Effect of Alternative Milling Techniques on the Yield and Composition of Corn Germ Oil and Corn Fiber Oil

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ABSTRACT

The effects of alternative corn wet-milling (intermittent milling and dynamic steeping (IMDS), gaseous SO_2 and alkali wet-milling) and dry grind ethanol (quick germ and quick fiber with chemicals) production technologies were evaluated on the yield and phytosterol composition (ferulate phytosterol esters, free phytosterols, and fatty acyl phytosterol esters) of corn germ and fiber oil and compared with the conventional wet-milling process. Small but statistically significant effects were observed on the yield and composition of corn germ and fiber oil with these alternative milling technologies. The results showed that the germ and fiber fractions from two of the alternative wet-milling technologies (the gaseous SO_2 and the IMDS) had, for almost all of the individual phytosterol compounds, either comparable or signficantly higher yields compared with the conventional wet-milling process. Also, both of the modified dry grind ethanol processes (the quick germ and quick fiber) with chemicals (SO_2 and lactic acid) can be used as a new source of corn germ and fiber and can produce oils with high yields of phytosterols. The alkali wet-milling process showed significantly lower yields of phytosterols compounds in germ but showed significantly higher yield of free phytosterols, fatty acyl phytosterol esters and total phytosterols in the fiber fraction.

Corn oil (obtained from corn germ) and corn fiber oil (obtained from corn fiber) are two valuable coproducts that are, or can be, produced in corn processing facilities. Corn oil is sold for $0.55 to $0.66/kg ($0.25 to $0.30 /lb) (wholesale price) and is mainly used as cooking oil and in or for other food applications. Commercial corn fiber oil-based products potentially can be sold for $17.6 to $22.0/kg ($8 to $10/lb) (based on the current market retail price of products similar to corn fiber oil). Corn fiber oil contains unique compounds; ferulate phytosterol esters (FPE), free phytosterols (St) and fatty acyl phytosterol esters (St:E) (Moreau et al 1996) and corn fiber oil can lower serum cholesterol levels in blood in laboratory animals (Moreau et al 1998) and, therefore, it has a nutraceutical value. The most abundant phytosterol in the FPE fraction in corn fiber oil is sitostanol, a fully-saturated stanol (Cater and Grundy 1998), which is considered to be more efficacious in cholesterol lowering ability than unsaturated phytosterols. However, recent clinical studies have shown that soy (unsaturated) phytosterol fatty acyl esters may be as efficacious as phytostanol fatty acyl esters in lowering serum cholesterol levels (Westrate and Meijer 1998). There are also reports that free phytosterols, when properly formulated, can lower serum cholesterol levels. Crude corn germ oil has a negligible amount of ferulate phytosterol esters (FPE) but has a significant amount of fatty acyl phytosterol esters (St:E) and free phytosterols (St) before refining. Moreau et al (1999b) showed that of the total FPE present in corn kernels, ≈90% is present in the fiber fraction and ≈3% in germ; of the total St present in corn kernels, ≈20% is present in the fiber fraction and ≈60% in the germ fraction; and of the total St:E present in corn kernels, 50% is in the fiber fraction and ≈40% is in the germ fraction.

Processing conditions have an effect on the yield and composition of oil from the fiber fractions of different grains. The conventional corn wet-milling process, with and without chemicals (SO_2 and lactic acid), have an effect on the ferulate phytosterol ester composition in the corn fiber oil (Singh et al 1999). Heat treatment (drying of fiber at high temperature before extraction) increases (≈10-fold) the yield of γ-tocopherol in corn fiber oil (Moreau et al 1999a) and increase the levels of tocotrienols, oryzanol, and other compounds in rice bran oil (Lane et al 1997).

The recent development of alternative corn wet-milling techniques such as the intermittent milling and dynamic steeping process (IMDS) (Lopes-Filho et al 1997), gaseous SO_2 (Eckhoff and Tso 1991; McKinney 1996), and the alkali wet-milling process (Du et al 1999; Eckhoff et al 1999) offer substantial technological improvements over the conventional wet-milling process and have potential for savings in energy and capital costs. Similarly, recent modifications of the dry grind ethanol process, such as the quick germ (Singh and Eckhoff 1996, 1997) and quick fiber processes (Singh et al 1999) for dry grind ethanol offer germ and fiber as new coproducts which can help reduce the cost of ethanol production. The germ and fiber from the dry grind ethanol industry also provide new sources of feedstock for recovery of cholesterol-lowering phytosterol compounds.

These alternative wet-milling and dry grind production technologies involve different chemical and milling parameters compared with conventional techniques and, therefore, might have an effect on the yield and composition of the nutraceutical compounds in the corn germ and fiber oil. In this study, the effects of alternative corn wet-milling and dry grind ethanol production technologies were evaluated on the yield and composition of corn fiber and germ oil and compared with the conventional wet-milling process which is currently the only bulk source of corn fiber for the corn processing industry.

MATERIALS AND METHODS

A yellow dent corn hybrid (3394) grown during the 1998 crop season at the Agricultural Engineering Farm, University of Illinois at Urbana-Champaign, was field dried to 14–16% moisture content and combine-harvested. Corn samples were hand-cleaned to remove broken corn and foreign material, packaged in plastic bags, and stored at 4°C until wet-milling. The whole kernel moisture content of the samples was measured using the 103°C convection oven method (AACC 2000).

Conventional wet-milling and gaseous SO_2 milling was done using the 100-g laboratory corn wet-milling procedure as outlined by Eckhoff et al (1996) and McKinney (1996), respectively, and both fine (cellular) and coarse fiber fractions (pericarp) were recovered separately. In the gaseous SO_2 procedure, the target flow

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rate of SO_2 to fill the sample bag was 597 mL/min for 2.5 min and the steep time was 14 hr. The IMDS procedure was done on 1-kg corn samples as described by Lopes-Filho et al (1997). The alkali wet-milling procedure used 100-g samples and followed Eckhoff et al (1999) through cracking and steeping and then the corn was milled according to Eckhoff et al (1996) using the steepwater as process water. The quick germ process was performed using the procedure outlined by Singh and Eckhoff (1996) and the quick fiber process was done as described in Singh et al (1999). Based on the poor yields of phytosterol compounds in the quick fiber process steeped without chemicals (Singh et al 1999), in this study the quick germ and the quick fiber processes were steeped with chemicals (0.2% SO_2 and 0.55% lactic acid). The collected germ and fiber samples were dried for moisture content determination using the two-stage, convection oven procedure (AACC 2000).

Dried fiber samples were ground to 20 mesh in a Wiley mill. Dried germ (1–2 g) and ground fiber samples (2–4 g) were placed in screw-top vials (55 mL) and 40 mL of hexane was added. The dried germ was homogenized using the Brinkman Polytron homogenizer. The tubes were shaken horizontally for 1 hr in a wrist-action shaker at room temperature. After extraction, the hexane extracts were filtered through a Whatman GF/A glass fiber filter fitted in a Buchner funnel with gentle vacuum. A part of the sample was removed for HPLC analysis as previously outlined by Moreau et al (1996). The rest of the solvent sample was dried under nitrogen and heat using an N-EVAP analytical evaporator (Organomation Associates Inc., Berlin, MA). The dried sample was transferred to 7.7-g vials in an 85:15 chloroform-methanol mixture and dried again under nitrogen to measure oil dry weight. The fiber yields, fiber oil recoveries, and yield of the three phytosterol compounds (FPE, St, and St:E) were compared with the fiber yield, fiber oil recovery, and yield of the three phytosterol compounds from the control sample (sample steeped with sodium meta-bisulfite and lactic acid).

Analysis of variance (ANOVA) and Duncan's multiple range test (SAS Institute, Cary, NC) were used for corn fiber oil and phytosterol compound yields. The level selected to show statistical significance was 5% ($P < 0.05$).

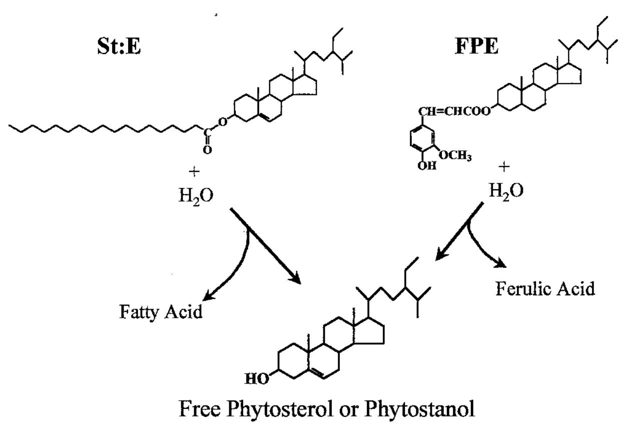

Fig. 1. Alkaline hydrolysis of phytosterol esters. St:E = fatty acyl phytosterol esters; FPE = ferulate phytosterol esters.

RESULTS AND DISCUSSION

Germ Yields and Germ Oil Composition

A germ yield of ≈5.0% with the conventional wet-milling process was comparable to the germ yield reported by Eckhoff et al (1996). Significant differences in germ yield and the phytosterol composition in the germ oil were observed with different alternative milling technologies. The germ yield for the gaseous SO_2 process was comparable to that of conventional wet-milling and the germ yield for the IMDS process was slightly higher (14.7%) than that of the conventional wet-milling process (Table I). The germ yield for the alkali wet-milling process was lower (by 35.7%) and higher (by 34.1%) for the quick germ processthan for the conventional wet-milling process. Lower germ yield in the alkali wet-milling process was probably due to the broken germ that was observed in the 100-g procedure which was difficult to recover. Higher germ yields from the quick germ process are due to some of the fiber fraction that comes along when the germ is recovered in the quick germ process (Singh and Eckhoff 1996).

Germ oil yields for the alkali wet-milling and the IMDS process were significantly lower compared with the conventional wet-milling process. Lower oil yields for the alternative corn milling processes were due to the shorter steep times in the alkali wet-milling (steep time of 1 hr) and IMDS (steep time of 5 hr) process compared with the conventional wet-milling process (steep time of 24 hr). Longer steep times allow the soluble proteins to leach out of the germ into the steepwater and therefore increase the concentration of the oil in the germ fraction (Watson 1984), which leads to higher extraction efficiency. Higher oil content with the gaseous SO_2 process and the quick germ process compared with the alkali wet-milling and the IMDS process were probably due to the longer steep time of 12–14 hr. The lack of diffusion of soluble protein in the steepwater after 12–14 hr of steep time would explain the comparable oil yield from the gaseous SO_2 process and the quick germ process to the conventional wet-milling process.

TABLE II
Percent Weight of Ferulate Phytosterol Esters (FPE), Free Phytosterols (St), and Fatty Acyl Phytosterol Esters (St:E) in Germ Oil and Total Phytosterol Compounds from Germ for Different Corn Processing Technologies

Corn Processing Technology	%wt of Oil FPE	St	St:E	Total
Conventional wet-milling	0.01 ± 0.00[a]	0.54 ± 0.02	0.44 ± 0.00	0.99
Gaseous SO_2 process	0.01 ± 0.00	0.60 ± 0.02	0.56 ± 0.05	1.16
Alkali wet-milling	0.01 ± 0.00	0.62 ± 0.01	0.43 ± 0.02	1.05
IMDS process[b]	0.02 ± 0.00	0.59 ± 0.02	0.48 ± 0.02	1.09
Quick germ process w/chem	0.01 ± 0.00	0.62 ± 0.00	0.54 ± 0.01	1.17

[a] Averages of two values ± standard deviation.
[b] Intermittent milling and dynamic steeping.

TABLE I
Yield of Germ, Germ Oil, and Ferulate Phytosterol Esters (FPE), Free Phytosterols (St), and Fatty Acyl Phytosterol Esters (St:E) Obtained from Different Corn Processing Technologies[a]

Corn Processing Technology	Germ Yield (%)	Oil Yield (%)	FPE (mg/100 g of corn)	St (mg/100 g of corn)	St:E (mg/100 g of corn)	Total Phytosterol (mg /100 g of kernels)
Conventional wet-milling	5.01d[b]	44.94a	0.17c	12.21b	9.85c	22.22
Gaseous SO_2 process	5.36cd	43.04a	0.16c	13.79ab	12.88b	26.83
Alkali wet-milling	3.22e	36.49b	0.08d	7.27c	4.99d	12.35
IMDS process[c]	5.75c	35.12b	0.47a	11.82b	9.65c	21.95
Quick germ process w/chem	6.72b	41.77a	0.32b	17.29a	15.08a	32.70

[a] All yields are averages of two values.
[b] Values followed by the same letter in the same column are not significantly different ($P < 0.05$).
[c] Intermittent milling and dynamic steeping.

TABLE III
Yield of Fiber, Fiber Oil, and Ferulate Phytosterol Esters (FPE), Free Phytosterols (St), and Fatty Acyl Phytosterol Esters (St:E) Obtained from Different Corn Processing Technologies[a]

Corn Processing Technology	Fiber Yield (%)	Oil Yield (%)	FPE (mg/100 g of corn)	St (mg/100 g of corn)	St:E (mg/100 g of corn)	Total Phytosterol (mg /100 g of kernels)
Conventional wet-milling	13.36a[b]	2.09b	7.75ab	3.19c	10.55c	21.48
Gaseous SO_2 process	13.19a	1.98bc	7.39bc	3.21c	10.91c	21.52
Alkali wet-milling	11.44a	7.51a	3.45d	7.65a	15.40a	26.49
IMDS process[c]	11.85a	1.82bc	8.03ab	3.70b	7.14d	18.87
Quick fiber process w/chem	11.90a	1.50cd	8.60a	2.88d	12.81b	24.30

[a] All yields are averages of two values.
[b] Values followed by the same letter in the same column are not significantly different ($P < 0.05$).
[c] Intermittent milling and dynamic steeping.

TABLE IV
Percent Weight of Ferulate Phytosterol Esters (FPE), Free Phytosterols (St), and Fatty Acyl Phytosterol Esters (St:E) in Fiber Oil and Total Phytosterol Compounds from Fiber for Different Corn Processing Technologies

Corn Processing Technology	%wt of Oil			
	FPE	St	St:E	Total
Conventional wet-milling	5.70 ± 0.23[a]	2.29 ± 0.11	7.78 ± 0.63	15.78
Gaseous SO_2 process	5.89 ± 0.30	2.47 ± 0.09	8.77 ± 0.39	17.13
Alkali wet-milling	2.21 ± 0.12	7.44 ± 0.90	10.48 ± 0.01	20.12
IMDS process[b]	3.74 ± 0.15	1.72 ± 0.05	3.32 ± 0.06	8.77
Quick fiber process w/chem	5.00 ± 0.17	1.68 ± 0.09	7.44 ± 0.25	14.11

[a] Averages of two values ± standard deviation.
[b] Intermittent milling and dynamic steeping.

The FPE recovery (mg/100 g of corn) was significantly higher for the IMDS process compared with the conventional wet-milling process or the other alternative milling processes. Visually, the germ fraction from the IMDS process was very pure (negligible fiber contamination). Therefore, contribution from fiber for FPE can be ruled out. Higher FPE recoveries with the IMDS germ samples is probably due to the extensive action of the steep chemicals on the milled corn fraction. In the IMDS process, milled corn (coarsely ground) is steeped with the conventional steep chemicals, unlike the conventional wet-milling process where whole corn is steeped. The FPE recoveries with the quick germ process was 88% higher compared with the conventional wet-milling process. As explained earlier, this is probably due to some contamination of fiber in the quick germ samples and the fiber's contribution to the FPE recovery. Low FPE recovery from the alkali wet-milled germ is probably due to the alkaline hydrolysis of the ferulate phytosterol esters to free phytosterols (Fig. 1) and loss of unrecoverable germ (broken germ) to the fiber fraction.

The recovery of St from germ (mg/100 g of corn) for the quick germ process was 42% higher compared with the conventional wet-milling process (Table I). All of the other alternative milling processes, except the alkali wet-milling process, were comparable to the conventional process for St recoveries. It is difficult to speculate why higher St recovery was obtained with the quick germ process compared with the conventional wet-milling process. The significant difference between the two processes is only in the steep time (24 hr for conventional wet-milling process and 12 hr for the quick germ process). Solubilization of nonpolar FPE in steepwater is probably negligible. Low St recovery with the alkali wet-milling process compared with the conventional wet-milling process was probably due to the loss of germ to the fiber fraction. The same trend as observed for St recovery was observed for the St:E recovery for all of the milling processes, except for the gaseous SO_2 process, for which the St:E recovery was significantly higher than in the conventional wet-milling process.

Of the total phytosterol compounds recovered from germ in the conventional wet-milling process, ≈55% came from the St fraction, ≈44% from the St:E fraction, and the remainder from the FPE fraction. The biggest contribution to the total phytosterol composition in the germ oil by the alternative milling processes was also made by the St fraction, followed by the St:E fraction, and the rest by the FPE fraction. The total phytosterol compounds from germ in mg/100 g of corn was highest for the quick germ process , followed by comparable amounts from the conventional, gaseous SO_2, and IMDS process. The lowest amount of the total phytosterol compounds came from the alkali wet-milling process (probably due to the lost germ fraction and ester sponification). The total percent weight of phytosterol compounds in the germ oil was ≈1.0% for all of the milling processes (Table II).

Fiber Yields and Fiber Oil Composition

Fiber yield for the conventional wet-milling process was comparable to the fiber yield reported by Eckhoff et al (1996). No significant differences in the fiber yield between the conventional and the alternative corn milling processes were observed. The fiber yield for the alkali wet-milling process also included the unrecovered germ fraction. Therefore, the actual fiber yield for the alkali wet-milling process was significantly lower compared with the conventional wet-milling process. Significant differences in the fiber oil yield and phytosterol composition were observed in the fiber oil with different milling technologies (Table III). Fiber oil yield for the gaseous SO_2 and the IMDS process were comparable to the conventional corn wet-milling process. Compared with the conventional wet-milling process, fiber oil yields for the quick fiber process was 28.2% lower. Low recovery of the fiber oil from the quick fiber samples compared with the conventionally wet-milled fiber samples was probably due to the shorter steep time of the quick fiber process. Unusually high (259.3% higher) fiber oil yield for the alkali wet-milling process compared with the conventional wet-milling process indicates the presence of unrecovered germ in the fiber fraction.

For FPE recovery from fiber in mg/100 g of corn, the gaseous SO_2 process, the IMDS process, and the quick fiber process had FPE recoveries comparable to the conventional wet-milling process. Significantly reduced (by 55.5%) FPE recovery from the fiber from the alkali wet-milling process compared with the conventional wet-milling process was due to alkaline hydrolysis of the ferulate phytosterol esters (FPE). Alkali hydrolyzes the ester bonds of the FPE releasing free phytosterols (St) (Fig. 1).

The St recovery from fiber from the alkali wet-milling process increased significantly (by 140%) compared with the conventional wet-milling process. This was largely because of the contamination of germ (unrecoverable germ) in the fiber fraction, and as explained above, due to the alkaline hydrolysis of FPE into St. Free phytosterol (St) recovery with the gaseous SO_2 process was comparable and St recovery with the IMDS process increased significantly (by 16%) compared with the conventional wet-milling process. Significantly lower recoveries of St were obtained from the quick fiber process compared with the conventional wet-milling process.

For St:E recovery from fiber, again, high recovery with the alkali wet-milling process compared with the conventional process was due to the contribution of the broken germ in the fiber fraction. Significantly higher recovery of St:E from the quick fiber process compared with the conventional and other alternative milling processes was observed. Significantly lower recoveries of St:E were observed for the IMDS process.

Of the total phytosterol compounds recovered from the fiber in a conventional wet-milling process, ≈49.0% came from the St:E fraction, ≈36.0% came from the FPE fraction, and the remainder came from the St fraction. The biggest contribution to the total phytosterol composition in the fiber oil by all of the alternative milling process was made by St:E, followed by FPE, and the rest by St, except for the alkali wet-milling process in which St was higher than FPE, and the IMDS process in which FPE was slightly higher than St:E. The total percent weight of the phytosterol compounds in the fiber oil for the conventional wet-milling process was ≈15.8%, and for the alternative milling processes ranged from 8.8% (for the IMDS process) to 20.1% (for the alkali wet-milling process) (Table IV).

The gaseous SO_2 process, the IMDS process, and the quick germ and quick fiber process (modified dry grind ethanol process) for both germ and fiber fractions performed either comparably or significantly higher in individual phytosterol recoveries (based on corn weight) compared with the conventional wet-milling process, except for the St:E recovery from fiber in the IMDS process and the St recovery from fiber in the quick germ process.

CONCLUSIONS

Overall, alternative wet-milling technologies (gaseous SO_2, IMDS, and alkali wet-milling) and modified dry grind ethanol processes (quick germ and quick fiber) had a significant effect on the yield and composition of corn germ and fiber oil compared with the conventional wet-milling process. The levels of almost all individual phytosterols in fractions from the gaseous SO_2 and the IMDS process were comparable to or better than the conventional wet-milling process. The alkali wet-milling process negatively affected the recovery of phytosterol compounds from germ. The modified dry grind ethanol processes, quick germ, and the quick fiber, were either comparable or significantly better compared with the conventional wet-milling process in phytosterol recovery.

The results suggest that the germ and fiber fraction from two of the alternative wet-milling technologies (gaseous SO_2 and IMDS) can be used for the recovery of corn germ and fiber oil without any negative effects on the yields of phytosterol compounds. Also, both of the modified dry grind ethanol processes (quick germ and quick fiber) can be used as a new source of germ and fiber and the oil extracted from them will have yields of phytosterols.

This study also suggests that there are significant levels of phytosterol compounds in the crude corn germ oil. However, currently during the refining process these phytosterols are significantly reduced. In view of the recently recognized health benefits of phytosterols, it would seem appropriate to alter refining technologies to preserve the natural phytosterols and derivatives to produce a more "healthy" oil. Phytosterol enriched, refined corn germ oil, prepared from the crude oils noted here could potentially sell for a premium price and bring more revenue back to corn refiners and ethanol producers.

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