Factors Affecting Oil Extraction/Water Adsorption in Sequential Extraction Processing of Corn

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ABSTRACT: The sequential extraction process (SEP) uses ethanol to extract oil and protein from cracked, flaked, and dried corn, and the dried corn simultaneously dehydrates the ethanol. Value-added co-products are possible, potentially making production of fuel ethanol more economical. The effects of solvent-to-corn (S/C) ratio, corn moisture content (MC), and number of extraction stages on ethanol drying, oil recovery, and protein loss during the simultaneous oil extraction/water adsorption step of SEP were evaluated. Extractions were carried out by using both aqueous ethanol and ethanol/hexane blends at 56°C. The S/C ratios tested were 3:1, 2:1 (control), 1.5:1, and 1:1 (w/w). More anhydrous ethanol, greater oil yields, and less co-extracted protein were obtained with higher S/C ratios. Less anhydrous ethanol and lower moisture adsorption capacities were obtained when the corn MC was ≥1.12%. Oil yields gradually decreased with drier corn, whereas protein loss increased when corn MC was <1.12%. Reducing the number of extraction stages from seven (original SEP) to five did not affect ethanol drying capability, oil yields, and protein co-extracted with oil. Using ethanol/hexane blends resulted in more anhydrous ethanol, higher oil yields, and less protein co-extracted with oil.

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KEY WORDS: Corn, corn oil, corn protein, ethanol, ethanol drying, extraction, maize, sequential extraction.

Sequential extraction processing (SEP) of corn is a promising new approach to fractionating corn for ethanol production (1,2). SEP uses ethanol to extract oil and protein from cracked, flaked, and dried corn, and the dried corn simultaneously dehydrates the ethanol (Scheme 1). Value-added co-products such as corn oil and protein products are possible, potentially making fuel ethanol production more economical.

A critical element of SEP that helps reduce processing costs is the integration of oil extraction and ethanol drying into a single step that replaces alternative more expensive means of recovering anhydrous ethanol with simple water adsorption using dried, flaked corn as adsorbent. Corn (grits, starch, meal, residue) and other biomass materials have been used to adsorb water from ethanol/water mixtures (85–90% alcohol) after partial distillation of fermentation beer to produce anhydrous ethanol (3–5). Robertson *et al.* (6) reported that improved energy efficiency could be achieved when another process is consolidated with water adsorption from ethanol, as is the case with intensive adsorption/fermentation (corn used to dehydrate ethanol later became the feedstock for fermentation) and simultaneous extraction of oil (7,8).

SEP recovers more than 90% of the oil from flaked corn and produces 99% ethanol (1). The oil extraction capability of SEP is obviously impressive, but the ethanol-drying step must be improved (i.e., an alcohol moisture content of ≤0.5%) before industry will consider SEP as a practical means of producing fuel ethanol. Ethanol-drying efficiency is increased by keeping the driving force (water activity differences between ethanol and corn) high, which is achieved by reducing the solvent-to-corn (S/C) ratio. Solvent evaporation costs also are reduced by using the minimum amount of solvent needed to recover adequate amounts of oil (leaving about 0.5% residual oil). Moisture-adsorbing capacity of the flaked corn could be increased by drying the corn to lower moisture contents, but only to the extent that energy costs are kept reasonable. This study evaluated the effects of S/C ratio, corn moisture content (MC), and number of extraction stages on ethanol drying, oil recovery, and protein loss during the simultaneous oil extraction/water adsorption step of SEP.

MATERIALS AND METHODS

Influence of S/C ratio. Batches (350 g) of soft dent corn (Pioneer 3394; Pioneer Hi-Bred International, Inc., Johnston, IA) were cracked and then flaked to 0.5-mm (0.02 in.) thickness by using a Roskamp rollermill (Model K; Roskamp Mfg., Inc., Waterloo, IA). The flakes were dried at 55°C in a forced-air convection oven to 1.12% (weight basis, wb) moisture for use in single-stage batch extractions. Each batch was individually sealed in a polyethylene bag and placed in a desiccator at ambient temperature until used. The moisture content of each corn batch was determined by Karl Fischer titration (9).

The S/C ratios evaluated were 3:1, 2:1 (control based on prior work), 1.5:1, and 1:1 (w/w). Aqueous ethanol and 70% ethanol/30% hexanes were the extraction solvents. Extractions were carried out using the batch system described by Miller *et al.* (10) with modifications. The long, narrow column (4 cm i.d. × 63 cm length, L/D ratio of about 15) was used as the vessel for flaked corn. The solvent percolated through the bed of flaked corn at 25 mL solvent/min flow rate,

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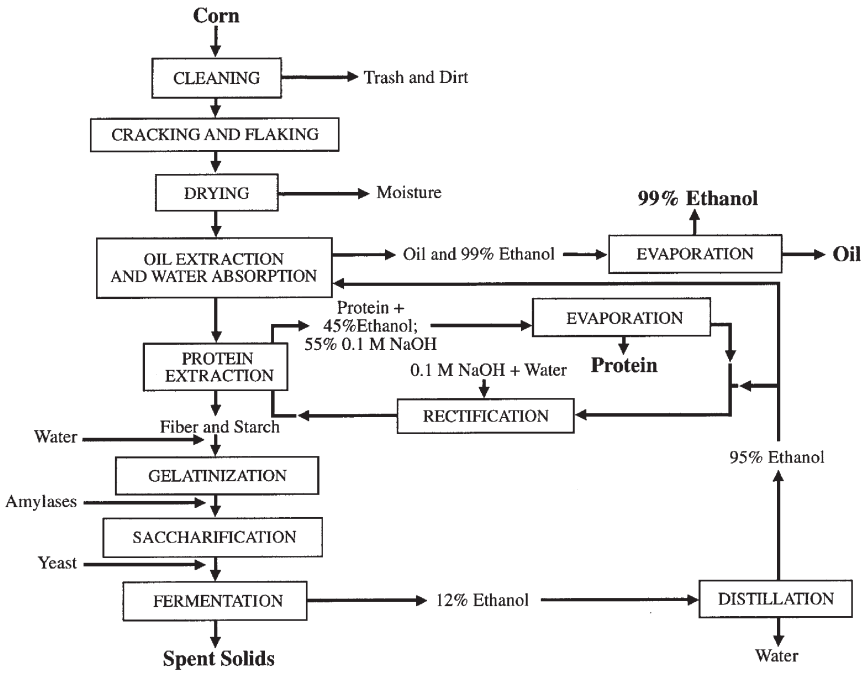

SCHEME 1

and the final solvent wash drained for 15 min before collecting miscella (extract) samples for analyses. Triplicate extractions were done for each S/C ratio.

The moisture contents of miscellas, marcs (solvent-laden, defatted corn), and air-dried defatted flakes were determined by Karl Fischer titration (9). To determine miscella oil content, a known weight of miscella was placed into a micro-Kjeldahl flask. The ethanol was evaporated by using a rotary evaporator. The mixture of oil and any solids was washed three times with 20-mL aliquots of hexanes. The hexane wash was removed by pipette to a preweighed beaker and then allowed to evaporate in a fume hood. The amount of oil in the beaker was weighed to calculate oil content. The solids remaining in the flask after hexane washing were analyzed for crude protein content by using AACC method 46–08 (11). The residual oil and crude protein contents of the air-dried defatted flakes were determined by using standard AACC methods 30-20 and 46-08, respectively (11).

Influence of corn moisture content. Soft dent corn was prepared as described in the previous section, except for the drying step where the flaked corn was dried at 55°C in a forced-air convection oven to the desired moisture content [2.5, 1.75, 1.12 (control), 0.75, 0.50%]. The mean value of three batches of flaked corn was used for each starting moisture content. The solvents and conditions for extraction and chemical analyses were similar to those described in the preceding section. The results from the previous experiments determined the S/C ratio to be used for this part of the study.

Influence of number of extraction stages. Soft dent corn was cracked, flaked, and dried to a moisture content that was determined by the results of experiments described in the preceding section. The full countercurrent system as described by Hojilla-Evangelista *et al.* (1,2) was used for this part of the study. Modifications, such as using the long, narrow corn vessel, and single-pass solvent percolation through the flake bed, were incorporated, following the improvements of Miller *et al.* (10). Extractions were carried out using aqueous ethanol in a system that used seven (control) and five stages following the procedure described by Hojilla-Evangelista *et al.* (1,2). The reduced number of extraction stages was based on data that revealed that a substantial amount of oil was extracted in the first two extraction stages, and that a significant amount of water also was adsorbed from ethanol during the same period of time (10). After steady state was achieved (usually after 15 batches of corn), miscellas, marcs, and air-dried defatted flakes were analyzed for moisture, crude free fat/residual oil, and crude protein contents by using standard methods described in the preceding sections.

Statistical analyses. Statistical analysis was performed by using the SAS7 Systems for Windows software (SAS Institute Inc., Cary, NC). Multiple ANOVA and Duncan's Multiple Range tests were performed on all data to determine differences among the treatments.

RESULTS AND DISCUSSION

Influence of S/C ratio. (i) Ethanol drying. As with previous SEP studies, the moisture content of the starting solvent was

TABLE 1
Effects of Solvent-to-Corn Ratio on Ethanol Drying During SEP[a,b]

	Aqueous ethanol				70% Ethanol/30% hexane			
	1.0	1.5	2.0	3.0	1.0	1.5	2.0	3.0
Starting solvent MC, %	7.3 ± .0	5.1 ± .0	4.0 ± .0	2.9 ± .0	4.6 ± .0	3.2 ± .0	2.3 ± .0	1.7 ± .0
Recovered miscella MC[c], %	3.0 ± .2^{a,b}	3.3 ± .4^{a}	3.0 ± .1^{a,b}	2.4 ± .2^{b}	1.1 ± .1^{c,d}	1.4 ± .4^{c}	0.5 ± .0^{e}	0.6 ± .0^{d,e}
Water removed, g/100 g solvent	4.3^{a}	1.8^{b}	1.1^{c}	0.5^{d}	3.5^{d}	1.8^{b}	1.8^{b}	1.1^{c}
Starting corn MC, %	1.2 ± .0	1.1 ± .1	1.1 ± .0	1.1 ± .0	1.1 ± .0	1.1 ± .0	1.1 ± .0	1.1 ± .1
Marc MC, %	5.0 ± .5^{a}	4.1 ± .1^{c}	3.5 ± .1^{d}	2.9 ± .1^{e}	4.6 ± .3^{b}	3.8 ± .0^{c,d}	3.6 ± .1^{d}	2.8 ± .1^{e}
Moisture adsorbed, g/100 g corn	3.8^{a}	3.0^{b}	2.4^{c}	1.8^{d}	3.4^{a,b}	2.7^{b,c}	2.5^{c}	1.7^{d}

[a]Abbreviations: SEP, sequential extraction processing; MC, moisture content.
[b]Values across columns followed by the same roman superscript letter are not significantly different ($P < 0.05$).
[c]Values were adjusted on oil-free, protein-free basis.

based on solvent hold-up in corn (40–60 g/100 g corn) and the amount of ethanol produced from one bushel of corn (35 g/100 g corn) (1,2); that is,

$$\text{starting solvent MC} = 100 - \frac{(W_s - \text{SH} - E) \times C_{\text{ah}} + (\text{SH} + E) \times C_{\text{aq}})}{\text{S/C ratio}} \quad [1]$$

where W_s = amount of starting solvent (g), SH = solvent hold-up (g), E = amount of ethanol produced from one bushel of corn (g), C_{ah} = concentration of anhydrous ethanol (0.992 weight basis, 99.5% volume basis), and C_{aq} = concentration of aqueous ethanol distilled from fermentation of corn starch (0.924 weight basis, 95% volume basis). SH and E remained constant whereas the amounts of incoming solvent changed when the S/C ratio was varied, resulting in different starting concentrations (or moisture contents) of incoming solvents (Table 1).

For all S/C ratios, the moisture contents of miscellas decreased after oil extraction, indicating the simultaneous drying of solvent (Table 1). With aqueous ethanol, drier miscellas were obtained with the 3:1 S/C ratio. This was attributed to the fact that there already was less moisture initially to adsorb from the solvent at this ratio than from the solvent at the 1:1 ratio. In addition, pumping the larger quantity of solvent for the 3:1 S/C ratio at the same flow rate as the solvents for the lower ratios allowed for longer contact time between flakes and solvent. There were no significant differences in the moisture contents of miscellas recovered from S/C ratios ≤2.

With 70% ethanol/30% hexanes, significant drying of ethanol occurred at S/C ratios ≥2. Miscellas recovered from the ethanol/hexanes blend were also considerably drier at all S/C ratios than were those from aqueous ethanol (Table 1), which was supported by Miller *et al.* (10), who reported that the ethanol/hexanes blend was more efficient in producing nearly anhydrous ethanol. Both extraction solvent and S/C ratio significantly affected the moisture content of the recovered miscella, with the solvent exerting far greater influence than the S/C ratio (F-values of 263 and 9, respectively).

The moisture content of flaked corn increased after oil extraction (Table 1), another indication of simultaneous drying of solvent. Moisture adsorption capacity increased when the S/C ratio was reduced. At the lower S/C ratio, the difference in water activities of ethanol and corn is large, corresponding to a high driving force and resulting in much higher moisture adsorption capacity. There were no significant differences in moisture adsorption capacities of flaked corn extracted with aqueous ethanol or with ethanol/hexanes within a given ratio (Table 1). As with miscella moisture contents, the effects of extraction solvent and S/C ratio on marc moisture contents were also statistically significant; however, S/C ratio had greater influence than did the solvent (F-values of 98 and 5, respectively).

(ii) Oil recovery. More oil was extracted by both aqueous ethanol and ethanol/hexanes when higher S/C ratios were

TABLE 2
Effects of Solvent-to-Corn Ratio on Oil Recovery During SEP[a]

	Aqueous ethanol				70% Ethanol/30% hexane			
	1.0	1.5	2.0	3.0	1.0	1.5	2.0	3.0
Starting corn oil content, % db	4.4 ± .1	4.4 ± .1	4.4 ± .1	4.4 ± .1	4.4 ± .1	4.4 ± .1	4.4 ± .1	4.4 ± .1
Residual oil in defatted corn, % db	1.7 ± .3^{a}	1.1 ± .1^{b}	0.7 ± .1^{c}	0.6 ± .1^{c,d}	0.8 ± .0^{c}	0.7 ± .1^{c}	0.4 ± .0^{d,e}	0.3 ± .0^{e}
Oil recovery, %	62 ± 7^{e}	74 ± 2^{d}	83 ± 2^{c}	85 ± 2^{b,c}	81 ± 1^{c}	84 ± 2^{c}	90 ± 1^{a,b}	93 ± 1^{a}

[a]Means across columns followed by the same roman superscript letter are not significantly different ($P < 0.05$). db, dry basis; for other abbreviation see Table 1.

TABLE 3
Effects of Solvent-to-Corn Ratio on Protein Extracted with Oil During SEP[a]

	Aqueous ethanol				70% Ethanol/30% hexane			
	1.0	1.5	2.0	3.0	1.0	1.5	2.0	3.0
Starting corn CP content, % db	8.9 ± .1	8.9 ± .1	8.9 ± .1	8.9 ± .1	8.9 ± .1	8.9 ± .1	8.9 ± .1	8.9 ± .1
Defatted corn CP content, % db	7.9 ± .5	8.6 ± .5	8.3 ± .1	8.1 ± .4	8.5 ± .3	8.2 ± .3	8.5 ± .5	8.8 ± .1
Protein loss, %	11 ± 5[a]	4 ± 5[a,b]	7 ± 1[a,b]	9 ± 5[a,b]	5 ± 3[a,b]	8 ± 3[a,b]	4 ± 6[a,b]	2 ± 1[b]

[a]Means across columns followed by the same roman superscript letter are not significantly different ($P < 0.05$). CP, crude protein. For other abbreviations see Tables 1 and 2.

used (Table 2). There was no significant difference between oil recoveries obtained at S/C ratios of 2 and 3, but there was a substantial reduction in the amount of oil extracted at S/C ratios of <2. Oil solubility in ethanol at elevated temperatures is enhanced as the solvent becomes more anhydrous (12), as was the case when S/C ratios were increased (Table 1). Ethanol/hexanes extracted significantly more oil than did aqueous ethanol (Table 2). This outcome was expected because aqueous ethanol extracts polar lipids whereas the blend of ethanol and hexanes is capable of extracting both polar and nonpolar lipids. The extraction solvent, S/C ratio, and the interaction of these two factors all had statistically significant effects on oil recovery (*F*-values were 88, 49, and 6, respectively).

(iii) Protein extracted with oil. In the current protocol for SEP, substantial amounts of zein are co-extracted during the oil extraction/water adsorption step. However, zein recovery and purification are difficult because of the presence of large quantities of oil. It would be preferable to shift zein extraction to downstream processes to simplify both the oil and protein recoveries. Therefore, in the oil extraction/water adsorption step, it is more desirable if little to no protein will be co-extracted.

There was no clear trend that described the effect of S/C ratios on protein extraction with oil. With aqueous ethanol, protein loss declined when S/C ratios were reduced from 3 to 1.5, but increased substantially with S/C of 1. The opposite result was observed for ethanol/hexanes (Table 3). Zein, specifically α-zein, is soluble in 95% ethanol (volume basis, or 92.4% by weight), and one would expect more zein to be extracted with oil as the alcohol approached this concentration. Ethanol/hexanes extracted less protein with oil than did aqueous ethanol (except in the case of 1.5 S/C ratio) (Table 3) because of less polarity. Even with the higher amounts of protein co-extracted with oil by using aqueous ethanol, protein loss was still markedly reduced, being less than half of that extracted in the original SEP (>22%).

As S/C ratio decreased, moisture adsorption capacity of the corn increased, but the recovered solvents had higher moisture contents. Oil recovery decreased and protein loss increased at the lower S/C ratios. No significant differences were detected between data obtained at S/C ratios of 3 and 2.

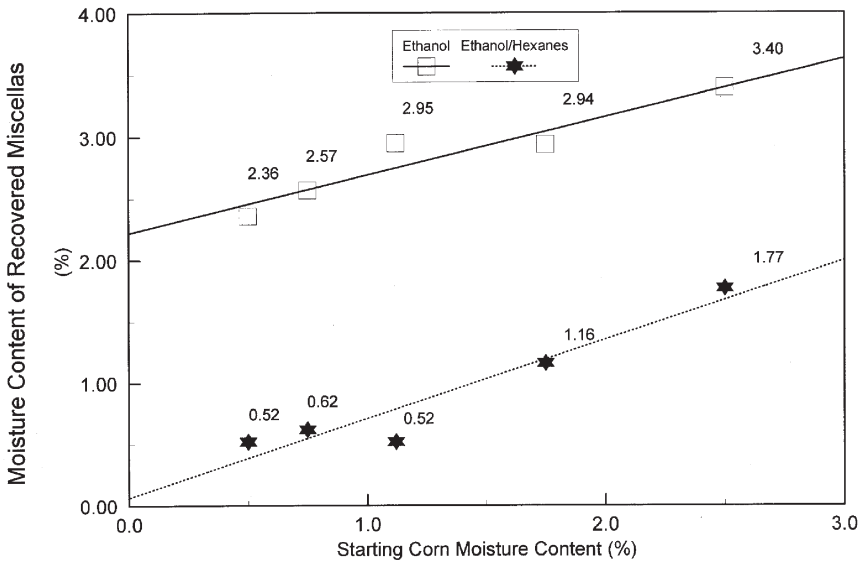

FIG. 1. Moisture contents of miscellas recovered from sequential extraction processing (SEP) of undegermed, flaked corn with varying starting moisture contents.

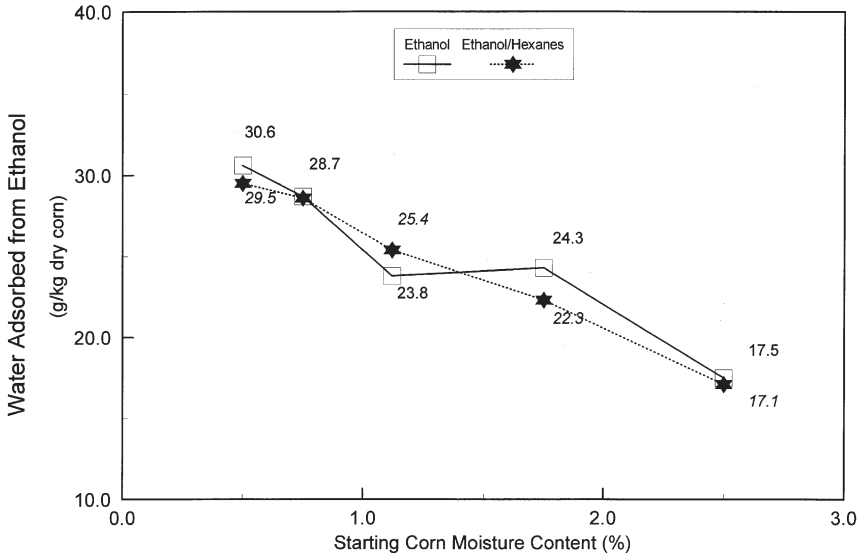

FIG. 2. Effect of starting corn moisture content on moisture adsorption capacities of flaked corn during SEP; see Figure 1 for abbreviation.

Based on these results, the original S/C ratio of 2 was retained.

Influence of corn moisture content. (i) Ethanol drying. Drier miscellas were recovered and higher moisture adsorption capacities were obtained when drier flaked corn was used for extraction (Scheme 1, Fig. 1). As water was removed from corn, more sites became available for adsorption of water from the solvent percolating through the flake bed. Miscella moisture contents were not significantly different at corn moisture contents ≤1.12%. More anhydrous miscellas (as low as 0.5% moisture) were reclaimed when ethanol/hexanes was the extracting solvent (Fig. 1). Moisture adsorption capacities of corn were similar for both solvents at the corn moisture contents evaluated (Fig. 2).

(ii) Oil recovery. Oil extraction apparently was unaffected by the extent of drying the flaked corn. Oil recoveries and miscella oil contents declined only gradually with decreasing corn moisture content (Figs. 3,4). Ethanol/hexanes extracted slightly more oil (5–8%) than did aqueous ethanol at all corn moisture levels.

(iii) Protein extraction. More protein was co-extracted with oil when drier flaked corn was used, with significant losses

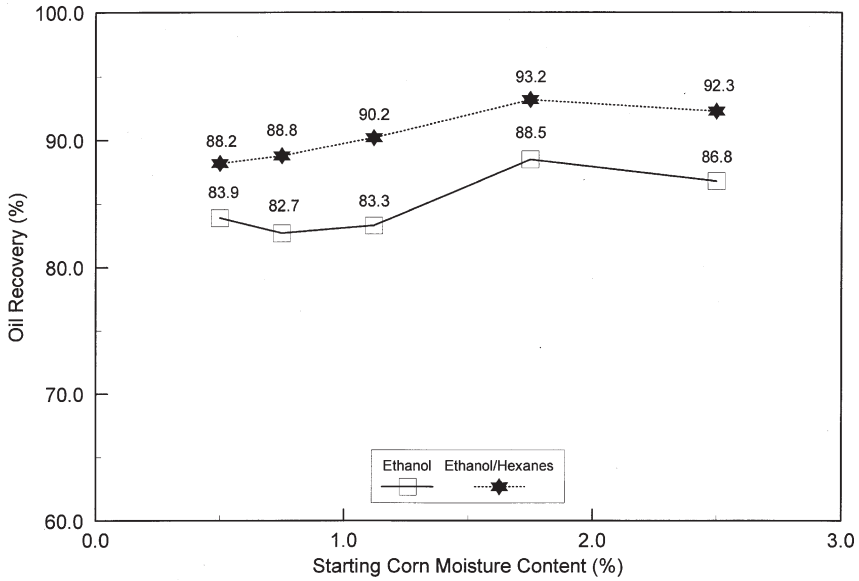

FIG. 3. Effect of starting corn moisture content on oil recovery during SEP; see Figure 1 for abbreviation.

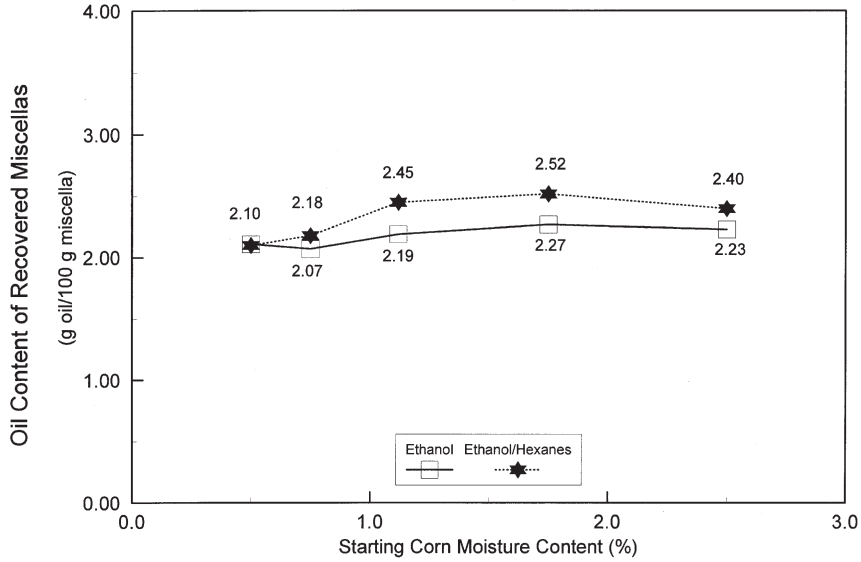

FIG. 4. Oil contents of miscellas recovered from SEP of undegermed, flaked corn with varying starting moisture contents; see Figure 1 for abbreviation.

observed at <1.12% moisture contents (Figs. 5,6). Drying the corn to <1% moisture may have created additional fissures in cells that allowed the protein to be more readily extracted. The less polar ethanol/hexanes blend extracted less protein than did aqueous ethanol (Figs. 5,6).

Drier miscellas were recovered and moisture adsorption capacities increased when drier flaked corn was used. However, protein co-extracted with oil increased significantly when the corn moisture content was <1.12%. Oil recovery was not affected by corn moisture content. Overall, there was no benefit to drying corn to <1.12%.

Influence of number of extraction stages. This part of the study used only aqueous ethanol to evaluate the effects of the number of extraction stages, because based on the results from the preceding sections, similar trends would have been obtained with 70% ethanol/30% hexanes. The system that used ethanol/hexanes also would produce significantly more anhydrous miscella and practically no protein co-extracted with oil, but oil recoveries between the two solvents would be similar.

(i) Ethanol drying. Slightly drier miscella (ethanol) was recovered from the system that used five extraction stages (Table 4), but the difference in miscella moisture contents

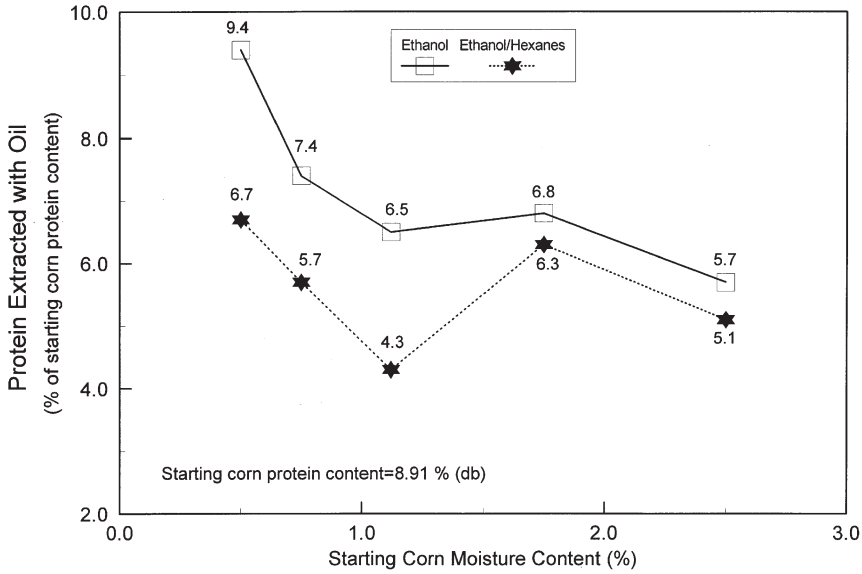

FIG. 5. Effect of starting corn moisture content on protein co-extraction with oil during SEP; see Figure 1 for abbreviation.

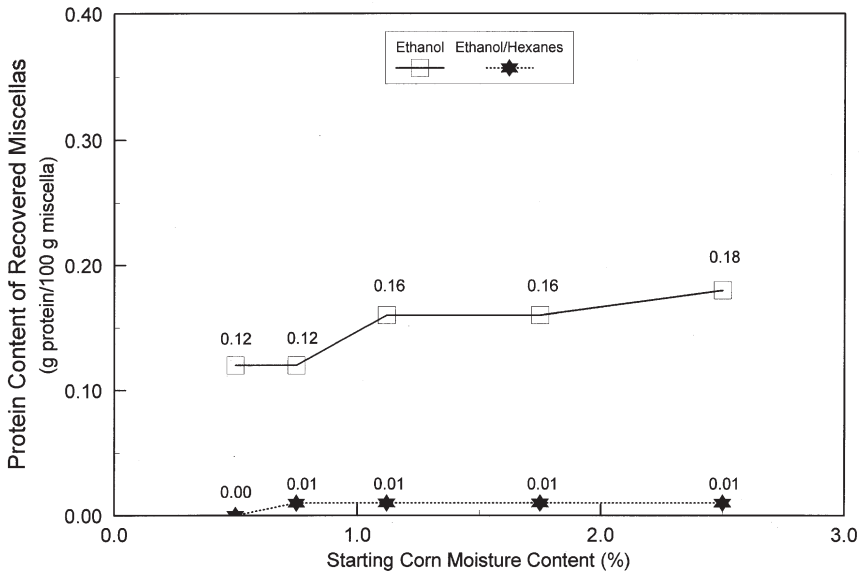

FIG. 6. Protein contents of miscellas recovered from SEP of undegermed, flaked corn with varying starting moisture contents; see Figure 1 for abbreviation.

from this system and the control (seven stages) was not significant. Similar moisture adsorption capacities were also obtained. In both systems, significant ethanol drying occurred in the first extraction stage where the oldest miscella came in contact with fresh, dry, flaked corn (Fig. 7). Little or no water adsorption appeared to take place in the subsequent extraction stages, indicating that the ability of corn to adsorb water from ethanol may have been exhausted after the first stage.

(ii) Oil recovery. Both systems extracted significant quantities of oil and had nearly identical oil recoveries of >95% (Table 4). The bulk of the oil (≅75%) was removed in the first extraction stage (Fig. 8), which used the driest miscella (Fig. 7). Oil solubility in ethanol is enhanced as the solvent becomes more anhydrous.

(iii) Protein extraction. Both extraction systems had substantially lower amounts of protein extracted with oil (>22%) when compared with the original SEP, even though protein loss with five extraction stages was twice as much as that of the control SEP system (seven stages) (Table 4). Most of the protein appeared to be extracted in the first two extraction stages (Fig. 9).

Reducing the number of extraction stages from seven in the original SEP to five did not adversely affect ethanol-drying capability, moisture adsorption capacity, or oil extraction efficiency. The amount of protein extracted with oil increased when five extraction stages were used during SEP, but this amount was still substantially less than that obtained from the original unmodified SEP system.

These results showed that greater S/C ratios and the use of drier flaked, defatted corn tended to produce more anhydrous recovered ethanol without adversely affecting oil yields, but they are also likely to increase the amount of co-extracted proteins, which would complicate the oil recovery process. The effects of these variables were not significant enough to warrant changing the S/C ratio and corn moisture content being used in the current SEP design (2 and 1.12%, respectively). The data also showed that it is possible to reduce the number of extraction stages from seven to five without adversely affecting ethanol-drying ability and oil yields or increasing protein co-extraction. Most important, this study revealed that the ethanol/hexanes blend was a far more efficient solvent for SEP than aqueous ethanol alone, producing nearly anhydrous recovered ethanol, high oil yields, and markedly less protein loss. Despite these benefits, caution should be taken when considering ethanol/hexanes as replacement for aqueous ethanol. The presence of hexanes will require additional complex

TABLE 4
Effects of Number of Extraction Stages on Ethanol Drying, Oil Recovery, and Protein Co-extracted with Oil During SEP[a]

	Number of extraction stages	
	5	7 (control)
Solvent: aqueous ethanol		
Fresh solvent MC, %	3.5 ± 0	3.7 ± 0
Recovered miscella MC[b], % (oil-free, protein-free basis)	2.2 ± 0.4	2.7 ± 0.1
Water removed, g/100 g solvent	1.3	1.0
Starting corn MC, %	1.1 ± 0	1.1 ± 0
Marc MC[b], %	3.4 ± 0.1	3.5 ± 0.1
Moisture adsorbed, g/100 g corn	2.2	2.4
Starting corn oil content, % db	4.4 ± 0.1	4.4 ± 0.1
Residual oil in defatted corn[b], % db	0.2 ± 0.1	0.1 ± 0
Oil recovery[b], %	96.1 ± 1.9	97.1 ± 0.6
Starting corn CP content, % db	8.9 ± 0.1	8.9 ± 0.1
Defatted corn CP content[b], % db	8.1 ± 0.5	8.5 ± 0.5
Protein loss[b], %	8.6 ± 5.6	5.4 ± 5.2

[a]For abbreviations see Tables 1–3.
[b]Mean values of 10 extractions at steady state.

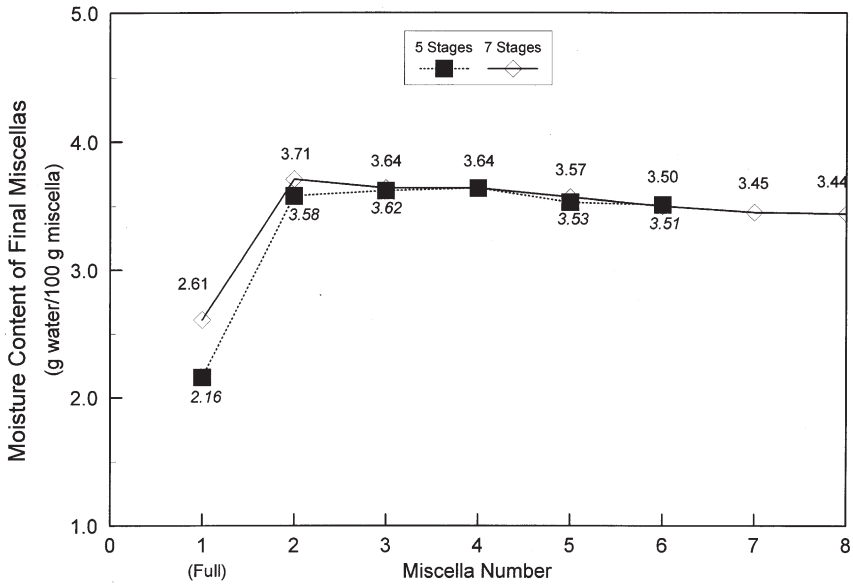

FIG. 7. Moisture contents of final miscellas from five-stage and seven-stage SEP of undegermed, flaked corn; see Figure 1 for abbreviation.

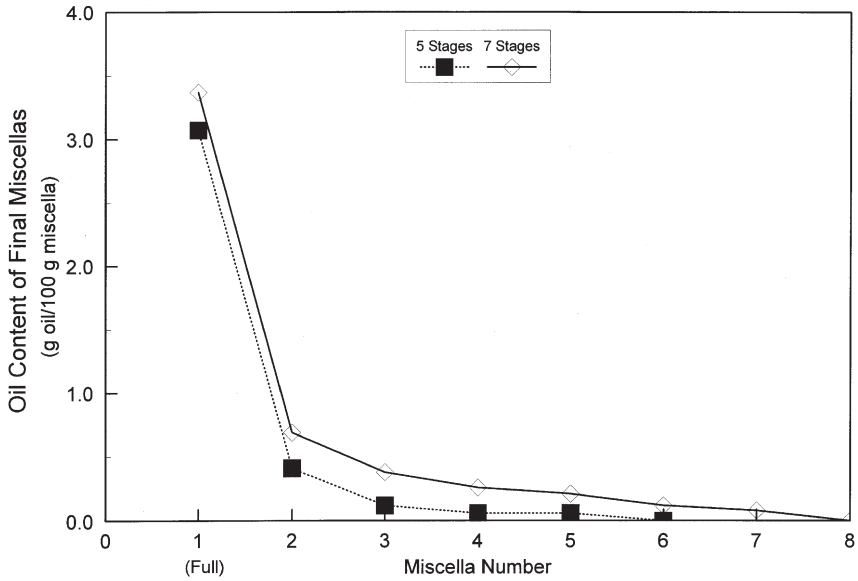

FIG. 8. Oil contents of final miscellas from five-stage and seven-stage SEP of undegermed, flaked corn; see Figure 1 for abbreviation.

separation and recovery methods in downstream operations, in addition to meeting and enforcing safety regulations that are otherwise not required if aqueous ethanol is the extracting solvent.

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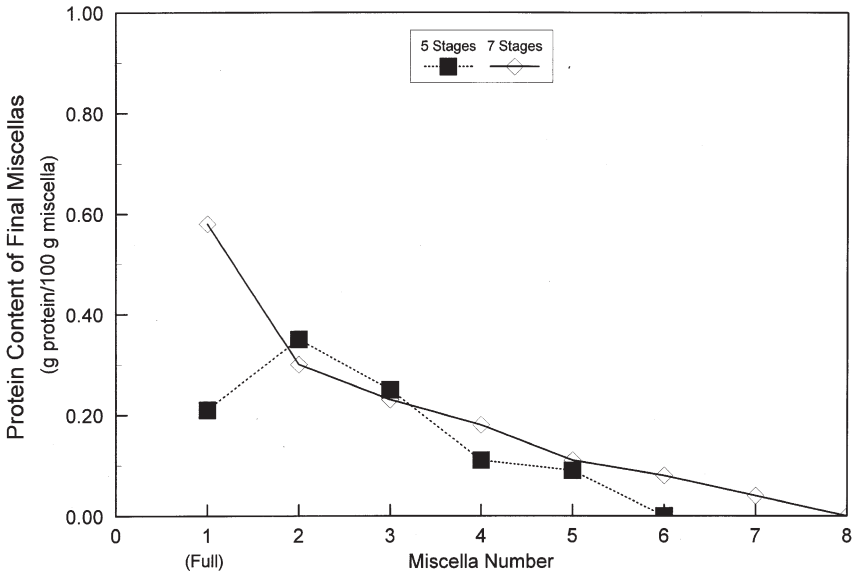

FIG. 9. Protein contents of final miscellas from five-stage and seven-stage SEP of undegermed, flaked corn; see Figure 1 for abbreviation.

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