



Short Communication

Pharmaceuticals and personal care products in chicken meat and other food animal products: A market-basket pilot study



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HIGHLIGHTS

- We examined chicken, ground beef and milk samples for pharmaceuticals and personal care products.
- A large fraction of the analytes were not present above detection limits in any of the samples.
- Acetaminophen was present at low, but measurable levels in all three milk samples tested.
- Measurable residues of tetracycline drugs were present in some milk samples at low levels.
- Caffeine and 1,7-dimethylxanthine were frequently found at low levels in chicken and milk.

ARTICLE INFO

Article history:

Received 17 January 2014

Received in revised form 25 April 2014

Accepted 26 April 2014

Available online xxxx

Editor: D. Barcelo

Keywords:

Antibiotics

Chicken

Ground beef

Milk

Pharmaceuticals

Residues

ABSTRACT

Pharmaceutical drugs are extensively used in industrial food animal production. We examined whether residues of veterinary antibiotics and other pharmaceuticals and personal care products (PPCPs) were detectable in a small market-basket sample of retail chicken ($n = 39$), ground beef ($n = 3$) and milk ($n = 3$) samples. High-performance liquid chromatography and tandem mass spectrometry were used to assess the concentration of 59 PPCPs and their residues in animal products. All samples of ground beef, milk, and 14 chickens were analyzed individually, while an additional 25 chicken samples were pooled and analyzed in groups of five. The majority of PPCPs were not detected in meat and milk samples. Caffeine was detected in two of three milk samples (0.4 ng/mL, 2.0 ng/mL) and in 10 of 19 individual and pooled chicken samples (median: 18.6 ng/g, range: 6.1–28.8 ng/g). Acetaminophen was detected in three of three milk samples (median: 1.5 ng/mL, range: 1.4–2.1 ng/mL). Antibiotics in the tetracycline class were detected in two of three milk samples (median: 1.0 ng/mL, range: 0.1–2.0 ng/mL) and did not exceed regulatory residue tolerances of 300 ng/mL. There are no regulatory residue tolerances for caffeine or acetaminophen in animal products. The acetaminophen detections in milk, however, raise questions about extra-label and unapproved use of pharmaceutical drugs in food animal production, as this drug is not approved for use in lactating dairy cattle or any other type of food animal production. Additional studies are needed to confirm our finding of PPCPs in meat and dairy products.

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1. Introduction

Antimicrobial drugs and other pharmaceuticals are extensively used in industrial food animal production (Love et al., 2011). Measurable residues of these compounds may remain behind in animal products if drug withdrawal periods are inadequate or are not observed or if drugs are used in a manner that is inconsistent with their labeling. Numerous studies have developed sensitive methods to detect antimicrobial residues (Berrada et al., 2008; Azzouz et al., 2011; Du et al., 2012; Lehotay et al., 2013).

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While some of the human health hazards associated with environmental pollution of pharmaceutical-contaminated livestock effluent have been previously characterized (Boxall, 2004), to the best of our knowledge, no peer-reviewed studies have examined commercially-available meat and milk for the potential presence of a wide array of antimicrobial classes and pharmaceuticals and personal care products (PPCPs).

The United States Department of Agriculture Food Safety and Inspection Service (USDA-FSIS) conducts surveillance for chemical residues in food animal products under the National Residue Program (NRP) for Meat, Poultry and Egg Products. This program analyzes selected animal tissues (but not necessarily muscle tissue) for a variety of chemical and pharmaceutical residues using various methods, including a 7-plate microbial inhibition assay, high performance liquid chromatography

(HPLC) as well as liquid chromatography and mass spectrometry (LC/MS/MS) (United States Department of Agriculture, 2012). In 2011, the USDA conducted 5006 tests for veterinary antibiotics for all domestic animal production under the NRP and found detectable levels of antibiotics in ~1% of samples (55/5006 samples) and violations in 0.16% of samples (8/5006 samples) (United States Department of Agriculture, 2013). Using a 7-plate microbial inhibition assay, detectable levels of antibiotics were found in four of 290 beef cattle samples (1.4%), two of 330 dairy cattle samples (0.6%), and five of 621 chicken samples (0.8%), with one beef cattle sample in violation for antibiotics and no violations in dairy cattle or chickens (United States Department of Agriculture, 2013). Tetracyclines, aminoglycosides, sulfonamides, and macrolides were the most common classes of antibiotics detected in all products (United States Department of Agriculture, 2013). Data reported as part of the NRP are limited in their ability to aid in characterizing human exposures to drug residues through meat consumption; sampling results are only reported in a categorized format that does not allow for the development of animal product residue distributions that could be used in exposure assessment. In addition, for many compounds, muscle tissue residues are not reported. Instead, less commonly consumed organ meats (e.g. livers, kidneys) are sampled to look for violative residues.

The US Food and Drug Administration (FDA) conducts analyses of liquid milk from the bulk tanks of dairy farms for chemical residues. In addition to requiring that the industry sample all bulk milk tanks for β -lactam antibiotic residues and report positive results to regulators (United States Food and Drug Administration, 2005), the FDA's surveillance program provides for chemical and microbial sampling of milk from bulk pickup tankers from randomly selected dairy production facilities (United States Food and Drug Administration, 2005). The chemical analyses performed in the FDA's milk surveillance are generally limited to detection of metals, pesticides and β -lactam antibiotics. Milk products with pharmaceutical residues that exceed FDA tolerance values are removed from the marketplace. Dairy producers found to be responsible for the contamination have their milk removed from the supply chain by the FDA until a representative sample of the producer's milk is no longer positive for drug residues (United States Food and Drug Administration, 2005). The most recent surveillance conducted by the FDA reported a prevalence rate of 0.03% for violative pharmaceutical residues in samples of unpasteurized bulk tank milk (United States Food and Drug Administration, 2012).

The biological significance of chronic, dietary exposures to trace levels of antimicrobials is unclear. Therapeutic use of antibiotics in a clinical setting may contribute to increased risk of obesity, type 1 diabetes, inflammatory bowel disease, allergies and asthma (Blaser, 2011; Trasande et al., 2012; Mårild et al., 2013). Subclinical doses of antibiotics in mice administered via drinking water result in changes to the gut microbiome that lead to weight gain and altered levels of hormones involved in metabolism (Cho et al., 2012). Research has not been conducted to determine whether drug residues in meat and dairy can affect the gut microbiome of mice or humans.

We previously published two studies (Love et al., 2012; Nachman et al., 2012) reporting drug residues in poultry feather meal, a rendered product comprised of sterilized and finely chopped poultry feathers. Chemical analyses of feather meal revealed residues of numerous antibiotics and personal care products; every sample had quantifiable levels of between two and ten antibiotics, and many had other pharmaceutical residues, including acetaminophen, diphenhydramine, and fluoxetine, the active ingredients in Tylenol, Benadryl and Prozac, respectively. The analyses also found residues of multiple fluoroquinolone drugs (which have been banned from use in poultry production since 2005) in the majority of samples tested. More recently, the authors detected antimicrobial arsenical drugs and their metabolites in chicken meat from poultry likely fed arsenical drugs (Nachman et al., 2013). Given the findings of these poultry studies, and the growing interest in both the scientific community and the public, a comprehensive further investigation of the potential presence of drug residues in retail meats is warranted.

The objective of this study was to examine whether measurable residues of veterinary antibiotics and other PPCPs persist in a small sample of retail animal products.

2. Methods

The study was conducted in two phases. Initially, 14 chicken breasts were collected from seven different supermarkets (Table 1) and frozen at -20°C , as part of a larger group of samples collected for a previous study (Nachman et al., 2013). Frozen chicken breast samples were thawed, homogenized in a blender with distilled Milli-Q water and re-frozen (a detailed description of the sample processing procedure is provided in Nachman et al., 2013). The volume of water added to the sample was recorded and a correction factor was applied to normalize the sample for the quantitation of concentrations of analyte residues. A 5 gram subsample of frozen homogenate was removed and shipped on dry ice to AXYS Analytical Laboratory (British Columbia, Canada) for PPCP analysis using high-performance liquid chromatography (HPLC) and tandem mass spectrometry (MS/MS) to assess the concentration of PPCPs and their residues by Environmental Protection Agency (EPA) Method 1694 (United States Environmental Protection Agency, 2007; Love et al., 2012). Method 1694 was employed for two reasons. First, the method is a panel that includes many pharmaceuticals approved for use by the FDA in the production of food animals. Second, our earlier examination of poultry feather meal employed the method and found residues of multiple target analytes in tested samples (Love et al., 2012), suggesting the appropriateness of the method in this context. A list of the 59 analytes is provided in Appendix 1. The method used for this analysis was an in-house validated tissue method based on EPA 1694 using the same extraction solvents. Multiple labeled standards are used and they are outlined in the published method (United States Environmental Protection Agency, 2007). Chicken samples were analyzed individually. Upon receiving negative results for the majority of analytes in the first phase of sampling, a decision was made to analyze pooled chicken samples for the second phase of analysis to improve the odds of detecting target analytes.

In the second phase, 25 chicken breast samples were collected from nine supermarkets (Table 1) and processed as described above. Processed samples were then pooled in five composite batch samples, each comprised of five individual samples. For each pooled sample, 5 g of frozen homogenate from each of five individual samples was thawed and blended to create a 25 g composite sample, which was subsequently re-frozen and shipped on dry ice. The individual samples included in each pool were from the same city (except for pooled sample #4, which contained four samples from Philadelphia supermarkets and one sample from a Baltimore supermarket). The unique identifiers of individual samples were recorded in the interest of being able to perform analysis on individual samples from any composite batch that returned a quantifiable analyte concentration in the analysis. All USDA Organic chicken samples were pooled into one batched composite sample labeled Batch 4. The results of our analysis of these pooled Organic samples, along with results from all other batches are presented in Table 1. In addition, two additional types of animal products: ground beef ($n = 3$ samples) and milk ($n = 3$ samples) were analyzed. Ground beef and milk samples were analyzed individually for PPCPs. The nature of ground beef and homogenized milk makes these products inherently "pooled" in a manner comparable to the chicken samples in batches 1–5; each sample represents a composite of tissue or milk from multiple animals. The milk samples were collected from two retail locations in Victoria, British Columbia and transported on ice to AXYS Analytical Laboratory for laboratory analysis. All milk samples were pasteurized, two were skim milk, and one was 1% milkfat. The ground beef samples were collected from two supermarket locations in Victoria, British Columbia, and included three different brands. These samples were collected in Canada because of the complications associated with shipping ground beef and milk samples across the international border. All of the ground beef and milk samples tested were conventionally produced; none indicated that they were antibiotic-free.

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