



Fractionation of distillers dried grains with solubles (DDGS) by sieving and winnowing

KeShun Liu *

Grain Chemistry and Utilization Laboratory, National Small Grains and Potato Germplasm Research Unit, USDA-ARS, 1691 S. 2700 West, Aberdeen, ID 83210, United States

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ABSTRACT

Four commercial samples of distillers dried grains with solubles (DDGS) were sieved. All sieved fractions except for the pan fraction, constituting about 90% of original mass, were then winnowed with an air blast seed cleaner. Sieving was effective in producing fractions with varying composition. As the particle size decreased, protein and ash contents increased, and total carbohydrate (CHO) decreased. Winnowing sieved fractions was also effective in shifting composition, particularly for larger particle classes. Heavy sub-fractions were enriched in protein, oil and ash, while light sub-fractions were enriched for CHO. For protein, the combination of the two procedures resulted in a maximum 56.4% reduction in a fraction and maximum 60.2% increase in another fraction. As airflow velocity increased, light sub-fraction mass increased, while the compositional difference between the heavy and light sub-fractions decreased. Winnowing three times at a lower velocity was as effective as winnowing one time at a medium velocity. Winnowing the whole DDGS was much less effective than winnowing sieved fractions in changing composition, but sieving winnowed fractions was more effective than sieving whole DDGS. The two combination sequences gave comparable overall effects but sieving followed by winnowing is recommended because it requires less time. Regardless of combinational sequence, the second procedure was more effective in shifting composition than the first procedure.

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1. Introduction

In recent years, increasing demand for ethanol as a fuel additive and decreasing dependency on fossil fuels have led to a dramatic increase in ethanol production from various cereal grains (corn, wheat, sorghum, rye, etc.). In the US, the majority of ethanol production is based on a dry-grind method using corn as a major raw material. Cereal grains contain 60–70% starch and 30–40% non-starch components (protein, fiber, oil, and ash). During a typical dry-grind method, grains such as corn, are ground to reduce particle size, and then mixed with water and thin stillage to produce a slurry. The slurry is cooked. Its starch is liquefied and saccharified with enzymes and fermented with yeasts to produce ethanol. After removal of ethanol by distillation, the remaining non-fermentables are centrifuged, dewatered, mixed and finally dried to produce a coproduct known as distillers dried grains with solubles (DDGS) (Bothast and Schlicher, 2005).

DDGS is a dry mix of particulate materials. Besides containing some unconverted starch, DDGS is a concentrated form of non-fermentable components from the original corn, including protein, oil, fiber, and ash (Rosentrater and Muthukumarappan, 2006). Marketing DDGS is critical to sustainability of a dry grinding plant, but

high fiber content of DDGS limits its uses to animal feed, mainly for ruminants. With a rapid increase in DDGS supply, there is a need to enhance the value of DDGS. One simple and economical approach is to dry fractionate DDGS. This could result in at least two types of products, a protein and oil enriched fraction and a fiber enriched fraction. The protein and oil enriched DDGS could be better used as feed for non-ruminant animals (including fishes) (Cheng et al., 2003), and as food ingredients (Rosentrater and Muthukumarappan, 2006). Fiber enriched fractions could be used for production of corn fiber oil, corn fiber gum, or cellulose-based ethanol (Buchana, 2002; Srinivasan et al., 2007). Thus, dry fractionation of DDGS not only enhances nutritional values of certain fractions but also would expand market shares. Furthermore, according to Belyea et al. (2004), DDGS with high oil (13%) and high protein (33%) contents is worth \$5–20 per ton more than regular DDGS. Thus enhanced DDGS could potentially sell at a higher price.

Several investigators have looked into different dry fractionation methods to enhance DDGS values, including dry milling followed by sieving (Wu and Stringfellow, 1982), sieving alone (Wu and Stringfellow, 1986; Liu, 2008), air aspiration (Singh et al., 2002), and a combination of sieving and elutriation (Srinivasan et al., 2005). These methods have met with varying degrees of success. Notably, Srinivasan et al. (2005) found that sieving the DDGS into various size categories and then elutriating sieved fractions of

* Tel.: +1 208 397 4162; fax: +1 208 397 4165.
E-mail address: Keshun.Liu@ars.usda.gov

larger size classes at appropriate air flow velocities was more effective than sieving alone in separating fiber from DDGS. Subsequently, a US patent application for the combined method (known as elusive process) as well as an elutriation apparatus was filed and later published (Srinivasan and Singh, 2006).

In this report, four DDGS samples were dry fractionated by sieving, winnowing and combinations of the two. Wind winnowing is an agricultural method developed by ancient cultures for separating grain from chaff. It is also used to remove weevils or other pests from stored grain. In its simplest form it involves throwing the mixture into the air so that the wind blows away the lighter chaff, while the heavier grains fall back down for recovery. In Ancient China, the method was improved by developing a rotary fan to produce an airstream. Although elutriation and winnowing are both based on density differences among particles and thus considered as air separation, the two work in different ways. In elutriation, separation of particles is carried out by means of an upward flowing stream of air in a vertical chamber (Srinivasan et al., 2005), while in winnowing a horizontal or slanted stream of air is used. Thus the latter would offer simplicity and a low cost option in equipment requirements and possibly an improvement in fractionation efficiency.

Furthermore, in the study by Srinivasan et al. (2005), a single series of four sieves plus a pan were used for sizing two different DDGS samples. Of the five size classes, only the three fractions with larger particle sizes were subjected to elutriation. The mass of these three fractions were about 60% and 43% of two DDGS samples used, respectively. From an economic and yield standpoint it makes more sense to dry fractionate as much of the DDGS sample as possible. In the present study, a series of three sieves plus a pan were selected for each of four different DDGS samples. After sieving, all sieved fractions except for the one retained on the pan, a total of about 90% of the original sample mass, were subjected to air separation.

2. Methods

2.1. Materials

Four DDGS samples were supplied from four dry grind ethanol plants located in two Midwest states: Iowa & South Dakota, and labeled as No. 1, 2, 3, and 4 according to plant numbers that were assigned sequentially upon the order of samples arriving in the laboratory. These plants processed commodity yellow dent corn available locally but might use different processing methods, which were commercial secrets to each plant.

2.2. Experiment 1: sieving followed by winnowing

2.2.1. Sieving

For each of the four DDGS samples, a set of three sieve sizes were determined that would yield 25–35% of the original sample mass in each of the three size-class fractions and roughly 8–12% in the pan fraction. The sieving procedure was according to an ASAE standard method (ASAE Standards, 2003). Basically, 100 g of DDGS sample, without any additional processing and moisture adjustment, was sieved with a series of three selected US standard sieves and a pan, fitted into a sieve shaker (DuraTap, Model DT168, Advantech Mfg. Co., New Berlin, WI), with shaking for 10 min. In the standard method, no word was mentioned about tapping during shaking. In this study, in order to improve sieving efficiency, the tapping option was used during shaking. The mass of material retained on each sieve as well as on the pan was determined and recorded. A total of four sieved size classes were obtained. The mass frequency (%) for material retained on each sieve size was

calculated. The sieving procedure was repeated three times for each sample to produce enough sieved fractions for subsequent air separation. This served as the first trial. The same 3-repeat procedure was then duplicated for a second trial.

2.2.2. Air separation (winnowing)

Winnowing was carried out by an air blast seed cleaner (Model ABSC, ALMACO, Nevada, Iowa), which was a tabletop model for cleaning small samples of seeds. The equipment consists of an electric fan, an air-intake control on the fan, an air separation chamber, a wood base, a 6-ft drop cord and switch, and two removable catch pans (Fig. 1). In addition, a collection chute for light particles was made in the author's laboratory so that they could be channeled downward to a collection pan, without much loss. A vibratory feeder was also constructed using a vibration device (such as a table top mill) and a stainless steel scoop. The device vibrated the scoop, which was fixed to the grinder at the handle area, and directed a powder sample to a precise location at the sample entry area of the winnowing machine. It was at this location where airflow velocity was measured using a hand-held anemometer. The airflow was regulated with a baffle plate that partially blocked the air intake on the blower.

During operation, the fan forced air into the air separation chamber (Fig. 1). A sample (whole DDGS or its sieved fraction) was delivered by the vibratory feeder, and entered into the air chamber where separation took place. The airflow caused the lighter particles to follow the air stream and exit from the chamber, where they were channeled downward to a catch pan. The heavy particles simply dropped down from the chamber, and were collected at the bottom by another catch pan.

Due to differences in density and shapes, four anemometer-measured airspeeds were selected for each sieved fraction, based on a criterion that they would increase the light fraction mass progressively from about 15% to about 80% of sieved fraction mass, with each increase in airflow speed causing a similar increase in the light fraction mass. For the 3 time-repeated winnowing procedure a single airflow velocity was chosen halfway between the two lowest airspeeds for the single air separation procedure.

For each trial, samples of each size-class (except for the pan sample), obtained by triplicate sieving, were mixed thoroughly, and then divided into six equal-weighted portions. One portion was analyzed for chemical composition and served as a control for winnowing. Four portions were subjected to one time air separation, each having its own airflow velocity measured by the hand-

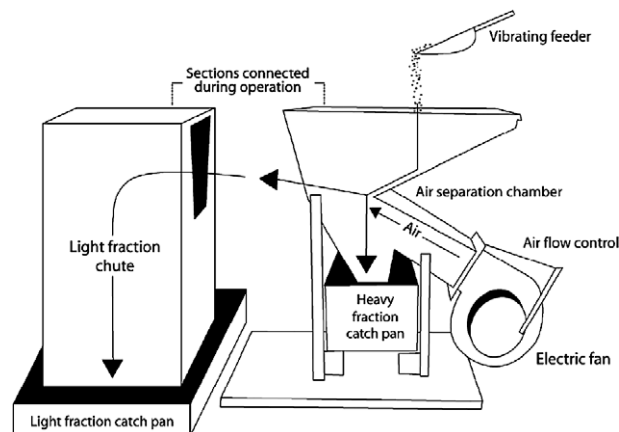


Fig. 1. Schematic drawing of an air blaster seed cleaner.

Table 1
Proximate composition of four DDGS samples.^a

DDGS samples	Moisture (%)	Protein (%)	Oil (%)	Ash (%)	Total CHO (%)
1	7.77 ± 0.22 c	30.66 ± 0.27 a	8.41 ± 0.28 b	4.39 ± 0.07 a	56.55 ± 0.09 ab
2	9.19 ± 0.07 b	30.62 ± 0.35 a	10.30 ± 0.51 a	3.84 ± 0.05 b	55.24 ± 0.11 b
3	10.73 ± 0.25 a	29.69 ± 0.04 a	10.19 ± 0.33 a	4.29 ± 0.04 a	55.83 ± 0.25 ab
4	9.08 ± 0.01 b	27.27 ± 0.28 b	11.50 ± 0.28 a	4.33 ± 0.07 a	56.90 ± 0.68 a
Minimum	7.77	27.27	8.41	3.84	55.24
Maximum	10.73	30.66	11.50	4.39	56.90
Average	9.19	29.56	10.10	4.21	56.13

^a Mean of duplicate measurements ± standard deviation; dry matter basis. CHO, carbohydrate. Column means bearing different letters were significant at $p < 0.05$.

Table 2
Dry fractionation of DDGS 1 and 2 by sieving and then winnowing: effects of sieve sizes and airflow velocities on fraction mass.^a

Type of fractionation	Symbols of fractions or sub-fractions	Particle size by US standard sieve No.	Particle size in microns (μm)	Sieved fraction mass (% whole sample)	Airflow velocity level	Airflow velocity (m/s)	Heavy fraction mass (% whole sample)	Light fraction mass (% whole sample)	Heavy fraction mass (% sieved fraction)	Light fraction mass (% sieved fraction)	
DDGS 1											
	Whole	Various		100.00							
Sieving	>25	>25	>710	33.91 a							
	25–45	25–45	710–355	30.73 b							
	45–80	45–80	355–180	26.62 c							
	<80	<80	<180	8.64 d							
Winnowing	>25 V1	>25	>710		I	3.35	29.47 a	4.44 d	86.89 a	13.11 d	
	>25 V2	>25	>710		II	5.14	23.51 b	10.40 c	69.33 b	30.67 c	
	>25 V3	>25	>710		III	6.93	16.97 c	16.93 b	49.95 c	50.05 b	
	>25 V4	>25	>710		IV	8.94	9.29 d	24.61 a	27.36 d	72.64 a	
	>25 R	>25	>710		3 Repeats	4.25	24.26 b	9.65 c	71.63 b	28.37 c	
	25–45 V1	25–45	710–355		I	2.23	26.68 a	4.04 d	86.84 a	13.16 d	
	25–45 V2	25–45	710–355		II	3.13	19.94 b	10.79 c	64.89 b	35.11 c	
	25–45 V3	25–45	710–355		III	4.02	10.06 c	20.67 b	32.73 c	67.27 b	
	25–45 V4	25–45	710–355		IV	4.69	5.83 d	24.90 a	18.97 d	81.03 a	
	25–45 R	25–45	710–355		3 Repeats	2.68	20.06 b	10.67 c	65.27 b	34.73 c	
	45–80 V1	45–80	355–180		I	0.89	22.26 a	4.36 d	83.58 a	16.42 d	
	45–80 V2	45–80	355–180		II	1.34	17.89 b	8.73 c	67.22 b	32.78 c	
	45–80 V3	45–80	355–180		III	1.79	14.93 c	11.69 b	56.02 c	43.97 b	
	45–80 V4	45–80	355–180		IV	2.23	5.85 d	20.77 a	21.97 d	78.03 a	
	45–80 R	45–80	355–180		3 Repeats	1.12	14.86 c	11.76 b	55.79 c	44.20 b	
	DDGS 2										
		Whole	Various		100.00						
	Sieving	>18	>18	>1000	33.21 a						
		18–25	18–25	1000–710	26.43 b						
		25–40	25–40	710–425	28.25 b						
<40		<40	<425	11.74 c							
Winnowing	>18 V1	>18	>1000		I	3.58	29.08 a	4.13 d	87.55 a	12.45 d	
	>18 V2	>18	>1000		II	5.14	24.60 b	8.60 c	74.03 b	25.97 c	
	>18 V3	>18	>1000		III	6.70	14.87 c	18.34 b	44.68 c	55.32 b	
	>18 V4	>18	>1000		IV	8.05	6.30 d	26.91 a	18.97 d	81.03 a	
	>18 R	>18	>1000		3 Repeats	4.34	25.03 b	8.17 c	75.39 b	24.61 c	
	18–25 V1	18–25	1000–710		I	2.68	23.37 a	3.06 d	88.41 a	11.59 d	
	18–25 V2	18–25	1000–710		II	3.80	19.55 b	6.87 c	73.99 b	26.01 c	
	18–25 V3	18–25	1000–710		III	4.92	13.22 c	13.21 b	50.02 c	49.98 b	
	18–25 V4	18–25	1000–710		IV	6.03	5.95 d	20.48 a	22.51 d	77.49 a	
	18–25 R	18–25	1000–710		3 Repeats	3.26	19.63 b	6.80 c	74.26 b	25.74 c	
	25–40 V1	25–40	710–425		I	2.46	23.48 a	4.77 e	83.17 a	16.83 e	
	25–40 V2	25–40	710–425		II	3.13	20.03 b	8.21 d	70.95 b	29.05 d	
	25–40 V3	25–40	710–425		III	3.80	13.30 d	14.94 b	47.08 d	52.92 b	
	25–40 V4	25–40	710–425		IV	4.69	5.85 e	22.40 a	20.74 e	79.26 a	
	25–40 R	25–40	710–425		3 Repeats	2.82	17.43 c	10.82 c	61.71 c	38.29 c	

^a Mean of duplicate measurements. Column means with different letters were significant at $p < 0.05$ among the same group of fractions or sub-fractions; m/s, meters per second.

held anemometer. The remaining 6th portion was subjected to three repeated air separation procedures, using a single airflow velocity. Both light and heavy fractions were collected for all separations and analyzed for chemical composition.

2.3. Experiment 2: winnowing followed by sieving

A reversed order of combining the two dry fractionation procedures was also carried out for the DDGS 4 sample only. Instead of

sieving followed by winnowing, the method proceeded with winnowing followed by sieving. One hundred gram of DDGS 4 was first separated by the air blast seed cleaner with four progressive air-speeds selected based on the same criterion stated in the above paragraph. The resulting four heavy fractions and corresponding four light fractions were then sieved based on the same sieving procedure described above. For each heavy fraction, two US standard sieves (mesh size 25 and 40) and a pan were selected for sieving, while for each light fraction, two US standard sieves with smaller holes (mesh size 40 and 70) plus a pan were used. Thus, for each light or heavy fraction, three sieved fractions were obtained.

2.4. Chemical analysis

The original DDGS samples, and all fractions or sub-fractions obtained through sieving, winnowing and their combinations were measured for contents of moisture, protein, oil, and ash. The original DDGS is termed as “whole” sample or fraction in contrast to

sieved or winnowed fractions. Moisture and ash contents were determined according to official methods (AOAC, 2002). The moisture content was used to convert concentrations of other components into a dry matter basis. The total nitrogen/protein content in samples was measured by a combustion method (AOAC, 2002), using a protein analyzer (Model FT528, Leco Corp. St. Joseph, MI). The protein content was calculated with a conversion factor of 5.75. The oil content was determined by an AOCS Official procedure (AOCS, 2005), using a fat analyzer (Model XT 10, Ankom Technology, Macedon, NY). However, instead of using petroleum ether, hexane was used as the extracting solvent. The total carbohydrate (CHO) was calculated for all types of samples, based on contents of protein, oil and ash, dry matter basis.

2.5. Statistical analysis

Data were treated with JMP software, version 5 (JMP, a business unit of SAS, Cary, NC, USA), for calculation of means and standard deviation, and for analysis of variance (ANOVA) in order to deter-

Table 3
Dry fractionation of DDGS 2 and 3 by sieving and then winnowing: effects of sieve sizes and airflow velocities on fraction mass.^a

Type of fractionation	Symbols of fractions or sub-fractions	Particle size by US standard sieve No.	Particle size in microns (μm)	Sieved fraction mass (% whole sample)	Airflow velocity level	Airflow velocity (m/s)	Heavy fraction mass (% whole sample)	Light fraction mass (% whole sample)	Heavy fraction mass (% sieved fraction)	Light fraction mass (% sieved fraction)
DDGS 3										
	Whole	Various		100.00						
Sieving	>18	>18	>1000	29.68 a						
	18–25	18–25	1000–710	28.51 a						
	25–40	25–40	710–425	29.99 a						
	<40	<40	<425	11.66 b						
Winnowing	>18 V1	>18	>1000		I	4.47	25.41 a	4.27 d	85.61 a	14.39 d
	>18 V2	>18	>1000		II	5.81	19.43 b	10.26 c	65.48 b	34.52 c
	>18 V3	>18	>1000		III	7.15	10.94 c	18.75 b	36.82 c	63.18 b
	>18 V4	>18	>1000		IV	8.49	5.44 d	24.25 a	18.32 d	81.68 a
	>18 R	>18	>1000		3 Repeats	5.14	20.61 b	9.08 c	69.43 b	30.57 c
	18–25 V1	18–25	1000–710		I	3.35	24.93 a	3.57 d	87.45 a	12.55 d
	18–25 V2	18–25	1000–710		II	4.47	19.29 b	9.21 c	67.67 b	32.33 c
	18–25 V3	18–25	1000–710		III	5.36	12.39 c	16.12 b	43.46 c	56.54 b
	18–25 V4	18–25	1000–710		IV	6.26	5.95 d	22.55 a	20.87 d	79.13 a
	18–25 R	18–25	1000–710		3 Repeats	3.80	18.86 b	9.65 c	66.16 b	33.84 c
	25–40 V1	25–40	710–425		I	2.68	24.74 a	5.25 d	82.48 a	17.52 d
	25–40 V2	25–40	710–425		II	3.35	19.66 b	10.33 c	65.54 b	34.46 c
	25–40 V3	25–40	710–425		III	4.02	13.34 c	16.65 b	44.47 c	55.53 b
	25–40 V4	25–40	710–425		IV	4.69	6.22 d	23.78 a	20.73 a	79.26 a
	25–40 R	25–40	710–425		3 Repeats	2.99	18.54 b	11.45 c	61.86 b	38.18 c
	DDGS 4									
	Whole	Various		100.00						
Sieving	>25	>25	>710	31.95 a						
	25–45	25–40	710–425	31.49 a						
	45–80	40–70	425–212	25.71 b						
	<80	<70	<212	10.58 c						
Winnowing	>25 V1	>25	>710		I	2.68	26.77 a	5.18 d	83.78 a	16.21 d
	>25 V2	>25	>710		II	4.02	20.95 b	11.00 c	65.56 b	34.44 c
	>25 V3	>25	>710		III	5.36	14.63 c	17.32 b	45.79 c	54.21 b
	>25 V4	>25	>710		IV	6.48	6.19 d	25.76 a	19.37 d	80.63 a
	>25 R	>25	>710		3 Repeats	3.35	21.67 b	10.28 c	67.84 b	32.16 c
	25–40 V1	25–40	710–425		I	2.01	27.11 a	4.37 d	86.11 a	13.89 d
	25–40 V2	25–40	710–425		II	2.91	20.24 b	11.24 c	64.31 b	35.69 c
	25–40 V3	25–40	710–425		III	3.80	11.42 c	20.06 b	36.29 c	63.71 b
	25–40 V4	25–40	710–425		IV	4.69	5.76 d	25.73 a	18.29 d	81.70 a
	25–40 R	25–40	710–425		3 Repeats	2.46	19.91 b	11.57 c	63.25 b	36.75 c
	40–70 V1	40–70	425–212		I	0.89	22.91 a	2.79 d	89.13 a	10.87 d
	40–70 V2	40–70	425–212		II	1.34	19.11 b	6.60 c	74.34 b	25.65 c
	40–70 V3	40–70	425–212		III	1.79	14.49 c	11.21 b	56.40 c	43.59 b
	40–70 V4	40–70	425–212		IV	2.23	5.65 d	20.06 a	21.99 d	78.01 a
	40–70 R	40–70	425–212		3 Repeats	1.12	13.47 c	12.24 c	52.46 c	47.54 b

^a Mean of duplicate measurements. Column means with different letters were significant at $p < 0.05$ among the same group of fractions or sub-fractions; m/s, meters per second.

mine the effect of sieving, winnowing, airflow rate, and their interactions. The Tukey's HSD (honestly significant difference) test was also conducted for pair-wise comparisons when there was a significant effect at $p < 0.05$ based on ANOVA.

3. Results and discussion

According to Liu (2008), DDGS consisted of particles varying in size and composition but close examination of DDGS showed that the particles can be grouped into three classes, flakes, granules, and aggregated granules. The flakes come mostly from tip cap and broken seed coat of corn kernels. The granules are mostly non-fermentable materials which are left from ground endosperm and germ. The aggregated granules are granules glued together, apparently by condensed distillers solubles added during the final stage of the process. Because all three types of particulates vary in size, shape and density, their partial separation by sieving and winnowing becomes possible. Since the flakes are mostly fiber, while granules or aggregated granules are mostly non-fiber components, their separation leads to differences in composition of fractions. This is the scientific basis for the dry separation of DDGS.

3.1. Chemical composition of DDGS samples

The four DDGS samples had similar but varying levels of chemical compositions (Table 1). The moisture content ranged from 7.77% to 10.73%, with an average value of 9.19%. On a dry matter basis, protein ranged from 27.27% to 30.66%; oil, 8.41% to 11.50%; ash, 3.84% to 4.39%; and total CHO, 55.28% to 56.85%. Since little simple sugars were expected to remain in DDGS after fermentation, since the majority of DDGS have a residual starch content around 5% (Belyea et al., 2004; Liu, 2008), the content of fibers (insoluble CHO) would be proportional to total CHO content in DDGS samples.

Among the four samples, DDGS 1 was rather unique. On the visual appearance, it had intact germs, while others did not. Apparently, DDGS 1 resulted from a modified method, in which germs were removed before liquefaction/saccharification (the steps convert starch to fermentable sugars) and were added back in a later step.

3.2. Effect of sieving

Because the four DDGS samples had different particle size distributions, for maximizing overall separation efficiency, different sieve series were used for each of DDGS samples (Tables 2 and

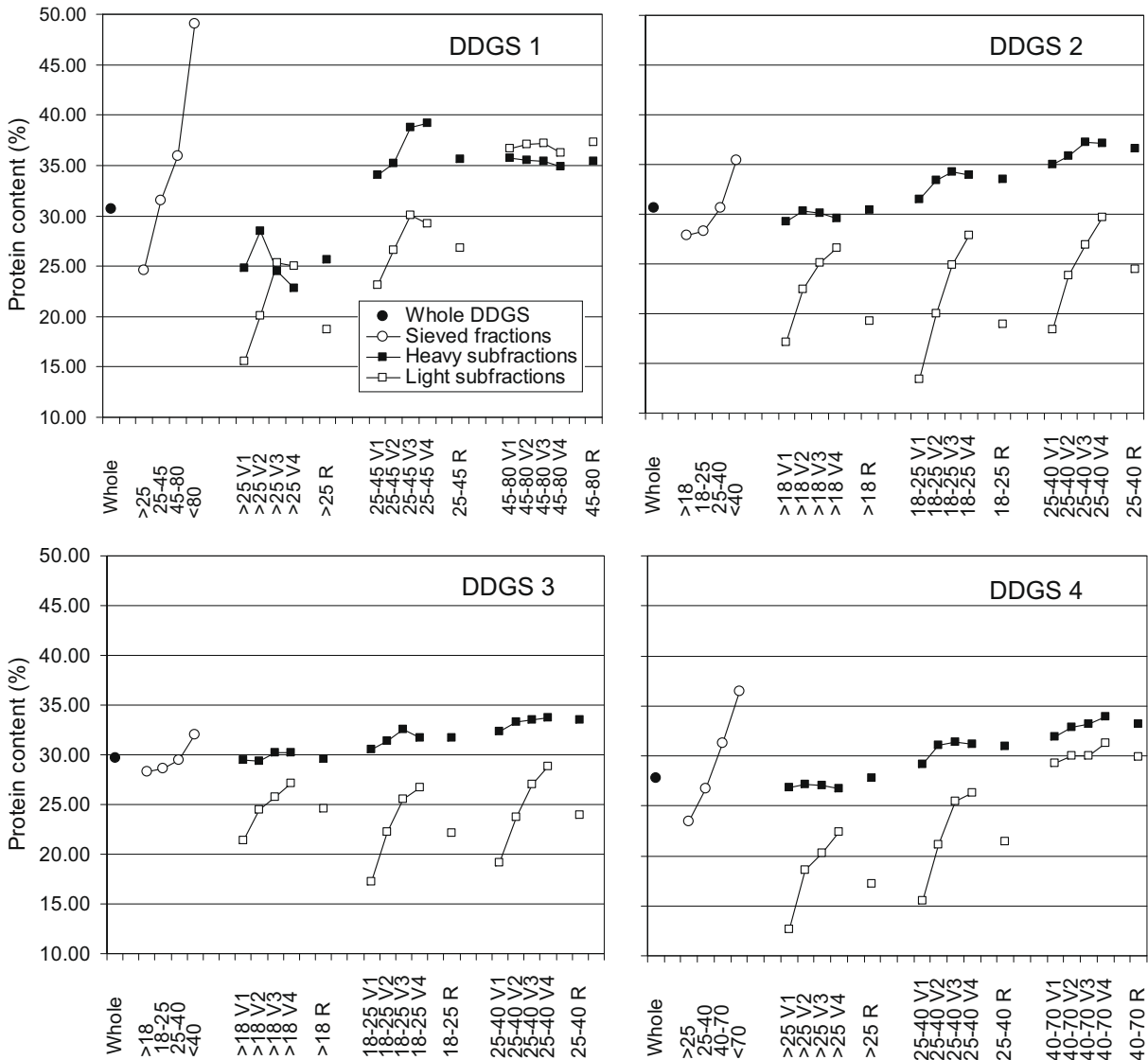


Fig. 2. Protein content (% dry matter) in whole and four sieved fractions, from each DDGS, and in five heavy and five light sub-fractions resulting from winnowing each of three sieved fractions (the pan fraction was excluded) at different airflow velocities. The symbols of fractions or sub-fractions in X-axis are defined in Tables 2 and 3.

3). For DDGS 1, sieves with mesh size 25, 45, and 80, plus a pan were used. For DDGS 2 and 3, sieves with mesh size 18, 25 and 40, plus a pan were used. For DDGS 4, sieves with mesh size 25, 40 and 70, plus a pan were used. These sets of sieve series were selected based on the criterion specified in the Method section. Regardless the difference in sieve series selected for sieving, all samples were sized into four sieved fractions.

Confirming the finding of Wu and Stringfellow (1986) and Liu (2008), in this study sizing by sieves was found effective in producing sieved fractions with varying levels of chemical composition (Figs. 2–5, the first cluster of the data sets on the X-axis for each DDGS sample). The effectiveness as well as the changing direction for a particular component as a function of increasing mesh size number depended on individual samples as well as components. In general, as the particle size decreased (that is, increasing mesh size No.), protein (Fig. 2) and ash (Fig. 4) contents increased, and the total CHO content (Fig. 5) decreased in all samples; oil content decreased in DDGS 1–3 but increased slightly in DDGS 4 (Fig. 3). Thus, the pan fraction had the finest particle size, and was highest in protein and ash and lowest in total CHO content. Sieving was most effective in producing fractions with varying composition for DDGS 1, and less effective for the other three samples. The highest increase in oil con-

tent observed with the fraction of >25 mesh from DDGS 1 was due to the presence of intact germ in this material.

There is a discrepancy in the literature regarding oil changes by sieving. Wu and Stringfellow (1986) reported that protein and ash contents increased, and lipid and neutral detergent fiber contents decreased as particle size decreased. Srinivasan et al. (2005) showed that fractions with smallest particle size had increased protein and fat contents and reduced fiber. This was true for both DDGS samples they studied. The current study with four DDGS samples indicated that the changing direction of the oil content as a result of sieving actually depended on DDGS samples, with majority of samples showing a decrease in oil content with reducing particle size.

3.3. Effect of air separation following sieving

To further fractionate DDGS for compositional shifting, all sieved fractions except for the one retained on the pan from each DDGS sample were subjected to winnowing at each of four increasing airflow velocities or to winnowing three times at one single velocity (Tables 2 and 3). The combined three sieved fractions had about 90% of the original sample mass.

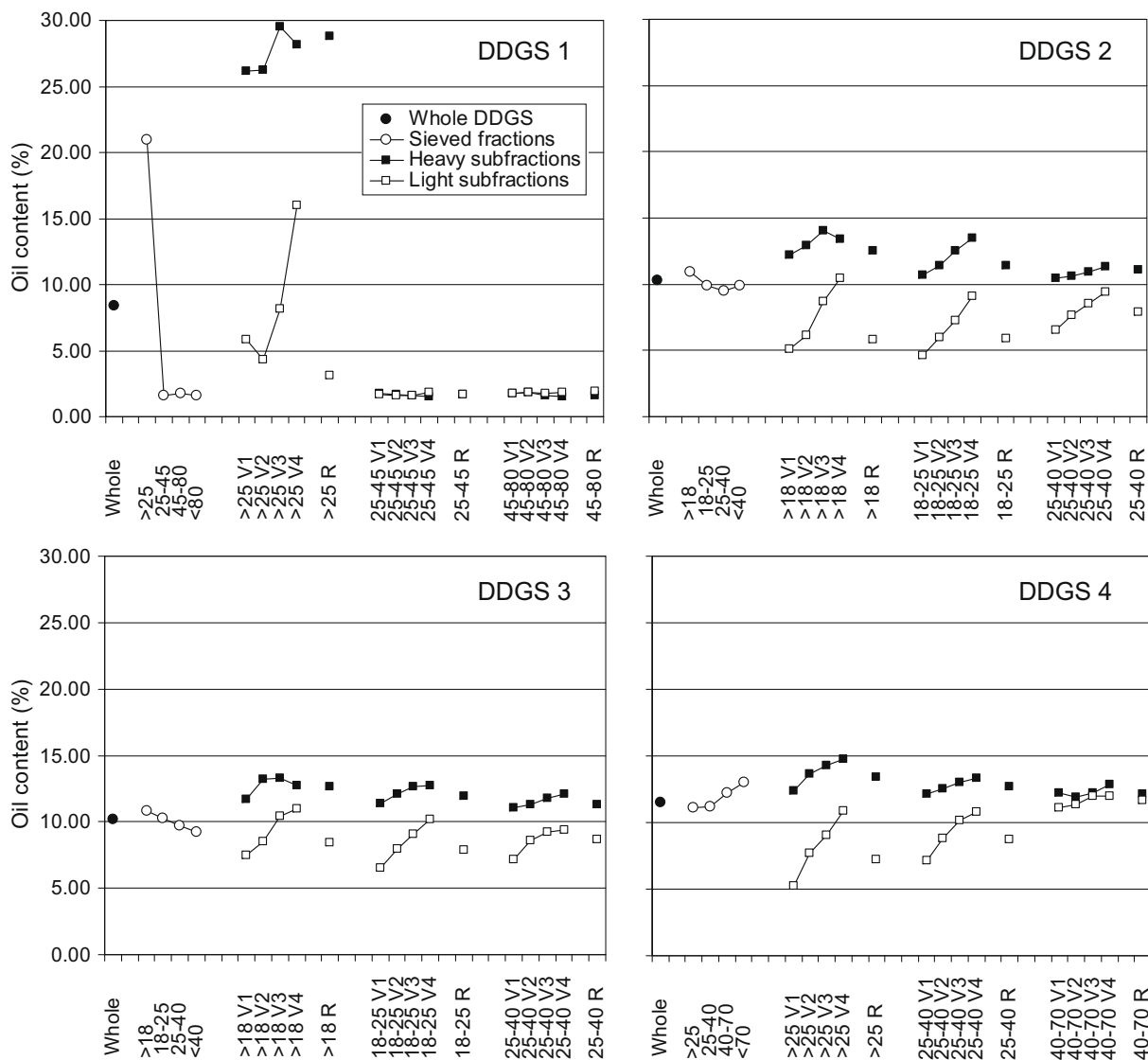


Fig. 3. Oil content (% dry matter) in whole and four sieved fractions, from each DDGS, and in five heavy and five light sub-fractions resulting from winnowing each of three sieved fractions (the pan fraction was excluded) at different airflow velocities. The symbols of fractions or sub-fractions in X-axis are defined in Tables 2 and 3.

Winnowing sieved fractions was also effective in fractionating DDGS since it caused differences in chemical composition between the heavy and light sub-fractions (Figs. 2–5). The larger this difference was, the more effective the process was. The difference in composition between heavy and light sub-fractions varied with DDGS samples, sieved fractions within a sample, airflow velocity, and individual chemical components. Protein (Fig. 2) was concentrated in all heavy sub-fractions except for those from the DDGS 1 sieved fraction of 45–80 mesh at all velocities and from the DDGS 1 sieved fraction of >25 mesh at velocity 3 and 4. Oil (Fig. 3) was also enriched significantly in heavy sub-fractions except for those from two sieved fractions of DDGS 1, 25–45 and 45–80 mesh, where no significant difference was observed between the light and heavy sub-fractions, at all velocities. Change in ash content as a result of air separation was rather dependent on DDGS source (Fig. 4). For DDGS 1, it was reduced in all heavy sub-fractions, but for DDGS 2–4, it was concentrated in all heavy sub-fractions. Total CHO was reduced in heavy sub-fractions of all sieved fractions, except for one sieved fraction of DDGS 1, having particle size of 45–80 mesh (Fig. 5).

As for the effect of particle size on winnowing efficiency, the larger the particle size of the sieved fraction, the larger the difference in chemical composition between the light and heavy

fractions. An explanation is that the winnowing machine separates heavy particles from light ones by density differences; the greater the difference in density among particles, the more effective the separation. It could not separate materials with little difference in densities. For sieved fractions with smaller particle size, density difference would diminish among particles. For the same reason, the pan fraction for all DDGS samples, which had finest particles, was not subjected to winnowing at all.

Airflow velocity also affected effectiveness of dry fractionation. Given a total amount of samples to be winnowed, as the velocity increased, there was an increase in the mass of a light fraction and a corresponding decrease in the mass of the heavy fraction (Table 2). This resulted in changes of not only concentration of a particular component, but also of the yield of a particular fraction. With respect to compositional changes, in general, as the velocity increased, protein, oil and ash contents in both heavy and light sub-fractions increased, but they increased at a faster rate in the light sub-fraction than the heavy sub-fraction (Figs. 2–4). Similarly, the total CHO content decreased in both the heavy and light sub-fractions but it decreased at a faster rate in the light sub-fraction than the heavy sub-fraction (Fig. 5). Here, the reference for “increased” or “decreased” is based of the level at the lowest airflow

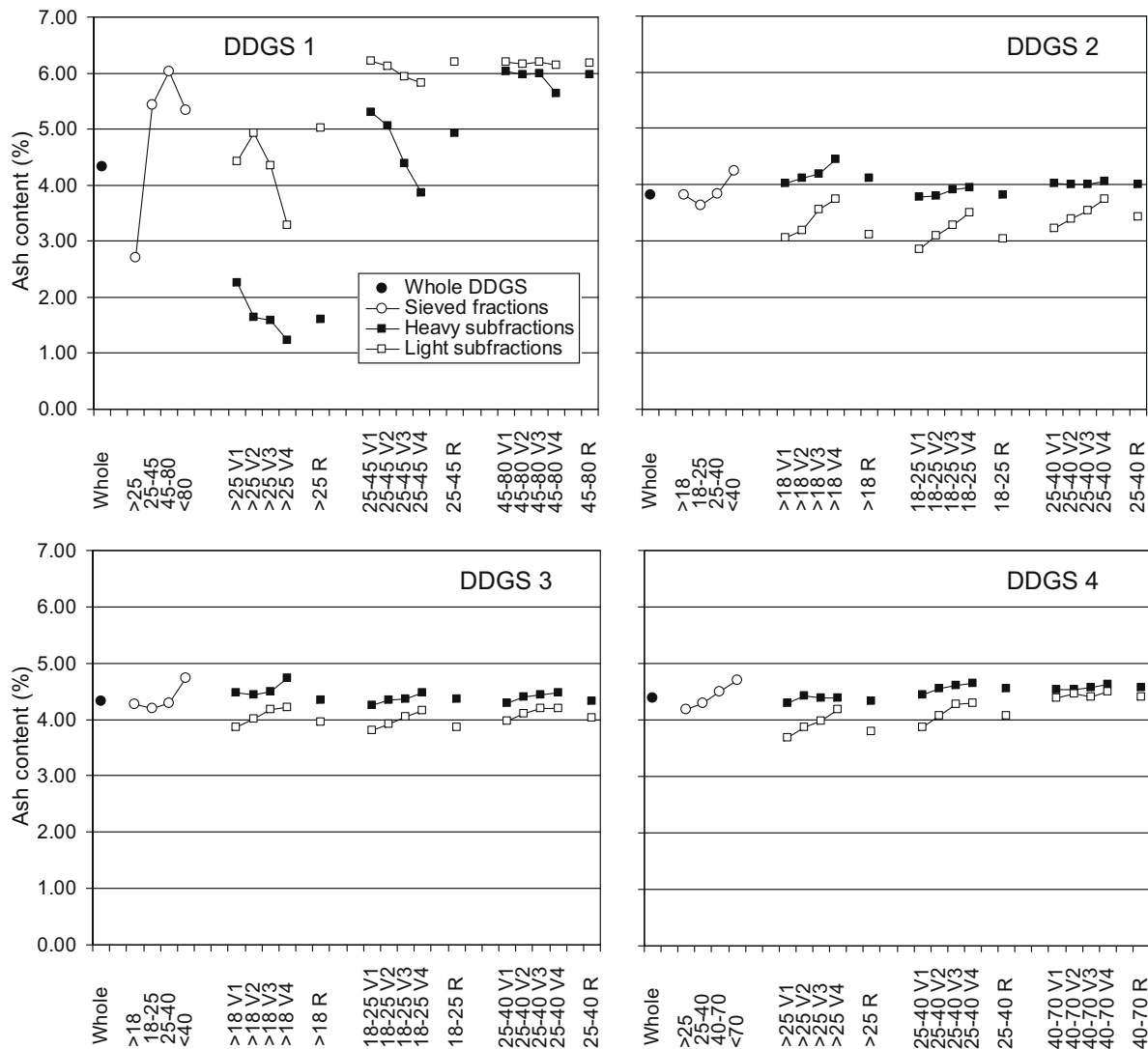


Fig. 4. Ash content (% dry matter) in whole and four sieved fractions, from each DDGS, and in five heavy and five light sub-fractions resulting from winnowing each of three sieved fractions (the pan fraction was excluded) at different airflow velocities. The symbols of fractions or sub-fractions in X-axis are defined in Tables 2 and 3.

velocity. It is not based on the level of the original sieved fraction or whole DDGS sample. Consequently, as the velocity increased, the compositional difference between the heavy and light sub-fractions decreased. Interestingly, three consecutive repeats of air separation at a lower velocity were as effective as a single air separation at a medium velocity.

DDGS consists mainly of flakes, granules and aggregated granules (Liu, 2008). Because the particulates come from different structural parts of corn kernels and because they differ in shape, the flakes would be less dense than the granular particulates. When an air current flows through sieved DDGS fractions, it would carry the flakes away along with some granular particulates of smaller size. As the airflow velocity increases, an increasing portion of granular particulates would be carried away into the light fraction. Because the force needed to carry smaller particles is lower than that for larger particles, as the size of material fed into the winnowing machine decreases, the operating air velocity would have to decrease in order to produce the same amount of lighter fraction.

Furthermore, sieving and winnowing did not necessarily cause parallel changes for some components measured. For example,

sieving caused protein and ash to increase and oil and total CHO to decrease in finer fractions of DDGS 1, while winnowing caused protein and oil to increase and ash and total CHO to decrease in the heavy sub-fractions. Srinivasan et al. (2005) showed that sieving as well as air separation by elutriation caused increases in both protein and oil contents in fractions with finer particle size or heavy sub-fractions. The current study with DDGS 4 confirmed their finding, but not with DDGS 1–3.

Since each procedure was duplicated, the standard deviation of measured parameters (mass and contents of chemical components) for each fraction or sub-fraction was calculated. On an average, in terms of relative standard deviation of the fraction mass measured, sieving gave 2.8%, winnowing, 2.5%, and the combination of the two, less than 5%. Therefore, the two fraction procedures and their combinations were rather reproducible.

3.4. Effects of winnowing followed by sieving

In previous discussion, sieving whole DDGS samples or winnowing sieved fractions were shown both effective in producing

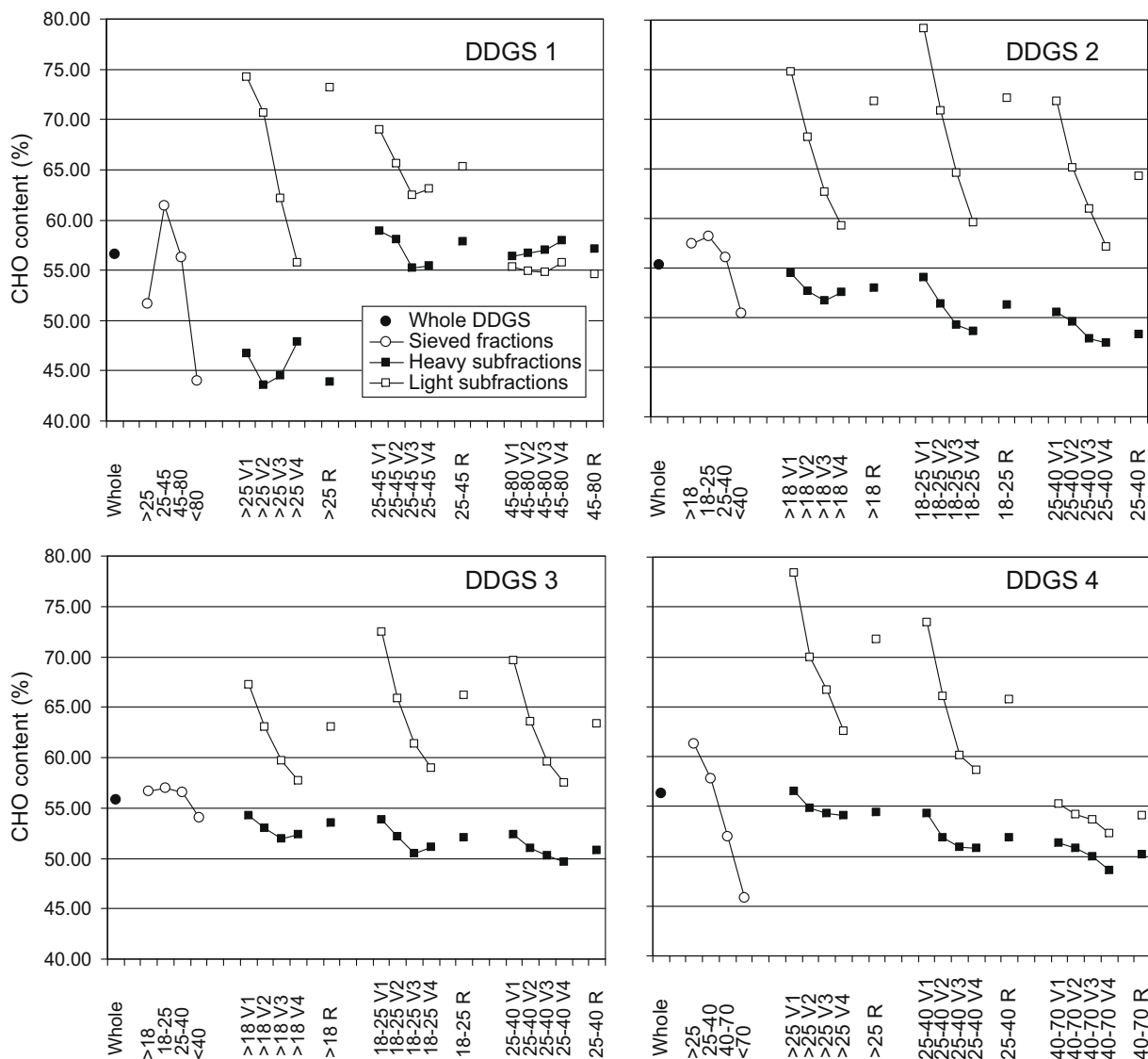


Fig. 5. Total carbohydrate content (% dry matter) in whole and four sieved fractions, from each of DDGS, and in five heavy and five light sub-fractions resulting from winnowing each of three sieved fractions (the pan fraction was excluded) at different airflow velocities. The symbols of fractions or sub-fractions in X-axis are defined in Tables 2 and 3.

fractions or sub-fractions with varying levels of chemical composition, and thus achieving the objective of enriching or reducing certain components. For comparison, a reverse order of the two procedures was tried on DDGS 4. Results show that winnowing whole DDGS sample apparently failed to produce fractions with substantial changes in chemical composition at any airflow velocities but sieving either heavy or light fractions obtained by winnowing caused significant changes in composition of resulting sieved sub-fractions (Table 4). This observation can be explained as follows: The size of particles in a whole DDGS sample varied greatly. So did density of particles. Without prior sieving into sized fractions, a fraction with similar density obtained through air separation would contain particles of different sizes. Since particle size was very much related to chemical composition (Liu, 2008), a fraction containing particles with varying size would have an average composition, close to that of whole sample. However, when this fraction was subsequently sieved according to particle size, difference in chemical composition among sieved sub-fractions would be exhibited.

3.5. Comparisons for effects of sieving, winnowing and their combinations

When expressed as percentage of original concentrations in the whole sample, sieved fraction or winnowed fraction, the maximum decrease and the maximum increase in concentrations of chemical components in DDGS fractions or sub-fractions due to sieving, winnowing and their combinations varied greatly with components,

DDGS samples and the combination order (Table 5). Except for DDGS 1, in either combination order (S + W or W + S), based on maximum changing span values, the second fractionation procedure was more effective in causing changes in contents of all four components (protein, oil, ash and total CHO) than the first procedure. Furthermore, the first procedure tended to increase protein, oil and ash contents to a larger extent in resulting fractions than to decrease these components, while the second procedure tended to decrease these components to a larger extent than to increase them in resulting fractions. For CHO, trends were just opposite under first and second procedures, respectively.

In general, the combination of the two procedures was most effective in shifting chemical composition for DDGS 1, least effective for DDGS 3. For DDGS 2 and 4, the effect was in between. Excluding DDGS 1, in terms of chemical components, the combined procedures led to more changes in protein, oil and total CHO contents than the ash content in the resulting fractions. Overall, for protein concentration, the maximum decrease was –56.4% in DDGS 2, the maximum increase was 60.2% in DDGS 1, and the maximum changing span was 109.5% in DDGS 1; for oil concentration, the maximum decrease was –81.4%, the maximum increase was 262.7%, and the maximum changing span was 344.1%, all found in DDGS 1; for ash concentration, the maximum decrease was –71.5%, the maximum increase was 39.2%, and the maximum changing span was 110.7%, all found also in DDGS 1; for total CHO concentration, the maximum decrease was –23.1% in DDGS 1, the maximum increase was 42.2% in DDGS 2, and the maximum changing span was 56.5% in DDGS 2. As mentioned earlier, DDGS 1

Table 4

Dry fractionation of DDGS 4 by winnowing and then sieving: effects of airflow velocities and sieve size on mass and chemical composition of resulting fractions.^a

Type of fractionation	Airflow velocity (m/s)	Winnowed fraction type	Particle size by US standard sieve No.	Particle size in microns (μm)	Fraction mass (%) blown fraction)	Fraction mass (% whole sample)	Protein in fraction (%)	Oil in fraction (%)	Ash in fraction (%)	Total CHO in fraction (%)
Whole						100.00	27.27	11.50	4.38	56.85
Winnowing	2.01	Heavy			75.49 a	27.90 a	11.54 bc	4.42 ab	56.77 a	
	2.01	Light			23.14 d	29.83 a	11.37 bc	4.34 ab	54.37 a	
	2.91	Heavy			58.82 b	28.15 a	11.48 bc	4.44 a	56.21 a	
	2.91	Light			39.45 c	29.45 a	10.85 bc	4.27 b	55.27 a	
	3.80	Heavy			36.08 c	28.84 a	12.08 ab	4.42 ab	54.65 a	
	3.80	Light			62.66 b	28.01 a	10.81 c	4.43 a	56.77 a	
	4.69	Heavy			20.09 d	28.02 a	12.87 a	4.34 ab	54.64 a	
	4.69	Light			78.63 a	28.66 a	10.90 bc	4.47 a	56.11 a	
Sieving	2.01	Heavy	>25	>710	40.27 b	30.40 a	25.79 e	11.69 b	4.23 c	58.29 b
	2.01	Heavy	25–40	710–425	38.03 bc	28.70 a	28.30 d	11.62 b	4.39 bc	55.69 c
	2.01	Heavy	<40	<425	21.51 d	16.24 b	33.15 b	12.39 ab	4.54 ab	49.91 e
	2.01	Light	>40	>425	17.95 d	4.14 d	14.60 f	6.77 c	3.82 d	74.81 a
	2.01	Light	40–70	425–212	46.63 a	10.79 c	30.85 c	11.90 ab	4.34 bc	52.91 d
	2.01	Light	<70	<212	35.66 c	8.26 c	36.76 a	12.63 a	4.69 a	45.92 f
	2.91	Heavy	>25	>710	46.08 a	27.10 a	25.32 d	12.36 b	4.26 d	58.06 b
	2.91	Heavy	25–40	710–425	39.42 b	23.19 a	29.60 c	11.91 c	4.48 c	54.01 c
	2.91	Heavy	<40	<425	15.09 e	8.89 c	33.30 b	12.26 b	4.58 b	49.86 d
	2.91	Light	>40	>425	31.04 c	12.25 c	18.68 e	8.24 d	3.98 e	69.10 a
	2.91	Light	40–70	425–212	45.18 a	17.82 b	31.36 bc	12.02 b	4.42 c	52.19 cd
	2.91	Light	<70	<212	23.47 d	9.25 c	36.63 a	12.67 a	4.70 a	46.01 e
	3.80	Heavy	>25	>710	60.61 a	21.86 c	27.24 d	12.80 a	4.31 d	55.65 b
	3.80	Heavy	25–40	710–425	32.97 d	11.89 d	30.46 c	12.53 a	4.52 bc	52.48 c
	3.80	Heavy	<40	<425	6.08 f	1.83 e	34.02 b	12.52 a	4.65 ab	48.81 d
	3.80	Light	>40	>425	46.32 b	29.65 a	22.92 e	9.01 b	4.17 e	65.54 a
	3.80	Light	40–70	425–212	38.51 c	24.13 b	31.68 c	12.10 a	4.46 cd	51.77 c
	3.80	Light	<70	<212	15.82 e	9.91 d	36.67 a	12.76 a	4.71 a	45.86 e
	4.69	Heavy	>25	>710	76.26 a	15.32 c	27.32 c	14.65 a	4.34 c	53.70 bc
	4.69	Heavy	25–40	710–425	20.92 d	4.20 e	30.81 b	13.40 ab	4.64 ab	51.15 bc
	4.69	Heavy	<40	<425	2.03 f	0.41 f	35.76 a	10.81 cd	4.56 ab	48.87 c
	4.69	Light	>40	>425	55.30 b	43.48 a	23.00 d	9.39 d	4.31 c	64.90 a
	4.69	Light	40–70	425–212	31.90 c	25.08 b	31.75 b	11.95 bc	4.48 bc	51.82 bc
	4.69	Light	<70	<212	12.57 e	9.88 d	36.81 a	12.78 b	4.72 a	45.68 d

^a Mean of duplicate measurements. Values in the same column of the same group with different letters were significant at $p < 0.05$.

Table 5

The maximum decrease, the maximum increase, and the maximum changing span in concentrations of chemical components in DDGS fractions or sub-fractions due to sieving, winnowing and their combinations.^a

Component	Method	DDGS sample	First fractionation procedure			Second fractionation procedure			Combined procedure		
			Maximum decrease due to S or W (percentage of whole sample)	Maximum increase due to S or W (percentage of whole sample)	Maximum changing span due to S or W (percentage of whole sample)	Maximum decrease due to W or S (percentage of fraction)	Maximum increase due to W or S (percentage of fraction)	Maximum changing span due to S or W (percentage of fraction)	Maximum decrease due to S + W or W + S (percentage of whole sample)	Maximum increase due to S + W or W + S (percentage of whole sample)	Maximum changing span due to S + W or W + S (percentage of whole sample)
Protein	S + W	1	-19.7	60.2	79.9	-36.9	24.4	61.4	-49.4	60.2	109.5
	S + W	2	-9.1	15.7	24.8	-52.8	21.4	74.3	-56.4	21.6	78.0
	S + W	3	-4.7	7.9	12.7	-39.9	14.6	54.5	-42.0	13.7	55.7
	S + W	4	-15.8	31.3	47.1	-46.0	17.5	63.5	-54.5	31.3	85.8
	W + S	4	-0.6	6.3	6.9	-51.1	30.9	82.0	-48.0	31.2	79.2
Oil	S + W	1	-81.4	150.0	231.4	-85.0	45.1	130.1	-81.4	262.7	344.1
	S + W	2	-4.1	6.3	10.4	-53.6	35.8	89.3	-55.1	36.6	91.7
	S + W	3	-9.4	6.0	15.3	-36.2	21.8	58.0	-35.9	30.7	66.6
	S + W	4	-3.4	13.0	16.5	-52.6	33.3	86.0	-54.3	28.7	83.0
	W + S	4	-3.2	15.4	18.5	-40.5	13.8	54.3	-39.3	31.2	70.6
Ash	S + W	1	-37.7	39.2	76.9	-54.3	82.8	137.1	-71.5	39.2	110.7
	S + W	2	-4.9	11.5	16.3	-21.6	16.6	38.2	-20.0	16.8	36.9
	S + W	3	-3.0	9.7	12.7	-9.6	10.9	20.5	-11.7	9.7	21.4
	S + W	4	-4.5	7.1	11.6	-12.0	8.2	20.2	-15.9	7.1	23.0
	W + S	4	0.1	4.1	4.1	-14.1	8.3	22.4	-11.7	9.6	21.3
Total CHO	S + W	1	-22.3	8.6	30.9	-15.8	43.6	59.4	-23.1	31.1	54.3
	S + W	2	-8.7	5.2	13.9	-16.4	36.2	52.6	-14.2	42.2	56.5
	S + W	3	-3.3	2.0	5.3	-12.2	27.3	39.5	-11.1	29.8	40.9
	S + W	4	-18.7	8.9	27.5	-11.8	27.9	39.7	-13.8	39.2	53.0
	W + S	4	-1.2	2.2	3.4	-19.2	37.6	56.9	-17.0	35.9	52.9

S, sieving; W, winnowing; S + W, sieving and then winnowing; W + S, winnowing and then sieving; CHO, carbohydrate.

^a Mean of duplicate values, expressed as percentage of original concentration in the whole sample, sieved fraction or winnowed fraction.

was a unique sample since it had intact germ. This also explained that for this sample the procedure had a maximum efficiency in changing its oil content.

With regard to the effect of procedure sequence, in the reversed order (W + S), although winnowing was not effective for producing two fractions having substantial difference from the whole fraction in chemical composition, the overall effect of winnowing and sieving was comparable to that by sieving followed by air separation. For example, the method of sieving followed by winnowing (S + W) produced a fraction having a 54.5% reduction in protein content from the original DDGS 4 sample, the maximum protein reduction possible, and another fraction had a 31.3% increase in protein content, the maximum increase possible, resulting in a maximum changing span of 85.8%. In comparison, the method of air separation followed by sieving (W + S) produced a fraction having a 48.0% reduction in protein content, the maximum protein reduction possible, and another fraction having a 31.2% increase in protein content, the maximum increase possible, resulting in a maximum changing span of 79.2%. However, in practice, the W + S order requires longer time to complete than the S + W order, the later combination sequence is thus recommended.

Using an elutriation apparatus they developed, Srinivasan et al. (2005) subjected the sieved fractions of 3 larger size categories (out of a total of 5 size classes) from each of two DDGS samples, a total of 59.7% and 42.9% of the original DDGS mass respectively, to air separation with varying air velocities. In this study, all sieved fractions except for the pan fraction (3 out of a total of 4 size classes) from each of four DDGS samples, a total of over 90% of original sample mass, were subjected to winnowing by the commercially-available air blast seed cleaner. However, unlike the present study, Srinivasan et al. (2005) did not include the reverse order of the two

procedures, that is, air separation followed by sieving. They also did not calculate the effect of individual and combined procedures on shifting chemical composition in terms of maximum decrease, maximum increase and/or maximum changing span. Therefore, no conclusion could be made with regard to which type of air separation was more effective, winnowing in this study or elutriation in the study of Srinivasan et al. (2005).

In conclusion, this study shows effectiveness of sieving, winnowing and their combinations in producing DDGS fractions with varying ranges of chemical compositions, including fractions enriched with protein and oil, and fractions enriched with CHO (presumably fiber as a major part). The effect was significant, particularly when the two procedures were combined. In terms of shifting component contents, sieving the whole fraction of DDGS was effective but sieving winnowed DDGS fractions was more effective. Winnowing the whole fraction of DDGS was ineffective, but winnowing sieved DDGS fractions was effective. The overall effect of winnowing followed by sieving was same as that of sieving followed by winnowing, but the later was less time consuming and thus is recommended as a choice for a combined method. Overall, dry fractionation would expand market shares, increase selling price, and improve end use values of DDGS.

References

- AOAC (Association of Official Analytical Chemists), 2002. AOAC Official Methods of Analysis. AOAC International.
- AOCS (American Oil Chemists' Society) Official Method, 2005. Rapid determination of oil/fat utilizing high temperature solvent extraction. Method Am 5-04.
- ASAE (American Society of Agricultural Engineers) Standards, 2003. Methods for determining and expressing fineness of feed materials by sieving. S319.3. St. Joseph, MI.

- Belyea, R.L., Rausch, K.D., Tumbleson, M.E., 2004. Composition of corn and distillers dried grains with solubles from dry grind ethanol processing. *Bioresource Technology* 94, 93–298.
- Bothast, R.J., Schlicher, M.A., 2005. Biotechnological processes for conversion of corn into ethanol. *Applied Microbiology and Biotechnology* 67, 19–25.
- Buchana, C.M., 2002. High value products from corn fiber. *Industrial Bioprocess* 24, 3–4.
- Cheng, Z.J., Hardy, R.W., Blair, M., 2003. Effects of supplementing methionine hydroxyl analogue in soybean meal and distiller's dried grain-based diets on the performance and nutrient retention of rainbow trout [(*Oncorhynchus mykiss* (Walbaum))]. *Aquaculture Research* 34, 1303–1310.
- Liu, K.S., 2008. Particle size distribution of distillers' dried grains with solubles (DDGS) and relationships to compositional and color properties. *Bioresource Technology* 99, 8421–8428.
- Rosentrater, K.A., Muthukumarappan, K., 2006. Corn ethanol coproducts: generation, properties, and future prospects. *International Sugar Journal* 108, 648–657.
- Singh, V., Moreau, R.A., Hickey, K.B., Belyea, R.L., Staff, C.H., 2002. Removal of fiber from distillers dried grains with solubles (DDGS) to increase values. *Transaction of the ASAE* 45, 389–392.
- Srinivasan, R., Singh, V., 2006. Removal of fiber from grain products including distillers dried grains with solubles. US Patent Application, Pub No. US 2006/0040024 A1, February 23.
- Srinivasan, R., Moreau, R.A., Rausch, K.D., Belyea, R.L., 2005. Separation of fiber from distillers dried grains with solubles (DDGS) using sieving and elutriation. *Cereal Chemistry* 82 (5), 528–533.
- Srinivasan, R., Dien, B.S., Rausch, K.D., Tumbleson, M.E., Singh, V., 2007. Fiber separated from distillers dried grains with solubles as a feedstock for ethanol production. *Cereal Chemistry* 84, 563–566.
- Wu, Y.V., Stringfellow, A.C., 1982. Corn distillers' dried grains with solubles and corn distillers' dried grain: dry fractionation and composition. *Journal of Food Science* 47, 1155–1157, 1180.
- Wu, Y.V., Stringfellow, A.C., 1986. Simple dry fractionation of corn distillers' dried grains and corn distillers dried grains with solubles. *Cereal Chemistry* 63 (1), 60–61.