The identification and falsification of fat-and-oil products are significant challenges in ensuring the quality and safety of food products. Amid the rapid growth of global production and consumption of oils and fats, coupled with increasing demands for quality, ensuring accurate identification of the components of the fat fraction has become a critical aspect for both producers and consumers.

The Customs Union "Technical Regulation on Fat and Oil Products". (TR CU 024/2011) establishes stringent requirements for the quality and safety of fat-and-oil products, including fatty acid composition. In a competitive market environment and with a growing share of imported products, there are significant risks of falsification, which may involve the addition of cheaper fats, alteration of the fat fraction composition, or substitution of one type of oil with another. This not only diminishes product quality but can also negatively affect consumer health.

Methods for identifying fat components and verifying their authenticity require high precision, reproducibility, and sensitivity. In laboratory practice, chemical methods, such as gas chromatography (GC), are widely used for qualitative and quantitative analysis of fatty acid composition. However, traditional GC methods have limitations in separating isomers and detecting low-concentration components. Therefore, the adoption of innovative methods, such as gas chromatography with a mass spectrometric detector (GC-MS), has become essential for enhancing the efficiency and accuracy of analyses.

Gas chromatography with a mass spectrometric detector offers high precision and sensitivity in fatty acid analysis. This method effectively identifies not only the primary fat components but also traces of falsification, such as trans isomers and unsaturated fatty acids that may be added to reduce production costs. The use of GC-MS also shortens analysis time and improves reproducibility, making it highly applicable for industrial control and scientific research.

Thus, improving methods for identifying and detecting falsification in the fat fraction of fat-and-oil products through innovative approaches, such as GC-MS, is of great importance for ensuring food safety and combating market falsification.

Research Aim and Objectives

The aim of this research is to improve methods for identifying and detecting falsification in the fat fraction of fat-and-oil products using the innovative method of gas chromatography with a mass spectrometric detector (GC-MS).

To achieve this aim, the following objectives were set:

- analyze the regulatory framework for identifying and detecting falsification of fat-and-oil products based on fatty acid composition;

- improve the existing sample preparation method for GC to enable its use in GC-MS;

- improve the GC method for identifying and detecting falsification of fat-and-oil products based on fatty acid composition using GC-MS;

- validate the improved GC-MS method to assess its suitability for identifying and detecting falsification of fat-and-oil products based on fatty acid composition;

- conduct testing and monitoring of the improved GC-MS method for identifying and detecting falsification of fat-and-oil products based on fatty acid composition.

Scientific Novelty

- A new accelerated sample preparation method for GC-MS has been developed to determine the qualitative and quantitative fatty acid composition across all types of fat-and-oil products.

- An innovative GC-MS method for identifying and detecting falsification of fat-and-oil products based on fatty acid composition has been improved.

- Regulatory and technical documentation has been developed:

1) Guidelines for conducting sample preparation of fat and oil products for the determination of fatty acid composition and transisomers of fatty acids, Almaty, 2023. – 8 p.;

2) Guidelines for the determination of fatty acid composition by gas chromatography with a mass spectrometric detector (GC-MS), Almaty, 2023. - 12 p.;

3) Utility model patent No. 5371, registration date: 05.11.2020 "Method of sample preparation for determining the quantitative fatty acid composition of milk by gas chromatography", Serikov M.S., Serikbayeva A.D., Myrzabayeva N.E., Toishimanov M.R., Nurgalieva M.T., Matkazy Zh.S.

4) DP–02–LLP-10 Kazakh Scientific Research Veterinary Institute LLP. A documented management system procedure within the framework of ISO 17025 and GLP Principles of Good Practice. Validation of analytical methods, Almaty, 2024. - 22 p.

Key Findings

1. A method of gas chromatography with a mass spectrometric detector (GC-MS) has been developed and improved for qualitative and quantitative identification of fatty acids across all types of fat-and-oil products, ensuring accuracy and reliability.

2. A new accelerated sample preparation method for GC-MS has been proposed, reducing analysis time and improving the accuracy of fatty acid composition determination.

3. Normative and technical documentation (validation procedures and guidelines) for effective conformity assessment of fat-and-oil product quality has been developed.

4. Validation of the GC-MS method has been conducted, confirming its suitability for identifying and assessing the quality of fat-and-oil products.

Practical Significance

The research results enable the implementation of the improved GC-MS method in laboratory practices of testing laboratories, conformity assessment centers, and enterprises engaged in quality control of fat-and-oil products. The methodology facilitates high-precision detection of falsified fats and determination of the fatty acid composition of products, improving product quality and reducing the risk of unsafe fats entering the Kazakhstan market.

Implementation of Research Results

The methodology has been tested, and the research results have been implemented in the testing laboratory of food safety at the Kazakhstan-Japan Innovation Center of the Kazakh National Agrarian Research University (KJIC KazNAIU) and other laboratories engaged in quality control of fat-and-oil products. The testing and monitoring of fat-and-oil products conducted within this dissertation revealed cases of product falsification and significantly improved the efficiency of quality control in conformity assessment.

Dissemination of Results

The research findings were presented and discussed at the conference dedicated to the 90th anniversary of the Kazakh National Agrarian Research University on June 5, 2020, focusing on food product quality control and innovations in analytical chemistry. Additionally, the research materials underwent testing in the food safety laboratory of KJIC KazNAIU and the testing center of the Kazakh Scientific Research Veterinary Institute LLP.

Validation of Research Results

The reliability of the results is confirmed through the use of modern analytical methods, validation of the GC-MS methodology, and comparison of data with standard methods. The analysis was conducted on a wide range of fat-and-oil product samples (n=41), ensuring the reliability and reproducibility of the findings.

Excel and Python were used to maintaining and analyzing the acquired test data Statistical calculations were performed: calculation of averages, standard and relative standard deviations (RSD), confidence intervals (95%) to assess accuracy, correlation and regression analysis to verify the relationship and linearity of the method, as well as calculation of detection limits (LOD) and quantitative determination (LOQ). The use of these tools has ensured high accuracy and efficiency of data processing.

Completion of Research Objectives

All research objectives have been fully accomplished. The GC-MS methodology has been successfully improved, validated, documented, and implemented in the practice of quality control of fat-and-oil products for conformity assessment.

Recommendations for Use

Recommendations have been developed for the implementation of the improved GC-MS method for identifying and assessing the quality of fat-and-oil products in quality control laboratories and food industry enterprises. The methodology is recommended for conformity assessment of fat-and-oil products and detection of falsified samples.

Assessment of Technical and Economic Efficiency

The implementation of the improved GC-MS method significantly reduces analysis time, enhances result accuracy, and minimizes errors in identifying fatty acid composition. This leads to resource savings and increased efficiency in quality control of fat-and-oil products. The methodology meets global standards and can be applied in conformity assessment laboratories and food industry enterprises in Kazakhstan.

Publications

1. Calibration of gas chromatography instruments for determining the fatty acid composition of food products. Serikov M.S., Nurgaliyeva M.T., M.R. Toishimanov, M.S. Serikov – Izdenister, Natyzheler. Research, Results, 2019.

2. Determination of falsification of powdered milk based on fatty acid composition using gas chromatography. Serikov M.S., Matqazy Zh.S., Collection of scientific papers by graduate students, dedicated to the 90th anniversary of the university, June 5, 2020, pp. 272-276.

3. Modification of the sample preparation approach for milk and dairy products to determine fatty acid profiles using gas chromatography. M.S. Serikov, M.T. Nurgaliyeva, A.D. Serikbayeva, A.S. Kononihin, Bulletin of Almaty Technological University, pp. 5-13.

4. Defining the Composition of Fat Phase in Foods, Maksat S. Serikov, Meruyet T. Nurgaliyeva, Karima A. Myrzabek, Maxat R. Toishimanov, Farida K. Baktybayeva, *Food Processing: Techniques and Technology* > Archive > Volume 52, Issue 4, 2022.

5. Species identification of the fatty acid composition of margarines and spreads, Maksat Serikov, Meruyet Nurgaliyeva, Assya Serikbayeva, Zhulduz Suleimenova, Karima Myrzabek, DOI: https://doi.org/10.21894/jopr.2024.0029, *Journal of Oil Palm Research*, published online: April 26, 2024.

6. Patent for a utility model No. 5371, registration date: November 5, 2020. "Method of sample preparation for determining the quantitative fatty acid composition of milk using gas chromatography," A.D. Serikbayeva, N.E. Myrzabekova, M.R. Toishimanov, M.T. Nurgaliyeva, Zh.S. Matqazy, M.S. Serikov.

Structure and Volume of the Dissertation

The dissertation comprises 178 pages of computer-typed text, includes 16 tables, 29 figures, and consists of an introduction, literature review, research materials and methodology, research findings, conclusion, and a list of 225 references.