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Comparison of Oven-drying Methods for Determination of Moisture Content in Feed Ingredients

J. Y. Ahn, D. Y. Kil[1], C. Kong, and B. G. Kim*
Department of Animal Science and Technology, Konkuk University, Seoul 143-701, Korea

ABSTRACT: An accurate assessment of moisture content in feed ingredients is important because moisture influences the nutritional evaluation of feedstuffs. The objective of this study was to evaluate various methods for moisture content determination. In Exp. 1, the weight loss on drying (LOD) of corn, soybean meal (SBM), distillers dried grains with solubles (DDGS), whey permeate, whey powder, spray-dried porcine plasma (SDPP), fish meal, and a mixed diet of these 7 ingredients were measured by oven drying at 135℃ for 2 h. Additionally, the samples were dried at 105℃ for 3, 6, 9, 12, or 15 h. The LOD contents of the DDGS, whey permeate, and whey powder measured by drying at 135℃ for 2 h were greater than the values measured by drying at 105℃ for 3 h ($p<0.05$). All samples except SDPP ($p = 0.70$) dried at 105℃ for 6, 9, 12, or 15 h caused more LOD compared with the samples dried for at 105℃ for 3 h ($p<0.05$). The LOD contents of the individual ingredients were additive when dried at 105℃ regardless of drying time. In Exp. 2, moisture contents of corn, SBM, wheat, whey permeate, whey powder, lactose, and 2 sources of DDGS (DDGS1 and DDGS2) were measured by the Karl Fischer method, oven drying at 135℃ for 2 h, and oven drying at 125℃, 115℃, 105℃, or 95℃ for increasing drying time from 1 to 24 h. Drying samples at 135℃ for 2 h resulted in higher moisture content in whey permeate (7.5% vs 3.0%), whey powder (7.7% vs 3.8%), DDGS1 (11.4% vs 7.5%), and DDGS2 (13.1% vs 8.8%) compared with the Karl Fischer method ($p<0.05$). Whey permeate and whey powder were considerably darkened as the drying time increased. In conclusion, drying samples at 135℃ for 2 h is not appropriate for determining the moisture content in whey permeate, whey powder, or DDGS as well as the mixed diet containing these ingredients. The oven-drying method at 105℃ for 5 to 6 h appears to be appropriate for whey permeate and whey powder, and at 105℃ for 2 to 3 h for DDGS. (**Key Words:** Dry Matter, Feed Ingredient, Karl Fischer Method, Loss on Drying, Moisture)

INTRODUCTION

Animal feeds contain 6 classes of nutrients including water, carbohydrates, proteins, fats, vitamins, and minerals. Among these, water, or moisture in feedstuffs is an important factor for the sale, purchase, transportation, and storage of feedstuffs. In addition, moisture determination influences the nutritional evaluation of ingredients when converting the nutrient contents to a dry matter (DM) basis (Thiex and Richardson, 2003). Thus, an accurate measure of the moisture content in feedstuffs is essential.

The DM contents (%) can be estimated by “100 (%) minus weight loss on drying (LOD) contents (%).” The oven-drying method for moisture determination has been widely used; this method measures the weight loss following heating of a sample. Although the oven method has been commonly used for estimating the LOD, this procedure has several limitations because the results of the LOD vary depending on the drying temperature and time. During the drying process volatile substances can be lost and unintended chemical reactions may occur (Mo and Tjornhom, 1978; Windham et al., 1987; Thiex and Van Erem, 1999).

Among oven-drying methods for moisture determination, a method that dries at 135℃ for 2 h (AOAC, 2005; method 930.15) is one of the most widely used procedures for feed moisture analysis due to its simplicity

* Corresponding Author: B. G. Kim. Tel: +82-2-2049-6255, Fax: +82-446-6255, E-mail: bgkim@konkuk.ac.kr
[1] Department of Animal Science and Technology, Chung-Ang University, Anseong 456-756, Korea.
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(Thiex and Richardson, 2003). However, this method may overestimate the moisture content of feeds or feed ingredients due to the loss of non-moisture compounds (Thiex and Van Erem, 1999; Thiex and Richardson, 2003).

Thus, the objective of this study was to evaluate different methods for determining the moisture content in feedstuffs.

MATERIALS AND METHODS

Experiment 1

Seven feed ingredient samples including corn, soybean meal (SBM), distillers dried grains with solubles (DDGS), whey permeate, whey powder, spray-dried porcine plasma (SDPP), and fish meal (FM) were ground (<1 mm) and analyzed for the LOD using a forced-draft oven (FC-PO-150, Dongseo Science Ltd., Seongnam, Korea). The methods used to determine of the LOD of the samples included drying at 135℃ for 2 h (AOAC, 2005; method 930.15), and drying at 105℃ for 3 h (Shreve et al., 2006; NFTA 2.2.2.5) in triplicate. Additionally, the samples were dried at 105℃ for 6, 9, 12, or 15 h in triplicate. The corresponding drying temperature and time applied for a mixed diet which was prepared for the additivity test of LOD and consisted of corn 20%, SBM 10%, DDGS 30%, whey permeate 20%, whey powder 10%, SDPP 5%, and FM 5%.

Experiment 2

Exp. 2 was conducted for further investigation of the LOD with various drying temperatures as well as drying time based on the results from Exp. 1. Eight ground samples (<1 mm) including corn, SBM, wheat, whey permeate, whey powder, lactose, and 2 sources of DDGS (DDGS1 and DDGS2) were dried at 135℃ for 2 h (AOAC, 2005; method 930.15) in triplicate. Additionally, the samples were dried at 95℃, 105℃, 115℃, or 125℃, and for each drying temperature, 1, 2, 3, 4, 5, 6, 9, 15, or 24 h of drying time was applied. The Karl Fischer (KF) method (AOAC, 2005; method 2001.12) was used as the reference method for the determination of actual moisture contents of feed ingredients. The KF method is designed to estimate the moisture content in samples by utilizing the quantitative chemical reaction of water with iodine and sulfur dioxide in the presence of lower alcohol and an organic base.

All dried samples were scored for color (L* for lightness, a* for redness, and b* for yellowness) with a CR-400 colorimeter (Konica Minolta Sensing Inc., Osaka, Japan). The reported value refers to the average of 3 measurements. Low L* scores represent a dark color, while high scores represent a light color (0 = black; 100 = white). High scores for a* and b* represent a high degree of redness and yellowness, respectively.

Calculations

For the additivity test in Exp. 1, the difference in LOD between the analyzed value and the predicted value based on the LOD of individual ingredients was calculated. The predicted LOD of a mixed diet was calculated by the following equation:

$$\begin{aligned}&\text{Predicted LOD of mixed diet}\\&= \text{LOD}_m \text{ of corn}\times0.2+\text{LOD}_m \text{ of SBM}\times0.1\\&+\text{LOD}_m \text{ of DDGS}\times0.3+\text{LOD}_m \text{ of whey permeate}\times0.2\\&+\text{LOD}_m \text{ of whey powder}\times0.1+\text{LOD}_m \text{ of SDPP}\times0.05\\&+\text{LOD}_m \text{ of FM}\times0.05\end{aligned}$$

where LOD_m is the measured LOD by the specific oven-drying method.

In Exp. 2, the KF method was used as a reference method to evaluate the accuracy of the oven methods. The difference between the LOD measured (AOAC, 2005; method 930.15) and the moisture measured by KF method was calculated by the following equation:

$$\begin{aligned}&\text{Difference}\\&= \text{\% LOD content measured (AOAC, 2005; method 930.15)} - \text{\% moisture content measured by KF method}\end{aligned}$$

The method that had the least percent deviation from the KF method was considered as the most accurate oven method for the determination of moisture in each ingredient (Ileleji et al., 2010). The percent deviation of LOD from the moisture content measured by the KF method was calculated by the following equation:

$$\text{Percent deviation, \%} = \frac{M_{OVEN} - M_{KF}}{M_{KF}} \times 100 \qquad \text{(Ileleji et al., 2010)}$$

where M_{OVEN} and M_{KF} are the moisture content measured by the oven method and the KF method, respectively.

Statistical analysis

Means were calculated using the LSMEANS statement of SAS (SAS Inst. Inc., Cary, NC, USA). The experimental unit was each dried sample, and significance was determined at an alpha of 0.05. Orthogonal contrasts were used to compare the moisture derived from various oven methods and the KF method for each ingredient or diet.

RESULTS

Experiment 1

The LOD measured by method 930.15 (AOAC, 2005) was greater than the values from National Forage Testing

Table 1. Moisture concentrations in feed ingredients and a mixed diet determined by various oven-drying methods (Exp. 1)

Item		Moisture (%)						SEM	p-value				
									135℃ 2 h[1]		105℃		
	Temperature (℃)	135	105	105	105	105	105		vs 105℃ 3 h[2]	vs 105℃[3]	Linear	Quadratic	3 h vs others[4]
	Drying time (h)	2[1]	3[2]	6	9	12	15						
Ingredient													
Corn		12.3	11.9	12.0	12.1	12.2	12.1	0.07	<0.01	<0.01	0.03	0.29	0.04
Soybean meal		10.7	10.3	10.4	10.4	10.5	10.5	0.03	<0.01	<0.01	<0.01	0.35	0.02
DDGS[5]		12.0	9.3	10.0	10.5	10.9	11.2	0.06	<0.01	<0.01	<0.01	<0.01	<0.01
Whey permeate		7.5	3.1	3.9	4.5	5.0	5.5	0.06	<0.01	<0.01	<0.01	<0.01	<0.01
Whey powder		8.9	3.0	3.7	4.4	5.0	5.4	0.15	<0.01	<0.01	<0.01	0.13	<0.01
SDPP[5]		7.8	7.5	7.6	7.5	7.6	7.3	0.04	<0.01	<0.01	0.07	<0.01	0.70
Fish meal		8.1	7.7	7.8	7.8	7.9	7.7	0.02	<0.01	<0.01	0.36	<0.01	0.05
Mixed diet[5]													
Predictied[6]		10.3	7.9	8.4	8.7	9.0	9.2						
Measured		10.1	7.8	8.4	8.7	9.0	9.0						
Difference		0.2*	0.1	0.0	0.0	0.0	0.2*	0.04					

SEM, standard error of the mean; DDGS, distillers dried grains with solubles; SDPP, spray-dried plasma protein.
[1] An oven method drying at 135℃ for 2 h (AOAC, 2005; method 930.15).
[2] An oven method drying at 105℃ for 3 h (Shreve et al., 2006; NFTA 2.2.2.5).
[3] A comparison between an oven method drying at 135℃ for 2 h and oven methods drying at 105℃ for 3, 6, 9, 12, and 15 h.
[4] A comparison between an oven method drying at 105℃ for 3 h and oven methods drying at 105℃ for 6, 9, 12, and 15 h.
[5] The mixed diet contained corn 20%, soybean meal 10%, DDGS 30%, whey permeate 20%, whey powder 10%, SDPP 5%, and fish meal 5%.
[6] Predicted weight loss-on-drying contents were calculated from the measured weight loss-on-drying contents of each ingredient.
* Measured values differ from predicted values (p<0.05).

Association (NFTA) 2.2.2.5 (Shreve et al., 2006) for all ingredients (p<0.01; Table 1). In addition, the method 930.15 (AOAC, 2005) also resulted in greater LOD compared with oven drying at 105℃ for all ingredients regardless of drying time (p<0.01). During the drying process at 105℃, the LOD of all ingredients except SDPP and FM linearly increased with drying time (p<0.01). The LOD contents of the individual ingredients were fairly additive. The measured LOD contents of the mixed diet dried at 105℃ for 3, 6, 9, or 12 h were not different from the predicted LOD of the mixed diet based on the measured LOD of individual ingredients.

Experiment 2

As shown in Table 2, the LOD contents in corn and wheat measured by method 930.15 (AOAC, 2005) were 12.1% and 9.6%, respectively, and those measured by NFTA 2.2.2.5 (Shreve et al., 2006) were 11.0% and 9.1%, respectively. These values were greater than the moisture content determined by the KF method (10.7% and 8.2% in corn and wheat, respectively; p<0.05). In SBM, the LOD measured by method 930.15 (AOAC, 2005) or by NFTA 2.2.2.5 (Shreve et al., 2006) was less than the value measured by the KF method (p<0.05). The oven method that resulted in the least deviation from the KF method was the method of drying corn at 95℃ for 24 h, SBM at 125℃ for 24 h, and wheat at 95℃ for 15 h.

The LOD contents in whey permeate, whey powder, and lactose derived from method 930.15 (AOAC, 2005) were 7.5%, 7.7%, and 5.1%, respectively, and those measured by NFTA 2.2.2.5 were 2.7%, 2.6%, and 1.2%, respectively (Table 3). These values were greater than the values measured by the KF method (3.0%, 3.8%, and 4.5% in whey permeate, whey powder, and lactose, respectively; p<0.05). As drying time increased, the LOD contents of lactose dried at 115℃ and 125℃ increased up to 5.1% which was the same as the value estimated by method 930.15 (AOAC, 2005). Lactose dried at 95℃ was estimated to be 0.1% to 0.3% of LOD contents and did not change as drying time increased. The oven methods drying at 105℃ for 6 h, 105℃ for 15 h, and 125℃ for 2 h had the least deviation from the KF method in whey permeate, whey powder, and lactose, respectively.

The LOD contents measured by method 930.15 (AOAC, 2005) were 11.4% and 13.1% for DDGS1 and DDGS2, respectively, and those measured by NFTA 2.2.2.5 were 8.7 and 9.0% in DDGS1 and DDGS2, respectively (Table 4). These values were greater than the moisture content of each ingredient measured by KF method (7.5% and 8.8% in DDGS1 and DDGS2, respectively; p<0.05).

The LOD contents measured by method 930.15 (AOAC, 2005) for all test ingredients were different from the moisture measured by the KF method (p<0.05; Figure 1). However, the difference in moisture between methods 930.15 (AOAC, 2005) and KF in corn, SBM, wheat, and lactose (1.4%, –1.2%, 1.4%, and 0.6% unit, respectively) was less than that of whey permeate, whey powder, DDGS1, and DDGS2 (4.5%, 3.9%, 3.9%, and 4.3% unit, respectively). The recoveries of moisture for method 930.15 (AOAC, 2005) were 113%, 90%, 117%, 113%, 250%,

Table 2. Moisture concentrations (%) in corn, soybean meal, and wheat determined by various oven methods and the Karl Fischer (KF) method (Exp. 2)[1]

Ingredient	Oven-drying method						KF method[3]
	Drying time (h)	Drying temperature (℃)					
		135[2]	95	105	115	125	
Corn[5, 6]	1	12.1±0.03	10.1±0.02	10.6±0.04	11.1±0.04	11.8±0.03	10.7±0.10
	2		10.1±0.01	10.9±0.04	11.1±0.01	11.8±0.03	
	3		10.0±0.01	11.0[4]±0.08	11.4±0.01	12.1±0.02	
	4		10.2±0.07	11.0±0.03	11.5±0.03	12.0±0.01	
	5		10.0±0.03	11.1±0.02	11.4±0.04	12.0±0.04	
	6		10.1±0.01	11.1±0.04	11.7±0.06	12.1±0.02	
	9		10.4±0.03	11.4±0.03	11.4±0.00	12.1±0.02	
	15		10.4±0.01	11.5±0.01	11.6±0.01	12.4±0.02	
	24		10.7*±0.01	11.7±0.07	11.3±0.08	12.6±0.01	
Soybean meal[5, 6]	1	10.3±0.05	8.9±0.03	9.6±0.04	9.7±0.03	10.0±0.03	11.5±0.08
	2		8.7±0.01	9.8±0.03	9.8±0.01	10.1±0.07	
	3		8.6±0.01	10.0[4]±0.02	9.9±0.02	10.2±0.04	
	4		8.6±0.02	9.9±0.02	9.9±0.04	10.2±0.07	
	5		8.9±0.02	9.8±0.03	10.1±0.03	10.3±0.06	
	6		9.0±0.02	9.8±0.03	10.1±0.02	10.3±0.05	
	9		9.2±0.06	9.7±0.02	9.9±0.05	10.4±0.03	
	15		8.9±0.01	10.2±0.02	10.0±0.04	10.5±0.03	
	24		9.1±0.03	10.2±0.01	9.9±0.02	10.8*±0.05	
Wheat[5, 6]	1	9.6±0.05	8.1±0.03	8.6±0.04	9.0±0.03	8.8±0.03	8.2±0.05
	2		7.9±0.01	8.7±0.03	9.1±0.01	9.0±0.03	
	3		7.8±0.01	9.1[4]±0.02	9.0±0.02	9.1±0.05	
	4		8.4±0.02	8.8±0.02	9.5±0.04	9.1±0.06	
	5		8.4±0.02	8.8±0.03	9.3±0.03	9.1±0.06	
	6		8.5±0.02	8.9±0.03	9.3±0.02	9.2±0.07	
	9		8.6±0.06	8.7±0.02	9.2±0.05	9.4±0.04	
	15		8.2*±0.01	9.4±0.02	9.4±0.04	9.6±0.05	
	24		8.3±0.03	9.7±0.01	9.4±0.02	9.5±0.09	

[1] n = 2 for KF and n = 3 for all oven methods.
[2] An oven method drying at 135℃ for 2 h (AOAC, 2005; method 930.15).
[3] The Karl Fischer method (AOAC, 2005; method 2001.12).
[4] An oven method drying at 105℃ for 3 h (Shreve et al., 2006; NFTA 2.2.2.5).
[5] The weight loss-on-drying measured by an oven method drying at 135℃ for 2 h (AOAC, 2005; method 930.15) differed from the moisture measured by the KF method (p<0.05).
[6] The weight loss-on-drying measured by an oven method drying at 105℃ for 3 h (Shreve et al., 2006; NFTA 2.2.2.5) differed from the moisture measured by the KF method (p<0.05).
* This method had the least deviation (%) from the KF method.

203%, 152%, and 149% for corn, SBM, wheat, lactose, whey permeate, whey powder, DDGS1, and DDGS2, respectively, with the KF method as the reference. The respective values with NFTA 2.2.2.5 were 103%, 87%, 111%, 27%, 90%, 68%, 116%, and 102%.

The lightness (L*) of dried whey permeate and whey powder decreased as drying time increased, particularly at high drying temperature (Figure 2).

DISCUSSION

Experiment 1

The greater LOD of DDGS measured by method 930.15 (AOAC, 2005) compared with that measured by NFTA 2.2.2.5 was in agreement with the data reported by Ileleji et al. (2010). The greater LOD by method 930.15 (AOAC, 2005) for DDGS, whey permeate, and whey powder compared with those measured by NFTA 2.2.2.5 indicated that this method might not be suitable for measuring the moisture contents of these ingredients.

The additivity of the LOD contents for the individual ingredients indicated that if the LOD content of individual feed ingredients were overestimated, the LOD content of a mixed diet containing the feed ingredients would also be overestimated. Although the LOD contents of the mixed diet measured by drying at 135℃ for 2 h (AOAC, 2005; method 930.15), and at 105℃ for 15 h, were significantly less than the predicted LOD of the mixed diet, LOD

Table 3. Moisture concentrations (%) in whey permeate, whey powder, and lactose determined by various oven methods and the Karl Fischer (KF) method (Exp. 2)[1]

Ingredient	Oven-drying method						KF method[3]
	Drying time (h)	Drying temperature (℃)					
		135[2]	95	105	115	125	
Whey permeate[5,6]	1	7.5±0.1	1.3±0.01	1.6±0.02	3.3±0.15	5.5±0.13	3.0±0.09
	2		1.4±0.03	2.3±0.03	3.4±0.05	6.1±0.05	
	3		1.5±0.13	2.7[4]±0.03	3.8±0.03	6.3±0.03	
	4		1.5±0.00	2.6±0.05	4.8±0.13	7.0±0.02	
	5		1.5±0.01	3.7±0.11	5.2±0.05	6.7±0.04	
	6		1.5±0.01	2.8*±0.03	5.8±0.03	7.4±0.05	
	9		1.6±0.01	3.5±0.02	6.3±0.01	8.0±0.03	
	15		1.6±0.01	4.4±0.01	6.4±0.01	9.7±0.13	
	24		1.7±0.00	4.8±0.03	6.8±0.01	12.9±0.17	
Whey powder[5,6]	1	7.7±0.06	1.7±0.01	2.0±0.00	2.9±0.06	5.8±0.14	3.8±0.08
	2		1.8±0.01	2.3±0.02	2.8±0.04	6.2±0.04	
	3		1.8±0.01	2.6[4]±0.02	3.1±0.03	5.9±0.04	
	4		1.9±0.03	2.6±0.02	4.2±0.08	7.2±0.02	
	5		2.0±0.03	3.2±0.07	4.5±0.04	7.0±0.04	
	6		1.9±0.00	2.7±0.02	5.5±0.04	7.4±0.07	
	9		2.1±0.01	3.1±0.02	5.9±0.01	7.8±0.02	
	15		2.2±0.03	3.5*±0.03	6.8±0.03	8.4±0.02	
	24		2.2±0.01	4.3±0.04	7.0±0.01	9.2±0.01	
Lactose[5,6]	1	5.1±0.01	0.3±0.16	0.3±0.03	1.8±0.08	3.8±0.11	4.5±0.05
	2		0.2±0.00	0.9±0.01	2.0±0.04	4.4*±0.05	
	3		0.1±0.01	1.2[4]±0.02	2.5±0.02	4.9±0.02	
	4		0.2±0.00	1.3±0.00	3.5±0.06	5.1±0.00	
	5		0.2±0.00	1.8±0.06	4.0±0.01	5.1±0.00	
	6		0.1±0.01	1.5±0.03	4.5±0.03	5.1±0.01	
	9		0.2±0.01	2.0±0.01	5.0±0.01	5.1±0.00	
	15		0.2±0.00	2.6±0.01	5.0±0.01	5.1±0.01	
	24		0.2±0.00	3.2±0.03	5.1±0.01	5.1±0.01	

[1] n = 2 for KF and n = 3 for all oven methods.
[2] An oven method drying at 135℃ for 2 h (AOAC, 2005; method 930.15).
[3] The Karl Fischer method (AOAC, 2005; method 2001.12).
[4] An oven method drying at 105℃ for 3 h (Shreve et al., 2006; NFTA 2.2.2.5).
[5] The weight loss-on-drying measured by an oven method drying at 135℃ for 2 h (AOAC, 2005; method 930.15) differed from the moisture measured by the KF method ($p<0.05$).
[6] The weight loss-on-drying measured by an oven method drying at 105℃ for 3 h (Shreve et al., 2006; NFTA 2.2.2.5) differed from the moisture measured by the KF method ($p<0.05$).
* This method had the least deviation (%) from the KF method.

difference between predicted and measured LOD of mixed diet was only 0.2% unit in both methods. This may be due to the highly precise LOD data in the present work. Within the triplicate samples of the LOD data, the values deviated very little among samples and the standard error of the mean for LOD in all ingredients ranged from 0.02 to 0.15.

Experiment 2

The KF method has been known to estimates the true moisture contents of samples (Thiex and Van Erem, 1999), and has been used as a reference method to evaluate other methods of measuring moisture (Isengard et al., 2006; Thiex, 2009; Ileleji et al., 2010).

Thiex and Van Erem (1999) reported that the moisture contents of corn measured by method 930.15 (AOAC, 2005) and the KF method were 11.82% and 12.34%, respectively. Although these values were statistically different ($p<0.05$), the percent difference was only –0.52% unit. This is in good agreement with the present data. Thiex and Van Erem (1999) also reported that the moisture measured by the KF method was greater than the LOD measured by method 930.15 (AOAC, 2005) for corn, soybeans, and barley. This suggests that method 930.15 (AOAC, 2005) may not overestimate the LOD of corn and soybean products for moisture determination because the bound water was difficult to remove from these ingredients by oven-drying methods. Therefore, although the LOD contents measured by method 930.15 (AOAC, 2005) are

Table 4. Moisture concentrations (%) in DDGS1, and DDGS2 determined by various oven methods and the Karl Fischer (KF) method (Exp. 2)[1]

Ingredient	Oven-drying method						KF method[3]
	Drying time (h)	Drying temperature (℃)					
		135[2]	95	105	115	125	
DDGS1[5,6]	1	11.4±0.02	6.9±0.04	7.2±0.03	8.4±0.03	8.1±0.08	7.5±0.02
	2		7.3±0.04	7.8±0.04	9.3±0.02	10.1±0.11	
	3		7.3±0.04	8.7[4]±0.06	9.2±0.06	11.3±0.15	
	4		7.8±0.03	8.9±0.06	9.4±0.05	11.4±0.07	
	5		7.6*±0.02	9.3±0.07	10.7±0.06	11.7±0.23	
	6		8.0±0.03	9.4±0.04	10.8±0.07	12.1±0.06	
	9		8.8±0.11	10.1±0.19	10.9±0.21	13.0±0.15	
	15		9.0±0.06	11.2±0.13	12.9±0.11	13.3±0.12	
	24		10.2±0.13	11.7±0.01	13.1±0.17	15.5±0.1	
DDGS2[5,6]	1	13.1±0.13	7.2±0.04	7.6±0.08	8.7*±0.03	10.2±0.15	8.8±0.05
	2		7.6±0.06	8.6±0.11	8.9±0.10	11.6±0.14	
	3		7.7±0.03	9.0[4]±0.08	9.3±0.01	11.8±0.15	
	4		8.3±0.10	9.4±0.05	10.9±0.13	13.2±0.05	
	5		8.1±0.17	10.0±0.07	11.6±0.15	12.2±0.09	
	6		7.9±0.03	9.7±0.20	11.7±0.34	13.6±0.10	
	9		8.9±0.12	11.2±0.04	12.4±0.19	14.4±0.08	
	15		9.0±0.14	11.7±0.15	12.8±0.15	15.0±0.18	
	24		10.4±0.15	12.5±0.12	13.5±0.06	15.9±0.07	

DDGS, distillers dried grains with solubles.
[1] n = 2 for KF and n = 3 for all oven methods.
[2] An oven method drying at 135℃ for 2 h (AOAC, 2005; method 930.15).
[3] The Karl Fischer method (AOAC, 2005; method 2001.12).
[4] An oven method drying at 105℃ for 3 h (Shreve et al., 2006; NFTA 2.2.2.5).
[5] The weight loss-on-drying measured by an oven method drying at 135℃ for 2 h (AOAC, 2005; method 930.15) differed from the moisture measured by the KF method ($p<0.05$).
[6] The weight loss-on-drying measured by an oven method drying at 105℃ for 3 h (Shreve et al., 2006; NFTA 2.2.2.5) differed from the moisture measured by the KF method ($p<0.05$).
* This method had the least deviation (%) from the KF method.

statistically different from those measured by the KF method in corn, SBM, and wheat in our study, method 930.15 (AOAC, 2005) may not result in a large deviation from the values obtained using the KF method in these ingredients or in a mixed diet containing these ingredients.

The large difference in the moisture contents of a milk product as determined by the method 930.15 (AOAC, 2005) and the KF method in the present study was in good agreement with the result in a previous study (Thiex and Van Erem, 1999). The authors also reported a large difference between the method 930.15 (AOAC, 2005) and the KF method for milk replacer.

Highly variable results of the LOD in whey permeate and whey powder may be caused by the Maillard reaction during the drying process. This is a chemical reaction between an amino group of an AA and a carbonyl group of a reducing sugar (Kim et al., 2012). As a result of the Maillard reaction, water is produced and evaporated during the drying process. As whey permeate and whey powder contain both amino groups and carbonyl groups, it is possible that the Maillard reaction occurs during drying process, especially at high temperature or during increased drying time.

The AOAC (2005) suggested that drying at 100℃ for 5 h in a vacuum oven (method 927.05) rather than method 930.15 should be used for moisture determination of whey permeate and whey powder. If one measures moisture in whey permeate, whey powder, or a mixed diet containing these ingredients using the method 930.15 (AOAC, 2005), the moisture contents of these samples may be overestimated.

Variability in the moisture content of DDGS has been previously reported (Thiex, 2009; Ileleji et al., 2010), and this may be largely attributed to the differences in the DDGS production procedure. The highly variable results of the LOD in DDGS could be caused by the evaporation of volatile matter during the oven-drying process. The DDGS are produced as a co-product from ethanol production from grains. Ethanol is derived from the fermentation process. Thus, DDGS may contain volatile compounds that could be evaporated with water when dried at high temperatures.

Thiex (2009) suggested that the NFTA 2.2.2.5 (Shreve

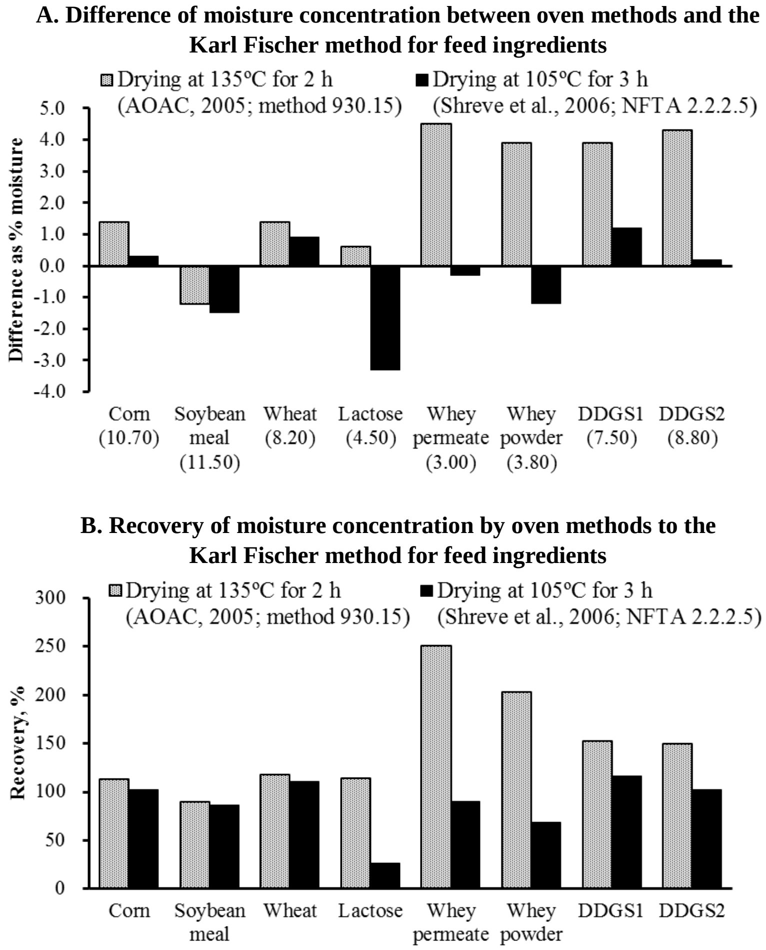

Figure 1. (A) Difference of moisture concentration between oven methods and with the Karl Fischer method, and (B) the recovery of moisture by oven methods to the Karl Fischer method in Exp. 2. The values in the parenthesis in Figure 1A represent the moisture concentration of each ingredient measured by the Karl Fischer method. DDGS, distillers dried grains with solubles.

et al., 2006) is appropriate to estimate the LOD contents of DDGS and that method 930.15 (AOAC, 2005) should not be used for the determination of moisture in DDGS because the NFTA 2.2.2.5 showed the least deviation from the KF method among oven-drying methods. Indeed, Thiex (2009) and Ileleji et al. (2010) reported that method 930.15 resulted in the highest LOD measurement out of all the methods assessed, which is consistent with the findings of the present study.

Drying time:		1 h	2 h	3 h	4 h	5 h	6 h	9 h	15 h	24 h
Whey permeate	L*	76.6	71.3	69.5	64.0	64.2	59.6	54.9	39.3	31.2
	a*	5.6	7.5	8.0	9.6	9.6	11.0	12.4	16.1	13.6
	b*	35.7	35.4	35.3	35.7	35.9	35.9	36.2	33.2	21.3

Drying time:		1 h	2 h	3 h	4 h	5 h	6 h	9 h	15 h	24 h
Whey powder	L*	57.1	54.0	53.6	49.1	49.1	46.1	45.4	36.7	33.3
	a*	12.6	12.3	12.3	11.7	12.0	11.9	11.9	13.7	13.8
	b*	35.0	31.8	31.2	29.0	28.5	27.6	27.4	27.5	24.6

Figure 2. Color changes of whey permeate and whey powder dried at 125°C (Exp. 2). L*, lightness; a*, redness; b*, yellowness.

CONCLUSION

In the current study, the oven method drying at 105°C for 5 to 6 h or at 105°C for 2 to 3 h was appropriate to determine of the moisture content in whey permeate and whey powder, or DDGS, respectively. Conversely, method 930.15 (AOAC, 2005) appeared to overestimate the LOD contents of whey permeate, whey powder, and DDGS compared with the KF method. Therefore, the AOAC (2005) method 930.15 should not be used to determine the moisture content of whey product and DDGS or of diets containing these ingredients

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