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NANOMATERIAL-BASED SENSORS FOR MILK ADULTERATION DETECTION: ADVANCES AND CHALLENGES

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Milk is a vital component of the human diet, valued for its nutritional benefits and versatility in various food products. However, the adulteration of milk poses a significant threat to consumer health and undermines the integrity of the dairy industry. Adulterants such as water, urea, detergents, and other contaminants can be intentionally or unintentionally added to milk, compromising its quality, safety, and authenticity, necessitating the development of analytical tools for the rapid identification of adulterants in milk products. Traditional and analytical methods lack the required selectivity, sensitivity, and compatibility with complex matrices. In response, researchers have turned to nanomaterial-based sensing systems as promising alternatives. This article explores the mechanisms and performances of various available nano sensors, analyzing their benefits, drawbacks, and applicability to milk products. Additionally, it highlights recent advancements in nano ensemble platforms for the detection of adulterants in milk products.

1. Introduction

The authenticity of milk and the presence of adulterants have become significant concerns in the dairy industry. Adulteration refers to the intentional or unintentional addition of substances to milk, compromising its quality, safety, and integrity. The identification and quantification of adulterants in milk products are crucial to safeguard consumer health, ensure fair trade practices, and maintain the reputation of the dairy industry. The adulteration of milk poses substantial risks to consumer health. Adulterants such as water, urea, formalin, hydrogen peroxide, and contaminants like antibiotics, pesticides, and heavy metals can be introduced during various stages of milk production, processing, and distribution[1], [2], [3], [4]. Consumption of adulterated milk can lead to acute and chronic health issues, including gastrointestinal disorders, allergic reactions, organ damage, and even long-term health risks[5], [6], [7], [8]. Ensuring the authenticity of milk products is, therefore, imperative for safeguarding consumer safety and well-being. It is crucial to understand the various types of milk adulteration and their implications to ensure the production and consumption of safe, high-quality milk products. Figure 1 illustrates the common types of milk adulterants and purpose of their mixing.

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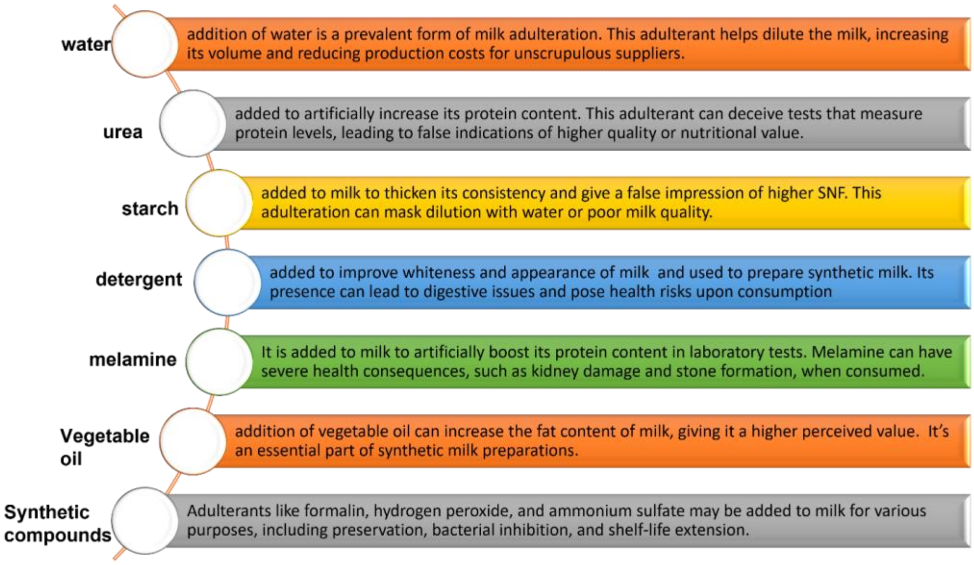

Fig. 1 Common milk adulterants and purpose of their mixing

Traditional methods which are used for milk adulteration detection including organoleptic evaluation chemical tests, physical tests, chromatographic techniques or microbiological tests[9], [10], [11]. Organoleptic evaluation involves visually examining and sensory assessing milk to observe its appearance, including factors such as color, consistency, and the presence of particles or sediment[12], [13]. Any deviations such as unusual color, excessive cloudiness, or abnormal texture may indicate potential adulteration. Sensory evaluation further involves tasting and smelling the milk to detect any off-flavors or odors that could be indicative of adulteration. Chemical tests for milk adulteration detection entail the addition of specific reagents or indicators to milk in order to identify the presence of adulterants[10], [14]. For instance, the introduction of iodine solution to milk can yield a blue color, indicating the presence of starch as starch reacts with iodine. Similarly, the addition of alcohol to milk may cause visible alterations such as curdling or precipitation, revealing the existence of adulterants like detergent or synthetic compounds. Another chemical test involves measuring the pH of milk to assess its acidity or alkalinity. Deviations from the normal pH range can serve as indications of potential adulterants. Physical tests for milk adulteration encompass measurements of freezing point and specific gravity[15]. The freezing point of milk is marginally lower than that of pure water. By measuring the freezing point, it becomes possible to detect added water since adulterated milk will exhibit a lower freezing point compared to pure milk. Additionally, determining the specific gravity of milk offers an indication of its density, which can undergo changes if water or other substances have been introduced. Chromatographic techniques are widely employed for milk adulteration detection. One such technique is Thin-Layer Chromatography (TLC), which entails the separation of milk components on a thin layer of adsorbent material. By comparing the separated spots with standard reference compounds, the presence of adulterants can be identified. Another commonly used technique is High-Performance Liquid Chromatography (HPLC), which separates milk components based on their chemical properties[1], [11]. HPLC can detect specific adulterants or contaminants by comparing

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retention times with known standards. Microbial analysis is a valuable approach for detecting harmful microorganisms, including bacteria, yeast, and mold, in milk[16]. This analysis involves assessing the presence and abundance of these microorganisms, which can indicate the overall hygiene and potential contamination of the milk sample. Elevated microbial counts in milk samples serve as indicators of poor hygiene practices during production, processing, or storage, raising concerns about the safety and quality of the milk. By conducting microbial analysis, potential health risks associated with microbial contamination in milk can be identified, allowing appropriate measures to be taken to ensure consumer safety. The traditional methods for milk adulteration detection, including chemical tests, organoleptic evaluation, physical tests, chromatographic techniques, and microbial analysis, have their own limitations. Chemical tests, while simple and widely used, can lack specificity and may not accurately detect certain adulterants. Organoleptic evaluation, relying on sensory perception, is subjective and can vary among individuals. Physical tests like freezing point and specific gravity measurements provide indirect indications of adulteration but may not always pinpoint specific adulterants[17]. Chromatographic techniques, such as Thin-Layer Chromatography (TLC) and High-Performance Liquid Chromatography (HPLC), require specialized equipment, trained personnel, and time-consuming procedures[18], [19]. Additionally, they may not cover the full range of potential adulterants. Microbial analysis, although important for identifying harmful microorganisms, does not directly detect chemical adulterants. Moreover, these traditional methods can be time-consuming, require multiple steps, and may lack the sensitivity required for detecting trace levels of adulterants. Therefore, the development and utilization of more advanced and specific techniques, such as nanomaterial-based sensors, are being explored to overcome these limitations and enhance the accuracy and efficiency of milk adulteration detection. Nanomaterial-based sensors are highly valued for their potential applications in food and dairy products. Nanomaterials possess unique optical, electrical, and magnetic properties that can be harnessed for a wide range of analytical applications, including extraction, sample preparation, and preconcentration of analytes in various sample types such as food, biological, and environmental samples. Additionally, nanomaterials have a large surface area-to-volume ratio, which enhances their interaction with analytes, leading to increased sensitivity and detection capabilities. Moreover, the easy surface functionalization of nanomaterials enables the attachment of specific receptors or ligands, enabling selective and targeted detection of adulterants in milk and dairy products[20], [21]. The distinctive properties of nanomaterials enable them to interact with analytes, enhancing their extraction efficiency and enabling the concentration of target analytes for subsequent analysis. Nanomaterials can also be integrated with specific analytical methods, such as spectroscopy, chromatography, or electrochemical techniques, to effectively monitor and quantify analytes of interest. The combination of nanomaterials with analytical methods provides improved sensitivity, selectivity, and detection limits, facilitating robust and accurate analysis of complex samples. This integration of nanomaterials with analytical methods opens up new possibilities for advanced and efficient analytical techniques in various fields, including food safety, environmental monitoring, and biomedical research. The development of portable nanosensors that can

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detect adulterants in dairy products without the need for sample preparation or pre-treatment has gained significant attention in recent years. Therefore, a comprehensive review of nanomaterial-based sensors is essential to provide guidance for future research endeavors focused on highly efficient and rapid detection of adulterants in dairy products. This review aims to survey the various nano ensemble-based platforms employed for the detection of dairy adulterants[5].

2. Nanosensors for Dairy Adulterant Monitoring

2.1 Nanomaterials

Nanomaterials are materials with unique properties and functionalities at the nanoscale, typically ranging from 1 to 100 nanometers in size[22]. They exhibit distinct physical, chemical, and biological characteristics compared to their bulk counterparts due to their small size and high surface-to-volume ratio. Nanomaterials can be classified into various types, including nanoparticles, nanotubes, nanowires, nanofilms, and nanocomposites as depicted in Fig. 2.

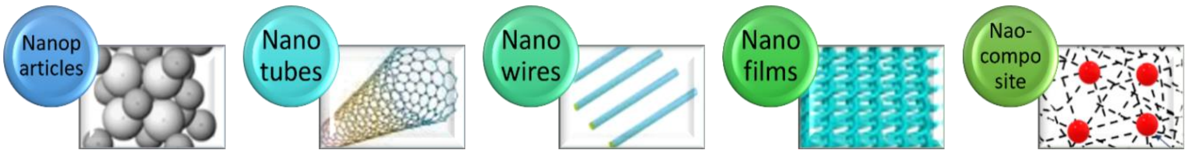

Fig. 2 Different types of nanomaterials

They can be composed of different materials, such as metals, metal oxides, carbon-based materials, polymers, and ceramics. These materials can be engineered and manipulated to exhibit specific properties and functionalities to suit diverse applications including milk adulteration detection. Metal nanoparticles, such as gold (AuNPs) and silver nanoparticles (AgNPs), are commonly utilized in milk adulteration sensors. They offer excellent optical properties, such as surface plasmon resonance, which enables sensitive detection of adulterants through colorimetric or spectroscopic methods. Carbon-based nanomaterials, including graphene, carbon nanotubes (CNTs), and carbon dots (CDs), are widely employed in milk adulteration sensors. These materials possess exceptional electrical properties, high surface area, and good biocompatibility, enabling efficient detection of various adulterants in milk samples. Quantum dots (QDs) are semiconductor nanocrystals with unique optical properties, such as tunable fluorescence emission. QDs are used in milk adulteration sensors to enable sensitive and multiplexed detection of different adulterants simultaneously, enhancing the analytical capabilities of the sensor[23][24]. Molecularly Imprinted Polymers (MIPs) are synthetic polymers designed to selectively recognize and bind specific target molecules. In milk adulteration sensors, MIPs can be used as recognition elements to detect and quantify adulterants with high selectivity and sensitivity. Metal-Organic Frameworks (MOFs are highly porous materials composed of metal ions or clusters coordinated with organic ligands[25] [23], [26], [27], [28]. MOFs have a large surface area and tunable pore structures, making them suitable for the adsorption and detection of adulterants in milk. Nanocomposites are hybrid materials composed of two or more types of nanomaterials[29].

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They combine the properties and functionalities of different nanomaterials, providing enhanced sensing capabilities for milk adulteration detection. These key nanomaterials offer unique properties, such as high sensitivity, selectivity, and stability, which make them well-suited for the development of milk adulteration sensors. By leveraging the advantages of these nanomaterials, researchers can design and fabricate innovative sensing platforms that enable rapid, accurate, and reliable detection of adulterants in milk products, ensuring consumer safety and product quality. When choosing nanomaterials for milk adulteration sensing, several criteria should be considered to ensure their suitability and effectiveness. The selection criteria for choosing nanomaterials are illustrated in the Fig. 3.

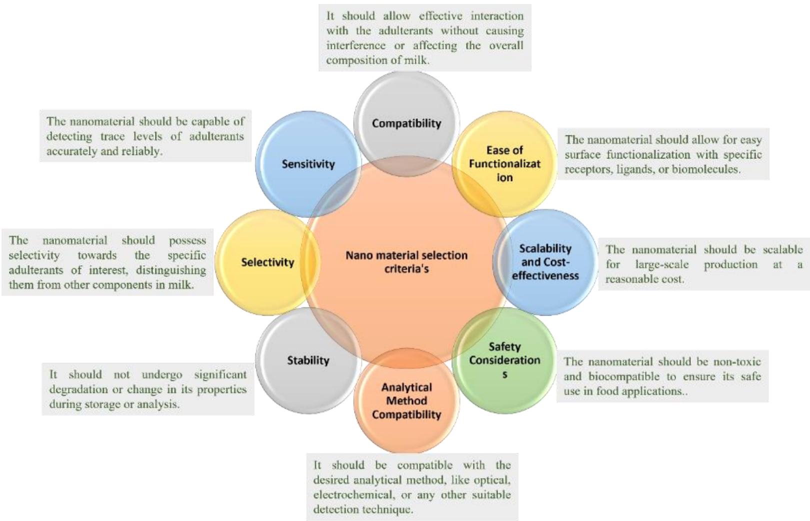

Fig. 3 Selection criteria of a nanomaterial

2.2 Nanosensors

Nanosensors are devices or systems that utilize nanomaterials to detect and quantify various analytes or substances with high sensitivity and selectivity. They are designed to operate at the nanoscale, utilizing the unique properties and interactions of nanomaterials for sensing applications. Nanosensors can be used in a wide range of fields, including healthcare, environmental monitoring, food safety, and industrial processes. The block diagram depicted in Fig. 4 illustrates the key components of nanosensors, which play a crucial role in their functioning and detection capabilities.

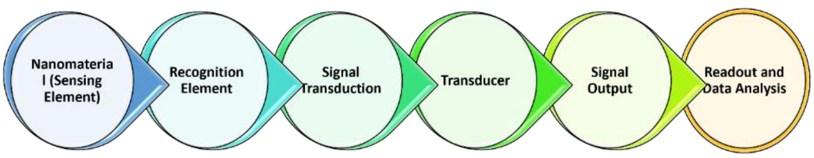

Fig. 4 Block diagram of a typical nanosensor

Fig. 5 classifies various types of nanosensors that can be utilized for detecting adulteration in milk. These nanosensors offer diverse sensing capabilities and can play a crucial role in ensuring the quality and authenticity of milk products.

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2.2.1 Optical Nanosensors

Optical nanosensors utilize optical signals, such as absorbance, fluorescence, or surface plasmon resonance, for the detection and quantification of adulterants in milk samples[5]. A common strategy in optical nanosensors involves the use of metal nanoparticles, such as gold or silver nanoparticles, which exhibit unique optical properties due to localized surface plasmon resonance. These nanoparticles can be functionalized with specific ligands or receptors that selectively bind to adulterants, resulting in a measurable change in the optical signal. By analyzing the intensity or wavelength of the optical signal, the concentration of the adulterant in the milk sample can be determined.[30] Alternatively, fluorescent nanomaterials, including quantum dots or carbon dots, are employed in optical nanosensors. Upon excitation, these nanomaterials emit fluorescence, and the presence of adulterants can modulate the fluorescence intensity or wavelength. The specific interaction between the nanomaterial and the adulterant leads to changes in the fluorescence signal, enabling sensitive detection and quantification of adulterants in milk. Optical nanosensors offer several advantages for milk adulteration detection, including real-time monitoring, high sensitivity even at low concentrations, and the potential for simultaneous analysis of multiple adulterants. Moreover, optical nanosensors can be integrated into portable and miniaturized devices, facilitating on-site and rapid analysis of milk samples. Nonetheless, challenges persist in the development and implementation of optical nanosensors for milk adulteration detection. These challenges encompass ensuring the stability and reproducibility of the nanosensor response, optimizing the sensor design to accommodate complex milk matrices, and addressing potential interference from components within the matrix. Continuous research and advancements in the field of optical nanosensors are imperative for their practical application in ensuring the authenticity and quality of milk products.

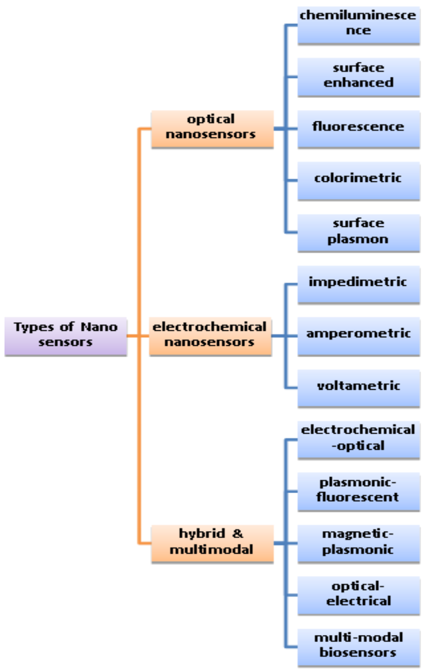

Fig. 5 Classification of nanosensors

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2.2.2 Electrochemical Nanosensors

Electrochemical nanosensors utilize electrochemical signals to detect and quantify adulterants in milk samples. Electrochemical nanosensor is based on the use of metal nanoparticles, such as gold or platinum nanoparticles, as sensing elements. These nanoparticles can be functionalized with specific receptors or ligands that selectively bind to adulterants in milk[23], [31], [32], [33]. The interaction between the adulterant and the nanomaterial generates a measurable electrochemical signal, such as changes in current or potential, which can be detected and correlated with the concentration of the adulterant. Alternatively electrochemical nanosensors use of carbon-based nanomaterials, such as graphene or carbon nanotubes. These nanomaterials offer excellent electrical conductivity and large surface area, allowing for efficient electron transfer and enhanced sensitivity in detecting adulterants[34], [35], [36]. Functionalization of these carbon-based nanomaterials with specific biomolecules or receptors enables selective and sensitive detection of adulterants in milk samples. Electrochemical nanosensors offer several advantages for milk adulteration detection, including rapid response, high sensitivity, and the potential for miniaturization and portable devices. They can be integrated into electrochemical platforms, such as electrochemical cells or biosensors, enabling real-time monitoring and on-site analysis of milk samples. However, challenges exist in the development of electrochemical nanosensors for milk adulteration detection. These challenges include optimizing the sensor design to improve sensitivity and selectivity, minimizing interference from matrix components in milk, and ensuring long-term stability and reproducibility of the sensor response. Continued research and advancements in electrochemical nanosensors are crucial for their practical implementation in ensuring the quality and authenticity of milk products. These nanosensors hold great promise for providing reliable and efficient detection methods for milk adulteration, contributing to consumer safety and confidence in the dairy industry.

2.2.3 Hybrid and Multi-Modal Nanosensors

Hybrid and multi-modal nanosensors combine different types of nanomaterials or sensing modalities to provide synergistic detection capabilities. In hybrid nanosensors, multiple nanomaterials with complementary properties are integrated to create a sensor platform with improved performance[5]. For example, a hybrid nanosensor may combine metal nanoparticles for optical sensing with carbon-based nanomaterials for electrochemical sensing. This combination allows for the simultaneous detection of adulterants using different sensing mechanisms, enhancing the overall sensitivity and selectivity of the sensor[37]. Multi-modal nanosensors, on the other hand, employ multiple sensing modalities to provide comprehensive and reliable detection of adulterants in milk. These nanosensors can integrate optical, electrochemical, and/or other sensing techniques into a single platform. For instance, a multi-modal nanosensor may combine fluorescence sensing, impedance spectroscopy, and surface-enhanced Raman spectroscopy (SERS) to simultaneously detect different types of adulterants in milk, achieving enhanced sensitivity and specificity. The integration of hybrid and multi-modal nanosensors offers several advantages for milk adulteration detection[32], [33], [38]. It allows for the complementary use of different sensing mechanisms, enabling

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more comprehensive analysis and reducing the chances of false positive or false negative results. Moreover, the combination of multiple nanomaterials or sensing modalities can compensate for individual limitations, leading to improved accuracy and reliability in detecting a wide range of adulterants in complex milk matrices.

3. Nano ensemble platforms

Nano ensemble platforms refer to integrated systems or structures that utilize nanomaterials in combination with other components to create highly efficient and sensitive detection platforms for various applications, including the detection of adulterants in milk. These platforms typically consist of multiple nanomaterials or nanocomposites with distinct functionalities, which work synergistically to enhance the sensing capabilities and performance[39]. Some common examples of nano ensemble platforms for milk adulteration detection include:

3.1 Nanoparticle-Embedded Matrices

These platforms involve the incorporation of nanomaterials, such as metal nanoparticles or quantum dots, into a matrix material, such as polymers or hydrogels. The nanomaterials act as sensing elements, while the matrix provides stability, compatibility, and support for the nanomaterials. The combination of the matrix and embedded nanoparticles enables enhanced detection sensitivity and selectivity for adulterants in milk[40].

3.2 Nanowire Arrays

Nanowire arrays consist of vertically aligned nanowires, such as silicon or metal oxide nanowires, that are functionalized with specific receptors or ligands for target adulterants[23]. The nanowires provide a large surface area for capturing the analytes, while the functionalization enhances the selectivity. The captured adulterants can be detected through various signal transduction mechanisms, such as electrical or optical measurements[41].

3.3 Nanoparticle-Modified Electrodes

In these platforms, electrodes, such as carbon electrodes or gold electrodes, are modified with functionalized nanoparticles, such as metal nanoparticles or semiconductor nanoparticles. The nanoparticles serve as sensing elements, and their functionalization enables specific detection of adulterants. The modification of the electrodes enhances the electrochemical response, leading to improved sensitivity and selectivity[42], [43].

3.4 Nanocomposite Films

Nanocomposite films are thin films composed of nanomaterials, such as metal nanoparticles, carbon-based materials, or nanoclays, dispersed within a polymer matrix. These films can be deposited onto surfaces or substrates to create sensing platforms for the detection of adulterants. The combination of different nanomaterials in the film allows for synergistic effects, enhancing the sensitivity and stability of the detection system[44], [45].

3.5 Hybrid Nanostructures

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Hybrid nanostructures involve the combination of different types of nanomaterials, such as metal nanoparticles, quantum dots, or carbon-based nanomaterials, into a single structure. These structures can have unique properties that arise from the synergistic effects of the combined nanomaterials. The hybrid nanostructures can be tailored for specific applications, including the detection of adulterants in milk, by incorporating appropriate receptors or ligands.

These are just a few examples of nano ensemble platforms used for milk adulteration detection. The integration of different nanomaterials or nanocomposites into well-designed structures or systems allows for enhanced sensitivity, selectivity, and stability, enabling efficient and reliable detection of adulterants in milk[46]. The development and optimization of nano ensemble platforms continue to advance, with researchers exploring novel combinations and architectures to further improve the performance and practical application of these platforms.

Conclusion

This article shed light on the significance of detecting adulteration in milk and the limitations of traditional methods in achieving accurate and efficient results. The potential of nanomaterial-based sensors as effective alternatives, focusing on optical and electrochemical nanosensors, as well as hybrid and multi-modal nanosensors is also discussed. These nanosensors offer numerous advantages, including high sensitivity, selectivity, real-time monitoring, and the potential for on-site analysis. They have the capability to detect a wide range of adulterants in milk, ensuring consumer safety and maintaining the integrity of the dairy industry. Continued research and advancements are necessary to optimize sensor design, improve sensitivity, and enhance selectivity. Furthermore, the integration of nanosensors into practical applications requires careful consideration of compatibility, scalability, and cost-effectiveness. Nanomaterial-based sensors hold great promise in revolutionizing the detection of adulterants in milk. Their development and implementation will contribute to the assurance of milk authenticity, safeguarding consumer health, and instilling confidence in the dairy industry. Further exploration and collaboration in this field will drive innovation, leading to the development of more efficient, reliable, and accessible nanosensors for milk adulteration detection.

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