



A new lab scale corn dry milling protocol generating commercial sized flaking grits for quick estimation of coproduct yield and composition

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ABSTRACT

The objective was to develop a 100 g lab scale dry milling protocol to determine effects of corn cultivars on coproduct fraction yields and composition. Six yellow dent, three colored and one white cultivar of corn were processed using 100 g samples to generate six coproducts, namely large grits, medium grits, small grits, fines, germ and pericarp. Compositional characteristics (crude protein, crude oil and neutral detergent fiber) of corn kernel and coproduct fractions were determined and compared against commercial samples. After increasing moisture to 23.5% on wet basis, corn kernels were passed through a degerminator. Sieving and aspiration steps were used to separate endosperm, germ and pericarp fractions without using a roller mill. True sized flaking or large grits were recovered which was not possible in earlier lab scale dry milling protocol. Coproduct yields were estimated with small coefficients of variability (< 10.0%). Crude oil content of large and medium grits for hard endosperm cultivars of yellow dent corn was < 1.1% (db). Grits from colored corn cultivars had higher crude oil and crude protein content compared to yellow corn cultivars. Hybrid effects were responsible for variations in coproduct yield and composition. Large grits yield was correlated with absolute density ($r = 0.89$) and test weight ($r = 0.85$). The protocol estimated coproduct yields with low standard deviations with respect to means and yielded true sized large grits. This new 100 g dry milling protocol should be helpful for industry in ascertaining dry milling characteristics of newly developed corn cultivars with small sample sizes.

1. Introduction

In addition to producing starch, grits, oil, distillers dried grains with solubles (DDGS), corn gluten meal and corn gluten feed for human and animal consumption (Somavat, 2017), corn processing industry also generates diverse industrial products like ethanol (Li et al., 2016; Zabed et al., 2016), biodiesel (Mata et al., 2012), biodegradable plastics (Pandey et al., 2017), cellulose nanofibrils (Valdebenito et al., 2017), zein (Dickey et al., 2001), furfural (Xiang and Runge, 2014), adhesives and plasticizers (Corn Refiners Association, 2016). These value-added coproducts enable traditional corn processing industry to generate additional revenue and be profitable despite fluctuations in prices of prime product like ethanol (Rosentrater, 2007).

Corn dry milling involves fractionating the kernel into different sizes containing endosperm, germ and pericarp. Based on particle size, endosperm fractions are classified further into flaking grits, cones, meal and flour. Large grits are used to make breakfast cereals and command

a high market price compared to other dry milling coproducts (Yuan and Flores, 1996). Unlike wet milling and dry grind processes where soft endosperm corn is preferred, hard endosperm corn is the choice for dry milling process as it yields greater proportion of large grits (Paulsen and Hill, 1985). Remaining endosperm fractions are utilized as brewery adjuncts and food ingredients. Germ and pericarp are combined with some endosperm fractions to make hominy feed, a low value coproduct (Rausch and Belyea, 2006). Corn dry milling caters to the needs of the human food sector; it is a relatively small industry and processes less than 2% of the total US corn (Corn Refiners Association, 2016). Generally dry milling characteristics of corn (fraction yields and their compositions) vary widely among corn cultivars (Rausch et al., 2009).

In the absence of necessary enzymes, humans cannot digest pericarp fiber; to prevent lipid oxidation and increase shelf life, < 1% fat is desired in endosperm fractions. Tempering corn is a vital operation whose purpose is to hydrate the germ, making it resilient against breakage during subsequent processing. Therefore, tempering

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conditions (time, temperature and moisture) and degermination method are important considerations and determine the effectiveness of the whole process (Rausch et al., 2009). Tempering is a vital dry milling operation; it helps in achieving differential hydration of corn kernels, making the germ resilient and facilitates effective germ and endosperm separation. Various methods used by researchers include two to three step tempering operations and tempering times of up to 16 h (Katta et al., 1997; Kirleis and Strohine, 1990; Pan et al., 1996; Strohine et al., 1986). In a lab scale study (12 kg samples), Peplinski et al. (1984) reported that a single-stage tempering method yielded large grits with 1.0–1.6% fat content. Effectiveness of a single stage tempering method was demonstrated in a series of pilot plant scale studies (40 kg samples) wherein a single stage 10 min tempering process was found to be sufficient for high flaking grit yields, although the fat content of flaking grits was not ascertained (Mehra and Eckhoff, 1997; Mehra et al., 2001). Rausch et al. (2009) reported a scaled down (1 kg), single stage tempering procedure which gave milling yields and crude fat contents comparable to those in the industry. However, due to a roller milling step required for germ separation, it was not possible to recover “true sized” large grits, although the process precisely quantified large grit yields.

Exploring the viability of using colored corns for anthocyanin recovery, purple and blue corn were fractionated using wet milling, dry milling and dry grind processes and coproduct yields were compared against conventional yellow dent corn (Somavat et al., 2016). Distribution and yield of anthocyanins in various coproduct streams were ascertained; in purple corn, pigments were concentrated in pericarp. Most pigments were recovered with pericarp in the dry milling process and with steepwater in the wet milling process. Pericarp recovery using dry milling was found to be the best process where pigment rich pericarp was recovered at the front end and remaining endosperm fraction, which remain unaffected, could be utilized further (Li et al., 2017). Our processing efforts were undertaken to assist the plant breeding group at the University of Illinois which concentrated on developing colored corn cultivars with higher pigment contents and adapting them to the climatic conditions of the Midwestern US. As a result, several types of colored corn were bred; often their harvested quantities ranged from a few hundred grams to a few kilograms.

Therefore, there was a need to dry mill smaller corn samples at 100 g scale and recover coproducts for milling characteristics and anthocyanin content determination. A study was conducted with 100 g samples and processing them using the 1 kg scale protocol (Rausch et al., 2009). Coproduct yields were comparable with the 1 kg process when post-temper drying time was reduced from 2 h to 30 min in 49 °C oven (Table 2). This demonstrated feasibility of a 100 g dry milling protocol; however, further adjustment of tempering time and post-temper drying duration was required. Another challenge was to adapt the process to recover large grits of size comparable to the commercial large grits so that not only yields, but also coproduct size from the protocol corresponded to those of industry.

2. Materials and methods

2.1. Materials

Ten corn cultivars including six yellow, three colored and one white corn were processed. Yellow corn included three hard endosperm cultivars (Hard1, Hard2 and Hard3), one high extractable starch (HE), one high amylose (HA) and one waxy cultivar (Waxy). All yellow corn and white corn were sourced from a major seed supplier. Colored corn cultivars included purple (Purple), blue (Blue) and red (Red). Purple corn was procured from a specialty foods vendor (Angelina's Gourmet, Swanson, CT). Jerry Peterson Blue Organic corn was purchased from Johnny's Selected Seeds (Fairfield, ME). Red corn was purchased from an online vendor (Amazon.com, Inc., Seattle, WA). Corn was cleaned using a 12/64" (4.8 mm) sieve for removal of broken corn and foreign

material (BCFM). Coproduct moisture contents were measured using an air oven at 135 °C for 2 h (Approved Method 44-15A, Association of Cereal Chemists International, 2010).

Kernel physical properties were determined using standard procedures. Test weight (TW) was measured using standard apparatus (Approved Method 55-10, Association of Cereal Chemists International, 2010) and was expressed in kilograms per hectoliter (kg/hL). Absolute density (AD) was determined using the ethanol column test (Hill et al., 1990). Thousand-kernel weight (TKW) was measured according to a method reported by Groos et al. (2003). All physical properties were measured in triplicate.

Compositional analyses of the corn and coproducts were done at a commercial analytical laboratory (Illinois Crop Improvement Association, Champaign, IL). Analyses included crude protein (Method 990.03, Association of Analytical Chemists, 2003), crude fat (Method 920.39, Association of Analytical Chemists, 2003) and neutral detergent fiber content (Van Soest et al., 1991). Starch contents of corn were measured using an acid hydrolysis method described by Vidal et al. (2009). All analyses were done in duplicate.

2.2. Moisture content determination and tempering

Corn sample moisture content was ascertained using an electronic moisture tester (GAC II, Dickey-John, Auburn, IL). The amount of water required to increase the kernel moisture content to 23.5% (wet basis) was calculated. Corn samples (100 g) and requisite amount of tap water at room temperature were added to 4 L cylindrical plastic bottles and sealed with caps. Corn samples were tempered by rotating the bottle continuously at 0.5 rpm for 30 min at room temperature until water was fully absorbed by corn kernels.

2.3. Preliminary 100 g study

Initial 100 g experiments were conducted with a hard endosperm cultivar (Hard1) using 1 kg protocol (Rausch et al., 2009). Corn samples (100 g) were mixed with water and tempered in plastic bottles for 20 min. Post tempering, the material was passed through a horizontal drum degerminator and conditioned in 49 °C oven for 2 h. After conditioning; sieving, roller milling and aspiration steps were used to separate large grits, small grits, fines, pericarp and germ fractions. Further experiments were conducted by reducing the post-temper drying times to 1 h and 30 min.

2.4. Modification study for 100 g dry milling protocol

Based on the insights gained from preliminary 100 g experiments using the 1 kg protocol, it was realized that tempering duration and post-temper drying time were the most important parameters which need to be adjusted. As a result, a 3 × 3 factorial design experiment was planned with three tempering times (20, 30 and 40 min) and three post-temper drying times (30 min, 1 h and 2 h). While tempering hard endosperm cultivars, 20 min tempering time was not sufficient and unabsorbed water remained in the tempering vessel. There was also a need to identify ideal post-temper drying time for 100 g samples. All experiments were done in triplicate using Hard1 cultivar of hard endosperm corn.

2.5. The 100 g dry milling protocol

After tempering to increase the kernel moisture content to 23.5%, corn was fractionated using a lab scale horizontal drum degerminator (15.2 cm diameter concentric roll, 1275 rpm and 375 W motor). Degermination process involved breaking of corn kernels by applying a shear force, so as to free endosperm fractions from germ, which largely remain intact due to relatively higher levels of hydration. Fractionated corn was conditioned in a convection oven at 49 °C for 1 h. Conditioned

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