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The effect of vitamin concentrates on the flavor of pasteurized fluid milk

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ABSTRACT

Fluid milk consumption in the United States continues to decline. As a result, the level of dietary vitamin D provided by fluid milk in the United States diet has also declined. Undesirable flavor(s)/off flavor(s) in fluid milk can negatively affect milk consumption and consumer product acceptability. The objectives of this study were to identify aroma-active compounds in vitamin concentrates used to fortify fluid milk, and to determine the influence of vitamin A and D fortification on the flavor of milk. The aroma profiles of 14 commercial vitamin concentrates (vitamins A and D), in both oil-soluble and water-dispersible forms, were evaluated by sensory and instrumental volatile compound analyses. Orthonasal thresholds were determined for 8 key aroma-active compounds in skim and whole milk. Six representative vitamin concentrates were selected to fortify skim and 2% fat pasteurized milks (vitamin A at 1,500–3,000 IU/qt, vitamin D at 200–1,200 IU/qt, vitamin A and D at 1,000/200–6,000/1,200 IU/qt). Pasteurized milks were evaluated by sensory and instrumental volatile compound analyses and by consumers. Fat content, vitamin content, and fat globule particle size were also determined. The entire experiment was done in duplicate. Water-dispersible vitamin concentrates had overall higher aroma intensities and more detected aroma-active compounds than oil-soluble vitamin concentrates. Trained panelists and consumers were able to detect flavor differences between skim milks fortified with water-dispersible vitamin A or vitamin A and D, and unfortified skim milks. Consumers were unable to detect flavor differences in oil-soluble fortified milks, but trained panelists documented a faint carrot flavor in oil-soluble fortified skim milks at higher vitamin A concentrations (3,000–6,000 IU). No differences were detected in skim milks fortified with vitamin D, and no differences were detected in any 2% milk. These results

demonstrate that vitamin concentrates may contribute to off flavor(s) in fluid milk, especially in skim milk fortified with water-dispersible vitamin concentrates.

Key words: milk, vitamin fortification, flavor

INTRODUCTION

Vitamin fortification of fluid milk has been in use in the United States since the 1930s, initially to prevent rickets (softening of bones) in children (Public Health Service, 1940). Vitamin D is essential for calcium absorption and is involved in the mineralization process required for bone growth. Vitamin D deficiency causes rickets in children and osteomalacia in adults (Ceglia, 2009). Recent studies also suggest that vitamin D plays a role in the prevention of prostate, breast, and colorectal cancers (Bouillon et al., 2006; Garland et al., 2006; Grant et al., 2007; Schwartz and Skinner, 2007). Vitamin A is needed for normal growth, vision, reproduction, and differentiation of epithelial cells. Vitamin A deficiency results in night blindness, xerophthalmia (progressive blindness caused by drying of the cornea of the eye), keratinization (accumulation of keratin in digestive, respiratory and urinary-genital tract tissues), and finally exhaustion and death (Zile and Cullum, 1983).

The United States Food and Drug Administration mandated in the 1990s that fortified fluid milks must be within 100 to 150% of label claims to address documented variability in vitamin amounts (Public Health Service, 1940). Reduced-fat and skim milks must be fortified with vitamin A at a minimum of 2,000 IU per quart (946 mL); fortification is optional for whole milk. All fluid pasteurized milk is fortified with vitamin D at a minimum of 400 IU per quart (946 mL) in the United States. Vitamin fortification is a standard procedure for pasteurized fluid milks in the United States, and vitamin concentrates are added to milk before pasteurization (PMO, 2015). Vitamin concentrates are available in oil-soluble and water-dispersible formulations (Murphy and Newcomer, 2001).

The standard shelf life recommended by manufacturers of commercial vitamin concentrates for fluid

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milk is 1 year at room temperature away from light. Recent work has addressed the stability of vitamin D in milk and other dairy products and has indicated that vitamin D is stable during processing and storage (Banville et al., 2000; Kazmi et al., 2007; Wagner et al., 2008; Hanson and Metzger, 2010; Tippetts et al., 2012). Vitamin A is unstable in the presence of heat, light, or acids (Yeh et al., 2017). Prolonged heating of milk, butter, or butterfat at high temperatures in the presence of oxygen can decrease vitamin A activity (Mousseron-Cadet, 1971; Hartman and Dryden, 1974).

The specific role of vitamin concentrates with respect to the flavor of fluid milk has not been addressed. A few studies have suggested that added vitamin A imparted a detectable off flavor, particularly in skim and low-fat milk (Weckel and Chicoye 1954; Whited et al., 2002). However, to our knowledge, no published studies have directly evaluated the specific role of the actual vitamin source and the carrier in their flavor contributions to fluid milk. Understanding the flavor contributions of vitamin sources to fluid milk can help the dairy industry strategically position vitamin fortification and enhance fluid milk quality. The objectives of this study were to characterize aroma-active compounds in vitamin concentrates used for fluid milk, and to determine the influence of vitamin A and D fortification on the flavor of milk.

MATERIALS AND METHODS

Experimental Overview

Two experiments (experiments 1 and 2) were included in this study. The purpose of experiment 1 was to determine the sensory profiles and key aroma-active volatile compounds in pure vitamin concentrates. To carry out this objective, we performed descriptive analysis, headspace extraction, and quantification of volatile compounds by GC-MS and gas chromatography-olfactometry (**GC-O**) on 14 different pure vitamin concentrates. Orthonasal threshold testing was then conducted on 8 key aroma compounds identified via GC-O. The purpose of experiment 2 was to determine possible vitamin flavor contributions to fluid milk fortified with vitamin A and D. To carry out this objective, representative vitamin concentrates from experiment 1 were selected to fortify skim and 2% fat milk to determine if trained panelists or consumers could detect flavor differences with and without vitamin fortification. We evaluated skim and 2% fat milk because they represent what most consumers purchase, and fat plays a protective role in vitamin degradation, light oxidation, and sensory thresholds of off flavors. If vitamin

fortification results in off flavor(s), it will be detected in reduced-fat or skim milk. We also conducted descriptive analysis, volatile compounds analysis, vitamin content analysis, and other proximate analyses.

Commercial Samples and Chemical Standards

Fourteen commercial vitamin concentrates (vitamins A and D) in both oil and water matrices were obtained in duplicate lots from multiple companies. All concentrates were within 45 d of manufacture, with >9 mo of remaining shelf life. Upon arrival, samples were stored at room temperature in the dark until analysis. All chemical standards were obtained from Sigma Aldrich (Milwaukee, WI).

Vitamin Analysis

Vitamin analysis was conducted in a laboratory with lighting that had UV filters to prevent light oxidation during extraction and analysis. Vitamin analysis for pure vitamin concentrates was conducted in accordance with the standard method performance requirements for vitamin A (AOAC International, 2012a) and vitamin D (AOAC International, 2012b) in pre-blends, premixes, and pure materials. The concentration of retinyl palmitate (vitamin A) and vitamin D₃ in the commercial vitamin premixes were confirmed by ultra-performance liquid chromatography (**UPLC**; Acquity H-Class; Waters Corporation, Milford, MA) with photodiode array (**PDA**) detection (325 nm for retinyl palmitate and 265 nm for vitamin D₃).

Vitamin A analysis for fortified fluid milk was conducted using AOAC method 2002.06 (AOAC International, 2007b). Briefly, retinyl palmitate from a 2 mL test portion of milk was extracted into 5 mL hexane (Sigma Aldrich) containing retinyl acetate (1.25 µg/mL; Sigma Aldrich, St. Louis, MO) as internal standard. Vitamin A palmitate was measured by UPLC with PDA detection (325 nm). The concentration of retinyl palmitate (µg/mL) was calculated using a relative response factor determined with calibration standards (0.01–5 µg/mL).

Vitamin D analysis was also conducted using AOAC method 2002.05 (AOAC International, 2007a). Briefly, vitamin D from a 15-mL test portion of milk was saponified by mixing with 10 mL of 50% (wt/wt) KOH (Sigma Aldrich) and 20 mL of ethanol (Sigma Aldrich) containing 2% (wt/vol) pyrogallol (Sigma Aldrich), followed by extraction with 20% (vol/vol) diethyl ether (Sigma Aldrich) in hexane containing vitamin D₂ (0.02 µg/mL; Sigma Aldrich) as internal standard. Vitamin D₃ was measured by UPLC with PDA detection (265

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