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**AN INTEGRATIVE REVIEW OF ANALYTICAL
TECHNIQUES USED IN FOOD AUTHENTICATION: A
DETAILED DESCRIPTION FOR MILK AND DAIRY
PRODUCTS**

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An integrative review of analytical techniques used in food authentication: a detailed description for milk and dairy products

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Orientadora: Djenaine de Souza

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“O correr da vida embrulha tudo, a vida é assim: esquenta e esfria, aperta e daí afrouxa, sossega e depois desinquieta. O que ela quer da gente é coragem.” – Guimarães Rosa

An integrative review of analytical techniques used in food authentication: a detailed description for milk and dairy products

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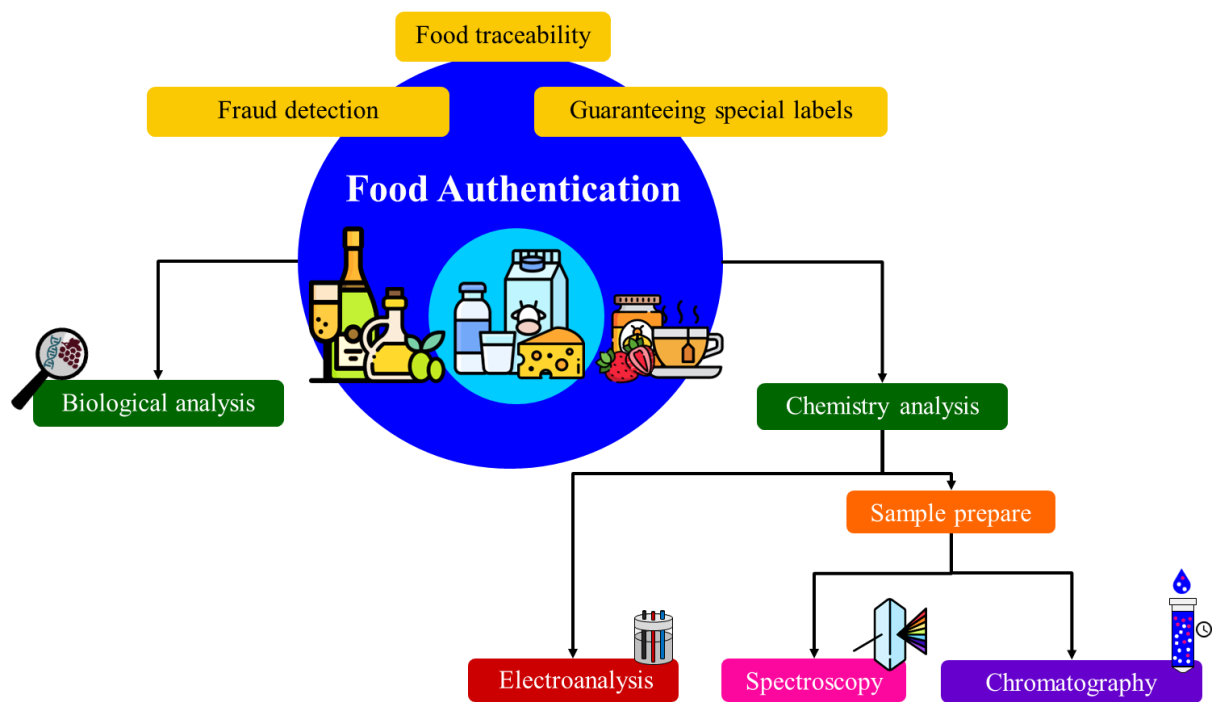
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Graphical abstract



Highlights

- Importance of authentication analysis of foods and beverages in food safety.
- Detection of adulteration and evaluation of traceability in foods and beverages.
- Overview of the use of analytical methods in food authentication analysis.
- Analytical techniques used in the authentication of milk and dairy products.
- Overview of adulteration and traceability in milk and dairy products.
- Perspectives in traceability and adulteration detection in foods are provided.

Abstract

In the last three decades, the research in food science and food authentication has increased due to numerous discoveries about nutritional needs, toxicities of food components, and the risks associated with improper nutrition and food adulteration. All research results followed the implementation of specific laws about the food components, evaluation of authenticity, and geographic origin. This way, the use of suitable analytical techniques for the identification and quantification of food components, as well as the detection of adulteration, falsification, deliberate substitution, and mislabeling of foods, have great importance in the industrial, scientific, and legislative context. This work is an integrative review of analytical techniques used in food authentication, indicating advantages and disadvantages as well as perspectives for direct, quick, and simple applications, guaranteeing the application of quality standards and ensuring food safety for consumers. Furthermore, this work shows that most previous papers published report the analysis of natural foods to evaluate the origin (traceability) and industrialized foods to detect adulterations and fraud. A focus on research to detect adulteration in milk and dairy products is presented due to the importance of these products in the nutrition of the world population. The main advances and the analytical approaches for the employ of modern analytical techniques in the identification and quantification of milk and dairy products adulterations were discussed, indicating trends and perspectives on food authentication.

Keywords

Foods Authentication; Foods Geographic Origin; Foods Adulteration, Analytical Detection, Milk and Dairy Products.

11. General Considerations

Food authentication is an essential step demanded by the food industries, legislative and supervisory agencies, scientific institutions, and consumers since it has occupied an important place in food safety, allowing the distribution of food and supplies between different parts of the world, guaranteeing consumers access to good quality food, and improving public health, since good quality food and with adequate nutritional value reduces the risks to consumers health (Galanakis, 2021). In addition, the worldwide increase in food production, with rapid and low-cost production, has resulted in fragile networks that are vulnerable to food fraud, and guarantee the authenticity and safety of food has become essential to ensure the health and quality of food on the market (Kitpipit et al., 2013).

The food authentication definition is related to procedures capable of evaluate the quality and verify if the specification of a given food complies with the label description. In addition, authentication makes it possible to confirm the origin, whether related to species, geography, or genetics and evaluate the adequate production method and the technologies used in the processing. Furthermore, authentication analysis by proper analytical procedures enables the identification of frauds such as adulteration, mislabeling, characterization, and misleading origin, which indicate crimes against public health and consumer faith (Danezis, Tsagkaris, Camin, et al., 2016; Spink, 2019; van Leeuwen et al., 2014).

Unfortunately, milk adulteration is a common practice in various countries, which results in health problems and death in humans (Ionescu et al., 2023). Milk is a substance of extreme nutritional importance due to its abundant source of high-quality proteins, including various amino acids essential in the growth and development of humans. For this, milk is a primary source of sustenance, providing the human body with many essential nutrients vital for maintaining optimum health (Cossignani et al., 2019). Rich in calcium, lipids, and saccharides, including lactose, an important energy source, milk can come from various animal sources. As a result of multiple factors, including the animal's diet and breed, as well as the methodologies employed during the processing phase, the nutritional composition of milk can fluctuate and differ. For this, some producers promote adulteration for more excellent added value and profit (Mafra et al., 2022; O'Sullivan et al., 2022).

Milk and dairy products are the main target samples when adulteration and fraud detection methods are developed. Furthermore, milk and dairy products require adequate identification of origin related to species due to religious or social habits. So, the authentication of milk and dairy products presents high scientific, industrial, and social relevance and has been searched in different fields such as Chemistry, Food Science, Medicine, and others.

2. Scope and approach

Food authentication requires interdisciplinary and intersectoral approaches and analytical detection methods that provide the feasibility of the necessary assessments and safety management and monitoring systems for all stages of the food production and supply chain (Spink, 2019).

In practice, the food authentication strategies involve the analysis and determination of compounds present and, mainly, some tracer compounds, followed by a comparison of obtained values with those established for genuine material. However, some aspects of food authentication present high complexity and require approaches and analytical tools more specific, among them the identification of geographic, genetic, or species origins, the organic or free-range agricultural management systems, or the application of irradiation, freezing, microwave heating as processing technologies (Danezis, Tsagkaris, Camin, et al., 2016).

Thus, this work aims to provide an integrative review with a current analytical approach for food authentication, emphasizing the use of analytical techniques to identify adulteration and perform the traceability of chemical components in processed and non-processed foods, discussing the advantages and disadvantages of previously published research. For this, relevant studies on analytical techniques, such as spectroscopic, chromatographic, and electroanalytical, used to detect fraud and verify the authenticity of foods were searched and highlighted. These data were obtained by a systematic search in *Science Direct*[®] and *Scopus*[®] databases using the keywords “*authentication*” AND “*food*”, “*authentication*,” AND “*beverage*”, from published papers from 2013 to 2023. Review articles were searched with the same search tools used for the analytical techniques published in the chemistry and food analysis field, which were employed in the bibliometric analysis to evaluate the field of research that is the focus of this article.

Special attention is paid to the authentication of milk and dairy products since they are essential food sources for children, sick people, and seniors, who are also commonly

involved in adulteration crimes involving foods. In the bibliometric analysis of milk and dairy products, the keywords used were “*authentication*” AND “*milk*”, “*authentication*” AND “*dairy products*”, “*traceability*” AND “*milk*”, “*traceability*” AND “*dairy products*”, “*adulteration*” AND “*milk*” and “*adulteration*” AND “*dairy products*”, considering secondary studies, without language limitation, from published papers from 2013 to 2023.

3. Food authentication

Food authentication presents intense scientific importance because it allows the evaluation of the composition, reliability, and guaranteed food safety for consumers and provides manufacturers, legislators, and consumers the certification of the origin and quality of foods and beverages. In practice, authenticity is performed to verify a few properties, such as if the description of the products meets the requirements of its legal denomination, or if others have not replaced the ingredients with lesser economic value to increase the profit from marketing, or evaluate the processing employed in its fabrication, or verify if the designation of origin, geographical or species origin is true (Montet & Ray, 2017; Qian et al., 2020).

In this context, some scientific and governmental organs developed guidelines for evaluate food quality, including authentication for fraud detection and traceability for evaluation origins. The Food Chemicals Codex (FCC) presents guidelines and technical specifications for asses the identity, quality, and purity of around twelve hundred food ingredients, and the United States Pharmacopeia (USP) provides reference materials to support these specifications. This way, any food ingredient legally marketed worldwide can be included in FCC. For this, it is recognized as the international standard used by other official food authorities worldwide, including Brazil and other countries of the Southern Common Market.

Codex Alimentarius (Food and Agriculture Organization (FAO) of the United Nations, 2023), created in 1983, has developed international harmonized food standards, guidelines, and codes of practice to protect consumer health and ensure equivalent practices in the food trade, which are internationally recognized and often form the basis for national legislation. *Codex Alimentarius* also presents guidelines to evaluate the food safety of ingredients or raw materials used to produce foods. Finally, the FSSC 22000 is a certification system with technical specifications for food safety specific to the food production sector, based on the internationally acclaimed ISO 22000 standard and

complemented by technical standards such as ISO TS 22002-1, considered for food manufacturing and ISO TS 22002-4, for packaging manufacturing. In addition, it is fully compatible with other standards, such as ISO 9001 and ISO 14001, which include alignment with high-quality production.

All over the world, milk and milk products represent a vital food class, consumed mainly by children and older adults due to their nutritional properties. In addition, milk is an essential ingredient in food production and is often the most common target of adulteration for economic reasons. The authentication of milk and dairy products is necessary to identify these adulterations and assess the animal origin of the milk since milk from undeclared species can pose health risks due to the presence of allergens. In addition, some populations have religious practices or personal choices that prevent them from drinking milk from specific species (Mafra et al., 2022). For this, governmental organs have several guidelines for assessing the authenticity of milk and dairy products.

The European Union, through Commission Regulation (*Commission Delegated Regulation (EU) 2020/692 of 30 January 2020 Supplementing Regulation (EU) 2016/429 of the European Parliament and of the Council as Regards Rules for Entry into the Union, and the Movement and Handling after Entry of Consignments of C*, 2020), establishes the conditions for the introduction of raw milk and milk products intended for human consumption into the European Union. Furthermore, the FAO also establishes the guidelines for export certificates of milk and dairy milk in the international trade that meet food safety and suitability requirements (Food and Agriculture Organization (FAO) of the United Nations, 2001, 2008). In Brazil, the Regulations for the Industrial and Sanitary Inspection of Products of Animal Origin (RISPOA) (*Decreto Nº 9.013, de 29 de Março de 2017 - Regulamenta a Lei Nº 1.283, de 18 de Dezembro de 1950, e a Lei Nº 7.889, de 23 de Novembro de 1989, Que Dispõem Sobre a Inspeção Industrial e Sanitária de Produtos de Origem Animal.*, 2017; Ministério da Agricultura e Pecuária, 2024) establish that the inspection of milk and dairy products covers everything from the health of the herd, obtaining the raw material, its analysis, and selection to the dispatch of the final product.

3.1. Food authentication for fraud detection

The research on food fraud has high relevance in food science, food chemistry, and criminology areas due to the adulteration, counterfeiting, substitution, and deliberate mislabeling of products can result in health problems for consumers and is characterized

as crimes against public faith and disrespect to general and specific legislations in each country (Spink, 2019). Besides, food and beverage fraud represents an economic problem and affects consumer confidence and the credibility of segments of the food industry, and in more extreme cases, can damage the export of products and the global economy.

Economically, food fraud cases are linked to the financial gain promoted by adulterations, carried out to improve the perception of the quality of products, or copy an established brand, or reduce the cost of manufacture or extend the shelf life of products (Abbas et al., 2018). This way, food fraud represents a preoccupation of legislators and the scientific community that evaluate mechanisms for prevent and identify food adulteration and other practices that could mislead consumers, who have the right to truthful information about their food (Montet & Ray, 2017).

Most adulterants are unknown and difficult to recognize using typical targeted screening methods, so food adulteration is a growing challenge for food manufacturers and analysts. The industry searches daily for non-targeted screening methods for food samples to provide proof of origin and prevent deliberate or accidental undeclared additions to these samples. Consumers, producers, and regulatory agencies have, since the 1980s, validated the authentication of food products as an essential quality criterion. Food inspection agencies consider authentication tests to be an important characteristic of food quality, allowing for the possibility of detection attempts for food fraud (Roberts et al., 2022). Already, consumers require transparency about the origin and content of their foods and beverages. In addition, food authentication represents a new starting point for addressing the issue of food integrity.

Fast and specific methods for detecting adulteration, verifying quality, and guaranteeing geographical origin and the type of production of food products are now widely sought for effective and safe application (Esteki, Simal-Gandara, et al., 2018). Efforts so far have ranged from very effective to ineffective, and a new approach will help assess and simplify what has become highly fragmented and disjointed (Barrere et al., 2020). Unlike food fraud, which the industry sees as unfavorable, food authentication can be positively positioned. If the food industry can prove that food is authentic, brand value, reputation, and profits can be enhanced (Theodoridis & Kraemer, 2018).

Most countries have specific legislation on food fraud. In Brazil, the legal basis for combating food fraud is described in Law 6437/1977 which configures infractions of federal sanitary legislation, establishes the respective sanctions and makes other

provisions (*Lei Nº 6.437, de 20 de Agosto de 1977. Configura Infrações à Legislação Sanitária Federal, Estabelece as Sanções Respectivas e Dá Outras Providências*, 1977).

Some scientific publications report that milk has been the most common target of fraud in raw materials for the food industry or even as a staple food for the nutrition of children and adults (Gimonkar et al., 2021; Handford et al., 2016). The most common types of fraud involve the addition of illegal products to increase the milk quantity (by addition of water and urea), or the reuse of deteriorated milk (by addition of caustic soda, hydrogen peroxide, and virgin lime), or the increase of shelf life due to reduction in the bacterial multiplication promoted by illegal compounds addition (Amsaraj et al., 2021; Balan, Dhaulaniya, Jamwal, Yadav, et al., 2020). These frauds can result in health problems and a loss of credibility in food industries, affecting product image and consumption of essential nutritional food (Breitenbach et al., 2018).

The global issue of milk adulteration was highlighted by incidents such as the large-scale adulteration of infant milk formulas with melamine in China in 2008. These incidents have raised concerns about food authenticity and the need for continuous quality monitoring (Gossner et al., 2009). Substances such as sucrose are added to milk to improve the total solids content and taste, compensating for the loss caused by water adulteration. Although sucrose adulteration does not pose a direct risk to human life, it can significantly negatively affect the economy and is considered fraudulent. The availability of qualitative and quantitative methods to detect added sugars in milk emphasizes the importance of comprehensive testing to ensure food authenticity and prevent food fraud (Balan, Dhaulaniya, Jamwal, Yadav, et al., 2020). The fat content of milk is also commonly adulterated for economic gain, to improve sensory characteristics, and, in some cases, to increase the added value of some products compared to others, such as margarine, a product characterized by being produced with vegetable fats and which commonly has less added value, but which can be marketed as butter. Analytical techniques are a powerful and effective tool for identify this and other milk and dairy product fraud types.

3.2. Authentication as a tool for guaranteeing special labels

Food authentication verifies the integrity of the special seals and labels, which manufacturers or traders, misleading consumers, and current legislation can often alter. Products with signs of origin, such as Geographical Indication (GI) or Appellation of Origin (AO) labels (Mosoti, 2006), agricultural labels, such as Organic Production or

Traditional Specialties Guaranteed (TSG) claims (Bigot et al., 2017), and seals related to the carbon footprint generated by the product (Kühne et al., 2023), are examples of labels that can add value or even win consumer preference. In addition, nutraceutical aspects are also commonly highlighted on food labels to broaden the consumer audience (Azman & Zaleha Sahak, 2014). Although strict regulations and controls are applied to these products, they are easily subject to fraud.

Food fraud and counterfeiting are facilitated by industrialization and globalization since most products are transformed, even minimally, to increase their shelf life or add value or flavor to the food. These processes, in turn, make food more susceptible to fraud, and the mechanisms and forms of fraud are constantly being updated and innovated, making the analysis of food authentication increasingly complex and difficult, and impossible to carry out using classical analytical methodologies alone (Johnson, 2014; Qian et al., 2020; Suman et al., 2021).

Some foods present special labels to guarantee the absence of some components, such as milk and dairy products or meat, in Halal and Kosher diets due to the context of religious dietary laws. Besides, special labels indicating organic milk production can have high relevance for consumers with high preoccupation regarding the sustainable supply chain of organic milk and dairy products (Piao et al., 2021; Woś et al., 2022).

3.3. Authentication as a tool for food traceability

According to the International Food Authenticity Assurance Organization (FAAO), food authenticity is the process of irrefutably proving that a food or food ingredient is in its original, genuine, verifiable, and intended form, as declared and represented (Food and Agriculture Organization (FAO) of the United Nations, 2018, 2021). Therefore, food is considered authentic or genuine when the product or its contents match the original condition and label information. So, authenticity refers to the guarantee that a product is genuine and has not been falsified or adulterated. At the same time, traceability links a food product to its processing methods and ingredients.

In practice, traceability refers to the ability to follow the path of a product from raw materials to the final food or to know its production, handling, processing, packaging, and shipping process, and is widely practiced in the certified food industry (Islam & Cullen, 2021; Qian et al., 2020). Traceability guarantees consumers the quality and origin of the food, compliance with regulatory compliance and environmental sustainability, and the eating habits of a specific consumer group (Galanakis, 2021). Furthermore, it can also

be used to detect and resolve problems, such as product quickly recalls without safety conformity (Manning & Soon, 2016). It can be verified using various techniques, such as certifications, authenticity labels, barcodes, and marking technologies.

Today, consumers are interested in the origin of primary food ingredients such as milk and dairy products. Therefore, identifying the origin of the milk (cow, buffalo, sheep, goat, donkey, and other animal origins) is essential for the dairy industry and guarantees the origin and quality of the milk for consumers, as well as reliability in the industry and guaranteeing the proper diet for nutritional needs.

Different animal species, including cows and goats, can produce milk that varies in nutrient content and composition. For example, goat's milk has a higher fat content than cow's milk, providing more specific fat-soluble vitamins. In addition, goat's milk exhibits a distinct protein profile, characterized by a high proportion of specific types of protein when compared to cow's milk. In addition, the lactose content found in milk can differ between species, with cow's milk containing a higher lactose concentration than goat's milk. Furthermore, the species of origin can influence the existence of specific bioactive compounds in milk, such as various types of antibodies and biological growth factors. In essence (Bruschetta et al., 2021; Ehsani et al., 2023; Qin et al., 2022), the species of origin plays a critical role in determining milk's composition and nutritional attributes, indicating the importance of traceability analysis in milk and dairy products.

4. Analytical methods for food authentication

The main purpose of food authentication analysis is to guarantee that the composition, purity, origin, and food processing methods are accurate and the content is authentic, correct, and precise (Food and Agriculture Organization (FAO) of the United Nations, 2018; Theodoridis & Kraemer, 2018). Authentication analysis reveals substitution, dilution, concealment, and mislabeling. A wide range of technologies is available, depending on the food, its characteristics, and its complexity in composition.

Various analytical techniques are used in food authentication, including chemical techniques such as chromatographic, spectroscopic, and electroanalytical, biological such as enzyme-linked immunosorbent assay (ELISA) and DNA-based, including PCR and DNA sequencing (Kitpipit et al., 2013; Lo & Shaw, 2018; Rohman et al., 2020).

Chemical techniques are used to identify specific components and quantify their food levels. The improvements in technologies applied to instrumentation for chemical analysis allow the detection of trace elements in complex chemical composition samples,

such as foods (Danezis, Tsagkaris, Brusic, et al., 2016; Danezis, Tsagkaris, Camin, et al., 2016). For example, the number and level of fatty acid content or fatty acid profile of olive oil are measured using gas chromatography coupled with mass spectrometry to verify the authenticity and origin of the food.

On the other hand, some foods do not contain chemical markers that allow unequivocal identification and, for this, require a global approach based on measure and evaluate a standard of properties of the complete composition of the product, for example, the authentication of organic eggs or sustainable palm oil. So, biological techniques, such as DNA tests and biomarkers, can provide specific information about the genetic composition of the food and quantify its proportion in the sample and can be used to trace and guarantee the geographical authenticity of the food. It has credibility because regulatory agencies recognize it and do not damage the sample so that it can be used for other purposes.

Biological techniques have recognition from regulatory agencies and do not damage the sample, which can be used for other purposes, including chemical analysis or marketing. However, food authentication presents disadvantages such as the need for technical knowledge and specialized equipment, the time-consuming sample extraction process, the high cost of equipment and specific reagents, and the presence of other components in the food matrix, which can affect the accuracy of the results, and the fact that it is a time-consuming method (Danezis, Tsagkaris, Camin, et al., 2016; Lo & Shaw, 2018; Mialon et al., 2023; Qian et al., 2020).

4.1. A bibliometric analysis in food authentication techniques

In recent years, the bibliometrics analysis has been used as a suitable tool for the analytical and systematized preparation of review publications, in which all information is based on parameter metrics that map and describe a field or area of research (Ceylan & Ceylan, 2024; Cvetkoska et al., 2023; Waltman & Van Eck, 2012). Bibliometric analysis can be carried out in various ways, with the combination of quantitative and qualitative variables being the most robust for generating a faithful description of the current state of a scientific area. Bibliometric networks or scientific maps help visualize and analyze a wide variety of information within a specific theme (Perianes-Rodriguez et al., 2016). The preliminary use of Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) (Page et al., 2021) protocol helps with the initial assessment of the actual state-of-art research from different fields.

The systematic search in *ScienceDirect*[®] and *Scopus*[®] database using as keywords the combination of words “*authentication*” AND “*food*”, “*authentication*” AND “*beverage*”, “*traceability*” AND “*food*”, and “*traceability*” AND “*beverage*” in the range from 2013 to 2023, considering reviews and research publications, and without limitation of language, was initially performed generating a database of information with 449 publications. All papers found were used in the PRISMA protocol, following some adaptations for evaluate the actual research about the analytical techniques employed in food authentication and assisting the authors in the identification, screening, eligibility, and inclusion of papers in bibliometric review. The resultant PRISMA diagram, indicating the total number of papers found in each step, is shown in Figure 1.

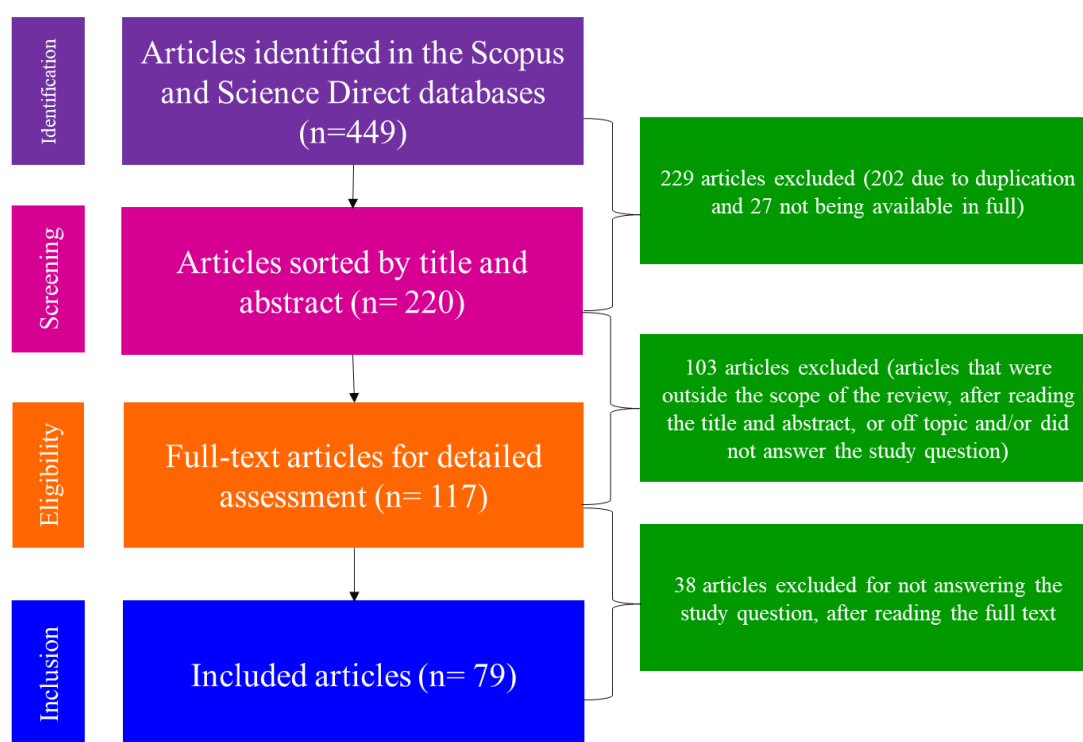


Figure 1: The PRISMA flow diagram used in the identification, screening, eligibility, and inclusion of review articles presented in this integrative review.

As shown in Figure 1, the first step (Identification) included all papers found with the search tools already described (449 papers found), followed by the second step (Screening), where the exclusion of repeated papers due to the use of two different databases (202 papers) or not available complete papers (27 papers) was performed. In the third step (Eligibility), after the lecture on the title and abstract of each paper, publications with no connection to the topic of interest were excluded. As an eligibility criterion, only studies that presented reviews on chemical and biological analytical methodologies applied to the authentication analysis, traceability, adulteration, and fraud

detection in food were screened. So, 103 papers were excluded because some of them dealt with traceability through physical labels without analytical technology, articles on management and administration strategies for traceability, or "smart packaging" with no association with chemical analysis of authentication food.

Finally, in the fourth step, the 117 papers selected were read entirely by authors, of which 47 were excluded because it were not within the scope of this paper (some experimental papers that were not filtered by the database filter and some reviews that deal with the more managerial aspect of food authentication), leaving only 79 papers to be discussed in the bibliometric review and shown in Table S1, Supplementary data.

The 449 papers found in the search of this work were analyzed using the VOSviewer tool (van Eck & Waltman, 2014) considering the co-occurrence variables for "indexed keywords", allowing a simple and basic visualization of bibliometric networks about food authentication (Sinha et al., 2021). The bibliometric networks produced by the VOSviewer tool consist of nodes and edges, of which nodes indicate quantity, so the larger the node, the greater the magnitude it represents, and edges represent connections between pairs of nodes (van Eck & Waltman, 2014). Furthermore, VOSviewer generates a table containing all quantitative information about the co-relation between data collected in the search (not shown) that were used in the interpretation of results, explained next.

Figure 2a shows the bibliometric network obtained by keyword search, where each keyword is represented by a node (rectangle with different colors), and the edges (lines with different colors) represent co-relation between keywords. The magnitude of a rectangle indicates the number of publications containing the corresponding term in their title or abstract. Keywords that frequently co-occur tend to be located close to each other within the visualization. VOSviewer categorized the terms into four groups, where only the group or cluster represented by the color orange appears to differ in the proportion of linked nodes (Donthu et al., 2021).

Furthermore, in Figure 2a, at the bottom right of the visualization, in red, called cluster 1 with 36 keywords, the standard scientometric terms in Food Analysis, such as food quality, quality control, chemical composition, and components related to food quality (fatty acid, vegetable oil, olive oil, plant oils, and chemical composition), which are represented by relatively small nodes, indicating the low incidence of reviews associating these keywords. Cluster 1 also contains keywords that indicate the analytical techniques most commonly used in food authentication, namely spectroscopic and

chromatographic techniques, with mass spectroscopy and chemometric techniques standing out, occurring 69 and 42 times, respectively.

In cluster 2, in green, with 36 keywords, represent the food most commonly listed, with samples of animal origin linked to the keyword's animal, meat, seafood, and milk, being the most cited, co-occurring, respectively, 41, 32, and 22 times. The indication of the most associated method in the indexed keywords refers to biological methods of authentication analysis, such as DNA, genomics, proteomics, polymerase chain reaction, and molecular biology.

Cluster 3, represented in blue, contains 34 keywords with the predominance of terms linked to adulteration identification and traceability in industrialized foods, such as fraud, authenticity, geographical origin, food products, food safety, crime, and accident prevention. Furthermore, keywords related to chemical analysis techniques, the third most common general term describing an analytical technique, are cited 30 times, behind only descriptors related to spectroscopic techniques and chemometric approaches. It is important to note that this cluster contains the keywords represented by the largest nodes, reinforcing that they are closely associated keywords. Furthermore, based on the approximation and density of the edges relating the terms in clusters 1 and 3, there is evidence of a strong association, i.e., reviews that used keywords from both clusters.

Finally, in cluster 4, represented in orange, 13 terms with strong co-occurrence are grouped. These include keywords related to stable isotope analysis and keywords related to samples of authentication interest, such as beverages and honey. The term geographical origins also appear in this cluster, indicating a strong correlation between reviews that have addressed this interest in food authentication and samples such as beverages and honey.

Figure 2b shows an overlay representation of the temporal progress of food authentication research. In this bibliometric network, the legend shows studies from 2013 to 2023 predefined in the search and collection of the articles used to build the database. The keywords represented in cooler tones and close to blue are more associated with studies from the beginning of the decade. In contrast, the keywords in warm tones and close to bright yellow represent keywords that appear in more recent studies. Interestingly, the general term authentication remains in the shade close to the green, indicating its recurring use. Among the terms in bright yellow, we can highlight procedures, which mention the development of new food authentication procedures, dairy products, chemometrics, spectroscopic techniques, and DNA barcoding. These terms may

386 indicate trends to be observed concerning food authentication. It is worth noting that
387 according to the bibliographic network, last year, there was a considerable volume of
388 reviews on food authentication publications in 2020.

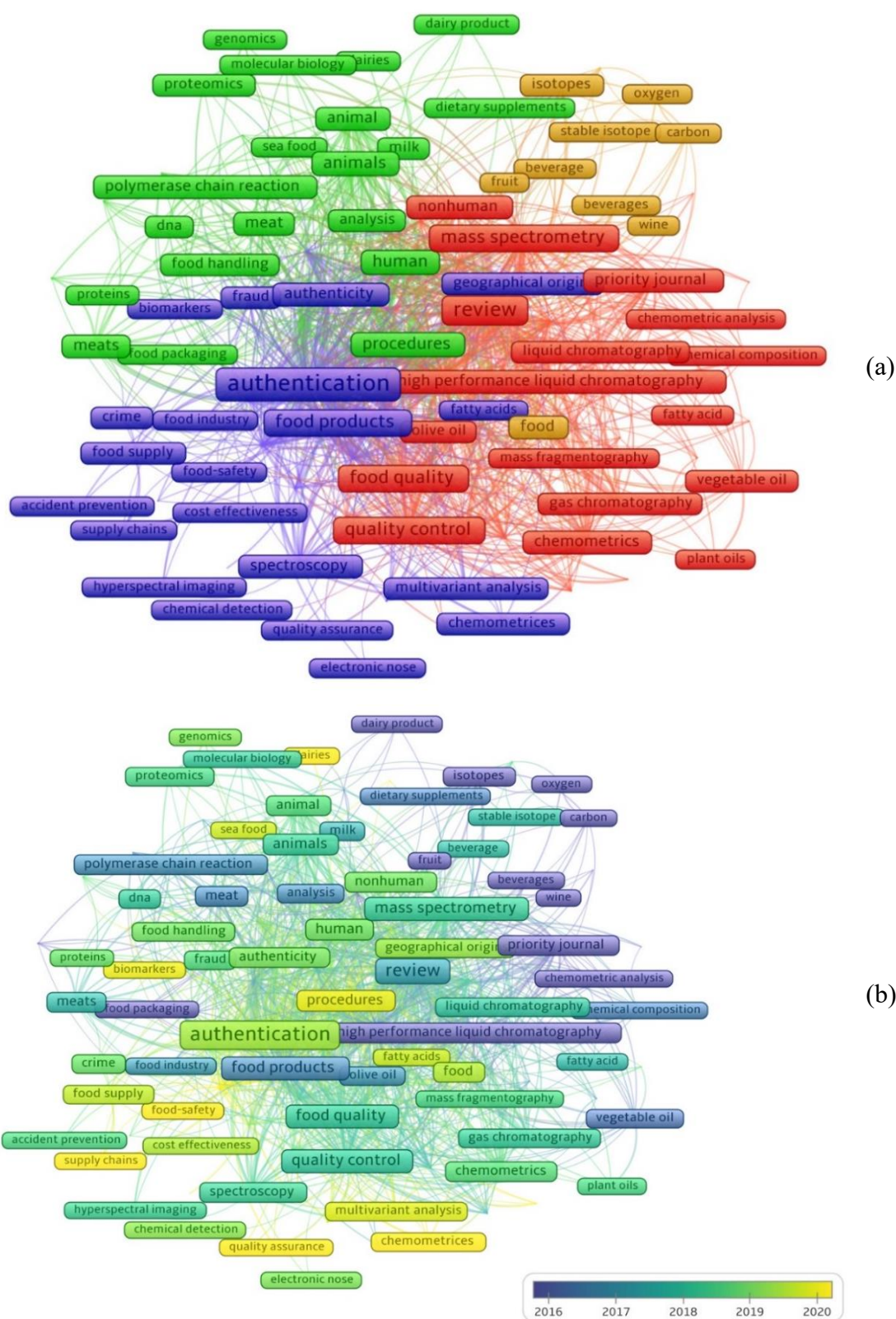
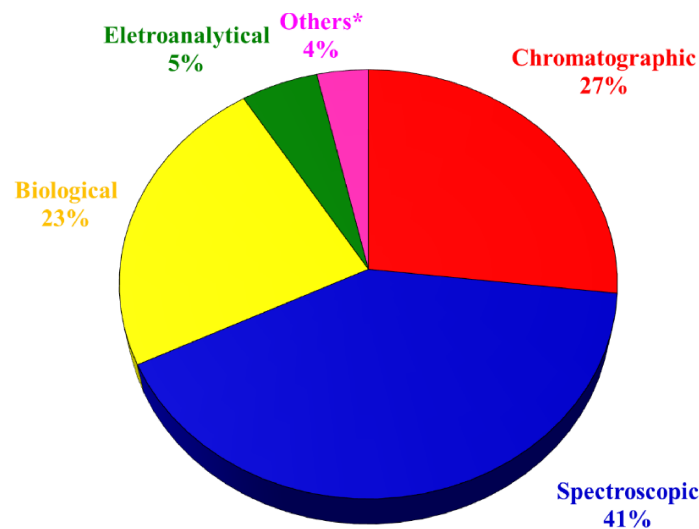


Figure 2: (a) VOSviewer visualization of a bibliometric network, where each node represents the number of times the keyword occurred, and lines between the nodes represent the co-occurrence between keywords. The bigger the node, the greater the occurrence of the keyword, and the thicker the link between nodes, the greater the occurrence of the co-occurrences between keywords. Each color represents a thematic cluster, wherein the nodes and links in that cluster can be used to explain the theme's (cluster's) coverage of topics (nodes) and the relationships (links) between the topics (nodes) manifesting under that theme (cluster). (b) the keywords indexed in nodes are represented according to the color temperature scale shown in the figure's legend to indicate the temporal evolution of the occurrence of the keywords. In this sense, warmer colors represent more recent areas of interest, and cooler color areas have been covered longer.

4.2. An overview of food authentication analytical techniques

The PRISMA diagram and bibliometric network derived VOSviewer tool with databases from the search in *Science Direct*[®] and Scopus[®], obtained according to the search tools described earlier, indicated 79 publications that revised (reviews publications only) the main techniques used in food authentication and/or applied these techniques in food authentication, according to the classification and distribution presented in Figure 3. Sometimes, the authors discussed more than one technique in the same review and presented applications in diversified types of samples, comparing the advantages, drawbacks, and applicability in food authentication analysis.



*Other techniques include calorimetric and gravimetric techniques.

Figure 3: Number of published papers in which food authentication techniques were reviewed by chromatography, spectroscopy, electroanalysis, biological, and other techniques. These data considered publications from *ScienceDirect*[®] and *Scopus*[®] in the years from 2013 to 2023 and were searched using the keywords "authentication" AND "food", "authentication" AND "beverage", "traceability" AND "food", and "traceability" AND "beverage".

As shown in Figure 3, 45 publications (41%) involved the employ of spectroscopic techniques such as fluorescence (O'Sullivan et al., 2022; Shi et al., 2020), Raman spectroscopy (Arroyo-Cerezo et al., 2021; Przykaza et al., 2021; Shi et al., 2020; Xu et al., 2020), mass spectroscopy (Castro-Puyana et al., 2017; Przykaza et al., 2021; Rohman et al., 2020; Shi et al., 2020; Valletta et al., 2021) and infrared spectroscopy (Moran et al., 2018; Przykaza et al., 2021; Shi et al., 2020; P. Wang & Yu, 2015; G. Zhang & Abdulla, 2022), which are, actually, analytical tools capable of detect and quantify traces of compounds even in small quantities of samples, resulting in specific information about the composition of food in a short space of time. Mass spectroscopy and infrared spectroscopy are helpful in adulteration detection, however, their disadvantages are high equipment costs, complicated analysis of the results, and the variation of compounds in the sample, which can alter the effectiveness depending on the type of food and the substances to be detected (Al-Naji & Zagrouba, 2020; Borràs et al., 2015; Galimberti et al., 2013, 2019; Montet & Ray, 2017).

Figure 3 shows that 30 publications (27%) discussed chromatographic techniques as suitable tools for food authentication. These techniques perform the separation of all the components of the sample according to physicochemical properties that allow differences in the time (retention time) in which each sample component passes through the column (stationary phase) when it is entrained by a mixture of organic solvents or inert gas (mobile phase). A suitable detector allows each sample component to be immediately identified and quantified with high precision and accuracy, even in small quantities. Chromatographic techniques more frequently reported were high-performance liquid chromatography with an ultraviolet-visible detector (Esteki, Shahsavari, et al., 2018), gas chromatography with a mass spectroscopy detector (Mialon et al., 2023), and ultra-performance liquid chromatography with a mass spectroscopy detector (Liu et al., 2022), among others.

The chromatographic techniques enable the selection of the most suitable method for food authentication due to its versatility, being highly applicable and recognized by the regulatory authorities. However, some disadvantages include the necessity of complex pretreatment and preparation of the samples before the chemical analysis, the high cost of reagents and equipment maintenance, and data interpretation requires a qualified and trained person (Fanali et al., 2016; Sherma & Rabel, 2018; van Leeuwen et al., 2014).

Among pretreatment of the food samples for food authentication analysis, in recent years, some procedures most effective have been developed food sector, which include cartridge solid phase extraction (CSPE), dispersive solid phase extraction (DSPE), online solid phase extraction (online SPE), pipette tip solid phase extraction (PT-SPE), magnetic solid phase extraction (MSPE), stir bar sorption extraction (SBSE), headspace solid phase microextraction (HS-SPME), direct SPME, membrane protected SPME and tube SPME, among others (J. Wang et al., 2023). Efficient sample pretreatment can avoid matrix interference and enrich targets. The extraction material plays a key role in these techniques, essential for increasing the accuracy and sensitivity of subsequent chromatographic detection techniques (Gonçalves-Filho & De Souza, 2022; Syafri et al., 2022).

Figure 3 shows that 23% of publications used biological techniques in food authentication analysis due to their versatility of applications and excellent selectivity, making them attractive and suitable for food authentication analysis (Agrawal et al., 2013). Some reports describe the application of DNA extraction and standard polymerase chain reaction (PCR) protocols for food authentication, presenting the critical role of small DNA markers with sufficient discriminatory power to enable authentication at the molecular level (Lo & Shaw, 2018). The main limitation of techniques based on the analysis of DNA markers is the need for robust methodologies that minimize the impact of DNA degradation and the insertion of inhibitors that can affect PCR amplification (Galimberti et al., 2013).

Amplified fragment length polymorphism (PCR-AFLP) and single-strand conformational polymorphism (PCR-SSCP) have been highlighted among the most widely used biological techniques for food authentication. Their applications can have some objectives, such as the authentication of foods claiming to be organic (Böhme et al., 2019), foods claimed to be consistent with halal production (Rohman et al., 2020), differentiation of animal species that give rise to meat products (Khalil et al., 2021), day products (Abbas et al., 2018; Baptista et al., 2021) and the so-called "blue foods" (Y.

Wang et al., 2023), and also vegetables and products derived from them (Francois et al., 2020; Wadood et al., 2020, 2022), authentication of foods with geographical indication appeal (Mialon et al., 2023; Singh et al., 2017). In addition, reports of the combined use of different techniques are standard to optimize the analytical results of authentication, associating the selectivity conferred by biological techniques with applications of electroanalytical techniques (Mohamad et al., 2022), chromatographic techniques (Saadat et al., 2022; Valdés et al., 2018) and spectroscopic techniques (Khalil et al., 2021; Wadood et al., 2022).

According to Figure 3, few publications (5 or 4.5%) reported electroanalytical techniques for food authentication. Electroanalytical techniques are based on measurements of electrical properties, such as current, potential, charge, and conductivity, which can be related to the identity and quantity of the compounds of interest. The use of voltammetric techniques, which involves the application of pulse potential difference and measurements of electrical currents, allows analytical results with sensitivity and sensibility similar to those obtained using chromatographic techniques (Almeida & De Souza, 2023; Martins et al., 2021; Megale & De Souza, 2023). So, some publications reported the use of voltammetric techniques in the food authenticity analysis (Arslan et al., 2021; Di Rosa et al., 2017; Liang et al., 2021; Mohamad et al., 2022; Negi et al., 2021; D. Zhang et al., 2024), but in a lower number than the observed of other types of techniques. Despite the simplicity of voltammetric analysis, the authors believe that a few publications were found, probably due to supervisory agencies not accepting these techniques as standard techniques, and food analysis requires official methods for identification and quantification of components and contaminants from raw materials and/or industrialized products.

Already in chromatographic analysis, the efficiency is related to the adequate choices of extraction methods for sample preparation (cow, buffalo, sheep, goat, donkey, and other animal origins or dairy products) in voltammetric analysis, the material used as a working electrode will affect the sensitivity, selectivity, precision, and accuracy in the analytical data. There are still no commercial electrodes capable of analyzing all the components in food samples with suitable analytical parameters.

Table S1 in *Supplementary data* presents some recent review publications in the field of Food Analysis, Food Chemistry, and Chemistry from bibliometric analysis of this work, which describe trends and perspectives in the use of the main analytical techniques for the analysis of food authenticity, including spectroscopy, chromatography, and

electroanalysis as essential tools in food traceability, fraud detection and the guarantee of special labels. So, was it possible to observe the predominance in the use of chromatographic techniques such as high-performance liquid chromatography, ultra-high-performance liquid chromatography, and gas chromatography, all using spectroscopic detectors (ultraviolet and visible, fluorescence, and mass spectrometry, among other) and extraction methods for sample preparation. Also, it can be observed that most publications reported the analysis of natural foods to evaluate the origin (traceability) and industrialized foods to detect adulterations and fraud.

4.3. Food authentication techniques: a comparison of performance and applicability

The reviews presented in Table S1, in Supplementary data, describe recent trends, perspectives, and approaches in the control of food quality and authenticity. The advantages, disadvantages, and applicability to different types of food can contribute to a more critical analysis of each technique's positive and negative points and relevance in choosing the best one for food traceability, fraud detection, and ensuring special labels are summarized in Table 1.

Table 1: Advantages and disadvantages of main techniques used in food authentication, according to reviews presented in Table S1, in Supplementary data, and discussed in this work.

Techniques	Advantages	Disadvantages
Chromatographic	It results in high precision, excellent sensitivity, versatility, wide application, and regulatory compliance.	It needs sample preparation steps and presents high cost, complexity, and long time for analysis.
Spectroscopic	It results in good sensitivity and specificity using chemometrics tools, versatility using reference materials, and analysis without pretreatment samples.	The instrumentation is expensive, and there is complexity in obtain reference materials due to matrix variation and limited application.
Biological	Are highly specific, with precise detection, applicability in traceability,	It presents high costs in instrumentation and biological materials and difficulties in the

	regulatory compliance, and are non-destructive.	stability of the results due to the complexity of the samples.
Electroanalysis	Voltammetry allows good- sensitivity results, has a great variety of materials for working electrode preparation, and minimized sample preparation.	Results depend on the working electrode materials used, and these techniques are not accepted by regulatory compliance.

Still, about the reviews presented in Table S1, in Supplementary data, the publications from 2013 to 2023 focused on analytical techniques for food authentication considering a wide variety of processed and non-processed foods, which 12% of the review focused on meat products (12%), followed by non-alcoholic beverages (11.5%), milk and dairy products (9.9%), alcoholic beverages (9.4%), and fish and seafood (8.4%), as shown in Figure 4. These foods are consumed worldwide and are of considerable importance in human nutrition. They even make up the diet of consumer groups sensitive to fraud and the quality problems that these foods may present, such as older adults and babies. Thus, proving the genuineness, quality, and safety of these foods is very important, and this is one of the factors that led these foods to be the most evaluated and compiled in the reviews analyzed in this study.

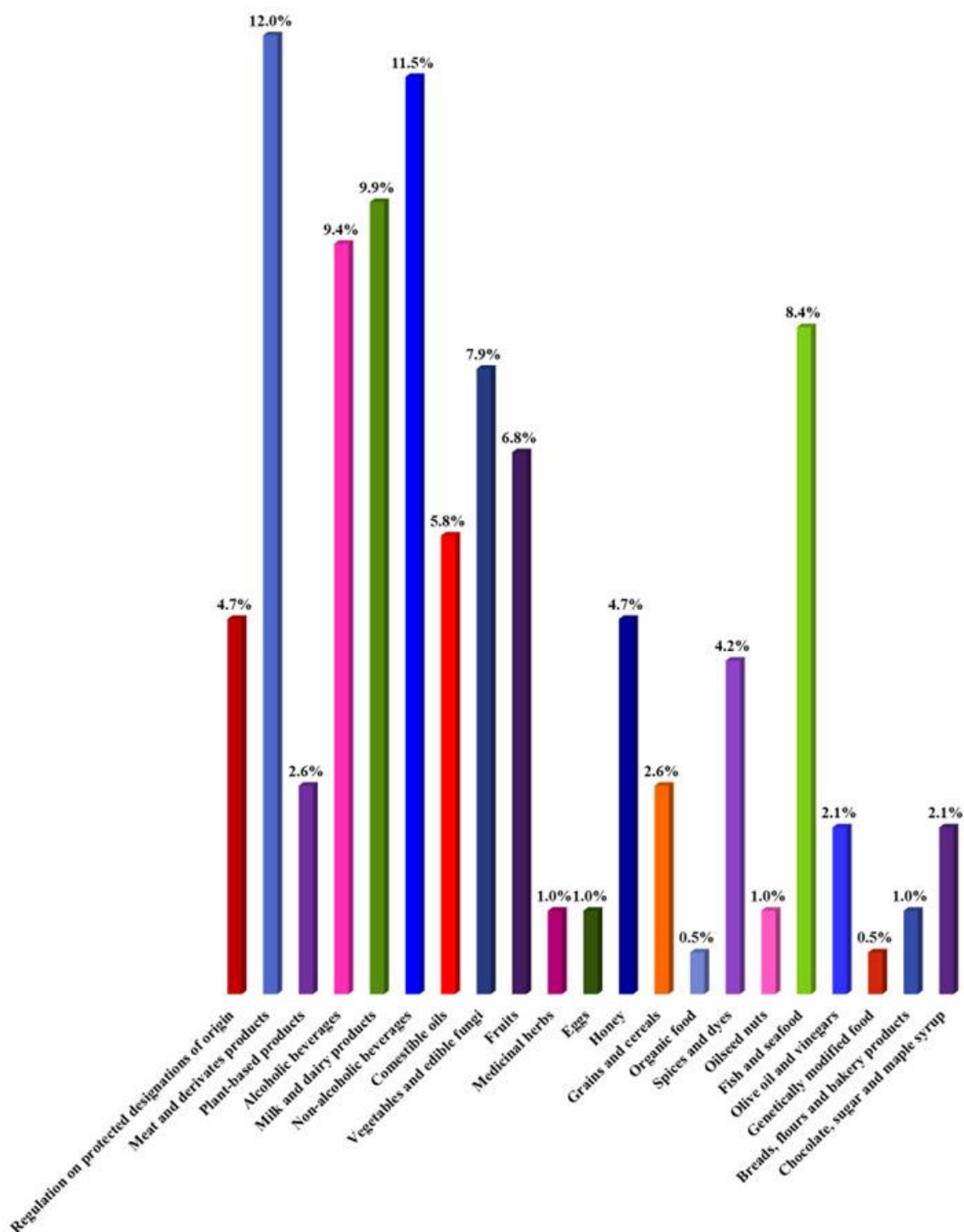


Figure 4: Perceptual of processed and non-processed foods evaluated in published reviews and discussed in this work. These data considered publications from *ScienceDirect*[®] in the years from 2013 to 2023 and were searched using the keywords "authentication" AND "food", "authentication" AND "beverage", "traceability" AND "food", and "traceability" AND "beverage".

4.4. State of the art of milk and dairy products authentication techniques: a simple bibliometric analysis

The demand for milk production and consumption has increased due to rapid urbanization and overpopulation, resulting in a discrepancy between supply and demand. To resolve this gap and generate profits, certain worldwide producers resort to illicit adulteration practices, such as an increase in the volume of milk and a change in its composition (Amsaraj et al., 2021; Balan, Dhaulaniya, Jamwal, Yadav, et al., 2020). Adulteration of cow's milk usually involves the addition of water, formaldehyde, hydrogen peroxide, or sodium hydroxide, and less often, but still relatively frequently, by the addition of whey (Aquino et al., 2014; Galvan et al., 2022; Lelis et al., 2022; Souza et al., 2011).

The addition of substances such as sucrose to milk improves the milk's total solids content and taste, compensating for the loss caused by water adulteration. Although sucrose adulteration does not pose a direct risk to human life, it can significantly negatively affect the economy and is considered fraudulent. The availability of qualitative and quantitative methods to detect added sugars in milk emphasizes the importance of comprehensive testing to ensure food authenticity and prevent food fraud (Balan, Dhaulaniya, Jamwal, Yadav, et al., 2020). The fat content of milk is also commonly adulterated for economic gain, to improve sensory characteristics, and, in some cases, to increase the added value of some products compared to others, such as margarine, a product characterized by being produced with vegetable fats and which commonly has less added value, but which can be marketed as butter.

In addition, milk adulteration results in alterations in all dairy products, which can lead to changes in the sensory properties of these products, requiring the additional use of food additives. In addition, adulteration in milk can result in the contamination of final products and the loss of reliability among consumers who believe that the consumption of dairy products is healthy and produced sustainably or respects individuals' religiosity.

The adulteration of milk and dairy products has emerged as the main target of fraudulent practices in Brazil, with an alarming 38% of cases reported between 2007 and 2017, since Brazil is one of the world's largest food producers, and for this plays a crucial role in the indirect nutrition of approximately 1.5 billion people worldwide. Consequently, this situation presents significant opportunities for fraudulent activities in the country (Tibola et al., 2018). Analytical techniques are a powerful and effective tool for determining this and other types of fraud (Ariza-Ortega et al., 2023).

The global issue of adulteration of milk and dairy products was highlighted by incidents such as the large-scale adulteration of infant milk formulas with melamine in

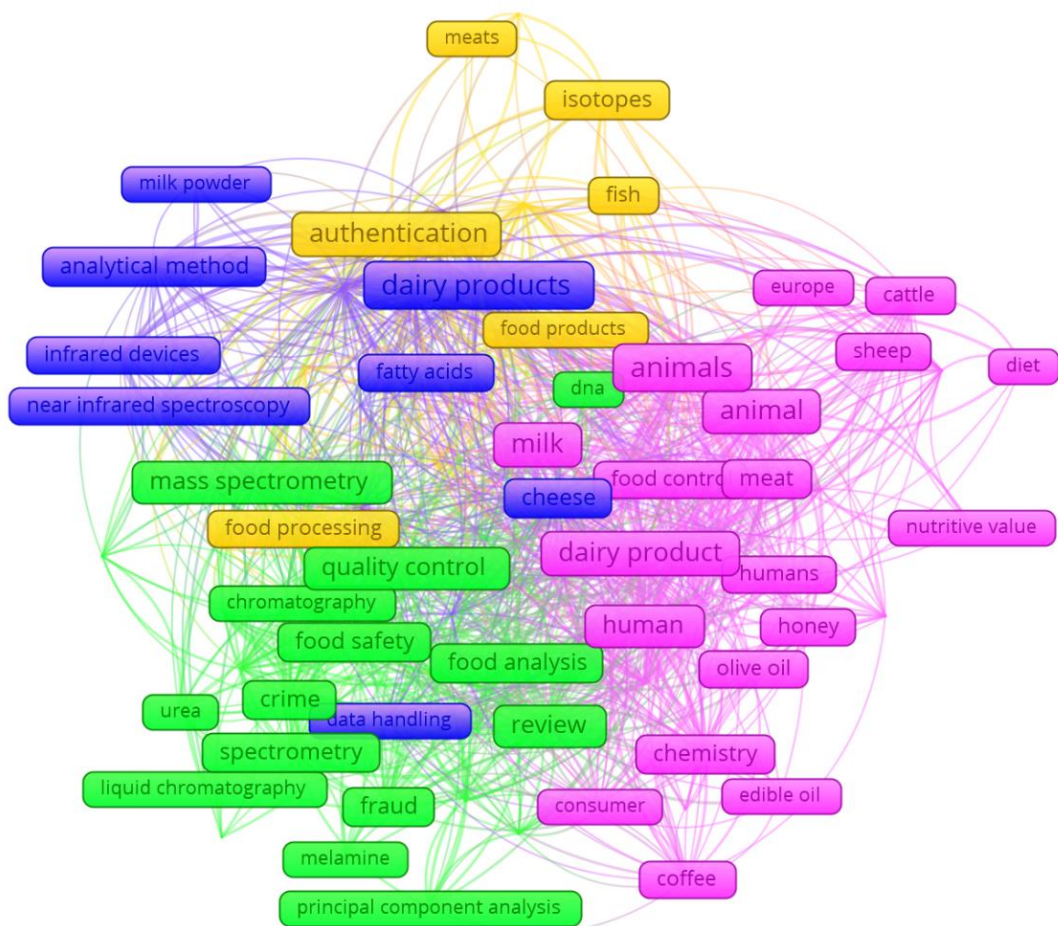
China in 2008. These incidents have raised concerns about food authenticity and the need for continuous quality monitoring (Gossner et al., 2009) and, consequently, the development and use of analytical tools for the identification and quantification of chemical components present in milk and dairy products, whether they are added intentionally, come from handling and production or result from the manufacturing process of dairy products.

The authors carried out a simplified bibliometric analysis to investigate the state-of-the-art developments and innovations in analytical methodologies evaluating the authenticity and determination of fraud and adulteration and performing the traceability of milk and dairy products from different animal species.

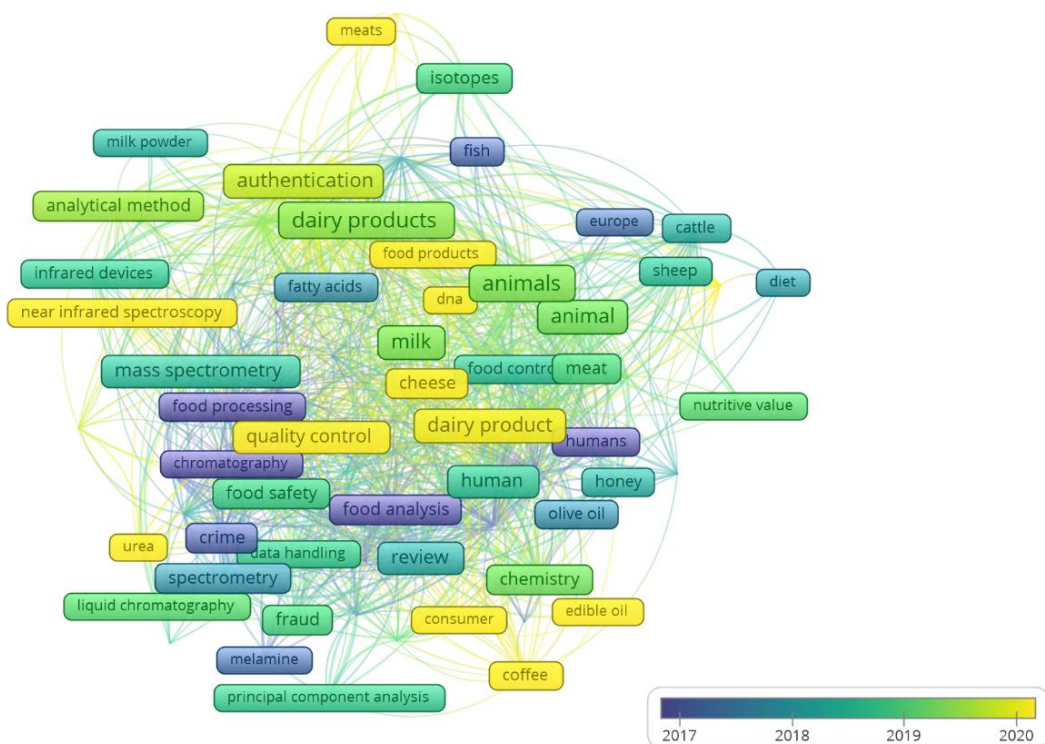
So, a scientific search was carried out as described in the *Scope and approach* section, using the *Science Direct*[®] and *Scopus*[®] database using as keywords the combination of the words "milk" OR "dairy products" AND "traceability" OR "authentication" OR "fraud" OR "adulteration", considering secondary studies published from 2013 to 2023, without language limitation, generating a database of 84 publications. The bibliometric maps were generated using the free VOSviewer[®] platform (van Eck & Waltman, 2014), considering the co-occurrence of indexed keywords that appear in the studies that make up the database and only considering words that occur at least three times.

As described in the previous topic, the dynamic visualization of a bibliometric map generated by VOSviewer[®] is based on the classification and association of terms in clusters generated according to the strength of the association link and the occurrence of the terms together in the same article. In this way, each cluster, represented by different colors, shows words with a strong co-occurrence, i.e., words commonly used as keywords indexed together. This type of map can then provide us with a view of the state-of-art in a field of research by indicating quantitatively and illustrating dynamically how a particular research field is organized.

Figure 5a shows the indexed keywords associated with the research field, which the authors will refer to as analytical techniques for authentication of milk and dairy products. So, the four clusters are strongly correlated, represented by the density of links between them, their proximity, and even their overlapping, indicating a strong co-dependence of the related themes in the different clusters.



(a)



(b)

Figure 5: (a) VOSviewer visualization of a bibliometric network for milk and dairy product authentication analysis, where each node represents the number of

times the related keyword occurred and the lines between the nodes represent the co-occurrence between keywords. (b) The color temperature scale, the warmer colors represent more recent areas of interest, and the cooler colors represent areas covered longer ago, showing the number of published research considering the years.

In cluster 1 from Figure 5, represented in fuchsia, in the bottom right-hand corner, it can see one of the study's main focus keywords, "*milk*" associated in the same cluster with different animal species, "*sheep*", "*bovine*", "*cattle*", and also with general keywords that indicate the association with analytical methodologies in "*procedure*" and "*chemical*". It is worth noting that, in this paper, the bibliometric analysis was performed to investigate the field of research without prior selections. As a result, terms appear in the clusters that at first indicate research that may fall outside the scope of the analysis, as is the case in this cluster where general food industry terms such as "*consumer*", "*diet*" and "*nutritive value*" are present, including other foods or classes of foods that are commonly the subject of authentication studies, such as honey, coffee, meat, and wine.

In the clusters represented at the lower ends of Figure 5, it can see cluster 2, in blue, where the keyword "*dairy products*" is associated with terms indicating chemical spectroscopic analytical techniques for food authentication through the terms "*analytical method*", "*infrared devices*" and "*near infrared spectroscopy*", in addition, once again, in this cluster, there is also a food that is outside the scope of the search, but which is also frequently the subject of authentication investigations, as previously discussed, represented by the keyword "*fish*". Cluster 3, in green, is also basically made up of keywords that refer to the analytical techniques used to analyze the authentication of dairy products; however, it indicates a more significant occurrence in this cluster of chromatographic techniques used in conjunction with detection by spectroscopic methods.

Considering the objective of this paper, clusters 2 and 3 from Figure 5a present meaningful and relevant evidence for the investigation of the field of study within the scope of this analysis. Among the analytical techniques reported as indexed keywords with the highest number of occurrences are "*mass spectrometry*" which occurs 8 times and is linked to other words 86 times, and in second place is "*near infrared spectroscopy*" with 5 occurrences and 44 links. This data provides indications reinforced in Figure 5b, where the two terms with the highest occurrence in cluster 4 are presented in tones that indicate the occurrence of the terms in more recent periods.

According to the scale shown in Figure 5b, "*mass spectrometry*" has been a keyword in the studies in the database for a more extended time, which is in line with the traditional nature of the method, mainly when associated with chromatographic separation techniques, which also appear in cluster 4. The keyword "*near infrared spectroscopy*", on the other hand, refers to a relatively recent method of food analysis, with research using this technology for the authentication of milk and dairy products being more recent, as indicated by its yellow representation in Figure 5b.

In addition, clusters 2 and 3 from Figure 5a provide evidence of the dairy products and analytes that have shown the most significant occurrence in the last ten years in the field of research, such as cheeses, powdered milk as products, analysis of melanin and urea as adulterants that have shown relevant occurrence.

Finally, in cluster 4 from Figure 5a, represented in yellow, there are again common interferences for the type of analysis conducted through the terms meats and fish, which are foods commonly investigated by approaches aimed at authentication of these foods. These interferences in this type of analysis are common since, in the case of reviews, some studies use the same analytical methodology, for example, based on the analysis of stable isotopes, to authenticate different food classes. As a result, these interferences appear in clusters 1 and 4. However, it is essential to note that the same approach can be used for the authentication and fraud investigation of different food classes, as seen in these clusters.

In the bibliometric map shown in Figure 5b, it is interesting to note that most of the research evaluated in this database is recent, which demonstrates the growing and recent concern with authentication analysis and fraud detection in dairy products. It is also important to note the trend of analytes investigated and used as adulterants, which are represented in yellow, indicating that they are analytes of recent interest, as is the case with urea (Amsaraj et al., 2021; Van Nguyen et al., 2023) (Amsaraj et al., 2021) and edible oils (Ariza-Ortega et al., 2023; Van Nguyen et al., 2023).

In recent years, various modern analytical technologies have been used to milk authentication from different animal species and dairy products. Table 2 presents a detailed list of recent studies on different milk and dairy products samples from the last five years, aiming to detect fraud and authenticate these products. Also, it shows the sample preparation steps employed, the detection method, some specific details in chemical analysis, and the analytical parameters obtained, such as recovery efficiency or accuracy, limit of detection, precision, and sensibility, among others.

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Table 2: Analytical parameters evaluated and sample preparation method of some research articles using different analytical chemistry methods for the authentication analysis of milk and dairy samples in the last ten years

Sample	Compound evaluated, or objective of analysis	Sample prepare	Analytical technique	Experimental parameters	Analytical parameters obtained	Reference
Goat milk	β -carotene as biomarker	LLE	ESI-MS	300 – 400 nm	Recovery efficiency: 98% LOD: 0.7 $\mu\text{g L}^{-1}$	(R. Zhang & Jia, 2022)
Commercial cow milk, organic cow milk, commercial goat milk	Detection of fraud in milk by the addition of whey	Not required	Fluorescence spectroscopy	295 nm	Predictive accuracy: 100%	(Lelis et al., 2022)
Infant milk formula	Determination of storage time	Not required	Micro NIR and benchtop NIR spectrometry	1158 – 2170 nm	Recovery efficiency: 89 – 100% micro NIR Recovery efficiency: 81.3 – 100% benchtop NIR	(R. Kang et al., 2022)
Bovine milk and cheese	Use machine learning methods for fraud detection by addition of whey in cheese	Not required	FTIR spectrometry	900 – 3000 cm^{-1}	Accuracy for classification and regression trees: 96.2 % Accuracy for multilayer perceptron neural networks: 97.8 % Sensity and specificity: > 95%	(Lima et al., 2022)
Yak organic and Jersey milk	Identify metabolites markets for assessing organic milk	LLE	UHPLC	HRMS Orbitrap	Candidates for molecular biomarkers: 85 and 34 differential metabolites in OYM and OJM	(M. Kang et al., 2023)

organic milk (OJM)	Quantify market trimethylamine N-oxide (OYM) and peptide Thr-Ala-Val (OJM) for assessing organic milk	LLE	UHPLC	MS/MS	Recovery: 993.06 – 2137.36 ng/g for Thr-Ala-Val and 110.41-135.17 ng/g for trimethylamine N-oxide	
Commercial bovine milk	Heat treatment indicators	Not required	FTIR spectrometry	3000 – 1000 cm ⁻¹	Accuracy of classification: 97%	(Y. T. Wang et al., 2021)
Raw bovine milk	Detection of formalin for fraud investigation	Not required	ATR-FTIR spectrometry	4000 – 400 cm ⁻¹	The range for detection of formalin as an adulterant: 0.5 – 5% v/v LOD: 0.5% Accuracy of classification: 100%	(Balan, Dhaulaniya, Jamwal, Amit, et al., 2020)
Human, cow, donkey, mare, goat and sheep milk	Identification of milk from different mammals	LLE	³¹ P NMR spectroscopy	202.45 MHz	Accuracy of classification: 90.3 – 100%	(Bruschetta et al., 2021)
Bovine milk	Distinction of A1 and A2 milk families across β -casein proteoforms	Not required	ATR-FTIR spectrometry	4000 – 650 cm ⁻¹	Confidence of classification of milk samples in clusters: 95%	(Daniloski, McCarthy, O’Callaghan, et al., 2022)
Raw bovine milk	Authentication of farming milk across fingerprint analysis	Not required	FTIR spectrometry	2000 – 10800 nm	Accuracy of classification of FTIR for validation: 73.5% Accuracy of validation for classification of FAs analysis: 77.3%	(Bergamaschi et al., 2020)

Ripened model-cheeses	Fingerprint analysis for authentication across volatile organic compound	Not required	NIR spectrometry	850 – 1048 nm	Accuracy of classification of GC for validation: 67.1% Accuracy of classification for VOCs analysis: 66.9%	
Semihard buffalo cheese	Assess the effect of heat treatment on buffalo cheese prepared with raw and pasteurized milk through different visual methodologies	Not required	Hyperspectral imaging	900 – 1700 nm	Accuracy of classification: 98,5% R^2_{cv} : 0.79 – 0.89 $RMSE_{cv}$: 0.25 – 1.99	(Darnay et al., 2022)
		Not required	Colorimetry	1094 – 1582 nm		
		LLE	Scanning Electron Microscopy	N.A.		
Raw bovine milk	Characterizing differences in milk protein β -casein structure of different genetic variants through combined methods	LLE	^1H NMR spectroscopy	9615 Hz	N.A.	(Daniloski, McCarthy, Markoska, et al., 2022)
		LLE	FTIR spectrometry	4000 – 600 cm^{-1}		
Dairy products of buffalo, goat and sheeo	Determination of cow DNA in buffalo, goat, and sheep dairy products	DNA extraction and dilution of samples	q-PCR	N.A.	LOD: 0.016 ng LOD cow milk in adulteration: 0.01 ng	(Giglioti et al., 2022)
Bovine milk	Authentication of bovine milk through three different miniaturized spectrometers	Not required	Hand-held spectrometer	1 – 44 nm	Specificity of prediction using different model: 53 – 100% Sensibility of prediction using different model: 50 – 100%	(Ehsani et al., 2023)
		Not required	Hand-held spectrometer	900 – 1700 nm		
		Not required	Hand-held spectrometer	1350 – 2500 nm		
Milk fermented	Discriminate peptide and molecular fingerprint	Evaporation	REIMS	MS	N.R.	(Murphy et al., 2021)
		LLE	MALDI	QTOF- MS	N.R.	

with different starter cultures						
Commercial goat milk	Authentication based on amino acids lysine, glutamine, proline and valine ratio	LLE	HPLC	PDA	Accuracy of classification: 100%	(M. Kang et al., 2022)
Commercial cheese	Authentication of Erzincan Tulum cheese through screening chemical composition profile	LLE	FTIR spectrometer	4000 – 650 cm ⁻¹	Accuracy of classification: 99%	(Menevseoglu et al., 2023)
Goat milk	<i>In-situ</i> detection of bovine milk in added for adulteration of goat milk	Not required	Hand-held NIR spectrometer	900 – 1650 nm	Accuracy of classification: 46.7 – 98.3%	(dos Santos Pereira et al., 2021)
Commercial butter and margarine	Authentication of butter and margarine through lipid profile, quantifying aliphatic, aromatic, carbonyl and sulfoxide compounds	Melted	FTIR spectrometry	4000 – 400 cm ⁻¹	N.R.	(Ariza-Ortega et al., 2023)
Bovine and goat commercial milk	Discrimination of fraudulent milks with added whey	Not required	Hand-held NIR spectrometry	900 – 1700 nm	Accuracy of prediction: 98.9%	(Galvan et al., 2022)
		Not required	EDXRF spectrometry	N.A.		
Commercial fat-filled milk (FMP) powders	Extensive routine authentication for identify freshness and stability indicators, for differentiated fresh FMP brands from	Not required	NIR spectrometry	850 – 2500 nm	Specificity of differentiation: 100% R²p: 0.83 – 1.00 RMSEP: 0.07 – 0.95	(Ejeahalaka et al., 2021)

	another and from aged samples					
Donkey milk	Authentication of donkey milk, against adulteration with bovine milk, and against deterioration due to freezing and unfreezing	Not required	FTIR spectrometry	4000 – 10000 cm ⁻¹	Accuracy of classification: 79.9 – 100%	(Di Donato et al., 2023)
Fat samples of bovine milk (cow cheese and cow butter)	Identify milk fat adulteration with non-milk fat	LLE	RP-HPLC	Refractometric detector	Range for detection of palm oil as adulterant: 1.5 – 33.5% Range linear for detector response vs. the milk fat: 2 – 15 mg/mL	(Van Nguyen et al., 2023)
Pure raw buffalo, goat and cow milk	Authentication of different types of milk including pure raw buffalo, goat and cow milk, and goat-cow and buffalo-cow milk mixtures	Not required	FTIR spectrometry	4000 – 650 cm ⁻¹	Accuracy for classification goat-cow: 93% Accuracy for classification buffalo-cow: 91% R²p: 0.99	(Sen et al., 2021)
Goat yogurt and cheese	Identify goat yogurt and cheese adulteration with cow milk	Not required	FTIR spectrometry	10000 – 4000 cm ⁻¹	Range linear for detector response vs. cow milk aduteration: 10, 15 and 20% Sensitivity and specificity values of calibration, cross validation and prediction: 100%	(Teixeira et al., 2021)
Bovine milk	Detection and quantification for discrimination of sucrose	Not required	AT-FTIR spectrometry	1070 – 980 cm ⁻¹	Accuracy of classification: 100%	(Balan, Dhaulaniya,

	adulteration in cow milk and pure milk				R²p: 0.99 RMSE: 0.20% LOD: 0.5% p/v	Jamwal, Yadav, et al., 2020)
Commercial buffalo yogurt	Detection of adulteration of buffalo yogurt with bovine milk	DNA extraction	qPCR	N.A.	Accuracy of classification buffalo: 96.8% Accuracy of classification bovine: 93.4% LOD: 0.015 ng μL^{-1}	(Marchetti et al., 2021)
Goat milk	Identification and quantification of goat milk adulteration with cow milk, and determination of fat and protein contents	Not required	FTIR spectrometry	4000 – 10000 cm^{-1}	Accuracy of classification: 100% LOD: 1.0154 g/100g RMSEP: 5.21 – 29.92	(Pereira et al., 2020)
Commercial pasteurized milk	Simultaneous detection of milk adulterants starch, urea and sucrose	Not required	FTIR spectrometry	400 – 4000 cm^{-1}	Accuracy of classification: 100% R²p: 0.97 – 0.99 RMSEP: 0.26 – 0.41	(Amsaraj et al., 2021)
Commercial bovine milk powder	Identify and quantify adulteration with added melanine	Not required	2D – COS FTIR spectrometry	400 – 4000 cm^{-1}	Spectroscopic band: 815 cm^{-1} LOD: 14.3 ppm Range linear for concentration of melanine: 0 – 35%	(Al-Lafi & AL-Naser, 2022)
Bovine milk powder	Detect and quantify adulterants such as corn starch and wheat flour in milk powder	Reduce particle size variation	FT-NIR and FT-MIR spectrometry	FT-NIR: 4050 – 11934 cm^{-1} FT-MIR: 657 – 3993 cm^{-1}	RMSEP corn starch: 0.74 and 1.69% w/w RMSEP wheat flour: 0.82 and 2.63% w/w	(Caballero-Agosto et al., 2024)
Commercial pasteurized milk	Rapid determination of milk acidity and detection of adulteration	Not required	Raman spectrometry	715–1812 cm^{-1}	RPD: 3.38 for PLSR and 6.31 for BP-ANN	(Huang et al., 2022)

					R²p: 0.90 for PLSR and 0.97 for BP-ANN	
Camel, donkey, goat, yak, bovine and sheep milk	Screening evaluation of milk of different animal species for determination of biomarkers and post authentication of these differents types of mik	Drying in spray drying, protein extraction and digestion	Shotgun proteomics with scheduled multiple reaction monitoring	QTOF- MS	Range of detection: 1 – 100% Range of quantitative recovery: 91.07 – 111.75%	(J. Zhang et al., 2024)
UHT milk, sterilized milk, yoghurt, butter, ice cream, cheese, milk powder, milk tablet, sweetened condensed milk, condensed milk, drinking yoghurt, hydrolyzed casein	An alternative approach for the identification of cow DNA in milk and dairy products can be achieved through the utilization of direct-asymmetric PCR and the CHA-FB system, also known as daPCR-CHA-FB	DNA extraction	Biosensor PCR based	Spectrofluorometer	Accuracy: 100% LOD: 1x10 ⁻⁴ ng	(Thanakiatkrai et al., 2024)
Commercial camel milk	Detection of allergen bovine derived β -lactoglobulin as an adulterant in camel milk	Centrifugation	Molecularly imprinted	AuNPs-rGO based MIP	Linear range: 4 – 100 ng mL ⁻¹ LOD: 3.58 ng mL ⁻¹	(Meng et al., 2024)

			electrochemical sensing			
Commercial UHT milk	Screening of commercial milk through use a electronic tongue	Not required	Electrochemical impedance spectroscopy	AgNPs-AuE	Prediction correlations: to 0.942, 0.94, and 0.96 for the prediction of lactose, urea and fat content.	(Perez-Gonzalez et al., 2024)

689 ATR-FTIR: Attenuated Fourier transform infrared. AgNPs-AuE: Silver nanoparticles – Gold electrode based. AuNPs-rGO: Nanocomposite of gold nanoparticles
 690 – reduced graphene oxide. ESI-MS: Electrospray ionization – mass spectrometry. FA: Fatty acid analysis. FTIR: Fourier transform infrared. HPLC: High-
 691 performance liquid chromatography. HRMS Orbitrap: High-resolution mass spectrometer orbitrap-based. LLE: Liquid-liquid extraction. LOD: Limit of detection.
 692 MALDI-QTOFMS: Matrix-assisted laser desorption ionization time-of-flight mass spectrometry. NIR: Near-infrared. PDA: Photo diode array detector. qPCR:
 693 Quantitative polymerase chain reaction. R^2_{cv} : Coefficients of determination during model cross-validation. R^2_p : Coefficient of determination of prediction. REIMS:
 694 Rapid Evaporative ionization mass spectrometry. RMSE_{cv} Root mean square error of model cross-validation. RMSEP: Root mean square error of prediction. RPD:
 695 Ratio of prediction to deviation. RP-HPLC: Reverse-phase high-performance liquid chromatography. Thr-Ala-Val: Treonine-Alanine-Valine. UHPLC: Ultrahigh
 696 performance liquid chromatography. VOC: Volatile organic compounds analysis. N.R.: Not reported. N.A.: Not applicable.

It can be observed in Table 2 that there is a predominance of methodologies that use spectroscopic techniques to authenticate dairy products, emphasizing the use of infrared spectrum methodologies, such as near-infrared spectroscopy (NIR), and Fourier-transform infrared spectroscopy (FTIR). NIR spectral information can be used to distinguish buttery cheeses from their comparable counterparts. Artisanal buttery cheese is vulnerable to adulteration with cheaper vegetable oils. In this sense, NIR technology can identify fraudulent activities and determine the presence of external fats. However, conventional analytical techniques for detecting fraud in buttery cheeses are expensive and time-consuming.

FTIR is a widely used technique in the identification of chemical contaminants in milk powder, including melamine, the main adulterant employed in milk. Some specific lines in the resultant spectrum (815 cm^{-1}) are related to the presence of melanin. Their intensity is evaluated by two-dimensional correlation spectroscopy (2D-COS) that allows the FTIR spectra of milk powder, with suitable precision, indicating an adequate approach in the analytical detection of food authentication, mainly in the milk powder adulteration (Al-Lafi & AL-Naser, 2022).

As shown in the bibliometric analysis in Figure 5 and confirmed in Table 2, some analytical techniques combine chemometrics tools, including mathematical and statistical methods, to extract meaningful and valuable information from chemical data, facilitating the analysis of complex data sets and identifying and elucidating patterns and trends in dairy products, specifically for authentication purposes. The use of chemometric techniques allows for the comprehensive analysis of various parameters in dairy products, including their composition, flavor, and geographical origin, helping to guarantee the authenticity of these products.

Chemometrics tools can identify specific markers or patterns that differentiate genuine dairy products from those adulterated or falsified. Incorporating chemometric analysis with spectroscopic techniques, such as near-infrared spectroscopy (NIR), allows the quality and authenticity of dairy products to be determined quickly and accurately. NIR spectroscopy is particularly valuable for providing pertinent information on the chemical composition of dairy samples, thus facilitating the detection of adulterants or contaminants. In addition, chemometrics allows for the development of predictive models that can classify dairy products based on their distinct characteristics, such as type of milk, breed or geographical origin. These models are invaluable in authentication, providing objective and reliable results (Z. Zhang et al., 2024).

In this context, Caballero et al. (Caballero-Agosto et al., 2024) used NIR allied to partial least squares regression (PLSR) models to detect and quantify adulterants, such as corn starch and wheat flour, in milk powder, allowing a valuable tool for trace and identify adulterated milk powder, particularly in developing and underdeveloped countries where monitoring and enforcement systems may be lacking. PLSR models allied to root mean square error of prediction (RMSEP) increased detection reliability, with a considerable contribution to food fraud detection, and have the potential to safeguard the quality and nutritional value of powdered dairy products, and can help maintain consumer confidence and protect public health.

Alternatively, portable NIR spectroscopy and chemometrics tools, such as partial least squares-discriminant analysis (PLS-DA), can identify adulteration. The established PLS-DA classification models demonstrated commendable efficiency in predicting adulteration in milk and dairy products, achieving an accuracy rate of 94.44% in identifying adulterated samples. In addition, the PLS prediction models performed exceptionally well in predicting adulterant levels (Medeiros et al., 2023).

In addition, Lelis et al. (Lelis et al., 2022) used infrared spectroscopy and fluorescence spectroscopy allied with chemometric tools to detect the fraudulent addition of whey to different types of milk. It was possible to conclude that fluorescence spectroscopy combined with chemometric tools can be used with good results in milk quality control, allowing the detection of fraudulent whey addition. The PLS-DA approach methodology showed the finest classification accuracy compared to Soft independent modeling of class analogy (SIMCA) to distinguish between genuine and adulterated milk, with 100% predictive accuracy, sensitivity, and specificity for whole and skimmed milk. However, the study found limitations in the methodology developed to quantify and classify milk samples according to the level of adulteration.

Huang et al. (Huang et al., 2022) employed Raman spectroscopy with chemometric tools to examine the acidity of milk, allowing results to accurately predict the milk acidity, with a strong correlation between predicted and measured values. This finding had significant practical implications for the dairy industry, as it offered a potential means of quickly and accurately assessing milk acidity that could be valuable for quality control, ensuring dairy products meet the required standards and regulations. In addition, using Raman spectroscopy and chemometric tools can provide a fast and non-destructive analysis method, allowing real-time monitoring of milk acidity during

production processes, assisting in the optimization of production parameters, and ensuring consistent product quality.

Fluorescence spectroscopy was used by Thanakiatkrai et al. (Thanakiatkrai et al., 2024) in the DNA identification from cow milk and dairy products prepared by cow milk. For this, they used direct-asymmetric PCR and catalyzed hairpin assembly with fluorescent biosensor (daPCR-CHA-FB) that indicated a direct correlation between the presence of synthetic cow DNA and the fluorescence signal, with an accuracy rate of 100% when tested on confirmed cow's milk samples. Notably, the assay did not exhibit cross-reactivity with nine alternative species. In addition, the assay's detection limit was determined at 1×10^{-4} ng of DNA. The test also demonstrated the assay's robustness by examining almost 250 market samples, revealing an observed fraud rate of 15.4%. Notable advantages of the assay include its rapid completion time of less than 2 hours, its independence from DNA purification processes, and its lack of dependence on expensive equipment. In addition, the simplicity and cost-effectiveness of the assay, attributable to its exemption from DNA purification requirements and the need for expensive machinery, make it accessible for routine use in laboratories and potentially for end users. However, it should be recognized that the current iteration of the assay offers only a semi-quantitative approximation of the contamination or adulteration of cow's milk.

The fat content of milk is a common target for adulteration to optimize economic gains from the marketing of milk. In their study, Van Nguyen et al. (Van Nguyen et al., 2023) evaluated the adulteration of milk fat with non-milk fat, using reverse-phase high-performance liquid chromatography (RP-HPLC) combined with chemometric techniques and a multidimensional vector model to assess the triacylglyceride fingerprint profile of the sample objectively. The proposed method detected adulteration at 1.5% to 100% concentrations.

The use of hyphenated techniques with the coupling of two or more analysis techniques to obtain an analytical tool faster and more efficiently than isolated techniques has also been reported for milk and dairy product authentication, with satisfactory results for complex analyses, as shown in the study conducted by Kang et al. (M. Kang et al., 2023). They used advanced chromatographic and spectroscopic techniques aligned with chemometric approaches based on non-targeted and targeted metabolomics to develop a methodology for accurately authentication organic Yak and Jersey cow milk. Additionally, these authors identify the Thr-Ala-Val peptide and D-biotin as metabolic markers to distinguish organic and non-organic Jersey milk. In addition, they determined

trimethylamine N-oxide as a metabolic marker for distinguishing organic and non-organic yak and Jersey milk. These findings provide critical information to assess the authenticity of organic milk. Furthermore, in the targeted strategy and using ultra-high-performance liquid chromatography (UHPLC) with tandem mass spectrometry (MS/MS) detector, the authors achieved recoveries of 993.06 - 2137.36 ng g⁻¹ for Thr-Ala-Val and 110.41-135.17 ng g⁻¹ for trimethylamine N-oxide, extremely low concentrations, indicating a very sensitive methodology for milk authentication.

Zhang et al. (J. Zhang et al., 2024) used high-pressure liquid chromatography coupled with quadrupole time-of-flight mass spectrometry (HPLC-Q-TOF-MS) and shotgun proteomics, a very sensitive bottom-up approach used to study complex mixtures of proteins, in development of a proteomics analytical method for the discriminate milk from 7 different species (camel, donkey, goat, sheep, buffalo, yak and cow) and provide an accurate detection methodology for milk species adulteration. This method could detect any adulteration in milk samples over a wide range from 1% to 100% and provide quantitative data on the proportion of cow's milk species in non-cow's milk samples. Its usefulness extends to assessing the authenticity of commercial dairy products and identifying incorrectly labeled milk samples. In addition, this technique is suitable for various dairy processing technologies, such as pasteurization, ultra-high temperature sterilization, spraying, and fermentation, due to the investigations were conducted into the stability of specific peptides under different processing treatments, including heat, pressure, and fermentation.

The high cost and, sometimes, low performance exhibited by conventional techniques, such as chromatography, to identify fraud and verify the authenticity of dairy products calls for exploring alternative methodologies that can resolve the issues of speed of analysis and total cost of analysis. One promising approach is using electroanalytical and optical methods in detection systems, which offer superior potential for identifying fraudulent activities and guaranteeing the authenticity of dairy products. However, electroanalytical techniques require adequate choices of working electrode materials to improve the analytical signals' sensitivity, selectivity, and reproducibility (Gonçalves-Filho & De Souza, 2022). Among milk and dairy products analysis using electroanalytical techniques, the papers found always use chemically modified electrodes prepared from a conductor or semiconductor material coated with a monomolecular, multi-molecular, ionic, or polymeric film, allowing in alterations in the electrochemical properties of the interface (Zoski, 2007).

The main modification for milk and dairy products authentication analysis is based on nanomaterials and biosensors. This is evidenced in the review of Himshweta and Singh (Himshweta & Singh, 2023), which focused on optical and electrochemical nanosensors to identify adulterants in dairy products. Detection systems based on nanomaterials provide more excellent selectivity and sensitivity than traditional methods, thus enabling more effective identification of adulterants in dairy products.

Electroanalytical techniques allied to nanosensors facilitate rapid results, enabling quick screening of dairy products to guarantee their authenticity and quality (Kalambate et al., 2024). In addition, nanosensors can be specifically designed to target and detect specific adulterants, thus increasing the accuracy and reliability of their identification. Using nanomaterials in electroanalytical detection systems allows the identification of adulterants in dairy products, which represents a challenge for conventional methods. In addition, nano-sensors offer the potential for miniaturization and portability, thus enabling local testing and monitoring dairy products for adulterants. The development of nanomaterial-based detection systems to detect milk adulteration contributes to the overall goal of protecting consumers against counterfeit products and the health risks associated with adulterated milk. In a recent review, Himshweta and Singh evaluated (Himshweta & Singh, 2023) the use of nanosensors in the identification of adulterants in dairy products, including milk from various animal species. It highlights the limitations of traditional methods in detecting adulterants in dairy products and emphasizes the need for comprehensive, rapid, and sensitive analytical tools. The review provides a comprehensive overview of nanomaterial-based detection systems as effective alternatives to traditional approaches for identifying adulterants in dairy products. In addition, the study discusses recent advances in the field of nanoarray platforms for the rapid detection of adulterants in dairy products.

The use of biosensors allied to some electroanalytical techniques has also been reported as a suitable approach with the potential to achieve sensitive and specific detection of adulterations and contaminations in milk. Previous methods described the application of graphene-based biosensors as a promising tool for milk analysis, as it offers increased sensitivity and rapid detection of several compounds relevant to the dairy industry. In addition, graphene-based biosensors offer remarkable advantages in sensitivity, speed, selectivity, miniaturization, stability, and durability for milk analysis, ensuring milk safety and quality. This detection method can have practical implications

for the dairy industry, facilitating timely intervention and control measures to protect the safety and quality of milk products (Bayrak & Gergeroglu, 2024).

Recently, Meng et al. (Meng et al., 2024) reported the use of a high-performance molecularly imprinted polymer (MIP) sensor prepared with gold nanoparticles (Au NPs) and reduced graphene oxide (rGO) for the determination of β -lactoglobulin, an adulteration marker in camel milk, which is used by consumers that suffering from milk allergies. The study's main findings include successfully identifying DNA aptamers targeting β -lactoglobulin and integrating them into the graphene-based biosensor. The sensor exhibited remarkable sensitivity and selectivity in detecting β -lactoglobulin, making it a promising tool for identifying milk allergens. In addition, the research explored the use of enzymatic digestion to eliminate protein templates from molecule-printed polymers, which could further increase the sensor's efficiency. In addition, this study provides information on the development of graphene-based biosensors for the identification of milk allergens.

An electronic tongue (ET) prepared from enzymes (including galactose oxidase, glucose oxidase, beta-galactosidase, urease and lipase) immobilized under silver nanoparticles supported in gold surface by Perez-Gonzalez et al. (Perez-Gonzalez et al., 2024) was employed by for the discrimination of milks with different characteristics. The methodology developed allowed for the development of satisfactory classification models, with the establishment of correlations between predictions acquired through analysis with the electronic tongue and the physicochemical parameters of the milk, measured experimentally. So, it can be seen that this methodology could be used developed in the classification and authentication of milk and dairy products.

All research evaluated in this paper was fundamental in the milk and dairy products analysis due to the majority of commercial qualitative tests for previous detection of fraud and adulterations in these products yielding a high rate of false-negative and false-positive results, allowing in the inadequate use of these products and consequently, economic damage and insufficient food safety for consumers.

5. Trends and perspectives in food authentication

Food adulteration is defined as the degradation of food quality by intentionally adding or subtracting any substance from the food. Among these, milk is a beverage that can be easily altered by diluting milk with water, adding solids to improve the low-value milk, adding proteins or harmful chemicals to increase the nutritional content or shelf life,

or even wholly disguising low-value milk as a higher-value product. Milk ohmic studies have been a promising and effective tool for better understanding milk properties, detecting adulteration in metabolic properties, and the amount of lipids and proteins (Qin et al., 2022). The application of traditional and more recent DNA-based methods for dairy product authentication has been discussed and analyzed since DNA quality is one of the main factors affecting molecular-based dairy product authentication, which can be influenced by the manufacturing process, extraction method employed, composition of the food matrix, among others (Baptista et al., 2021).

Biological techniques employing high-performance ohmic technologies, such as genomics and proteomics, have been introduced to address sustainability in fisheries and aquaculture production. Proteomics serves to understand developmental biology, physiology, disease/stress, and species recognition, enabling authentication and traceability to ensure food safety, identifying the presence of pathological species in foods, and disease diagnosis in raw materials (Jaiswal et al., 2023).

Furthermore, biological techniques provide an effective strategy for the authentication, traceability, and control of the quality and safety of seafood using proteomic approaches since protein molecules are closely related to texture and allergenicity properties, as well as being related to the species, place of production, processing and storage procedures. Qualitative and quantitative analysis of particular protein biomarkers has contributed to quality control and nutritional assessment of foods (Y. Wang et al., 2023).

Chromatographic techniques allied to chemometrics tools can be applied throughout the food chain to verify the nature or origin of food. Several of these techniques have been evaluated to guarantee authenticity and detect adulteration. Among these, chromatographic fingerprinting, coupled with multivariate analysis and chemometric data, allowed the development of the latest combined chromatographic techniques for evaluation in various foods, such as oils and fats, honey, dairy products, beverages, meat products, among others (Esteki, Simal-Gandara, et al., 2018)

Furthermore, chromatographic techniques allied to DNA-based methods combined with multivariate analysis have shown high precision, sensitivity, and selectivity, allowing the evaluation of multiple components of foods, especially for food products containing animal and vegetable fats. Already, spectroscopic techniques have been used to establish the geographical origin of food products with a fast response, low cost, non-destructive character, and the possibility of being miniaturized, being favorable

and very important for the authentication and traceability of foods (Abbas et al., 2018; Valdés et al., 2018).

Modern analytical techniques such as isotope ratio mass spectrometry and inductively coupled plasma mass spectrometry, combined with multivariate analysis, have been used to detect the authenticity of rice and its derivatives. Other techniques, such as DNA-based methods and ohmic analysis, also have great applicability in identifying these products (Wadood et al., 2022).

Chromatographic, spectroscopic, sensor-based, and molecular techniques have advantages and limitations concerning the limit of detection and their suitability for detecting specific types of adulterants (natural or synthetic). However, these techniques have been used to detect the presence of different adulterants, including plants and chemicals, because the adulteration by foreign materials reduces food quality and can lead to severe complications in public health (Kumari et al., 2021).

Intelligent packaging can help monitor the quality and integrity of food, but its downside is its high cost. As such, extrusion-based 3D printing has emerged as a cost-effective solution for creating packaging from materials considered safe and suitable for food, as permitted by regulatory bodies. 3D printing to manufacture intelligent packaging makes it possible to develop highly sensitive, self-indicating, and multifunctional intelligent components using non-toxic, biocompatible materials cheaper than conventional manufacturing methods, making it easier to identify fraud (Tracey et al., 2022).

Electroanalytical techniques allied to chemically modified electrodes, such as nanosensors and biosensors, provide excellent sensitivity and selectivity in food authentication, including the identification of adulteration and fraud in milk and dairy products. Finally, in recent years, electronic tongues (ET) have presented a powerful tool for food analysis, including milk and dairy product authentication, due to their fast analysis when allied to chemometric tools.

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Supplementary Data

for

An integrative review of analytical techniques used in food authentication: a detailed description for milk and dairy products

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1493 **Table S1:** Recent review publications in Food Analysis, Food Chemistry, and Chemistry about the main analytical techniques to analyze food
1494 authenticity. These data considered publications from ScienceDirect® in the years from 2013 to 2023 and were searched using the
1495 keywords “authentication" AND "food", "authentication" AND "beverage", "traceability" AND "food", and "traceability" AND
1496 "beverage".

Class of analytical techniques	Specific analytical techniques reviewed	Foods reported	Reference
Spectroscopic, calorimetric, biological,	Fluorescence spectroscopy, digital image colorimetry, ultra-violet spectroscopy	Application of sensors in packaging of various foods	(Tracey et al., 2022)
Chromatographic	Gas chromatography, high performance liquid chromatography, liquid chromatography, ultra-high performance liquid chromatography	Oils and fats, honey, dairy products, beverages, products meat	(Esteki, Simal-Gandara, et al., 2018)
Biological, spectroscopic, chromatographic, electroanalytical	Metabolomic, genomic, proteomic, lipidomic, glycomic, transcriptomic, raman spectroscopy, liquid chromatography, gas chromatography, fluorescence spectroscopy, biossensor, laser direct infrared, nuclear magnetic resonance spectroscopy, digital image colorimetry, ultra-high performance liquid chromatography, high performance liquid chromatography	Milk from different animal species	(Qin et al., 2022)

Chromatographic	Gas chromatography, ultra-high performance liquid chromatography, high performance liquid chromatography	Rice, milk , fish , teas , plant-based products, vegetables	(J. Wang et al., 2023)
Chromatographic	High performance liquid chromatography	Natural waters, milk, cooked food, fish, wheat, rice, corn, beab, chili powder, chili sauce, duck eggs, vegetables	(Fernández-Ramos et al., 2014)
Biological, chromatographic, spectroscopic, calorimetric	Gas chromatography, liquid chromatography, hight performance liquid chromatography, genomic, near infrared spectroscopy, mass spectrometry, ultraviolet-visible spectroscopy, nuclear magnetic resonance spectroscopy, differential scanning calorimetry, fourier transform infrared spectroscopy	Meats, fish, dairy products, nuts, vegetable oils	(Valdés et al., 2018)
Biological, chromatographic, spectroscopic	Metabolomic, genomic, gas chromatography, liquid chromatography, nuclear magnetic spectroscopy, near infrared spectroscopy, mid infrared spectroscopy, raman spectroscopy, mass spectrometry, fourier transform infrared spectroscopy	Milk, cheese, meat and honey with geographical indication (GI)	(Abbas et al., 2018)
Electroanalytical, biological,	Electrochemical biosensors, liquid chromatography, gas spectroscopy, mass spectrometry, metabolomic	Foods with geographical indication (GI)	(Bianchi et al., 2018)

chromatographic, spectroscopic			
Spectroscopic, chromatographic	Assesses flow injection analysis coupled to high-resolution mass spectrometry and high-resolution mass spectrometry	Paprika powder with geographical indication (GI)	(Monago-Maraña et al., 2022)
Spectroscopic, chromatographic	Ultraviolet-visible spectroscopy, near infrared spectroscopy, fourier transform infrared spectroscopy, raman spectroscopy, nuclear magnetic resonance spectroscopy, liquid chromatography	Meats, honey, greens, vegetables, teas, fish, with and without geographical indication (GI)	(Castro-Puyana et al., 2017)
Spectroscopic	Nuclear magnetic resonance spectroscopy	Honey, beer, condiments	(Kuballa et al., 2018)
Chromatographic, spectroscopic	Fluoresce spectroscopy, ultra-high performance liquid chromatography, high performance liquid chromatography, gas chromatograph, inductively coupled plasma atomic emission spectroscopy, mass spectrometry, nuclear magnetic resonance spectroscopy	Maple syrup with geographical indication (GI)	(Gad et al., 2021)
Chromatographic, spectroscopic	Liquid chromatography, gas chromatography, mass spectrometry, raman spectroscopy, ultraviolet fluorescence spectroscopy, inductively coupled	Tequila with geographical indication (GI)	(Pérez-Caballero et al., 2017)

	plasma atomic emission spectroscopy, nuclear magnetic resonance spectroscopy, ion chromatography, fourier transform infrared spectroscopy, ultraviolet-visible spectroscopy		
Chromatographic, spectroscopic	Gas chromatography, liquid chromatography, mass spectrometry, nuclear magnetic resonance spectroscopy, fourier transform infrared spectroscopy	Alcoholic beverages	(Luo et al., 2020)
Chromatographic, spectroscopic, calorimetric, biological, electroanalytical	Electronic nose, isotope ratiomass spectrometry, liquid chromatography, gas chromatography, differential scanning calorimetry, near infrared spectroscopy, mid infrared spectroscopy fourier transformed infrared spectroscopy, raman spectroscopy, nuclear magnetic resonance spectroscopy, genomic, ultraviolet fluorescence spectroscopy, mass spectrometry	Camellia oil	(Shi et al., 2020)
Chromatographic	Gas chromatography, high performance liquid chromatography	Pomegranate, raspberry, soybean, tea, meat, oil, and others	(Cuadros-Rodríguez et al., 2016)

Spectroscopic, chromatographic, electroanalytical	Gas chromatography, fourier transform infrared spectroscopy, hyperspectral scattering image, mass spectrometry, liquid chromatography, near infrared spectroscopy, nuclear magnetic resonance spectroscopy, ultra high performance liquid chromatography, high performance liquid chromatography, mid infrared spectroscopy, electronic nose, electronic tongue, ultraviolet-visible spectroscopy, conductivity-meters, density-methods, refractometry, computer vision systems	Wines, beer, extra virgin olive oil, teas, orange juice, fruit juices, milk, tomato juice, apples, meats, fish, coffee, milk, soy flour	(Borràs et al., 2015)
Spectroscopic	Raman spectroscopy	Non-alcoholic drinks, meat, potatoes	(Arroyo-Cerezo et al., 2021)
Biological	Adenine triphosphate bioluminescence-based methods	Milk, yogurt, cheese, orange juice, meat, broccoli, potatoes, tomatoes	(Bottari et al., 2015)
Biological	DNA barcoding	Dairy products, edible plants, meat products	(Galimberti et al., 2013)
Biological	Genomic based methods	Milk, blood, cheese, dairy products	(Baptista et al., 2021)
Biological	Genomic based methods	Fish, meat products, fruits, vegetables, cheese, olive oil, wine, ginseng	(Lo & Shaw, 2018)

Biological, Spectroscopic, electroanalytical	Genomic based methods, electrochemical biosensors, colorimetry, raman spectroscopy	Meat and meat products	(Khalil et al., 2021)
Chromatographic, biological, spectroscopic, electroanalytical	Liquid chromatography, gas chromatography, raman spectroscopy, ultraviolet-visible spectroscopy, mid infrared spectroscopy, near infrared spectroscopy, fourier transform infrared spectroscopy, nuclear magnetic resonance spectroscopy, hyper spectral imaging spectroscopy, mass spectrometry, immunological techniques, electrophoretic techniques, genomic, electronic nose, electronic tongue, electrochemical biosensors	Pepper, condiments	(Negi et al., 2021)
Biological, spectroscopic, chromatographic, electroanalytical	Mass spectrometry, metabolomic, genomic, gas chromatography, liquid chromatography, nuclear magnetic resonance spectroscopy, near infrared spectroscopy, mid infrared spectroscopy, electronic nose, electronic eye, electronic tongue	Fruits and derivatives, meat and meat products, dairy products, honey, eggs, oils and fats, fish, spices, coffee, chocolate, teas, bakery products, vinegar, wine and other beverages	(Danezis, Tsagkaris, Brusic, et al., 2016)
Biological, chromatographic, spectroscopic	Genomics, proteomics, liquid chromatography, gas chromatography, mass spectrometry, nuclear magnetic resonance spectroscopy, near infrared	No specific food sample	(Danezis, Tsagkaris, Camin, et al., 2016)

	spectroscopy, mid infrared spectroscopy, ultraviolet fluorescence spectroscopy, electronic nose, electronic eye, electronic tongue, raman spectroscopy, fourier transform infrared spectroscopy		
Biological, spectroscopic, chromatographic	Genomic, lipidomic, proteomic, gas chromatography, liquid chromatography, mass spectrometry, nuclear magnetic resonance spectroscopy, ultraviolet-visible spectroscopy, raman spectroscopy, fourier transform infrared spectroscopy, near infrared spectroscopy, mid infrared spectroscopy,	Plant-based products, dyes in food, drinks, postmortem viscera, seafood, beverages	(Saadat et al., 2022)
Spectroscopic	Ultraviolet-visible spectroscopy, near infrared spectroscopy, fourier transform infrared spectroscopy, raman spectroscopy, nuclear magnetic resonance spectroscopy	Fish with different consumer labels	(Freitas et al., 2020)
Biological	DNA barcoding	Honey, saffron	(Galimberti et al., 2019)
Electroanalytical, spectroscopy, chromatography	Electronic nose, electronic tongue, electronic eye	No specific food sample	(Di Rosa et al., 2017)

Spectroscopic, chromatographic, biological	Mid infrared spectroscopy, raman spectroscopy, near infrared spectroscopy, nuclear magnetic resonance spectroscopy, ultraviolet-visible spectroscopy, gas chromatography, high performance liquid chromatography, mass spectrometry, stable isotope measurements, genomic	Fish, wine, fruit, meat, agrifood	(Galvez et al., 2018)
Electroanalytical	Aptamer-based electrochemical sensors	Corn, beer, milk, fish	(Mohamad et al., 2022)
Biological, spectroscopic	Genomic, stable isotope measurements, mass spectrometry, nuclear magnetic resonance spectroscopy	Indian foods with geographical indication (GI)	(Dandage et al., 2017)
Spectroscopic, chromatographic, electroanalytical	Nuclear magnetic resonance spectroscopy, hyperspectral imaging spectroscopy, electronic nose, electronic tongue, electronic eye, ultraviolet-visible spectroscopy, near infrared spectroscopy, gas chromatography, liquid chromatography, mid infrared spectroscopy, fourier transform infrared spectroscopy, electrochemical biosensors, raman spectroscopy	Meats, fruits, vegetables, allegedly functional foods, medicinal foods, mushrooms, beverages, oils	(Zhou et al., 2020)

Spectroscopic	Laser-induced breakdown spectroscopy	Various foods with geographical indication (GI)	(Markiewicz-Keszycka et al., 2019)
Spectroscopic	Mass spectrometry, gas chromatography, liquid chromatography, nuclear magnetic resonance spectroscopy, raman spectroscopy, near infrared spectroscopy, mid infrared spectroscopy, fluorescence spectroscopy	Fish, meat, fruit	(Valletta et al., 2021)
Biological, chromatographic	Mass spectrometry, metabolomic	Tomato, wine, cereals, fruits, vegetables, mango, teas, fish, beef liver, oranges	(Castro-Puyana & Herrero, 2013)
Biological, chromatographic, electronalytical, spectroscopic	Metabolomic, genomic, gas chromatography, liquid chromatography, nuclear magnetic resonance spectroscopy, electronic nose	Korean food with geographical indication (GI)	(Singh et al., 2017)
Chromatographic, spectroscopic, biological	High performance chromatography, nuclear magnetic resonance spectroscopy, liquid chromatography, mass spectrometry, gas chromatography, fourier transform infrared spectroscopy, raman spectroscopy, lipidomic	Oils and fat	(Przykaza et al., 2021)

Chromatographic, spectroscopic	Gas chromatography, liquid chromatography, nuclear magnetic resonance spectroscopy, infrared spectroscopy, laser-induced breakdown spectroscopy	Honey	(Zhang & Abdulla, 2022)
Spectroscopic, chromatographic, biological, electroanalytical	Gas chromatography, liquid chromatography, high performance liquid chromatography, ultra-high performance liquid chromatography, mass spectrometry, mid infrared spectroscopy, near infrared spectroscopy, raman spectroscopy, nuclear magnetic resonance spectroscopy, isotopes ratio mass spectrometry, hyperspectral imaging, inductively coupled mass spectrometry, genomic, electronic nose, electronic tongue, electronic eye	Meat, milk, oils, rice, honey, pistachios, oregano, coffee, fish, butter, powdered milk, hazelnuts, peanuts, chocolate, alcoholic beverages, fruits	(McVey et al., 2021)
Chromatography, electroanalytical, spectroscopy	High performance liquid chromatography, electronic tongue, ultraviolet-visible spectroscopy, ultra-high performance liquid chromatography, electrochemical sensors, raman spectroscopy, gas chromatography, isotope ratio mass spectrometry, electronic nose, liquid chromatography, infrared spectroscopy	Teas, kombucha, tea wine and other derivatives	(Liang et al., 2021)
Biological	Proteomic	Protein - rich foods	(Gallardo et al., 2013)

Biological	Proteomic	Fish	(Jaiswal et al., 2023)
Spectroscopic	Near infrared spectroscopy, mid infrared spectroscopy	Allegedly functional and nutraceutical foods	(Nagy et al., 2022)
Spectroscopic	Raman spectroscopy	Meat, seafood, milk and dairy products, oils and fats, beverages, honey, grains and derivatives	(Xu et al., 2020)
Biological	Proteomic	Seafood	(Y. Wang et al., 2023)
Spectroscopic	Isotope ratio mass spectrometry	Chinese food with geographical indication (GI)	(Li et al., 2023)
Spectroscopic, biological	Metabolomic, mass spectrometry, fourier transform infrared spectroscopy, nuclear magnetic resonance spectroscopy	Alcoholic beverages	(Tabago et al., 2021)
Chromatographic, biological	Genomic, gas chromatography, nuclear magnetic resonance spectroscopy, liquid chromatography, proteomics, metabolomics	Seafood, meat, milk and dairy products, wine and other beverages, GMOs, gelatins	(Böhme et al., 2019)
Chromatographic, spectroscopic,	Ultra-high performance liquid chromatography, high performance liquid chromatography, liquid chromatography, gas chromatography, isotope ratio	Vegetables, fruits and derived products	(Wadood et al., 2020)

biological, electroanalytical	mass spectrometry, mass spectrometry, near infrared spectroscopy, fourier transform infrared, nuclear magnetic resonance spectroscopy, genomic, electronic tongues, electronic nose		
Spectroscopic, chromatographic	Isotope ratio mass spectrometry, nuclear magnetic resonance spectroscopy, mass spectrometry, near infrared spectroscopy, electronic tongue, mass spectrometry, high performance liquid chromatography, gas chromatography, liquid chromatography	Tea with geographical indication (GI)	(Shuai et al., 2022)
Chromatographic, spectroscopic, electroanalytical	Near infrared spectroscopy, raman spectroscopy, fluorescence spectroscopy, nuclear magnetic resonance spectroscopy, electronic tongue, electronic nose, colorimetric sensor array, high performance liquid chromatography, gas chromatography, mass spectrometry, pH meter, mid infrared spectroscopy	Alcoholic beverages	(Arslan et al., 2021)
Spectroscopic, chromatographic, electroanalytical	Fourier transform infrared spectroscopy, electronic nose, electronic tongue, raman spectroscopy, electron spin resonance, electronic eyes, gas chromatography, genomic, high performance liquid chromatography,	Herbs, condiments	(Xu et al., 2023)

	near infrared spectroscopy, hyperspectral imaging spectroscopy, atomic spectroscopy, mass spectrometry		
Spectroscopic, chromatographic, electroanalytical	Mass spectrometry, electronic nose, gas chromatography, liquid chromatography, high performance liquid chromatography	Baijiu with geographical indication (GI)	(Hong et al., 2020)
Spectroscopic	Ultraviolet-visible spectroscopy, near infrared spectroscopy, nuclear magnetic resonance spectroscopy, mid infrared spectroscopy, fourier transform infrared spectroscopy, hyperspectral imaging, mass spectrometry	Oils, cereals, vegetables, beverages, condiments, fruits, fruit juice, coffee, fish, cheese, honey, meat	(Riedl et al., 2015)
Chromatographic, spectroscopic, biological, classical instrumental techniques	High performance liquid chromatography, fourier transform infrared spectroscopy, precipitation, proteomic	Raw materials and derivatives of animal origin	(Rohman et al., 2020)
Spectroscopic, chromatographic, biological	Stable isotope ratio mass spectrometry, high performance liquid chromatography, near infrared spectroscopy, visible spectroscopy, volatilomic, genomic, gas chromatography, mid-infrared	Meat, dairy products	(Prache et al., 2020)

	spectroscopy, nuclear magnetic resonance spectroscopy spectroscopy		
Spectroscopic, chromatographic	Stable isotope ratio mass spectrometry, gas chromatography, high performance liquid chromatography, near infrared spectroscopy	Raw materials and derivatives of animal origin with the name "organic"	(Prache et al., 2022)
Spectroscopic, chromatographic, electroanalytical, biological	Isotope ratio mass spectrometry, near infrared spectroscopy, gas chromatography, ultra-high performance liquid chromatography, raman spectrometry, electronic nose, genomic, volatonic	Rice	(Wadood et al., 2022)
Spectroscopic	Near infrared spectroscopy	Medicinal herbs with geographical indication (GI)	(P. Wang & Yu, 2015)
Spectroscopic	Mass spectrometry, isotope ratio mass spectrometry	Wine, vinegar	(Ríos-Reina et al., 2021)
Spectroscopic	Spectroscopic fluorescence and FT-Raman	Milk, dairy products	(O'Sullivan et al., 2022)
Spectroscopic, chromatographic, biological	Near infrared spectroscopy, raman spectroscopy, nuclear magnetic resonance spectroscopy, ultra-high performance chromatography, genomic	Powdered milk	(Farag et al., 2022)

Chromatographic, spectroscopic	Fourier transform infrared spectroscopy, gas chromatography, nuclear magnetic resonance spectroscopy, near infrared spectroscopy	Essencial oils	(Syafri et al., 2022)
Chromatographic, spectroscopic, biological	Isotope-ratio mass spectrometry, high performance liquid chromatography, nuclear magnetic resonance spectroscopy, metabolomic	Fruits, vegetables and plant-based products	(Francois et al., 2020)
Biological, spectroscopic, chromatographic, eletroanalytical	Metabolomic, high resolution mass spectrometry, electronic nose, electronic tongue, nuclear magnetic resonance spectroscopy, fourier-transform infrared, near and mid infrared, gas chromatography, liquid chromatography	Wine, rice, olive oil, spices, honey	(Mialon et al., 2023)
Spectroscopic	Nuclear magnetic resonance spectroscopy, near-infrared spectroscopy, fourier transform infrared spectroscopy, fluorescence spectroscopy, hyperspectral, raman spectroscopy, ultra-violet spectroscopy,	Wine, meat, milk, oils, juices	(Esteki, Shahsavari, et al., 2018)
Chromatographic, spectroscopic, electroanalytical	Gas chromatography, nuclear magnetic resonance spectroscopy, electronic nose	Meat and meat products	(Lytou et al., 2019)

Spectroscopic, chromatographic	Gas chromatography, enzymatic assays, capillary electrophoresis	Fermented drinks of Mexican with geographical indication (GI)	(Robledo-Márquez et al., 2021)
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