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REVIEW ARTICLE

GLOBAL LANDSCAPE OF MILK POWDER QUALITY CONTROL: A COMPREHENSIVE REVIEW OF CONTEMPORARY SCIENTIFIC METHODOLOGIES, STANDARDS, AND ADVANCED DETECTION TECHNOLOGIES

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Abstract

Milk powder is a critical global commodity for nutrition and food security. Due to complex international supply chains, it remains a primary target for intentional economic adulteration, highlighting a need for rapid, nondestructive quality control beyond traditional, destructive reference methods. This manuscript provides a global review of contemporary quality control, synthesizing analytical methodologies from the IDF, ISO, and Codex Alimentarius. We systematically categorize traditional analytical techniques alongside state of the art nondestructive methods, emphasizing the integration of Fourier Transform Infrared (FTIR), Near Infrared (NIR), and Raman spectroscopy with advanced chemometrics. Our synthesis reveals a paradigm shift toward real time, field deployable diagnostics essential for mitigating food fraud across the farm to table continuum. Furthermore, the paper addresses international regulatory disparities, advocating for a hybrid validation approach that reconciles classical standards with emerging digital screening systems.
Keywords: Artificial intelligence, chemometrics, dairy authentication, milk powder, Process Analytical Technology (PAT).

INTRODUCTION

Milk powder is a highly complex, processed biological fluid matrix engineered into a shelf stable dehydrated form consisting of proteins, lipids, carbohydrates (primarily lactose), vitamins, and macro-minerals[1]. On a global scale, the trade velocity and volume of milk powder are unparalleled within the dairy industry, serving as an irreplaceable base ingredient for infant formula, recombined liquid milk, confectionery, and functional processed foods. Because raw liquid milk contains approximately 87% water, its transition into powder requires intense thermal dehydration, rendering the final product highly vulnerable to thermal degradation, chemical variations, and cross contamination if processing conditions fluctuate[2].

The contemporary dairy supply chain has expanded into an intricate web crossing multiple international borders, introducing distinct vulnerabilities. Historical events, such as the catastrophic 2008 melamine adulteration crisis, illustrated how easily localized regulatory failures propagate into global public health emergencies[1]. Consequently, modern quality control frameworks must fulfill a dual role: verifying standard macro-nutritional parameters and identifying non-intentional contaminants or highly sophisticated, intentional economic adulterations designed to evade traditional validation metrics.

This comprehensive review reconciles the classical analytical paradigms mandated by bodies such as the International Dairy Federation (IDF) and the International Organization for Standardization (ISO) with contemporary digital, non-destructive methodologies. By analyzing international standard limits, direct nutritional profiling, physical functionality tests, and advanced spectroscopic arrays, this manuscript maps out a holistic operational landscape for modern food safety professionals operating under global parameters.

Global regulatory frameworks and trade standards

International standardizations ensure that multi-ton batches of milk powder maintain transactional transparency and safety criteria regardless of origin. Three primary bodies dictate global trade parameters.

The Codex Alimentarius, jointly run by the FAO and WHO, establishes global baseline safety limits, maximal residue limits (MRLs) for veterinary drugs, pesticides, and heavy metals, as well as strict microbiological criteria for powdered formulas. Meanwhile, ISO/IDF Technical Committees jointly draft validation and reference methodologies, providing the exact chemical protocols required to verify physical and compositional compliance. Finally, regional regulatory bodies such as the European Food Safety Authority (EFSA), the United States Food and Drug Administration (FDA), and China's State Administration for Market Regulation (SAMR) impose specific secondary requirements that often necessitate enhanced precision profiles.

Table 1: Global compositional and quality specification matrix for commercial milk powders.

Quality/compositional parameter	Whole Milk Powder (WMP) standard	Skimmed Milk Powder (SMP) standard	Primary global reference method
Moisture content	≤ 5.0% m/m	≤ 5.0% m/m	ISO 5537 / IDF 26 (Drying oven, 87°C)
Milk fat content	≥ 26.0% and < 42.0% m/m	≤ 1.5% m/m	ISO 1736 / IDF 9 (Mojonnier Gravimetric)
Crude protein content	≥ 34.0% m/m (in MSNF*)	≥ 34.0% m/m	ISO 8968-1 / IDF 20-1 (Kjeldahl Nitrogen)
Insolubility index	≤ 1.0 mL (spray dried)	≤ 1.0 mL (spray dried)	ISO 8156 / IDF 129 (Centrifugation sediment)
Titratable acidity	≤ 18.0 mL	≤ 18.0 mL	ISO 6091 / IDF 86 (Potentiometric titration)
Scorched particles	Disc B (≤ 15.0 mg)	Disc B (≤ 15.0 mg)	ADPI Standard Filtration Method

*MSNF: Milk Solids Not Fat

Direct Analytical methods for compositional validation

1. Moisture and water activity architecture

Moisture control dictates the preservation thermodynamics of milk powder. High moisture levels facilitate enzymatic activity, localized lactose crystallization, and rapid microbiological deterioration. The global golden standard, ISO 5537/IDF 26, specifies a vacuum drying oven operating at 87℃ under a continuous desiccant air current to isolate unbound moisture without inducing lactose pyrolysis or structural decomposition. Alternative industrial procedures leverage rapid thermogravimetric moisture balances or the traditional oven method at 102℃. Modern industrial protocols also utilize automated systems to capture real time validation data[3,4].

Beyond moisture content, monitoring water sorption isotherms and glass transition temperature profiles remains critical to avoid downstream product caking during intercontinental shipping transit[5].

Table 2: Analytical performance profiling of modern non-destructive spectroscopic modalities.

Spectroscopic Array	Target compounds/ analytes	Typical Limit of Detection (LOD)	Primary chemometric engine	Process integration level
Benchtop NIR	Moisture, fat, total protein, lactose	0.1–0.5% mass fraction	Partial Least Squares Regression (PLSR)	At-line/online continuous loops
FT-MIR/ATR	Exogenous sugars, urea, plant proteins	0.05–0.2% mass fraction	PLS-Discriminant Analysis (PLS-DA)	Laboratory screening arrays
Conventional Raman	Chemical components, polymorphs	~ 100–500 ppm	Principal Component Analysis (PCA)	At-line/raw material screening
SERS (Au/Ag)	Melamine, cyanuric acid, antibiotics	10–100 ppb	Support Vector Machines (SVM)	Specialized toxicology labs
Hyperspectral Imaging	Spatial adulteration, distribution mapping	≥ 0.05% spatial blend	Artificial Neural Networks (ANN)	Post-packaging quality checks

2. Nitrogen profile and protein discrimination

The Kjeldahl nitrogen digestion method (ISO 8968-1) remains the legally binding reference technique for structural protein validation[6]. This process breaks down organic material using concentrated sulfuric acid in the presence of a copper or selenium catalyst to convert organic nitrogen into ammonium sulfate. Subsequent alkalization with concentrated sodium hydroxide releases volatile ammonia gas, distilled directly into a boric acid receiving solution, which is quantified via precision acidimetric titration. While highly reproducible, the Kjeldahl protocol is blind to the source of nitrogen, converting total elemental nitrogen into crude protein via a standardized empirical multi conversion factor (F=6.38 for dairy). This specific blind spot creates vulnerabilities to non-protein nitrogen (NPN) adulterants. To combat this, advanced high-performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) and ultra-high performance liquid chromatography quadrupole time of flight mass spectrometry (UHPLC-Q-TOF-MS) setups are increasingly cross referenced in high stakes regulatory environments to guarantee authentic amino acid footprints and differentiate individual structural caseins or plant protein blends[7-9].

3. Total fat and fatty acid profiling

Fat determination dictates the energy density, stability, and susceptibility to oxidative rancidity of Whole Milk Powder (WMP). The official reference protocol is the Mojonnier alkaline extraction method (ISO 1736 / IDF 9). Samples undergo digestion with ammonium hydroxide to dissolve the protective caseinate macrostructures, followed by ethanol addition to precipitate polar carbohydrates. The non polar lipid fractions are subsequently isolated via a solvent mixture of diethyl ether and petroleum ether. The organic solvent layer is separated, evaporated, and the remaining lipid mass is quantified gravimetrically. In regular processing checks, this is often cross validated with standard Babcock extraction methodologies or the Gerber butyrometric method to satisfy regulatory audit metrics[10,11].

4. Carbohydrate and lactose mapping

Lactose represents the dominant carbohydrate fraction in dairy powders. Accurate tracking of intact lactose versus its degradation derivatives is central to verifying thermal processing history and technical purity. High performance liquid chromatography (HPLC) coupled with refractive index detection or mass spectrometry provides clear quantitative mapping of oligo-saccharides and structural sugars[12,13]. For trace level compliance validation, standardized multi laboratory enzyme-linked or colorimetric assays are deployed to pinpoint free glucose, galactose, or lactoferrin markers[14-16]. Advanced nuclear magnetic resonance spectroscopy (^{1}H-NMR) and capillary zone electro-phoresis also allow simultaneous, direct tracking of intact lactose alongside secondary organic acids generated via metabolic or heat induced breakdown pathways[17,18].

5. Macro mineral and ash characterization

The elemental and inorganic mineral matrix comprising calcium, magnesium, phosphorus, sodium, and potassium is quantified to ensure nutritional compliance, particularly in specialized infant formulations. Traditional validation utilizes precision chelatometric titration arrays alongside gravimetric total ash combustion at 550°C[19]. In premium safety workflows, these manual titration setups are replaced by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) or Atomic Absorption Spectroscopy (AAS) to accurately determine core mineral content and screen for hazardous trace heavy metal contamination[19,20].

Physical functionality and powder hydrodynamics

The physical behaviors of milk powder upon reconstitution dictate its industrial utility. Variations in particle size distribution, superficial fat content, and spray drying thermal history alter powder hydro-dynamics and downstream storage stability[21].

1. Wettability and surface mechanics

Wettability describes the capability of powder particles to overcome structural surface tension and absorb water molecules through their outer capillaries. Standard validation protocols monitor the precise timeline required for 10 grams of spray dried powder to sink beneath a static water mirror stabilized at exactly 25°C. Modern process laboratories complement this binary timing mechanism with optical goniometers that record the dynamic water droplet contact angle relative to compacted powder tablet structures. Crucially, the surface wettability is heavily dictated by particle morphology, particle size distribution, and the presence of free surface fat on the powder edge[22].

2. Insolubility index and dispersibility mechanics

The Insolubility Index (ISO 8156) evaluates the percentage of structural matter that remains permanently undissolved due to thermal denaturation of proteins (specifically β-lactoglobulin) during manufacturing. Reconstituted milk is subjected to standardized high-shear mechanical agitation, followed by high-velocity centrifugation[23]. The resulting structural precipitate volume is quantified directly in graduated sedimentation tubes, where values exceeding 1.0 mL signal sub-optimal heat management within the spray dryer or prolonged storage degradation. This is supported by modified Niro or dynamic funnel setups to measure total physical dispersibility and flow indices[24]. On the production floor, these indices are frequently paired with automated electro-potentio-metric pH profiling arrays to monitor ion release kinetics during reconstitution loops[25].

Modern instrumental alternatives and non-destructive screening

While standard wet chemical procedures deliver baseline legal proof, they are inherently destructive, resource-heavy, and incompatible with high-throughput online Process Analytical Technology (PAT). The current scientific frontier relies heavily on vibrational spectroscopy paired with advanced chemometrics.

1. Near Infrared (NIR) and Mid Infrared (MIR) spectroscopy

NIR spectroscopy (operating between 780 to 2500 nm) measures overtone and combination molecular vibrations of fundamental chemical bonds, including C-H (lipids), N-H (proteins), and O-H (moisture/ lactose). Mid-Infrared (MIR) systems capture funda-mental rotational-vibrational fingerprints, trac-king highly specific structural markers like the glycosidic linkage bands of sucrose at 996 cm^{-1} and 1052 cm^{-1} to flag illegal sugar additions[1]. Online applications use specialized mid-infrared loops to track fat crystallization arrays directly inside industrial spray drying columns[26].

2. Raman and Surface Enhanced Raman Spectroscopy (SERS)

Raman spectroscopy measures inelastic scattering events following monochromatic laser excitation. This technology maps structural heterogeneity across dry matrices without requiring sample dissolution. Integrating silver or gold nanoparticle arrays (SERS) yields strong localized electromagnetic field enhance-ments via Surface Plasmon Resonance (SSPR) mechanisms. These advancements reduce limits of detection (LOD) for chemical toxins, industrial plastics, and antibiotic trace residues down to low parts per billion (ppb) thresholds.

3. Chemometric engines and data processing pipelines

Because raw spectroscopic readouts consist of highly overlapping spectral curves, raw data cannot be read directly. Modern systems feed raw signals through standardized mathematical preprocessing steps, including Standard Normal Variate (SNV) trans-formation, Multiplicative Scatter Correction (MSC), and Savitzky-Golay polynomial differentiation to eliminate background physical light scattering and instrumental drifting variances. Unsupervised models, such as Principal Component Analysis (PCA), are initially applied to map out sample groupings. Subsequently, robust supervised classification archite-ctures, such as Support Vector Machines (SVM), Random Forests (RF), and convolutional Long Short Term Memory (LSTM) network algorithms, are deployed to perform real-time, quantitative validation checks on composition and detect potential adulterants or rapidly determine total energy values and caloric profiles[27].

4. Emerging digital and identification technologies

Beyond vibrational spectroscopy, the digitization of the dairy supply chain is being accelerated by innovations in portable diagnostics and immutable data logging.

Microbiological control and consumer protection

Due to the processing susceptibility of powdered matrices and its primary use in vulnerable demographics, rigorous microbiological parameters are strictly enforced globally. Pathogens such as *S. enterica* and *C. sakazakii* pose critical contamination risks because they can survive for extended periods inside dry powder matrices.

Traditional isolation workflows rely on culture based enrichments using selective media, such as Buffered Peptone Water (BPW) followed by Rappaport-Vassiliadis Soya (RVS) broth, which require up to 5 days confirming results. Modern manufacturing facilities run parallel real time quantitative Polymerase Chain Reaction (qPCR) screening configurations capable of detecting bacterial target DNA sequences within a 12 to 24 hour window. Matrix Assisted Laser Desorption Ionization Time of Flight Mass Spectrometry (MALDI-TOF MS) is also utilized for rapid, precise identification of isolated bacterial colonies based on ribosomal protein profiling.

DISCUSSION AND FUTURE PERSPECTIVES

The transition of the global dairy industry toward high throughput, real time quality control represents a paradigm shift necessitated by the increasing complexity of international supply chains. While traditional wet chemical methodologies (Kjeldahl, Mojonnier, and gravimetric drying) remain the legal "gold standards" due to their high reproducibility and historical forensic value, they are inherently limited by their destructive nature, requirement for hazardous chemical reagents, and delayed feedback loops. The integration of Process Analytical Technology (PAT), specifically NIR and Raman spectroscopy, offers a compelling solution to these bottlenecks. However, the industry wide adoption of these advanced modalities is currently hampered by the requirement for extensive, matrix specific calibration models. Because milk powder matrices vary significantly based on protein to fat ratios and thermal histories, chemometric models such as the PLS-DA and ANN architectures discussed often suffer from portability issues when transferred across different industrial processing lines.

A significant challenge identified in this review is the existing "digital divide" in global dairy safety. While high income nations have largely transitioned toward automated, IoT-integrated quality control suites, many emerging markets still rely heavily on manual laboratory testing. Furthermore, the future of food fraud mitigation lies beyond laboratory walls. The convergence of portable, handheld spectroscopic devices with blockchain-enabled ledger systems could soon allow field inspectors to perform real time verification of product authenticity at any point in the supply chain, ensuring that the farm to table continuum remains secure and transparent.

Ultimately, regulatory bodies such as the ISO/IDF and Codex Alimentarius are under pressure to formalize these nondestructive techniques into standardized reference frameworks. The move toward "hybrid" validation protocols where a baseline of rapid, non-destructive screening is periodically confirmed by high-accuracy reference laboratory methods appears to be the most pragmatic trajectory for the next decade. As genomic and proteomic identification tools, such as MALDI-TOF MS, continue to drop in cost and complexity, we can anticipate a future where both chemical and microbiological safety markers are tracked in near real time, drastically reducing the risk of global public health crises such as those experienced in the past two decades[1,9].

CONCLUSION

The global landscape of milk powder quality control is undergoing a necessary evolution. By reconciling the chemical precision of classical analytical paradigms with the agility of modern machine learning driven spectroscopy, the industry is better positioned to defend against both accidental contamination and intentional economic adulteration. Achieving global nutritional security, however, will require continued investment in the standardization of these advanced detection technologies, ensuring they are accessible, interoperable, and rigorous enough to protect the most vulnerable segments of the global population.

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AUTHOR'S CONTRIBUTIONS

Mugabo C: conceptualisation, literature survey, data organisation, writing original draft.

DATA AVAILABILITY

The related author can provide the empirical data supporting the study's conclusions upon request.

CONFLICT OF INTEREST

There are no conflicts of interest in regard to this project.

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