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Investigation and Risk Assessment of Heavy Metal Contamination in Broiler Feed Ingredients in Shandong Province, 2015: Postprint

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Abstract

This study aimed to investigate the contamination status of arsenic, lead, cadmium, chromium, and mercury in broiler feed ingredients in Shandong Province in 2015 and assess the contamination risk of feed ingredients. The experiment collected eight types of feed ingredients used by different feed manufacturers in Laiyang (Yantai), Wendeng (Weihai), Laixi (Qingdao), Zhucheng (Weifang), Juxian (Rizhao), Yishui (Linyi), Xiajin (Dezhou), and other locations, including corn, soybean meal, wheat bran, peanut meal, cottonseed meal, corn gluten meal, dried distillers grains with solubles (DDGS), and trace element premix. Inductively coupled plasma atomic emission spectrometry (ICP-AES) was employed to determine the contents of cadmium, lead, and chromium, while atomic fluorescence spectrometry (AFS) was used to detect arsenic and mercury. The detection rate, average content, exceedance rate, ratio of highest content to average content (HC/AC), and coefficient of variation were calculated. The results showed that the detection rates of arsenic, lead, cadmium, chromium, and mercury in feed ingredients were 32.29%, 7.29%, 12.50%, 100.00%, and 100.00%, respectively; the average contents in positive samples were 0.21, 1.27, 2.12, 3.48, and 0.02 mg/kg, respectively; the exceedance rates were 0.00, 1.04%, 1.19%, 54.76%, and 9.52%, respectively; the HC/AC values were highest for arsenic, lead, and cadmium in trace element premix, chromium in cottonseed meal, and mercury in peanut meal; the coefficients of variation were highest for cadmium in trace element premix, mercury in peanut meal, arsenic in soybean meal, and lead and chromium in cottonseed meal. In conclusion, chromium and mercury contamination in broiler feed ingredients was severe; arsenic, cadmium, chromium, and lead in trace element premix, chromium and lead in cottonseed meal, and mercury in peanut meal and soybean meal were prone to severe contamination. Therefore, it is recommended to establish maximum limits for multiple heavy metal elements in premixes and meal-type feeds.

Full Text

Investigation and Risk Assessment of Heavy Metal Pollution in Broiler Feed Ingredients in Shandong Province, 2015

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Abstract: This study investigated the contamination levels of arsenic (As), lead (Pb), cadmium (Cd), chromium (Cr), and mercury (Hg) in broiler feed ingredients in Shandong Province during 2015 and assessed their associated risks. Eight types of feed ingredients used by various feed manufacturers were collected from Laiyang (Yantai), Wendeng (Weihai), Laixi (Qingdao), Zhucheng (Weifang), Juxian (Rizhao), Yishui (Linyi), and Xiajin (Dezhou), including corn, soybean meal, wheat bran, peanut meal, cottonseed meal, corn gluten meal, distillers dried grains with soluble (DDGS), and trace mineral premix. Inductively coupled plasma atomic emission spectrometry (ICP-AES) was employed to determine Cd, Pb, and Cr concentrations, while atomic fluorescence spectrometry (AFS) was used for As and Hg analysis. Detection rates, mean concentrations, exceedance rates, maximum-to-average concentration ratios (HC/AC), and coefficients of variation were calculated. The results showed detection rates of 32.29%, 7.29%, 12.50%, 100.00%, and 100.00% for As, Pb, Cd, Cr, and Hg, respectively. Mean concentrations in positive samples were 0.21, 1.27, 2.12, 3.48, and 0.02 mg/kg, with exceedance rates of 0.00%, 1.04%, 1.19%, 54.76%, and 9.52%, respectively. The highest HC/AC values were observed for As, Pb, and Cd in trace mineral premix, Cr in cottonseed meal, and Hg in peanut meal. The greatest coefficients of variation were found for Cd in trace mineral premix, Hg in peanut meal, As in soybean meal, and Pb and Cr in cottonseed meal. In conclusion, Cr and Hg pollution was most severe in broiler feed ingredients, with trace mineral premix being particularly prone to serious contamination by As, Cd, Cr, and Pb; cottonseed meal by Cr and Pb; and peanut meal and soybean meal by Hg. Therefore, establishing maximum limits for multiple heavy metals in premixes and oilseed meals is recommended.

Keywords: broiler chickens; feed ingredients; arsenic (As); lead (Pb); cadmium (Cd); chromium (Cr); mercury (Hg)

Introduction

Rapid industrialization and urbanization have intensified environmental pollution, with industrial and domestic wastewater discharge, sewage irrigation, and toxic pesticides introducing heavy metals into soil, water, and air [?]. In February 2011, China' s State Council approved the *Comprehensive Heavy Metal Pollution Prevention and Control Plan (2011-2015)*, which prioritized the management of As, Pb, Cd, Cr, and Hg contamination. Reports indicate that 300 million mu of farmland (approximately 20 million hectares) nationwide is threatened by heavy metal pollution, representing one-sixth of China' s total cultivated land area, with 12 million tons of grain contaminated annually [?]. A 2013 survey by Song et al. [?] found that 16.67% of China' s arable land suffered from heavy metal contamination, with these pollutants migrating through the food chain into cereals, forage, and livestock. In 2015, Yuan et al. [?] reported severe exceedances of Cr and Pb in layer feed from Sichuan Province (11.1% and 18.2%, respectively). Furthermore, 2012 monitoring by the Ministry of Agriculture revealed that over 70% of feed and feed ingredients from enterprises across 30 provinces exceeded standards for As, Pb, Cr, Cd, and other heavy metals [?]. Inadequate quality control and outdated detection methods for feed ingredients have led to frequent heavy metal poisoning incidents in animals, posing serious threats to livestock production and food safety—an issue that has attracted significant attention from government authorities and researchers [?]. Therefore, strict inspection and control of heavy metal contamination in feed ingredients are essential measures for ensuring feed and product safety while reducing environmental pollution, as well as for identifying pollution sources.

Shandong Province ranks first nationally in total animal husbandry output value, with its broiler industry generating over 30 billion RMB annually and serving as a pillar sector. As China' s second-largest feed production region with 1,460 feed enterprises, Shandong is also a major producer of soybean meal, cottonseed meal, wheat bran, and DDGS. However, research on heavy metal contamination in Shandong' s feed supply remains limited. Pan et al. [?] found As concentrations in pig feed from Shandong ranging from 17.1 to 34.1 mg/kg—8.5 to 17.0 times the national standard limit. Wang et al. [?] reported that beef cattle feed in North China (including Shandong) exceeded Cr and Pb standards by 83.33% and 66.67%, respectively, while dairy feed exceeded Cr standards by 60% and layer feed exceeded Pb standards by 53.85%. All livestock manures contained heavy metals above permissible levels, with pig and broiler manure showing the most severe contamination, confirming that feed is the primary source of heavy metals in manure. This study aims to investigate the concentrations of As, Pb, Cd, Cr, and Hg in broiler feed ingredients from Shandong Province in 2015, assess contamination risks, and provide reference data for feed production, livestock farming, and environmental protection.

1.1 Main Instruments and Reagents

The study utilized an OPTIMA 8000 inductively coupled plasma mass spectrometer (PerkinElmer, USA) and an AFS-920 dual-channel atomic fluorescence spectrophotometer (Beijing Jitian Instrument Co., Ltd.). Concentrated nitric acid and perchloric acid (both analytical grade) were purchased from Tianjin BASF Chemical Co., Ltd., while standard solutions for As, Cr, Hg, Pb, and Cd were obtained from Beijing Century Aoke Biotechnology Co., Ltd.

1.2 Sample Collection

From March to November 2015, samples were collected according to the national standard *Feed Sampling* (GB/T 14699.1-2005) [**?**]. Eight feed ingredients were obtained from various feed manufacturers in Laiyang (Yantai), Wendeng (Weihai), Laixi (Qingdao), Zhucheng (Weifang), Juxian (Rizhao), Yishui (Linyi), and Xiajin (Dezhou), including corn, soybean meal, wheat bran, peanut meal, cottonseed meal, corn gluten meal, DDGS, and trace mineral premix. Twelve samples of each ingredient were collected (96 total). Approximately 2 kg of each raw sample was reduced to about 200 g using the quartering method, ground to pass through a 20-mesh sieve (except premix), sealed in labeled bags, and stored for analysis.

1.3 Sample Preparation

Samples were stored at -20 °C and equilibrated to room temperature before processing. After mixing and grinding, samples were passed through a 40-mesh sieve. Exactly 1.000 g of each sample was transferred to a 100 mL Erlenmeyer flask, mixed with 15 mL of acid solution (nitric acid:perchloric acid = 4:1, v/v), covered with a watch glass, and cold-digested overnight in a fume hood. The following day, samples were heated on an electric hotplate at 130-140 °C until white fumes appeared and the volume was reduced to 1 mL. After cooling, digests were transferred to 10 mL volumetric flasks and diluted with ultrapure water.

1.4 Detection Conditions

Inductively coupled plasma atomic emission spectrometry (ICP-AES) was used to determine Cd, Pb, and Cr concentrations (GB/T 24875-2010) at analytical wavelengths of 267.716, 220.353, and 228.802 nm, respectively. Atomic fluorescence spectrometry (AFS) was employed for As (GB/T 5009.11-2014) and Hg (GB/T 13081-2006) analysis at wavelengths of 193.7 and 253.7 nm, respectively.

1.5 Quality Control

Standard curves were prepared before sample analysis, with correlation coefficients between heavy metal concentrations and absorbance values exceeding 0.99. Linear ranges were 1-1,000 g/kg for As and Cd, 100-10,000 g/kg for Cr, 0.001-1 g/kg for Hg, and 10-2,000 g/kg for Pb, demonstrating excellent linear relationships. Recovery tests yielded 96.14%-102.00% recoveries, and repeatability tests showed relative standard deviations (RSD) of 0.4%-2.6%, confirming the reliability and feasibility of both detection methods.

1.6 Sample Analysis and Calculations

Samples were analyzed for five heavy metals according to the specified conditions, with a standard reference material inserted every 10 samples to monitor instrument stability. Concentrations were calculated from standard curves, and samples exceeding the linear range were reanalyzed after appropriate dilution.

Detection rates and mean concentrations were calculated for positive samples. Heavy metal concentrations in trace mineral premixes were adjusted to a 1% inclusion rate according to manufacturer recommendations. Maximum concentrations were identified for each metal in positive samples. Exceedance rates for As and Pb in trace mineral premixes were calculated according to the *Hygienical Standard for Feeds* (GB 13078-2001) [?], while Cr, Cd, and Hg have no specified limits. For other ingredients, exceedance rates for all five heavy metals were calculated based on the *Maximum Levels of Contaminants in Foods* (GB 2762-2012) [?].

1.7 Risk Assessment

Coefficients of variation (standard deviation/mean) and maximum-to-average concentration ratios (HC/AC) were calculated for each heavy metal across samples. These metrics, along with maximum concentrations, were used to evaluate the degree of variation and contamination risk. The coefficient of variation measures both individual differences and the distribution density of measurements. Smaller values indicate lower dispersion and risk, while larger values signify greater variability and higher risk probability. Therefore, integrating maximum concentration, coefficient of variation, and HC/AC provides a robust approach for assessing severe contamination risks.

2.1 Chromium in Broiler Feed Ingredients

Chromium was detected in 100% of samples across all ingredients: corn, wheat bran, soybean meal, peanut meal, cottonseed meal, corn gluten meal, DDGS, and trace mineral premix. Mean concentrations were 0.95, 1.05, 0.93, 3.89, 4.08, 4.50, 1.92, and 10.43 mg/kg, respectively, with maximum concentrations of 1.93, 2.49, 2.21, 9.00, 24.00, 17.84, 10.12, and 19.71 mg/kg. Exceedance rates were 30.76% for corn, 25.00% for wheat bran, 16.67% for soybean meal, 100.00% for peanut meal, 81.82% for cottonseed meal, 90.91% for corn gluten meal, and 46.15% for DDGS.

2.2 Mercury in Broiler Feed Ingredients

Mercury was detected in 100% of all samples, with mean concentrations of 0.02 mg/kg across most ingredients (0.01 mg/kg in wheat bran and corn gluten meal). Maximum concentrations reached 0.04 mg/kg in corn, 0.02 mg/kg in wheat bran, 0.05 mg/kg in soybean meal, 0.08 mg/kg in peanut meal, and 0.02 mg/kg in cottonseed meal, corn gluten meal, DDGS, and trace mineral premix. Exceedance rates were 15.38% for corn, 0% for wheat bran, 8.33% for soybean meal, 8.33% for peanut meal, 18.18% for cottonseed meal, 0% for corn gluten meal, and 15.38% for DDGS.

2.3 Arsenic in Broiler Feed Ingredients

Arsenic detection rates were 30.77% in corn, 33.33% in wheat bran and soybean meal, 25.00% in peanut meal, 36.36% in cottonseed meal, 27.27% in corn gluten meal, 15.38% in DDGS, and 58.33% in trace mineral premix. Mean concentrations were 0.17, 0.08, 0.05, 0.24, 0.22, 0.15, 0.03, and 0.42 mg/kg, respectively, with maximum concentrations of 0.33, 0.19, 0.12, 0.33, 0.44, 0.17, 0.03, and 1.08 mg/kg. No exceedances were observed for any ingredient.

2.4 Lead in Broiler Feed Ingredients

Lead was detected in 0% of corn, wheat bran, peanut meal, corn gluten meal, and DDGS samples, but in 8.33% of soybean meal, 18.18% of cottonseed meal, and 33.33% of trace mineral premix samples. Mean concentrations were 0.00 mg/kg for most ingredients, 0.09 mg/kg for soybean meal, 0.26 mg/kg for cottonseed meal, and 1.46 mg/kg for trace mineral premix, with maximum concentrations of 0.00, 0.00, 0.09, 0.00, 0.52, 0.00, 0.00, and 3.58 mg/kg, respectively. Only cottonseed meal showed exceedances at 9.09%.

2.5 Cadmium in Broiler Feed Ingredients

Cadmium detection rates were 0% in corn, wheat bran, peanut meal, and corn gluten meal, 8.33% in soybean meal, 9.09% in cottonseed meal, 7.69% in DDGS, and 75.00% in trace mineral premix. Mean concentrations were 0.00 mg/kg for most ingredients, 0.11 mg/kg for soybean meal, 0.03 mg/kg for cottonseed meal, 7.50 mg/kg for DDGS, and 1.52 mg/kg for trace mineral premix, with maximum concentrations of 0.00, 0.00, 0.11, 0.00, 0.03, 0.00, 7.50, and 2.67 mg/kg, respectively. Only DDGS showed exceedances at 7.69%.

2.6 Comprehensive Risk Assessment

Maximum concentrations revealed the highest values for As (1.08 mg/kg) and Pb (3.58 mg/kg) in trace mineral premix, Cr in cottonseed meal (24.00 mg/kg), Cd in DDGS (7.50 mg/kg), and Hg in peanut meal (0.08 mg/kg). HC/AC ratios were highest for As (2.58) and Pb (2.46) in trace mineral premix, Cr in cottonseed meal (5.89), Cd in trace mineral premix (1.75), and Hg in peanut meal (4.05). Coefficients of variation were greatest for As in soybean meal (1.01), Pb in cottonseed meal (1.35), Cr in cottonseed meal (1.67), Cd in trace mineral premix (0.54), and Hg in peanut meal (0.97).

Integrating maximum concentrations, HC/AC ratios, and coefficients of variation indicates that trace mineral premix is prone to severe contamination by As, Cd, Cr, and Pb; cottonseed meal by Cr and Pb; and peanut meal and soybean meal by Hg. These findings warrant heightened attention to heavy metal pollution in these ingredients.

Overall, the study found detection rates of 32.29% (31/96), 7.29% (7/96), 12.50% (12/96), 100.00% (96/96), and 100.00% (96/96) for As, Pb, Cd, Cr, and Hg, respectively. Mean concentrations in positive samples were 0.21, 1.27, 2.12, 3.48, and 0.02 mg/kg, with exceedance rates of 0.00% (0/96), 1.04% (1/96), 1.19% (1/84), 54.76% (46/84), and 9.52% (8/84). These results demonstrate widespread and substantial heavy metal contamination in broiler feed ingredients, consistent with findings by Yuan et al. [?].

Chromium is an essential trace element that regulates carbohydrate, lipid, and protein metabolism, with dietary supplementation shown to improve economic returns [?, ?]. However, Cr is also a toxic pollutant that actively migrates through the environment, dispersing through air, water, and soil where crops absorb and accumulate it [?]. Due to its high toxicity and non-biodegradability, Cr bioaccumulates through food chains. Water-soluble hexavalent chromium is among the eight most hazardous chemicals to human health, one of eight recognized carcinogenic metal compounds, and one of three priority pollutants identified by the U.S. Environmental Protection Agency (EPA) [?]. Consequently, China' s *Maximum Levels of Contaminants in Foods* (GB 2762-2012) [?] limits Cr to 1.0 mg/kg in cereals and legumes, while the *Hygienical Standard for Feeds* (GB 13078-2001) [?] sets a 10 mg/kg limit for complete feeds. This study' s finding of 100% Cr detection and a 54.76% exceedance rate (46/84) in feed ingredients, with a mean concentration (3.48 mg/kg) exceeding cereal/legume limits, indicates severe and ubiquitous Cr contamination. According to China Powder Equipment Network statistics, approximately 600,000 tons of chromium slag are discharged annually, with a cumulative total of 6 million tons, of which less than 17% undergoes detoxification or comprehensive utilization [?]. Additionally, 2005 environmental statistics show the leather industry alone discharges 180 million tons of chromium-containing wastewater annually, suggesting widespread water and soil contamination in feed production regions. The highest Cr exceedance rates, mean concentrations, and maximum concentrations were found in peanut meal, cottonseed meal, corn gluten meal, trace mineral premix, and DDGS, indicating that contamination primarily originates from protein-rich oilseed meals and trace mineral premixes.

Mercury is a persistent, mobile, highly bioaccumulative, and toxic pollutant with significant adverse effects on animal health and ecosystems. China is both a major global producer and consumer of mercury, using approximately 900 tons in 2000—70% of which was imported—out of a worldwide production of 2,000 tons [?]. Although total environmental Hg emissions are substantial, only atmospheric emissions can be quantitatively estimated, with at least 643 tons released in 2007 from sources including coal-fired boilers (33%), power plants (19%), non-ferrous metals (18%), and cement production (14%) [?]. National standards limit Hg to 0.02 mg/kg in cereals and legumes and 0.1 mg/kg in complete feeds [?, ?]. This study' s detection of Hg in 100% of samples at a mean concentration of 0.02 mg/kg and a maximum of 0.08 mg/kg, with a 9.52% exceedance rate (8/84), demonstrates extensive and serious Hg contamination linked to China' s large-scale usage and emissions. Although exceedance rates were low for individual ingredients, elevated mean concentrations in corn, peanut meal, cottonseed meal, and DDGS, and high maximum concentrations in corn

(0.04 mg/kg), soybean meal (0.05 mg/kg), and peanut meal (0.08 mg/kg) far exceeded food contamination limits, highlighting the need for vigilant monitoring of specific ingredients.

Cadmium, arsenic, and lead are common contaminants in food and feed, with China being a major global producer. Mining and smelting of non-ferrous metals constitute primary pollution sources [?]. National standards limit Cd, As, and Pb to 0.1–0.2, 0.5, and 0.2 mg/kg, respectively, in cereals and legumes, and As and Pb to 10 mg/kg in premixes [?, ?]. This study' s lower detection and exceedance rates for Cd, As, and Pb compared to Cr and Hg suggest relatively lighter contamination in Shandong' s feed ingredients. However, trace mineral premix showed the highest detection rates, mean concentrations, and maximum concentrations for Pb and As, as well as the highest Cd detection rate, underscoring the need for focused attention on heavy metal contamination in premixes.

Maximum concentrations and HC/AC ratios reflect sample-to-sample variation but do not fully capture overall distribution patterns or individual differences. The coefficient of variation serves as a risk indicator that measures both inter-individual differences and the shape of the distribution curve. While various metrics exist for assessing variability (range, mean deviation, standard deviation), the coefficient of variation (standard deviation/mean) is essential for comparing datasets with different units or means. Smaller values indicate lower dispersion and risk probability, whereas larger values signify greater variability and higher risk. This study' s integrated analysis of maximum concentrations, HC/AC ratios, and coefficients of variation confirms that trace mineral premix is susceptible to severe As, Cd, Cr, and Pb contamination; cottonseed meal to Cr and Pb; and peanut meal and soybean meal to Hg contamination. These findings demand heightened vigilance regarding heavy metal pollution in these ingredients.

In summary, this investigation revealed severe Cr and Hg contamination in broiler feed ingredients, with trace mineral premix showing particularly heavy metal loads. The analysis identified trace mineral premix, cottonseed meal, peanut meal, and soybean meal as high-risk ingredients for severe contamination by specific heavy metals. These results strongly support the establishment of maximum limits for multiple heavy metals in premixes and oilseed meals.

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