Survey of PCDD/Fs in Animal Feed and Feed Additives

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Introduction

Food safety is a high priority in many countries. Food from animal origins has been contaminated with polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans (PCDD/Fs) in the U.S in 1997 and 2002, and in Europe during the past few years.[1,2,3] Intensive studies point out that animal feed and feed additives are a major source of contamination. In 2001, a feed which was produced and distributed in Spain contained choline chloride premix (manufactured in Belgium) and was contaminated with high levels of PCDD/Fs.[4] Highly contaminated lime (with 2.5 million pg I-TEQ/kg) has been added to wet peel for neutralization of citrus pulp production in Brazil, 1997.[5] The citrus pulp containing about 5-10 ng I-TEQ/kg of PCDD/Fs was distributed on the global market as a feed material for ruminants. Currently, Korea imports 75% of its animal feed and feed additives. It is a global problem that has to be monitored in each country. Hazard Analysis Critical Control Point (HACCP) should be introduced to the production of animal feed. The levels of PCDD/Fs in feed and feed additives must be thoroughly assessed in order to find the most strategic points to ensure animal feed safety. This study presents the results of the survey related to contamination of PCDD/Fs in animal feed and feed additives in Korea.

Methods and Materials

Samples were provided from producers or distributors in Korea. There were 32 samples, including: 7 different samples of fish meal, 2 samples of animal fats, shell powder, calcite, cholin chloride, sodium bicarbonate, calcium phosphate, and others. These were collected between February and August 2004. An automatic shaker using hexane and acidic silica extracted the oily samples over a two-hour period. The other samples were extracted using a Soxhlet extractor over 18 hrs with toluene/acetone (9/1, v/v). ^{13}C-PCDD/Fs were spiked into the samples as internal standards before the extraction. An isotope dilution method was used for analysis based on U.S. EPA method 1613B. Clean-up was performed by silica, alumina, and carbon columns using a Power-Prep^{TM} (FMS Inc., U.S.) automated column procedure. The extract was analyzed by HR-GC/MS (Autospec Ultima, Micromass Co., U.K.) equipped with a DB5MS capillary column (50m x 0.25mm I.D., 025µm film thickness, J&W Scientific, U.S.). A single analysis of each sample was made.

Results and Discussion

Table 1 presents the sum of concentrations of PCDD/Fs in animal feed and feed additives. Where two or more samples were available, the results were averaged. The ranges of concentrations are not presented because the sources of samples were different for the same kinds of feeds and feed additives. The PCDD/Fs in fish oil showed the highest level of 23.33 ng/kg, and animal fats were second with a level of 11.37 ng/kg. The level of PCDD/Fs in fish meal ranged from 0.5 ng/kg to 12.1 ng/kg. Based on the results of this study, feed of animal origin must be given priority for maintaining the safety of animal feed. A sample of DL-methionine showed only OCDD of 8.28 ng/kg. It may indicate that the source of OCDD is anthropogenic.

The determined TEQ levels of individual samples are shown in Figure 1. Out of 32 individual samples, 27 showed levels of less than 0.5 ng WHO-TEQ/kg. The levels of PCDFs were much higher in feed of animal origin. Only low levels of PCDDs were found in feeds of plant origin and feed additives. Eljarrat et al. (2002) reported that levels of PCDD/Fs in samples of animal origin were higher than the levels in samples of mineral origin with the exception of kaolin. The total contribution of PCDDs was greatest in the kaolin samples.[6,7] Limestone showed only 1,2,3,4,7,8-HxCDD at 0.04 ng WHO-TEQ/kg in this study. Congeners with the highest TEFs, 2,3,7,8-TCDD and 1,2,3,7,8-PeCDD, were present at the highest levels. Fish oil showed the highest level of PCDD/Fs with 4.68 ng WHO-TEQ/kg, and the most significant congener was 2,3,4,7,8-PeCDF.

Figure 2 presents the congener profiles of the samples showing high concentrations of PCDFs. The concentrations of 2,3,4,7,8-PeCDF in fish meals, fish oil, and shell powder and 1,2,3,4,7,8-HxCDF in animal fat were the highest levels. It indicates that the source of contamination is different between feed of fish origin and other animal origin. 2,3,7,8-TCDF was found in fish meal-1 and fish oil. 1,2,3,4,6,7,8-HpCDF, 1,2,3,4,7,8-HxCDD, 1,2,3,4,6,7,8-HpCDD, and OCDD were often found in low levels from the feed of animal origin. 2,3,7,8-TCDD and 1,2,3,7,8-PeCDD were not detected in any samples. More data is needed to get a general contribution rate of PCDD/Fs from feed and feed additives to meat and dairy products. This survey, however, showed that the primary source of PCDD/Fs in feed was of animal origin.

Table 1. Concentrations of the sum of PCDD/Fs in animal feed and feed additives

Ingredient	Concentration (ng/kg)	Ingredient	Concentration (ng/kg)
Meat meal	2.16 (n=2)	Vitamins	3.0
Blood flour	0.86	Minerals	0.43
Blood meal	1.40	Calcite limestone	0.45 (n=2)
DL-Methionine	8.28	Limestone	0.37
Animal fat	11.37 (n=2)	Salt	0.44
Fish meal	4.38 (n=7)	Ethoxyquin	ND
Shell powder	5.34 (n=2)	Cholin chloride	1.63 (n=2)
Fish oil	23.33	Sodium bicarbonate	3.10
Cotton seed	1.16	Bicalcium phosphate	1.22
Bit pulp pellet	ND		

ND = not detected, n=number of samples, not replicate

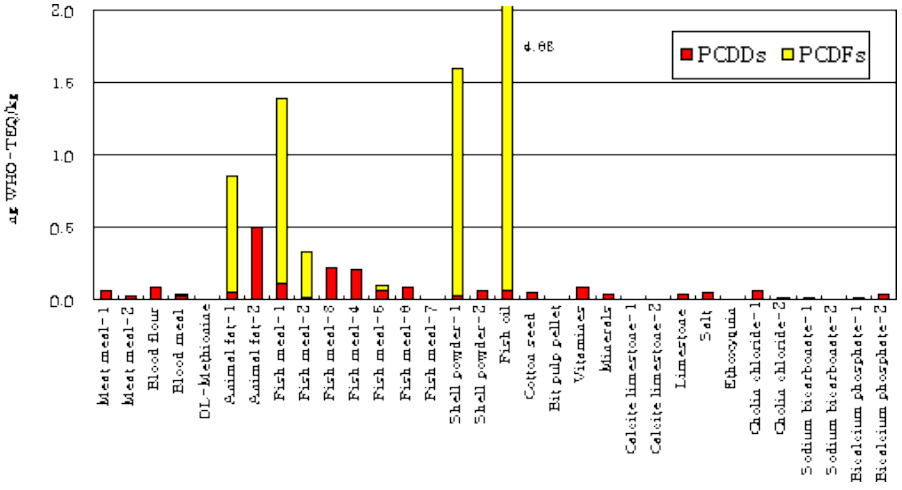

Figure 1. TEQ levels of PCDD/Fs in feed and feed additives.

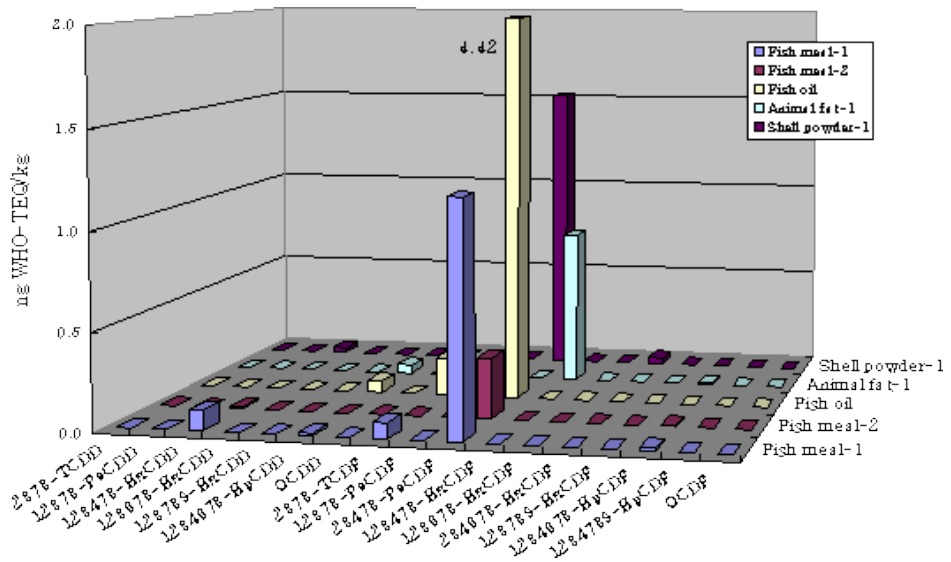

Figure 2. Congener profiles of the samples showing the intensive PCDFs.

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