Kansas Agricultural Experiment Station Research Reports

Volume 0
Issue 2 *Dairy Research (1984-2014)*

Article 92

1988

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Abstract

The amount of free fatty acids present in cheese is important to dairy processors because these compounds make a significant contribution to the overall flavor. In this study, the results obtained using three relatively rapid methods of determining free fatty acids concentrations in cheese were compared to those acquired by using a more laborious but accurate gas chromatographic technique. One method, the Extraction-Titration Method, was found to be superior to the others because of its simplicity and reliability. In addition, the values obtained by this method were found to closely correlate with short chain fatty acid concentrations of cheese as determined by gas chromatography.; Dairy Day, 1988, Kansas State University, Manhattan, KS, 1988;

Keywords

Kansas Agricultural Experiment Station contribution; no. 89-107-S; Dairy; Free fatty acids; Cheese; Gas chromatography

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THE EVALUATION OF RAPID METHODS FOR MONITORING FREE FATTY ACID LEVELS IN CHEESE

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Summary

The amount of free fatty acids present in cheese is important to dairy processors because these compounds make a significant contribution to the overall flavor. In this study, the results obtained using three relatively rapid methods of determining free fatty acids concentrations in cheese were compared to those acquired by using a more laborious but accurate gas chromatographic technique. One method, the Extraction-Titration Method,was found to be superior to the others because of its simplicity and reliability. In addition, the values obtained by this method were found to closely correlate with short chain fatty acid concentrations of cheese as determined by gas chromatography.

Introduction

The accurate determination of free fatty acid concentrations in cheese is important because of the significant contributions these compounds make to aged cheese flavor. Free fatty acids accumulate in cheese during the aging period as a result of the action of particular enzymes known as lipases, which are produced by the microorganisms involved in the aging process. Extensive activity of lipases on the fat of cheese will result in high concentrations of free fatty acids and may lead to off-flavors, a condition referred to as lypolyzed flavor. The short chain fatty acids, such as butyric acid, are thought to be most responsible for this defect in aged cheese flavor.

The determination of free fatty acids in cheese can be performed in several ways. The Acid Degree Value has been applied to cheese because it is a standard method for monitoring the fatty acid content in milk and cream. Detergent is used to disperse the protein, resulting in the liberation of the milk fat, a portion of which is withdrawn, dissolved in solvent, and quantitated by neutralizing with base. A major problem with this method is that the more water-loving short chain fatty acids are likely to be poorly represented in the fat portion withdrawn from the treated milk. The Copper Soap Method utilizes a nonpolar solvent system to extract the copper salts of fatty acids, which are measured by their absorption of ultraviolet radiation. This method also has been reported to incompletely extract and quantitate short chain fatty acids. Utilization of a more water-loving solvent system to recover short chain fatty acids results in the simultaneous extraction of organic acids, predominantly lactic acid, which is present in cheese in high concentrations as a result of bacterial fermentation. The Extraction-Titration method utilizes hydrochloric acid to liberate lipids for extraction into a solvent system and silicic acid to absorb interfering phospholipids. Acid washes are performed to remove organic acids, and the fatty acids are quantitated by neutralizing with base.

Most of the rapid methods described here were developed primarily to determine free fatty acids in milk. Dairy processors require rapid and inexpensive methods of determining free fatty acid concentrations in cheese as an index of sharpness or lypolyzed flavor. The objective of this study was to assess how accurately these rapid techniques would reflect short chain, long chain, and total free fatty acid concentrations in cheddar cheese as compared to the more time consuming but accurate gas chromatographic method.

Procedures

Mild, sharp, and extra sharp cheddar cheeses from the same processor were purchased at a local supermarket and frozen at -20 C until analysis. A gas chromatographic method was used as the way of obtaining the most accurate free fatty acid profile of the cheese. The gas chromatograph is an instrument that heats compounds until they are volatile and separates them on a long small diameter column of adsorbent material. Using this instrument, the researcher is able to quantitate each fatty acid individually. The three rapid methods simply give an indication of the total amount of free fatty acids in cheese without differentiating them by chain length.

The rapid methods employed were the Acid Degree Value method, the Copper Soap method, and the Extraction-Titration method. The basic concept involved in each method was described in the introduction. The values obtained from each of these techniques were compared to the results of the gas chromatographic profile for short chain fatty acids (4-10 carbons long), long chain fatty acids (12-18 carbons), and total fatty acids for mild, sharp and extra sharp cheeses. A statistical computer program was used to obtain correlation coefficients to describe the relationship between the values obtained by rapid methods and the gas chromatographic methods. For example, a correlation coefficient near 1.0 means that the rapid method is able to effectively reflect the pattern of free fatty acid levels as determined by gas chromatography for mild, sharp and extra sharp cheeses. Conversely, a lower value of 0.5 would indicate that the rapid method is doing a relatively poor job of monitoring free fatty acid concentrations for cheese of differing ages.

Results and Discussion

The results obtained with Acid Degree method showed the least correlation with the gas chromatographic data, particularly for butyric acid (C_4) and short chain fatty acids in general (Table 1). Thus, this method is not recommended for monitoring free fatty acid concentrations as an index of cheese flavor. The Extraction-Titration Method yielded values that closely correlated with the concentrations of butyric and other short chain fatty acids as determined by gas chromatography. The mean values obtained with the Copper Soap Method did not correlate with the short chain fatty acid levels as well as those obtained with the Extraction-Titration Method. The inefficient extraction of the more water-loving short chain fatty acids by the relatively nonpolar solvent system used with the Copper Soap Method is the most likely explanation for this lack of correlation. We found the Copper Soap Method to be more complicated and less reliable than the Extraction-Titration Method. The Copper Soap Method, however, was more effective in reflecting the total and long chain fatty acid concentration of cheese.

The Extraction-Titration Method was found to be a simpler and more reliable method of monitoring free fatty acid levels in cheese and was the most effective method of those tested at reflecting short chain fatty acid concentrations. Since the latter class of fatty acids plays an important role in the production of lypolyzed flavor in other dairy products, this method may have an important advantage for analyzing free fatty acids as an index of cheese flavor.

Table 1. Correlation of Values Determined by Gas Chromatography with Those Determined by Other Methods for the Fatty Acid Concentration of Cheddar Cheese of Various Ages

Method	Correlation Coefficients (r)[a]			
	Total	Short Chain[b]	C_4	Long Chain[c]
Acid Degree Value	0.739	0.561	0.470	0.750
Extraction-Titration	0.804	0.911	0.885	0.756
Copper Soap	0.908	0.807	0.726	0.898

[a]All values significant at $p < 0.01$.
[b]Short chain = C_4 - C_{10}
[c]Long chain = C_{12} - C_{18}