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Validation of Selective Ion Flow Tube Mass Spectrometry for Fast Quantification of Volatile Bases Produced on Atlantic Cod (*Gadus morhua*)

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Selective ion flow tube mass spectrometry (SIFT-MS) is a direct mass spectrometric technique that allows qualitative and quantitative analysis of a large number of volatile organic compounds. Because of its speed and ease of use, this nondestructive technique could be considered as a practical tool for quality control. This research focuses on the possibilities of direct headspace sampling by SIFT-MS for the quantification of the volatile basic nitrogen content (TVB-N) of fish fillets. These volatile bases [trimethylamine (TMA), dimethylamine (DMA), and ammonia] give additional information in conjunction with the sensory scoring and microbiological analysis about the quality of the fish fillets. This research validates in a first part the SIFT-MS method for the quantification of the volatile bases in mixed cod samples. With regard to the investigated linearity, repeatability, reproducibility, recovery, limit of detection, and limit of quantification, SIFT-MS appeared to be an adequate technique for measuring volatile bases spiked on cod. In the second part of this research, the technique was validated for the analysis of volatile bases on cod fillets during a storage experiment under ice. A good correlation was obtained between the proposed direct headspace sampling and traditional methods. The sensitivity of the SIFT-MS method can be improved when cod fillets are made more alkaline (pH>11) during sampling.

KEYWORDS: SIFT-MS; Atlantic cod (*Gadus morhua*); TVB-N; TMA-N

INTRODUCTION

Quality estimation of raw fishery products is nowadays a necessity for good functioning quality systems in fish-processing companies. Fish and fishery products are very susceptible to irreversible quality losses during storage due to chemical, but mainly microbiological, degradation. To assess the degree of spoilage of fish and fishery products, fast, accurate, objective, and nonambiguous measurements are needed. An often used parameter to determine fish spoilage, next to sensory scoring, is the quantification of typical odorous basic nitrogen compounds present in the fish tissue. $(CH_3)_3N$ (trimethylamine or TMA), $(CH_3)_2NH$ (dimethylamine or DMA), and NH_3 (ammonia) are products of autolytical and microbiological degradation and are collectively known as the total volatile basic nitrogen (TVB-N) fraction.

Facultative anaerobic bacteria such as *Shewanella putrefaciens*, *Acinetobacter*, and *Photobacterium phosphoreum*, recovered on iced fish, are able to reduce trimethylamine-*N*-oxide (TMAO) to TMA (*1*). TMAO has an osmoregulatory function in marine fish species, and the amount of TMAO present in the muscle tissue depends on the species, season, and marine environment (*2*). TMA is known to have a typical "fishy" odor. Some gadoid fish species are able to reduce TMAO in an autolytic pathway to DMA and formaldehyde (*3*, *4*). When bacterial growth is reduced, as under freezing conditions, production of DMA is an important factor involving quality losses and is considered as a frozen storage index (*5*). Ammonia is generally a bacterial degradation product, produced by the decarboxylation of amino acids in the fish muscle tissues (*1*). Ammonia is also released in Elasmobranch fish species, even in the early stages of storage, because of a fast enzymatic ammonia production originating from ureum (*6*).

The determination of the TVB-N in a fish sample is known to be the most common chemical parameter applied, because of its simplicity to evaluate the microbiological spoilage degree of fish and meat products. According to European directive 95/149/EEC, this indicator can be used if sensorial methods raise doubts about the freshness of the food product. Critical limits have been set for groups of seafood species and are expressed in milligrams of TVB-N per 100 g of tissue, for example, 35 mg of TVB-N/100 g of tissue and 15 mg of TMA-N/100 g of tissue for cod stored under ice (*1*, *7*). Fresh cod normally has <20 mg of N/100 g of TVB and 3 mg of N/100 g of TMA (*1*). Generally, the most common methods for TVB-N estimation are based on steam distillation of an alkalized sample (*8*−*10*). TMA analysis is

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