



Article

Analysis of Modern Methods for Controlling Moisture in Compound Feed and Metrological Capabilities of the Dielectric Method

Avezov Nodirbek Egambergonovich¹, Mansurbek Qurbanbayev²

1. Doctor of Philosophy (PhD) in Technical Sciences
2. Chief Specialist at the Khorezm Branch of "UzNIM" State Enterprise

Abstract: The article presents a comparative analysis of modern methods for moisture control—one of the key indicators determining product quality and shelf life in compound feed production. The operating principles, accuracy, speed, and application areas of thermogravimetric, Karl Fischer, infrared drying, near-infrared spectroscopy (NIRS), microwave, conductometric, and dielectric methods are examined. The physical foundations of the dielectric method, its metrological characteristics, factors influencing measurement results, and issues regarding measurement uncertainty estimation are covered in detail. Recommendations are provided for improving the normative and technical framework for calibrating and verifying dielectric moisture meters.

Keywords: compound feed, moisture content, dielectric method, dielectric permittivity, moisture meter, metrological support, calibration, measurement uncertainty, thermogravimetric method, quality control.

1. Introduction

The sustainable development of the livestock and poultry sectors largely depends on the quality of the forage base. Compound feed is a complex multi-component product whose nutritional value, shelf life, and technological properties are directly determined by its moisture content. When moisture content exceeds the standard limit, mold fungi develop in the product, mycotoxins accumulate, the granulation process is disrupted, and the shelf life of the product is sharply reduced. Conversely, an over-dried product leads to increased energy consumption and economically unjustified mass loss [1]. Therefore, in accordance with current standards, the moisture content of compound feed is generally required to be maintained within the range of 12–14.5%. Under production conditions, rapid, reliable, and metrologically grounded moisture control is an essential prerequisite for managing the technological process. Today, there are dozens of moisture determination methods available, which differ significantly from one another in terms of accuracy, speed, cost, and automation capabilities. The aim of this article is to conduct a comparative analysis of modern moisture control methods for compound feed and to reveal the metrological capabilities of the dielectric method widely used in production, the sources of uncertainty arising from its application, and ways to improve its metrological support [2], [3].

Comparative Analysis of Moisture Control Methods

Moisture determination methods are conventionally divided into two major groups: direct and indirect methods. In direct methods, the water in the sample is separated by physical or chemical means and its quantity is measured directly; in indirect methods, a physical quantity functionally related to moisture content is measured. The

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thermogravimetric method is adopted as an arbitration method in international and national standards. The essence of the method consists of drying the sample to a constant mass at a temperature of 103–130°C and calculating the moisture content based on the mass difference [4]. Its main advantage is high accuracy and universality; its disadvantage is the long duration of the analysis, sample destruction, and methodological errors caused by the loss of volatile substances during the drying process. Therefore, the thermogravimetric method serves not for operative management of the technological process, but as a reference method for laboratory control and calibration of other methods. The Karl Fischer titration method is based on the selective chemical reaction of water with iodine, enabling the determination of exact water content with high accuracy (up to $\pm 0.05\%$). However, due to the high cost of reagents, requirement for qualified personnel, and complexity of sample preparation, this method is mainly used in the compound feed industry for research purposes and dispute resolution [5]. The infrared (IR) drying method is an accelerated variant of the thermogravimetric method, performed using analytical balances equipped with an IR radiator. Analysis time is reduced to 5–15 minutes, with an accuracy of ± 0.2 – 0.5% . The method is convenient for express laboratory control, but the result is sensitive to the choice of drying regime, requiring validation of the regime against the arbitration method for each product type. Near-infrared spectroscopy (NIR) is based on measuring absorption bands characteristic of O–H bonds in water molecules within the 780–2500 nm range. Modern NIR analyzers simultaneously determine protein, fat, and fiber alongside moisture in 30–60 seconds without destroying the sample, and can be installed online in the production stream. The main metrological problem of this method is the dependence of chemometric calibration models on the product formulation: when the formulation changes, the need arises to recalibrate the model [6]. Furthermore, the high cost of the equipment is not always acceptable for small enterprises. The microwave method is based on measuring the attenuation and phase shift of electromagnetic waves in the centimeter and decimeter ranges within a moist material. It stands out for its ability to operate in streams with thick layer depths and its resistance to dust, making it promising for continuous monitoring in elevators and granulation lines. Disadvantages include the complexity of the equipment and sensitivity to density variations. The conductometric method utilizes the dependence of a material's electrical conductivity on moisture content [7]. The instruments are simple and inexpensive, but the measurement result is strongly dependent on salt composition, temperature, and density, with sensitivity dropping sharply at low moisture levels (below 8%). For this reason, conductometric moisture meters are primarily used as a rough estimation tool. The above analysis shows that the dielcometric method occupies a special place in providing a compromise between laboratory accuracy and production speed: owing to its speed, relatively low instrument cost, and sufficient accuracy, it has become the most widely used express method in compound feed enterprises [8].

2. Materials and Methods

Physical Principles of the Dielcometric Method

The dielcometric method is based on measuring the complex dielectric permittivity of a moist material. The relative dielectric permittivity of water ($\epsilon \approx 81$) is approximately 16 to 40 times greater than that of the main components of dry organic material ($\epsilon \approx 2$ – 5). Due to this, even a slight change in the moisture content of the material leads to a significant shift in the capacity of the measuring capacitor. Measurements are typically carried out in the frequency range of 1–30 MHz, because in this range the dipole polarization of free and bound water is clearly manifested, while the effect of ionic conductivity remains relatively small. In practice, dielcometric moisture meters consist of a capacitive sensor (a cylindrical or planar capacitor) and a measuring generator. When a sample is placed in the sensor chamber, the capacitance of the capacitor

changes—and consequently, the generator frequency or the parameters of the resonant circuit shift; this variation is converted into moisture units via a calibration curve. Modern instruments are equipped with a temperature sensor, allowing the microprocessor to automatically apply temperature corrections, while individual calibration curves for different product types are stored in the memory.

3. Results and Discussion

In accordance with GOST 29027 requirements, the primary metrological characteristics of moisture meters designed for solid and bulk substances include the measurement range, the limit of permissible absolute error, repeatability of measurement results, and additional errors due to temperature and density [9]. Modern dielectric moisture meters intended for compound feed typically cover a measurement range of 5–40%; the limit of the main absolute error in the operating range is ± 0.5 –1.0%, and repeatability is 0.2–0.3%. It should be noted that while these indicators fully meet technological control requirements, they are insufficient for arbitration measurements [10]. The main factors influencing dielectric measurement results can be identified as follows. First, sample density and granularity: the degree of compaction of the material in the sensor chamber directly affects capacitance, which is why most instruments strictly regulate sample dosing and compaction procedures or provide automatic mass measurement. Second, temperature: due to the temperature coefficient of dielectric permittivity, every 10°C change can introduce an additional uncorrected error of up to 0.3–0.5% [11]. Third, non-uniform moisture distribution throughout the material and the proportion of bound water: because surface and internal moisture are not balanced in freshly dried products, the measurement result becomes time-dependent. Fourth, formulation composition: variations in mineral additives, salt, and fat content lead to shifts in the calibration curve. The listed factors impose special demands on the metrological support of dielectric moisture meters. Within the system ensuring uniformity of measurements, such instruments must undergo periodic verification (calibration), which is usually performed in two ways: using reference materials (certified samples with known moisture levels) or by comparison with the arbitration thermogravimetric method [12]. In the second approach, the readings of the instrument under verification are compared with values determined in a drying oven according to ISO 6496. Here, the temperature field uniformity in the drying oven chamber is of decisive importance, because if the reference method itself is not metrologically validated, the entire verification chain loses reliability. This situation directly relates to the metrological certification of drying ovens and thermostats, forming an essential link in ensuring the integrity of the measurement traceability chain [13]. When evaluating measurement uncertainty based on the GUM (JCGM 100:2008) methodology, it is advisable to include the following components in the uncertainty budget for dielectric measurements: uncertainty of the calibration curve (which includes the reference method uncertainty), Type A uncertainty due to repeatability, residual uncertainty of the temperature correction, uncertainty related to density and dosing, and uncertainty of sample representativeness (sampling from a batch). Practical calculations show that in production conditions, expanded uncertainty ($k = 2$) typically falls within the range of 0.6–1.2%, with the largest share often attributed to the sampling and calibration stages. This implies that beyond improving the instrument itself, standardizing the sampling procedure serves as a vital source of metrological reserve [14]. For testing laboratories accredited under ISO/IEC 17025:2017 requirements, using the dielectric express method requires method validation—that is, statistically confirming its agreement with the arbitration method for a specific product range. For this purpose, it is recommended to perform regression analysis based on at least 30–50 pairs of parallel measurements, evaluating systematic bias and the standard deviation of differences [15]. At the national level, within the framework of the procedure for certifying measurement methodologies

(O'z DSt 8.010), it is advisable to formalize methodologies tailored to the product nomenclature of each enterprise.

4. Conclusion

The analysis conducted allows for the following conclusions. First, there is no universal method for controlling moisture content in compound feed: while the thermogravimetric method serves as a reference due to its high accuracy, the dielcometric and NIR methods are the primary tools for rapid technological control. Second, in terms of the cost-to-speed and accuracy ratio, the dielcometric method is the most optimal solution for small- and medium-capacity enterprises, as its accuracy level of $\pm 0.5\text{--}1.0\%$ satisfies technological regulation requirements. Third, to fully realize the metrological potential of the method, it is necessary to automate temperature and density corrections, periodically update calibration characteristics across product types, and strictly regulate the sampling procedure. Fourth, because the traceability of dielcometric moisture meters is ensured via the arbitration drying method, metrological certification of the temperature field in drying ovens must be considered a key factor determining the reliability of the entire measurement chain. As a direction for future research, investigating the frequency-temperature dependencies of dielectric characteristics for various compound feed formulations can be identified, alongside developing a methodology to reduce calibration uncertainty based on these findings.

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