Variability in the Vitamin D3 Content of 2% Milk from a Nationwide United States Department of Agriculture (USDA) Sampling

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Abstract

Objective: The objective of the study was to assess the vitamin D3 content of 2% retail milk in the United States and its variability. Milk contains little natural vitamin D but is usually supplemented with vitamin D3 for retail sale in the US. The fortification target is 400 IU (10 mcg) per quart (25% DV per 8 oz serving or 42 IU/100g). Materials and Methods: In 2001, the USDA sampled and analyzed vitamin D3 in milk from 12 statistically selected supermarkets in the United States. In 2007, sampling was repeated, obtaining milk from 24 different supermarkets selected statistically to represent the nation. Vitamin D3 was quantified by comparison of the UV peak areas with standards and corrected for recovery. Vitamin D2 and vitamin D3 are baseline resolved. *In some cases, the milk purchased in one state was actually produced in another state.

Background

• A review of North Carolina dairies published in 1996 found that vitamin D3 was added at 9 different points in the processing of milk at various dairies1, including at the raw milk tanker or the homogenizer.
• When vitamin D3 is added in a lipid soluble form prior to separation of the cream, low-fat and skim milks may be under-fortified and higher fat products over-fortified.
• In 2004, the FDA provided guidance2 on the fortification of milk suggesting that a metering system be used when adding vitamin D3 after cream separation and before homogenization to allow for adequate mixing.

Methodology

• Milk samples obtained from 12 locations in 2001 and 24 different locations in 2007 (Fig. 1).
• 5.0 mL of sample with an internal standard (D3)4, 20 minutes at 60°C, and extracted with hexane.
• Hexane extract was washed with dilute methanol and dried.
• Sample re-suspended in hexane/methylene chloride, applied to HPLC ZORBAX SIL column (5µ), vitamin D fraction collected, and dried.
• Sample re-suspended in hexane/methylene chloride/alcohol (85/15/0.2), applied to HPLC ZORBAX SIL column, vitamin D fraction collected, and dried.
• Sample was applied to a Vydate ODS column in acetonic/methylene chloride (75/25).
• Vitamin D3 quantified by comparison of the UV peak areas with standards and corrected for recovery. Vitamin D3 and vitamin D2 are baseline resolved.

Results

• Only 33% of the vitamin D3 in 2% milk samples from 2001 fell within 100 to 150% of label value (400 IU [16 µg] per qt) (Figure 3a).
• 83% of the vitamin D3 in 2% milk samples from 2007 fell within 100 to 150% of label value (Figure 3b).

Conclusions

• More than twice as many of the 2% milk samples from 2007 met the label levels for vitamin D3 than the 2% milk samples collected in 2001.
• It is possible that the guidance from the FDA in 2004 may have had some impact on how the vitamin D3 is added to the milk.
• Some dairies still have quality control problems with vitamin D3 fortification of milk as evidenced by 12.5% of samples below the expected range.
• The accuracy and precision of the analyses, using the validated methodology, gives confidence in these estimated values for vitamin D3 levels in milk.

Additional Planned Research

• Another sampling of milk from four of the original dairies was planned for 2008 – CA3, CO1, MD1 and FL2 (see Fig. 3b). This additional sampling may give some indication as to whether the level of fortification with vitamin D3 by these dairies is consistent.
• Analysis of skim, 1%, whole, and 1% chocolate milk sampled along with the 2% milk is ongoing.

References