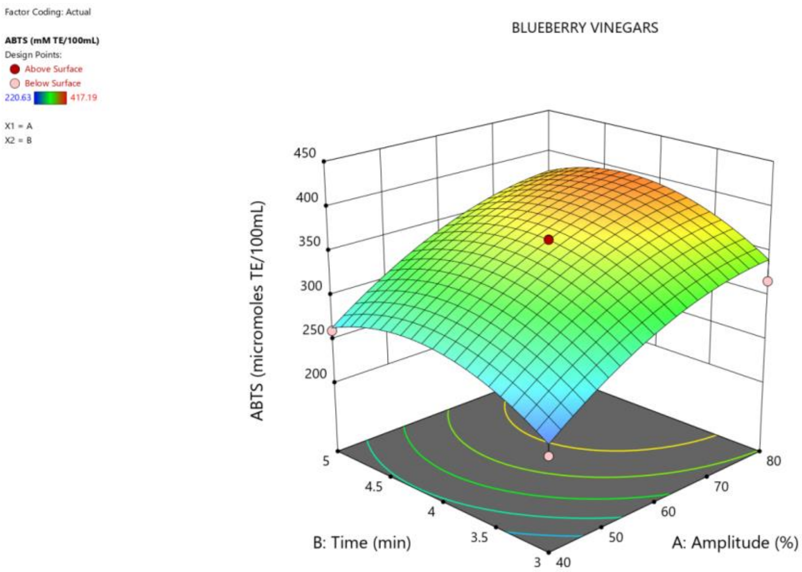


Figure 8.16 Response surface showing the effect of time (minutes) and amplitude (A%) of ultrasound treatments on the antioxidant potential of blueberry vinegars: ABTS test

At the end of the optimization process, ABTS was found to be 363.69 μmol TE/L and DPPH was 217.102 μmol TE/100 mL with a treatment duration of 3.96 minutes and an amplitude of 78.50% (Figure 8.18).

Figure 8.18 Contour plots indicating the predicted optimal values for all responses (predicted parameter values for an amplitude of 78.50% and a treatment time of 3.96 minutes). CI (%) for CTP (612.715; 643.31), for CTA (22.4769; 23.1216), for ABTS (366.732; 416.689), and for DPPH (218.104; 240.227).

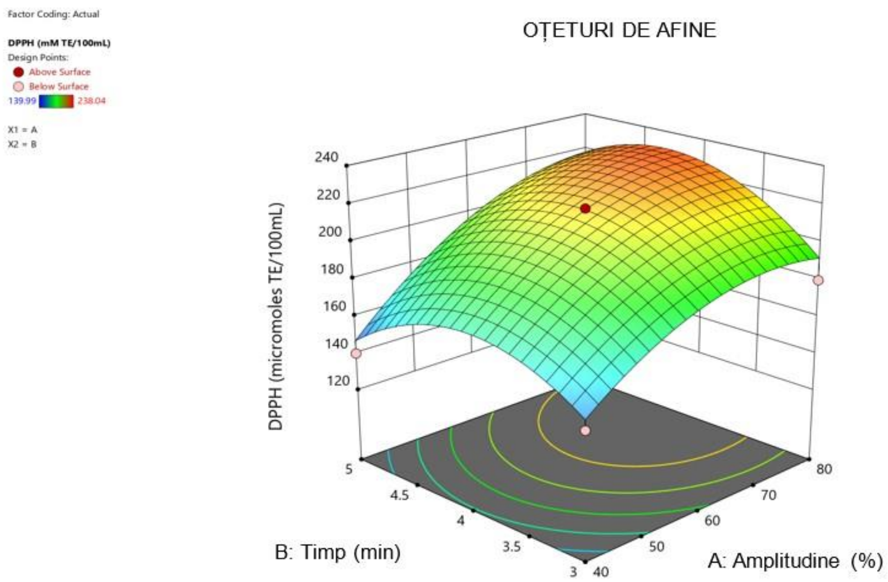


Figura 8.17 Response surface showing the effect of time (minutes) and amplitude (A%) of ultrasound treatments on the antioxidant potential of blueberry vinegars: DPPhH

The lowest DPPH value (μmol TE/L) was determined in the sample treated for 5 minutes at an amplitude of 40% in application 13 (139.1 μmol TE/100 mL); the highest DPPH value was detected in the sample treated at an amplitude of 80% for 4 minutes (238.9 μmol TE/100 mL). Figure 8.18 shows that is likely to achieve a satisfactory range of antioxidants by applying an amplitude of 78.50% and a treatment duration of 3.96 minutes. This is likely due to the high sensitivity of phenols to these conditions.

8.7.5 HPLC analysis of phenolic compounds

In this study, four phenolic compounds (ellagic acid, gallic acid, ferulic acid, and chlorogenic acid) were identified in several samples selected based on the results obtained using the optimization model. The levels of these antioxidants were measured in vinegars produced from substrates treated with ultrasound at amplitudes of 80% and 60% for 4 and 5 minutes, as well as from untreated substrates. Data were expressed as the mean of three determinations ± standard deviation. Values with different letters differ significantly according to the ANOVA and Duncan's test ($p < 0.05$).

The highest levels of all phenolic compounds tested by HPLC were found in the vinegar sample A804. This vinegar was produced using an amplitude of 80% for 4 minutes, with these treatment parameter values being very close to those recorded after applying the optimization model (A 78.5%, 3.96 minutes).

8.8 Conclusions on the influence of ultrasound treatment as a pre-treatment for fruits used in vinegar production

In the vinegar production process, it is desirable to adopt procedures that facilitate color extraction and flavor dissolution. By applying ultrasound technology, the continuous extraction of qualitatively relevant compounds from exocarp is pursued, exploiting the phenomena triggered by the propagation of ultrasound in a solid-liquid medium, such as fruit must. Given the high concentrations of biologically active substances from fruits, accelerating the kinetics extraction through a physical process can be strategic.

The experiments design represents a structured methodology for planning and designing a sequence of tests. Analysis of variance (ANOVA) was used to identify significant input variables for a particular response. The predictive model developed can help achieve proper results for improving productivity.

The optimization function of the design software indicated that an amplitude of 78.50% and a treatment time of 3.95 minutes were selected for maximizing the extraction of total polyphenols and anthocyanins.

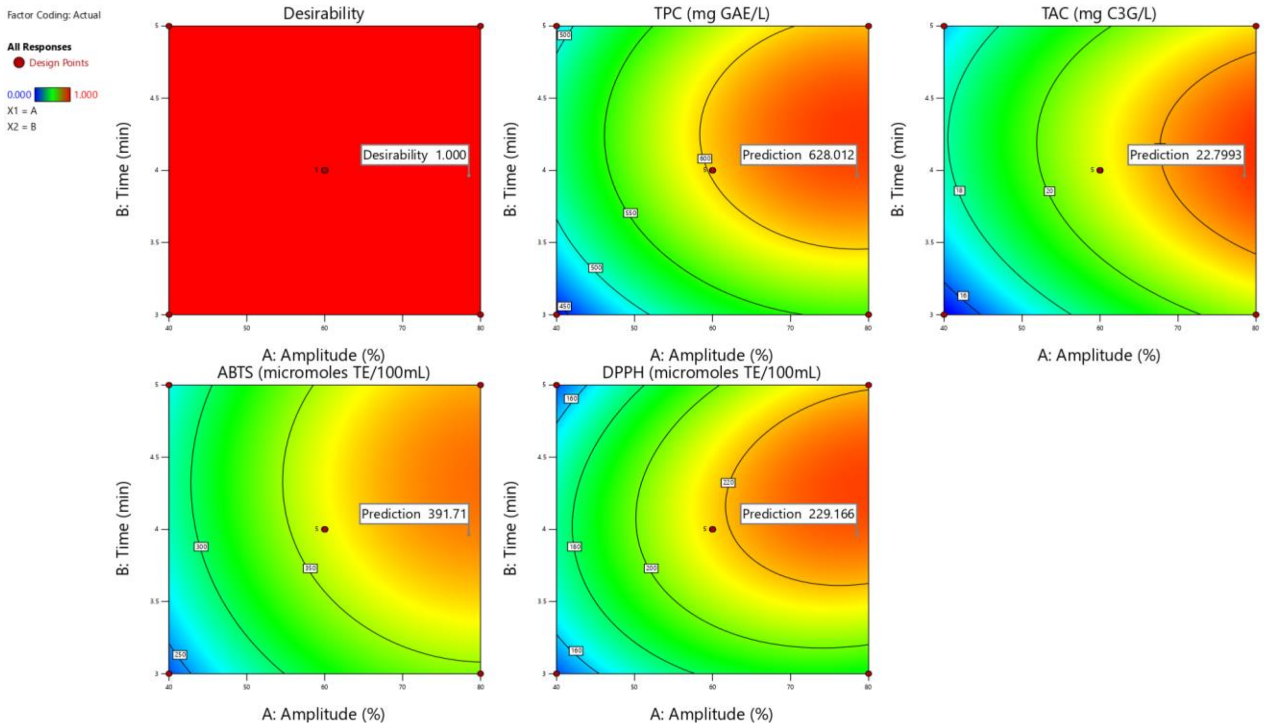


Figure 8.18 Contour plots indicating the predicted optimal values for all responses (predicted parameter values for an amplitude of 78.50% and a treatment time of 3.96 minutes). CI (%) for CTP (612.715; 643.31), for CTA (22.4769; 23.1216), for ABTS (366.732; 416.689), and for DPPH (218.104; 240.227).

Testing the experimental model refers to estimate the effects of ultrasonication in the early stages of vinegar production, specifically the pre-maceration treatment of crushed fruits, to estimate the impact of kinetic color and phenolic compound extraction, while also evaluating the potential for reducing maceration-fermentation times. Tests conducted immediately after ultrasound treatment showed an increase of total polyphenol content and monomeric anthocyanins, which was more evident as the treatment time and amplitude percentage increased.

The research results present data on the content of phenolic compounds and anthocyanins correlated with antioxidant capacity (determined by two types of tests) of various types of vinegars obtained from blueberry, blackberry, and raspberry juices used in different percentages.

The activity of collecting of free radical as ABTS and DPPH may be attributed to phenolic substances and anthocyanins, which are present in significant concentrations in vinegar variants obtained from 60% berry fruit juices inoculated with 0.5% acetic bacteria. The highest values of these compounds were identified in the blueberry vinegar obtained from the substrates with the percentages mentioned above.

The values of collecting activity of ABTS and DPPH were significantly different among the tested vinegar variants ($p < 0.05$); the variant with the highest CTP and CTA values presenting the greatest percentage of free radical inhibition.

The results indicated that all vinegar variants obtained from blueberry and blackberry juices showed good antioxidant potential, higher than that of apple vinegar.

Berry fruit vinegars represent an interesting source of phenolic compounds and anthocyanins and could contribute to the daily intake of antioxidants. These biologically active compounds with potential functional and antioxidant benefits could also improve the sensory properties of vinegar. The data presented in this study could be of interest to nutritionists, food scientists, and food producers.

Experimental results indicated that vinegar samples obtained using 0.5% acetic bacteria and 60% berry fruit juices in the substrate had the highest average values for CTP, CTA, and antioxidant activity. However, all these variants had the lowest values for the acetic acid content. A significant positive correlation was noted between CTP, CTA and antioxidant capacity, and a negative correlation between CTP, CTA and acetic acid content in vinegar samples. The highest acetic acid content (4.94%) was identified in vinegars produced from substrates containing 40% raspberry juice and inoculated with 1% acetic bacteria, while vinegars obtained from substrates with 60% blueberry juice inoculated with 0.5% bacteria exhibited the highest CTP (180.83 mg GAE/L), CTA (96.87 mg/L), and antioxidant potential (65.90% DPPH radical inhibition, 1.54 mM TE/L).

Chapter 9: Implementation of food safety management system with ultrasound treatment as a processing step in vinegar production

9.1 Establishment of preliminary operational programs (Good Manufacturing Practices - GMP and Good Hygiene Practices - GHP) for vinegar production

Preliminary programs will be implemented considering the context, space size, and activities carried out. These preliminary programs will be established prior to conducting a hazard analysis. The results of a safety and quality risk analysis in the vinegar production process are presented. This analysis was developed according to HACCP principles, identifying risks associated with each stage of the process. Preventive measures are proposed to manage these risks, including potential critical factors and the establishment of critical limits or necessary control measures. The focus of the analysis was on identifying critical control points (CCPs) for safety and critical points (CPs) for quality in vinegar production. For each identified CCP or CP, critical factors and necessary control actions are specified to ensure compliance and quality of the final product.

Chapter 10: General conclusions and summary of contributions

1. Personal and Original Contributions

2. Future Development Directions

3. Final Conclusions

1. Personal and original contributions

In line with the thesis title, the general objective of this research is to identify technological and managerial factors applicable to ultrasound treatment in vinegar production, factors that lead to increased extraction of biologically active compounds and reduced processing times.

Scientific contributions can be evaluated through the author's input from various research perspectives, which are concretized in:

A. Synthesis contributions:

1. Identification of the current state of research in the field of fermentative food technology;

2. Identification of the most relevant scientific works in the field of fermentative food technology, specifically vinegar technology, published in specialized literature, and the most significant trends in the field through bibliographic analyses performed in international databases;

3. Analysis of fermentation in food industry as an ecological alternative for improving nutritional value;

4. Analysis of the advantages of food fermentation technologies, including environmental and health benefits;

5. Critical analysis of fermented products and their relationship with health;

6. Analysis of the recovery of food waste through fermentation, presenting a future perspective on fermentation.

B. Theoretical and experimental contributions:

1. Analyzing published scientific articles on vinegar technology, grouping six research directions: microorganisms involved, chemical composition, health benefits, production technologies, adjunctive medicines, and vinegar residues;

2. Demonstrating the potential of using ultrasound in vinegar technology and highlighting that previous research on the effects of sound waves was conducted at a laboratory level or using pilot devices, with ultrasonication parameters not systematized and optimized;

3. Determining the content of phenolic compounds and anthocyanins correlated with antioxidant capacity (determined by two types of tests) of various types of vinegar obtained from blueberry, blackberry, and raspberry juices used in different percentages;

4. Research on evaluating the total polyphenol content (TPC), total anthocyanin content (TAC), and antioxidant capacity (using ABTS and DPPH free radicals) of experimental vinegar types obtained through the fermentation process juices from berries, treated with ultrasound;

5. Designing, testing, and analyzing the experimental model for increasing the extraction of biologically active compounds in fruit juice using DESIGN-EXPERT® VERSION 12 TRIAL software to

optimize the number of experiments and operating conditions and to obtain maximum information with the minimum number of samples;

6. Identifying and selecting the solution to maximize the extraction of total polyphenol content using the optimization function of the design software: amplitude 78.50%, treatment time 3.95 minutes. The TPC values were the highest (643.2 mg GAE/L);

7. Identifying and selecting the solution to maximize anthocyanin content using the optimization function of the design software: amplitude 80%, treatment time 4 minutes. The highest TAC value was 23.29 mg C3GE/L;

8. Performing ANOVA analysis for process optimization to increase ABTS and DPPH levels. At the end of the optimization, ABTS was found to be 363.69 (μmol TE/L) and DPPH 217.102 (μmol TE/100 mL) with a treatment time of 3.96 minutes and amplitude of 78.50%;

9. Evaluating the possibility of reducing maceration-fermentation time by identifying samples that recorded the highest values for total polyphenol and anthocyanin content at the end of maceration;

10. Specifying the significant influence of the predictors used (amplitude %, treatment time, and maceration duration), both individually and in interaction, on the model;

11. Outlining the possibility of using ultrasound treatment in a vinegar production unit due to the increased rate of extraction of biologically active compounds and antioxidant activity, as well as the potential for reducing maceration-fermentation duration;

12. Managing ultrasound treatment as an additional technological operation that can be integrated into the vinegar production process and identifying ultrasound treatment as a critical control point (CCP) due to the risk of acoustic amplifier damage (safety risk);

13. Highlighting the use of ultrasound treatment as a solution to improve management in vinegar production technology by reducing maceration-fermentation time, enhancing the quality of final products, and obtaining an innovative product - vinegar with enhanced nutritional and sensory characteristics.

C. Contributions with a curriculum scientific nature

1. The current state of research in fermentative food technologies, namely vinegar production;

2. Preparation of scientific research reports within the doctoral research program;

3. Completing the PhD thesis.

D. Novelty of the doctoral thesis

The novelty of the doctoral thesis consists of:

1. Thematic and object of theoretical investigations;

2. Comparative analysis of fruit vinegar production technologies;

3. Establishing optimal ultrasound parameters for maximizing polyphenol and anthocyanin extraction.

E. Practical utility of research results

The research results provide scientific, educational, and practical utility, as highlighted by the contributions made and the following aspects:

- Scientifically, the studies contribute significantly to fundamental knowledge by developing and deepening research on the use of ultrasound in food industry;
- Educationally, the results are of interest and utility both in terms of the results themselves and as well as of the research methods and procedures applied;
- Practically, the research provides a scientific basis for knowledge accumulated through practical experience and offers concrete solutions regarding optimal ultrasound conditions for efficient extraction of anthocyanins and polyphenols from fruits;
- From the applicative point of view, the research brings on the one hand a scientific foundation of the knowledge gained through practical experience, and on the other hand it offers concrete solutions regarding the optimal ultrasonication conditions for an efficient extraction of anthocyanins and polyphenols from fruits.

F. Valorization and dissemination of research results in the scientific academic environment

The valorization and dissemination of research results in the scientific academic environment have been achieved through:

- Publication of 7 scientific papers and articles as a first and co-author in scientific journals and international conferences

Publications in ISI Journals:

1. **Cristina Padureanu**, Carmen Liliana Badarau, Alina Maier, Vasile Padureanu, Mirabela Ioana Lupu, Cristina Maria Canja, Geronimo Raducu Branescu, Oana-Crina Bujor, Florentina Matei, Mariana-Atena Poiana, Ersilia Alexa, Anisor Nedelcu, ULTRASOUND TREATMENT INFLUENCE ON ANTIOXIDANT PROPERTIES OF BLUEBERRY VINEGAR, Fermentation 2023, 9(7), 600; https://doi.org/10.3390/fermentation9070600, (FI 3,3)
2. Alina Maier, Vasile Padureanu, Mirabela Ioana Lupu, Cristina Maria Canja, Carmen Liliana Badarau, **Cristina Padureanu**, Ersilia Alexa, Mariana-Atena Poiana, OPTIMIZATION OF A PROCEDURE TO IMPROVE THE EXTRACTION RATE OF BIOLOGICALLY ACTIVE COMPOUNDS IN RED GRAPE MUST USING HIGH-POWER ULTRASOUND, Sustainability 2023, 15(8), 6697; https://doi.org/10.3390/su15086697, (FI 3,3)
3. Mirabela Ioana Lupu, Cristina Maria Canja, Vasile Padureanu, Adriana Boieriu, Alina Maier, Carmen Liliana Badarau, **Cristina Padureanu**, Catalin Croitoru, Ersilia Alexa, Mariana-Atena Poiana, INSIGHTS ON THE POTENTIAL OF CAROB POWDER (CERATONIA SILIQUA L.) TO IMPROVE THE PHYSICO-CHEMICAL, BIOCHEMICAL AND NUTRITIONAL PROPERTIES OF WHEAT DURUM PASTA, Appl. Sci. 2023, 13(6), 3788; https://doi.org/10.3390/app13063788, (FI 2,2)
4. Alina MAIER, Vasile PADUREANU, Mirabela LUPU, Cristina CANJA, Geronimo Raducu BRANESCU, **Cristina PADUREANU**, Mariana-Atena POIANA, Effect of High-Power Ultrasound Treatment on Bioactive Compound Content and Chromatic Characteristics of Red Wines, Vol. 81 No. 1 (2024): BULLETIN OF UNIVERSITY OF AGRICULTURAL SCIENCES AND VETERINARY MEDICINE CLUJ-NAPOCA. FOOD SCIENCE AND TECHNOLOGY, https://journals.usamvcluj.ro/index.php/fst/article/view/14845
5. Alina MAIER, Vasile PăDUREANU, Cristina CANJA, Mirabela Ioana LUPU, Geronimo Răducu BRĂNESCU, **Cristina PĂDUREANU**, Mariana-Atena POIANĂ, **Influence of High-Power Ultrasound Treatment on Red Wine Quality Parameters,** Scientific Papers. Series B. Horticulture, Vol. LXVIII (1), p.X-Y, (În Horticulture Section of the International

Conference"Agriculture for Life, Life for Agriculture",); https://horticulturejournal.usamv.ro/index.php/scientific-papers/current-issue, (FI 0,3)

Publications in BDI

1. **Cristina Padureanu**, Alina Maier, Cristina Maria Canja, Vasile Padureanu, Oana-Crina Bujor, VALORISATION OF GRAPE MARC BY-PRODUCTS IN BEVERAGE INDUSTRY: THE CASE OF FORTIFIED GRAPE JUICE, Proceedings of 22nd International Multidisciplinary Scientific GeoConference SGEM 2022, Volume 22, doi: 10.5593/sgem2022V/6.2/s25.46
2. **Cristina Padureanu**, Anișor Nedelcu, Alina Maier, Vasile Padureanu, Mirabela Ioana Lupu, Carmen Liliana Badarau, THE TOTAL CONTENT OF POLYPHENOLS AND THE ANTIOXIDANT PROPERTIES OF SEVERAL BERRY VINEGARS, Bulletin of the Transilvania University of Braşov, Series II: Forestry ◘ Wood Industry ◘ Agricultural Food Engineering ◘ Vol. 15(64) No. 1 – 2022; https://doi.org/10.31926/but.fwiafe.2022.15.64.1.11

- Preparation of scientific research reports as part of the scientific training program;
- Completion of the PhD thesis.

3. Final Conclusions

Eligible target group for this research includes:

- Researchers
- Engineers and technicians working in the field of fermentation food technology
- Professionals responsible for improving food technology management systems
- Engineers developing equipment for food industry
- Other interested parties

Fermented functional foods have emerged as a subsector of functional food industry, which has steadily grown in recent years. Fermentation has many potential health benefits such as: the supply of beneficial lactic acid bacteria, helps metabolism, increased nutritional availability, improved mood and behavior, but also benefits for cardiovascular health.

Dairies and meat are the most popular fermented products widely consumed. On the other hand, alongside dairy and meat, fermented fruits and vegetables are well known for their worldwide consumption and health benefits.

The awareness of benefits of the alternative proteins has recently expanded the consumption of fermented products that have as basic ingredients insects and seaweed. Furthermore, to maximize the bioconversion efficiency of food waste, it is necessary to optimize food waste and the use of cutting-edge biotechnological methods.

Innovative techniques, such as precision fermentation, are suitable for producing desired proteins, lipids, and carbohydrates, as they allow generation of molecules with similar compositions with their homologues conventionally grown.

In the future, fermentation industries will operate continuously with automation, leading to reduced human involvement and errors prevention. In future fermentation processes, inexpensive materials such as plastic, cement, or ceramics may replace steel.

Artificial Intelligence will be responsible for controlling fermentation processes, thus providing more efficient, sterile processes with reduced energy, water, and labor requirements.

Another future research dirtection could be the use of innovative technologies such as high hydrostatic pressure, ultrasound, microwaves, pulsed electric fields, etc., and their effects on the production of high-quality fruit vinegar. Some of these have been tested for grape vinegar production, and the results have been promising.

The goal of this research was to test ultrasound technology on different fruit varieties, potentially grown in various regions, to observe the influence of ultrasound on the extraction process of different compounds during vinegar production.

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