Detection Methods for Wheat Freshness: A Review

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Abstract: The quality of post-harvest wheat inherently declines over time during storage, posing significant challenges in its preservation. Accurately and promptly identifying the wheat freshness is not merely a complex scientific endeavor requiring urgent attention; it is also a pragmatic necessity tied to ensuring the safety of grain storage operations. This article offers a comprehensive overview of the currently prevalent research detection methodologies for wheat freshness, meticulously summarizing the fundamental detection principles and preliminary research findings associated with each approach. Furthermore, it engages in a rigorous analysis and discussion of these methods, culminating in insightful thoughts and recommendations for future research endeavors in this technological domain. The article aims to furnish grain industry professionals and researchers with novel, enlightening, and invaluable references to guide their explorations in related fields.

Keyword: Grain Storage; Wheat; Detection Methods; Storage Duration; Freshness Degree

1. Introduction

China stands as the world's preeminent wheat producer, assuming a pivotal position in global and domestic grain consumption patterns, reserves management, and trade dynamics. Its substantial production and storage capacity of wheat underscores this status [1-2]. Over the recent decade spanning from 2015 to 2022, wheat output in China has consistently surpassed the milestone of 130 million tons, culminating in a remarkable figure of 138 million tons in 2022 alone, indicative of the country's robust agricultural prowess [3].

As the duration of storage elongates, the internal framework of the wheat gradually undergoes relaxation, accompanied by a decline in enzyme activity, respiratory capability, and overall vitality. This gradual degradation process ultimately results in the deterioration of the grain [4]. Notably, pertinent research has illuminated [5-6] that initially, the culinary and baking qualities of wheat enhance with extended storage, peaking around the four-year mark, before the baking quality commences a downward trend. Consequently, the prevailing consensus within the grain industry is that wheat kept in storage for over five years under standard conditions ceases to be viable for either continued storage or consumption, necessitating prompt intervention through the reserve rotation mechanism. Failure to do so may give rise to substantial waste and pose challenges to food security.

However, due to the diverse specific conditions of wheat storage across different regions, a simplistic, uniform, and fixed implementation of rotation based solely on time cycles can also lead to unnecessary waste of human, material, and financial resources. Therefore, there is a practical need in the grain storage industry for precise detection of wheat freshness. The wheat freshness is a comprehensive concept that scientifically reflects the harvest years of wheat and the degree of quality deterioration during storage. It involves various aspects such as the appearance characteristics, chemical properties, and enzymatic activities of wheat. By comprehensively considering these factors, we can scientifically judge the freshness of wheat, providing important references for wheat storage and processing.

By actively integrating theories from other relevant disciplines and expanding the knowledge boundaries of grain information detection and analysis, we explore effective and feasible precise detection methods for freshness, aiming to robustly support safe grain storage early warning decisions and effectively enhance the efficiency of grain storage operations. In summary, the issue of detecting wheat freshness is crucial for grain storage safety and even overall food security. It holds extremely important practical significance and theoretical research value.

Over the years, numerous researchers have diligently delved into this domain, yielding fruitful outcomes that have significantly contributed to the field. Leveraging our extensive practical scientific research experience, we are dedicated to meticulously examining and analyzing existing methodologies and pertinent studies. Our objective is to provide a solid foundation for future enhancements to these methods and inspire the development of more comprehensive and innovative methodologies, thereby advancing the overall landscape of this research area.

According to their principles and application methods, the detection can mainly be divided into sensory the detection methods for wheat freshness can mainly be divided into sensory detection methods, chemical detection methods, and other novel electronic information detection technologies, which is shown in Figure 1. Among them, chemical methods and electronic information detection technologies are the focus of this review, which will be introduced in the third and fourth parts respectively. The fifth part presents the conclusion and future research directions.

2. Sensory Detection Method

Sensory detection method mainly through the human visual, olfactory, taste and other sensory organs of wheat appearance, smell, taste and other evaluation, in order to preliminarily determine the freshness of wheat.

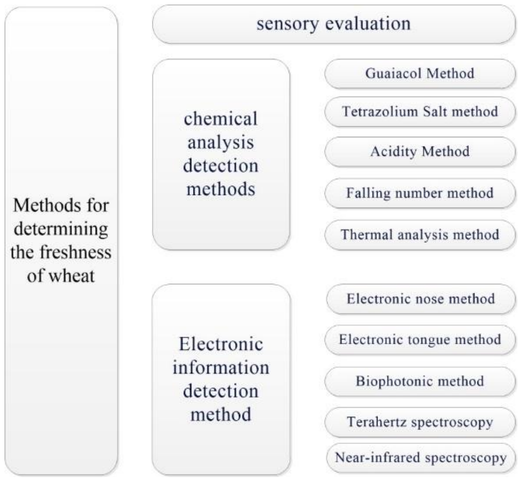

Figure 1. Research Methods for Determining the Freshness of Wheat

The evaluation rules [7] clearly outline that wheat is deemed suitable for continued storage solely when its color, odor, gluten water absorption capacity, and tasting score values concurrently fulfill the "suitable for storage" criteria specified in Table 1. In cases where the color and odor are within normal parameters, yet either the gluten water absorption or tasting score falls under the "slightly unsuitable for storage" threshold in Table 1, the wheat is classified as slightly unfit for storage and necessitates prompt rotation out of storage. Conversely, if any of the parameters—color, odor, or tasting score—meets the "heavily unsuitable for storage" standard in Table 1, the wheat is immediately deemed heavily unfit and must be scheduled for immediate removal from storage facilities.

According to the "Rules," the determination of wheat suitability for storage relies heavily on human sensory evaluation, which is subject to considerable subjectivity. Moreover, the storage quality indicators only determine whether wheat is suitable for storage, and it is difficult to establish an objective quantifiable standard as a basis for judging new wheat [8].

3. Chemical Analysis Detection Methods

The chemical detection method evaluates the freshness of wheat by measuring changes in certain chemical components or chemical reactions within the wheat, such as Guaiacol Method, Tetrazolium Salt Method, etc.

3.1 Guaiacol Method

The principle of the guaiacol method is an indirect freshness test for wheat based on the properties of peroxidase, which is widely present in various animals, plants, and microorganisms. In the presence of hydrogen peroxide, the peroxidase in wheat can catalyze the formation of oxygen atoms from hydrogen peroxide, causing guaiacol to oxidize and change color. Wheat from different storage years contains varying amounts of peroxidase, resulting in distinguishable colors.

Table 1. Wheat Storage Quality Determination Indicators

Item	Suitable for Storage	Slightly Unsuitable for Storage	Heavily Unsuitable for Storage
Color, Odor	Normal	Normal	Basically Normal
Gluten Water Absorption (%)	≥180	<180	-
Tasting Score Value (points)	≥70	≥60and<70	<60

However, the guaiacol method is influenced by many factors when measuring peroxidase activity, such as incubation time, pH value, temperature, etc. This results in poor accuracy and reproducibility of the test results. He et.al [9] used this method for rapid freshness identification of wheat and found the following issues: large errors often occur in multiple parallel treatments of the same sample; poor grain coloration; and the reaction proceeds indefinitely. Additionally, the method involves a slight difference between the color development time and the maintenance time, making it difficult to accurately control, especially for wheat samples with similar storage durations, where the differences in color intensity are not significant, leading to crude results unsuitable for quantitative assessment.

3.2 Tetrazolium Salt Method

For grain kernels or seeds with vitality, once they have absorbed enough water, the dehydrogenase enzymes within the embryo cells regain activity. With proper reaction treatment, the embryo tissue of the grain kernels or seeds can be stained red. However, in the embryo cells of non-viable grain kernels or seeds, dehydrogenase does not catalyze and therefore the embryo tissue does not undergo a staining reaction. This is the basic principle of the tetrazolium salt staining method. Yang et.al applied the tetrazolium salt method to determine the freshness of wheat [10]. The results showed that the tetrazolium salt staining method has several advantages: clear color indication, simple operation, rapid processing, no need for expensive instruments, and the ability to quantitatively express the degree of wheat aging. Ren et.al [11] improved experimental conditions and proved that the tetrazolium salt staining method can distinguish whether new wheat within 2 months has completed maturation, as well as wheat of the same variety but with different storage years (up to 2 years).

However, this method has a long staining time, the results are complex to determine, and detailed microscopic examination is required to assess the staining of various tissue structures in the embryo, making it difficult to apply in practice [12].

3.3 Acidity Method

Wheat and rice are considered an acidic food. Its acidity mainly stems from the decomposition of phosphoric acid, fats, proteins, and carbohydrates, resulting in substances such as fatty acids, phosphoric acid, acid phosphates, lactic acid, acetic acid, amino acids, etc. [13]. The acidity method is a technique used to identify the storage duration or freshness of wheat grains by measuring differences in their acidity values, which includes the acidity indicator method and the fatty acid value method.

The acidity indicator method uses the color change of acid-base indicators to determine the freshness of grains. The principle is that wheat stored for a longer time will have a lower pH value due to hydrolysis compared to wheat stored for a shorter period; thus, the freshness of wheat can be determined by the color change mechanism of acid-base indicators. Research [11] shows that the acidity indicator method can effectively differentiate between fresh and old wheat, but it cannot provide a quantitative measure of the storage duration.

During storage, the most rapid and noticeable quality change in wheat occurs in lipid substances. Wheat produces free fatty acids due to hydrolysis. Studies have shown that under certain storage conditions, the fatty acid value of wheat tends to increase with the number of years stored [14-15].

However, factors such as the quality and storage conditions of wheat can affect changes in the fatty acid value. Additionally, there are several factors that can influence the measurement of the fatty acid value, such as: the extraction time of solvents; the variation in the standing time after filtration; the national standard method for measuring fatty acid value is time-consuming, and there's variability in the visual identification of the titration endpoint. As a result, the standardization of the operator's work is critical to the accuracy of the measurement results, with subjective factors having a significant impact.

3.4 Falling Number Method

Falling number is an international recognized indicator of alpha-amylase activity [16]. The higher the alpha-amylase activity, the lower the falling number. Conversely, the lower the activity, the higher the number. The falling number value is defined as follows: In a viscometer tube, a certain amount of wheat flour and water mixture is immersed in a water bath to obtain a paste. The paste undergoes liquefaction under the action of alpha-amylase; the time (s) required for the stirrer to pass through this paste and freely fall a specific distance from the start of immersion in the water bath is the falling number value of the wheat [17-18]. Multiple studies have shown a good correlation between the change in falling number value and the storage duration of wheat. As storage time increases, the falling number value rises, reflecting the deterioration of wheat quality during storage and the length of storage [19-20]. Currently, as the testing process of this method involves a variety of instruments and equipment, requires deep human intervention, and some steps are currently irreplaceable, such as the operation of measuring the sedimentation time of the stirrer, there have been no reports of related automated products; this limits the application of this method in rapid detection and on-site testing research. Additionally, the results of this method are easily influenced by temperature and moisture content in wheat [21]. There is currently no specific or clear quantitative model proposed to describe the correlation between storage duration and falling number value; these aspects require further in-depth research.

3.5 Thermal Analysis Method

Thermal analysis is a technique for studying certain physical and chemical changes that occur in materials as they are heated or cooled. Zhan [22] used thermal analysis techniques such as thermogravimetry (TG), differential scanning calorimetry (DSC), and dynamic thermomechanical analysis (DMA) to study wheat at different storage times. The results showed that DMA has a good ability to measure storage time, with certain quantitative relationships between related parameters and storage time, and a high correlation coefficient (>0.8). In 2021, Liang et.al [23] combined nuclear magnetic resonance technology with DSC technology. The results showed that long-term storage of wheat affected by environmental factors would lead to an increase in bound water content, a decrease in free water content, and a decrease in gelatinization temperature, among other regular responses. However, they did not provide a clear temporal domain characteristic description related to the storage duration of wheat or other scientific conclusions. Additionally, this method requires grinding the wheat into flour or pressing it into tablets. The process demands a high level of specialization, and quantitative standards for the quality of the ground flour and tablet requirements have not been provided. The measurement process is relatively complex and not suitable for rapid on-site determination.

4. Electronic Information Detection Method

Electronic information detection methods primarily involve the use of modern electronic and information processing technologies to rapidly and accurately assess the freshness of wheat. This includes the use of electronic noses, optical sensors, terahertz waves, etc.

4.1 Electronic Nose Method

The electronic nose, also known as an odor scanner, is an instrument composed of a selective array of electrochemical sensors and appropriate identification methods. It can quickly identify the odor information emitted by the sample being tested, indicating the hidden characteristics of the sample. It is a rapid food detection instrument developed in the 1990s [24]. Zhao et.al [25] used an electronic nose to classify wheat stored for different times and at different temperatures. The study showed that the content of volatile components in wheat (hexanol, octanol, pentadecane, hexadecane, etc.) was significantly correlated with the fatty acid value, falling number, tasting score, and gluten water absorption rate. Zhang et.al [26] used an electronic nose to compare the concentrations of volatile gases released by wheat from five different aging years (2000-2004). Preliminary results show the feasibility of applying the electronic nose method to the production year labeling. When the electronic nose identifies the volatile odors of wheat, it does not require a complicated sample pretreatment process and has advantages such as low cost, simple testing, quick response, and being environmentally friendly. However, its detection accuracy can be constrained by factors such as the manufacturing process of the sensors, sensitive film materials, and data analysis methods. Therefore, further in-depth research into targeted system manufacturing processes, materials, and analysis methods is needed to ensure the robustness of the system's detection.

4.2 Electronic Tongue Method

The electronic tongue is a novel intelligent taste sensing system, also known as an artificial taste system or taste sensor. It is based on bionic principles and consists of a multi-sensor array and multivariate statistical analysis subsystems. By simulating human taste, it can perform qualitative and quantitative analyses of complex solution samples. It has the advantages of simple operation, rapid detection, portability, good reproducibility, and strong objectivity [27-28]. Guo et.al [29-31] utilized a self-developed three-electrode electronic tongue system to test samples of wheat from five different production years (harvested between 2013 and 2017). They applied Wavelet Packet Transform (WPT) for data preprocessing and feature extraction of the collected wheat samples, followed by using the CELEDAT classification algorithm based on ensemble learning to conduct classification research on the wheat samples from the different production years. The results showed that the system achieved the best classification accuracy of 96.6%, with an F1-score value of 0.963, preliminarily demonstrating the feasibility of using the electronic tongue detection method to detect different production years of wheat. However, as the researchers mentioned, this study only conducted preliminary qualitative discriminant analysis on wheat aged for different years and requires further deepening and specification.

4.3 Biophotonic Method

Biophoton emission (BPE) also known as ultraweak luminescence (UWL) is a ubiquitous phenomenon of living systems. Biophotons originate from non-localized coherent electromagnetic fields within living matter, offering a comprehensive indicator of the fundamental characteristics of biological systems with high sensitivity. They can reveal subtle changes inside biological systems and demonstrate the faint influences of the external environment [32]. Common methods for detecting biophoton emission signals include spontaneous biophoton emission, water-stimulated biophoton emission, and delayed luminescence. Bao et.al [33] conducted an analytical study on the delayed luminescence information of wheat samples from different varieties and years. The results showed that there were differences in the ultraweak luminescence intensity of wheat samples of the same variety across production years and viability, to some extent validating the feasibility of using biophoton analysis technology to assess the storage quality of wheat.

Gong et.al [34-35] tested the spontaneous biophoton data of wheat samples from five different storage years (2015-2019), calculated features such as the multiscale permutation entropy, mean value, variance, and standard deviation of the data, and constructed a feature space. They then established a classification model using a BP (Back Propagation) neural network to attempt exploring the quantification and standardization issues of the freshness of wheat grains. The results showed that MPE could serve as a distinctive feature to identify the freshness of wheat, and the accuracy rate could reach over 90% via a BP neural network. The research findings preliminarily verified the feasibility of using spatial domain features of wheat biophoton emission data for the quantitative standardization of wheat grain freshness.

4.4 Terahertz Spectroscopy

The terahertz wavelength ranges from 0.03

mm to 3 mm, situated between infrared and microwave on the electromagnetic spectrum. Compared with other bands, terahertz radiation possesses unique characteristics such as instantaneousness, broad bandwidth, coherence, low energy, penetrability, and absorption. Its detection capabilities can effectively fill the gaps left by other technologies, obtaining sample information that is unattainable through other means. The main components of agricultural products, such as water, proteins, fats, and starches, exhibit rich absorption features in the terahertz spectral region [36]. In recent years, scholars have conducted meaningful research attempts using terahertz spectroscopy to address the challenge of identifying aged wheat. Wang and others collected attenuated total reflection Terahertz (ATR-THz) spectral data from wheat samples with different degrees of aging under controlled conditions and conducted research on developing a rapid nondestructive identification model for aged wheat, as well as its validation and evaluation. The absorption coefficient spectrum PLA-DA (Partial Least Squares Discriminant Analysis) model achieved an identification rate of over 81%, with external validation sample recognition not falling below 73%; the refractive index spectrum PLA-DA model had an identification rate above 76%, with external validation sample recognition exceeding 84%. The research findings suggest that ATR-THz technology has potential for nondestructive identification of aged wheat [37]. Ge et.al [38] attempted to use THz time-domain spectroscopy (THz-TDS) to sample the terahertz time-domain spectral data of wheat samples from four different storage years (2007~ 2011), and calculated the refractive index and absorption coefficient of wheat from different storage years in the 0.2THz-2THz range. The experimental data showed that there were differences in the refractive index and absorption coefficient among the wheat samples stored in different years.

4.5 Near-infrared Spectroscopy

Apart from the visible light region, the near-infrared spectral region, with a wavelength range of 780-2526 nm, is the earliest area of electromagnetic waves that people have studied and understood. Typically, the near-infrared spectrum is divided into two bands: short-wave (780-1100 nm) and long-wave (1100-2526 nm). Near-infrared spectroscopy involves associating the calibrated data of a substance's near-infrared absorption spectrum with the sample's reference data to establish a relationship model between the sample's absorption spectrum and its components. This serves the purpose of classification, identification, and detection in applied research. Near-infrared spectroscopy technology, with its advantages of low cost, speed, precision, and non-destructiveness, has been widely used in various research applications for wheat quality testing [39]. Yang et.al [40] selected six wheat samples from different production years to test the infrared spectra of the wheat and compared the results with those from the tetrazolium salt staining method. They preliminarily concluded that the transmittance near 3300 and 1650 cm-1 decreased with the extension of storage years. Liu [41] used Fourier Transform Infrared Spectroscopy (FTIR) to study wheat stored for different times. The results showed that the spectra of samples from the same variety but different years were similar, yet there were differences in the aspect of absorption intensity ratios, which changed with the increase of storage time. Wu et.al [42] used a Fourier Transform Infrared Spectrometer to collect near-infrared spectral data of 45 wheat seed samples at the beginning of natural aging, and after 4, 7, and 9 months. They used the standard deviation of the spectral data as model sample data and employed a multi-class support vector machine method to conduct research on classifying and qualifying the four degrees of natural aging in wheat seeds by developing classification models. The research results indicated that this approach could rapidly determine the degree of short-term natural aging in wheat seeds, offering a convenient detection method for nondestructive monitoring and exploitation of changes in physiological characteristics during seed storage.

However, the above studies did not involve an analysis of the impact of variety factors. Additionally, this method is quite sensitive to the moisture content of the samples; that is, the inherent free water content in wheat grains could directly affect the results obtained by infrared spectroscopy. At the same time, moisture content is also an important quality indicator for wheat samples. Yet, the corresponding literature has not clearly presented an analysis of how these factors might influence the detection results.

5. Conclusions and Future Studies

This article reviews the existing methods for detecting wheat freshness and summarizes the advantages and disadvantages of each method. In summary, the complexity of implementation, research conclusions, and method limitations of the detection methods reviewed in the literature are summarized in Table 2.

Underpinned by extensive practical experience, the theory emphasizes that wheat grains are dynamic, non-rigid colloidal systems, embodying an organic unity where their essence arises from the intricate interplay of physicochemical and biochemical properties inherent in their constituent elements. The attributes of each indicative factor are interconnected rather than isolated, signifying that the overall vitality of wheat grains is holistically influenced by the intertwined effects of both internal and external factors. Contemporary research endeavors that narrowly focus on simplistic amalgamations of existing indicators or isolated detection techniques fall short in comprehensively assessing the total quality of wheat.

Table 2. Summary of Existing Detection Methods

	Method	Implementation complexity	Research conclusions	Limitations
Chemical analysis detection	Guaiacol	★★★★	● Suitable for rapid qualitative assessment of freshness	● The color development time and the duration of the result are minimally different, making the outcome difficult to control. ● Large detection errors, poor reproducibility
	Tetrazolium Salt	★★★	● Simple operation, fast, and capable of quantitative expression.	● Staining time is relatively long, not suitable for on-site use.
	Acidity	★★★★	● A positive correlation between the fatty acid value of wheat and the storage period.	● Factors affecting the determination of fatty acid value are also numerous, making the results difficult to control.
	Falling number	★★★★	● Good correlation with the storage period of wheat.	● Requires significant human intervention and some steps are currently irreplaceable. ● The quantitative relationship with storage duration is unclear.
	Thermal analysis	★★★	● The relevant parameters show a certain quantitative relationship with storage time.	● Requires a high level of specialization, not suitable for on-site measurement.
Electronic information detection	Electronic nose	★★	● Application to storage period calibration has certain feasibility.	● Robustness needs to be improved. ● Further quantitative analysis and research are needed.
	Electronic tongue	★★★★	● Application to storage period calibration has certain feasibility.	● Research needs further deepening.
	Near-infrared spectroscopy	★★★	● The transmittance of specific spectral bands decreases with the extension of years. ● For the same variety, the spectral absorption intensity of samples from different years increases with storage time.	● The research did not involve the analysis of factors such as variety. ● The method is quite sensitive to the moisture content of the samples.
	Terahertz spectroscopy	★★★	● In specific spectral bands, there are differences in both the refractive index and absorption coefficient of wheat samples stored in different years.	● Pretreatment is complex, not suitable for on-site testing.
	Biophotonic	★★	● Have some feasibility	● The research needs to be further deepened.

Many current studies are still at the stage of qualitative analysis, with insufficient clear data characteristic analysis or understanding. Most research applies mainstream analysis limited to a single aspect of data time-domain characteristic analysis, merely conventional applications of classical statistical methods. However, there are few reports on the integration of various transform domain data analysis methods in modern data analysis, which will inevitably lead to insufficient and wasteful information mining and utilization, and is not conducive to complete, accurate and stable quantitative analysis. Therefore, the full exploration of time domain, spatial domain, frequency domain (transform domain), and other information from wheat storage quality data, as well as targeted multi-domain joint representation data analysis techniques, are gaps that urgently need to be filled in this field of research.

In conclusion, detecting the wheat freshness is crucial for ensuring food safety and quality. Although various detection methods have been developed over the years, there is still a need for further research to improve their accuracy, efficiency, and accessibility. Future studies could focus on the following aspects:

(1) Selecting a highly sensitive data source rooted in fundamental principles, capable of mirroring the wheat's holistic vitality or improving the sensitivity and specificity of existing detection methods, especially for early-stage degradation or slight changes in wheat freshness.

(2) Developing Multimodal Integration techniques to integrate information from different sources (e.g., spectroscopy, electronic nose, machine vision) and enhance the overall performance of detection systems.

(3) Investigating the influence of environmental factors (e.g., temperature, humidity) on the reliability and stability of detection methods, and developing adaptive algorithms to compensate for these effects.

(4) Exploring the potential of novel detection technologies, such as hyperspectral imaging and nanotechnology, in assessing wheat freshness more accurately and efficiently.

(5) Establishing standardized protocols and databases for evaluating and comparing different detection methods under various conditions, facilitating the transfer and application of research findings in industry practice.

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