0273-2289/01/94/0041/$12.25

Fermentation and Costs of Fuel Ethanol from Corn with Quick-Germ Process

Frank Taylor,*,[1] Andrew J. Mcaloon,[1] James C. Craig, Jr.,[1] Ping Yang,[2] Jenny Wahjudi,[2] and Steven R. Eckhoff[2]

[1]Eastern Regional Research Center, U.S. Department of Agriculture,† Agricultural Research Service, 600 East Mermaid Lane, Wyndmoor, PA 19038, E-mail: ftaylor@arserrc.gov; and [2]University of Illinois at Urbana-Champaign, 1304 W. Pennsylvania Avenue, Urbana, IL 61801

Received October 1, 2000; Revised February 1, 2001; Accepted February 1, 2001

Abstract

The Quick-Germ process developed at the University of Illinois at Urbana-Champaign is a way to obtain corn oil, but with lower capital costs than the traditional wet-milling process. Quick-Germ has the potential to increase the coproduct credits and profitability of the existing dry-grind fuel ethanol process, but the fermentability of the corn remaining after oil recovery has not been tested. Therefore, a series of pilot scale (50 L) fermentations was carefully controlled and monitored with unique methods for standard inoculation and automatic sampling. It was found that the concentration of suspended solids was significantly reduced in the Quick-Germ fermentations. When compared at the same concentration of fermentable sugars, the fermentation rate and yield were not statistically different from controls. When Quick-Germ was integrated into a state-of-the-art dry-grind fuel ethanol process, computer simulation and cost models indicated savings of approx $0.01/L of ethanol ($0.04/gal) with the Quick-Germ process. Additional savings associated with the lower suspended solids could not be quantified and were not included. However, the savings are sensitive to the price of corn oil.

Index Entries: Quick-Fiber; ethyl alcohol; cost estimation; cornstarch; oil; dry-grind process; wet milling; distiller's dried grains.

*Author to whom all correspondence and reprint requests should be addressed.

†Mention of brand or firm name does not constitute an endorsement by the U.S. Department of Agriculture over others of a similar nature not mentioned.

Introduction

Owing in large part to federal and state tax incentives, fuel ethanol production has amounted to approx 1% of the total US gasoline supply over the past 5 yr. Ethanol and methyl tertiary butyl ether (MTBE) may be used interchangeably to make clean-burning reformulated gasoline. Currently, about three-fourths of reformulated gasoline contains MTBE and one-fourth ethanol. However, contamination of groundwater by MTBE may lead to discontinuation of its use and possibly to significant expansion of the fuel ethanol industry in the near future. Currently, >90% of fuel ethanol in the United States is produced from corn either by the dry-grind fuel ethanol process or as an extension of traditional corn wet milling. The dry-grind process has recently gained a larger share of the market, in part owing to the lower capital investment for new plants. On the other hand, wet milling produces more valuable coproducts such as edible corn oil and gluten meal. To lower the cost of fuel ethanol further, hybrid processes having lower capital costs than traditional wet mills but yielding more valuable coproducts than currently produced in most dry-grind plants offer clear advantages. The need for new coproducts is accentuated by the falling price of distiller's dried grains with solubles (DDGS), the only coproduct of typical dry-grind ethanol plants, from $0.09 to $0.04/lb over the past 5 yr. Carbon dioxide and perhaps glycerol may be recovered in some dry-grind plants, but the markets for these products are limited, and new hybrid processes and products are needed.

One possibility is the Quick Germ process, a variation of wet milling under development at the University of Illinois at Urbana-Champaign (UIUC). The traditional wet-milling process begins by soaking the corn in water (steeping). This softens the corn kernels so that the components (germ, starch, fiber, and protein) can be separated. Starting with corn at the same moisture content (15%) as is normally supplied to the dry-grinding or wet-milling process, researchers at UIUC have shown that the germ containing the valuable oil can be recovered after a shorter steeping step than is practiced in wet milling *(1)*. Fermentation of Quick-Germ corn offers an attractive process alternative, but until now, fermentation of Quick-Germ corn has not been investigated. For example, removing the oil could create a foaming problem, because normally the oil in whole-corn fermentation suppresses foam. Also, removing some of the nonfermentables should make the fermentation process, and subsequent steps, more efficient, but the magnitude of this improvement has not been assessed.

Also under investigation at UIUC is Quick-Fiber, a process to recover corn fiber, consisting primarily of the outer covering of the corn kernel, called the hull, pericarp, or bran. Corn fiber can be processed into corn fiber gum, a substitute for imported gum arabic *(2)*, and is also a source of corn fiber oil, a dietary ingredient that has been shown to lower serum cholesterol levels in humans *(3)*. Removing both germ and hull will leave a fermentation feedstock that is even lower in nonfermentables, but again the actual fermentation of this material has not been studied. Therefore, the present study was undertaken to measure the rate and yield of fermentation of Quick-Germ corn, Quick-Fiber corn, and whole ground corn. The cost savings on integrating Quick-Germ into a computer model of a state-of-the-art dry-grind fuel ethanol process were estimated.

Materials and Methods

A series of eight preliminary 3-L fermentations was carried out to establish standard methods and procedures for the 50-L fermentations. Samples of Quick-Germ corn and controls were prepared at UIUC according to the 1-kg batch procedures previously described *(1)*. The 4-L batches were shipped overnight either frozen in dry ice or nonfrozen. Some of the nonfrozen samples but none of the frozen samples were degraded during shipment by the action of contaminating microorganisms. All subsequent runs for the 50-L fermentations were frozen before shipment.

Yellow dent corn of a single hybrid (Pioneer 3394) was stored in a 190-L (55-gal) sealed steel drum at ambient conditions. Corn was mixed in the drum prior to sampling, and random samples of 30 kg were removed and cleaned with a seed cleaner having a 4-mesh screen. Cleaned corn (25.5 kg each test, five replicates for three conditions) was weighed, stored in a plastic container, and equilibrated to room temperature before soaking. Three samples of corn of approx 20 g each were taken for determination of moisture content *(4)*.

For the control, corn samples (25.5 kg) were milled with a hammer mill having a screen size of 1 mm. The ground corn was mixed with 64 L of water and ground again with a disk mill to further reduce the particle size. The amount of water added was calculated so that the solids concentration in the control, Quick-Germ, and Quick-Fiber samples was about the same. The material was then frozen and shipped to USDA's Eastern Regional Research Center (ERRC) for fermentation.

For Quick-Germ, corn samples (25.5 kg) were soaked with 55 L of water and 0.5% lactic acid for 12 h at 59°C in a steep tank (vibra-screw) *(1)*. The soak water and soaked corn were transferred into two 95-L (25-gal) plastic buckets separately. The soaked corn was ground using a Bauer mill (Bauer Bros.) while pumping part of the soakwater into the mill to prevent plugging and heat build-up using a peristaltic pump (Cole-Palmer). The speed of the disks in the mill was adjusted to obtain the appropriate force on the corn to release most of the germ without damaging it.

A Dorr-Oliver, 3-in., Type NZ germ cyclone was set up by placing it on top of a 95-L (25-gal) stainless steel feed tank supported by four legs. The soak water left from milling was poured into the feed tank and a centrifugal pump (Dorr-Oliver) was used to recirculate the water from the feed tank to the cyclone. Ground corn was added gradually to maximize mixing in the feed tank and to prevent plugging in the hydrocyclone. The slurry density was adjusted to 1.066 kg/L (9°Be).

The overflow was collected, washed using a 200-mesh vibratory screen, and dried overnight in a 49°C oven. The underflow of the hydrocyclone was transferred back into the feed tank to maintain a high level of liquid in the tank to facilitate pumping of the feed. At the end of each run, the underflow was further ground by a disk mill to reduce the particle size, frozen, and shipped to ERRC for fermentation.

For Quick-Fiber, the same amount of corn was soaked at the same condition in 50 L of water, and grinding was performed in the same way as for Quick-Germ. The slurry density was about 1.082 kg/L (11°Be). After drying in the oven, the overflow from each run was sent through an aspirator to separate the germ and fiber. Germ and fiber moisture were determined using a two-stage oven procedure, method 45-15A *(4)*. The germ, fiber, and total yield were calculated and compared across every sample. At the end of each run, the underflow was further ground by a disk mill to reduce the particle size, frozen, and shipped to ERRC for fermentation.

The average starch content, expressed as the percentage of total corn dry solids, was 66% for the controls, 72% for the Quick-Germ runs, and 75% for the Quick-Fiber runs. The average nonfermentable suspended solids of the starting material, expressed as the percentage of total corn dry solids, was 18% for the controls, 14% for the Quick-Germ runs, and 10% for the Quick-Fiber runs. When received, the shipments were placed in a freezer and kept frozen until fermented. Each batch was thawed, transferred with some additional cold tap water to a 70-L fermentor, heated to 55°C, and adjusted to pH 6.0 with 10% sodium hydroxide. To liquefy the mash, the temperature was gradually increased while continuously (1 mL/min) adding 45 mL of Spezyme Delta AA (alpha-amylase from Genencor, Elkhart, IN) diluted with 150 mL of distilled water.

When the temperature of the liquefied mash reached 80°C, it was sampled for density and total solids (by drying overnight in a 70°C vacuum oven), rapidly cooled to 55°C, and adjusted to pH 4.4 with sulfuric acid. Then 75 mL of Optidex L-300 (Genencor glucoamylase) was added, and saccharification was continued for 2 to 3 h at 55°C. Ammonium chloride (75 g) was added, and the temperature was set to 35°C (actual temperature between 34 and 35°C). The fermentor was inoculated with 1.67% by volume of an exponentially growing culture of *Saccharomyces cerevisiae*, ATCC 4126 (American Type Culture Collection, Manassas, VA), having an optical absorbance of 0.35 at 600 nm (3.1 g/L of dry yeast). Optical absorbance was corrected for the absorbance of the media and dead cells in the inoculum by extrapolating the exponential growth curve to time zero.

The initial fermentation volume varied from 43 to 52 L, since the quantity of material shipped and the amount of water added were different for each run. The amount of corn dry solids in the fermentor varied from 9 to 16 kg, and the concentration of corn dry solids varied from 210 to 300 g/L. The pH during fermentation was maintained above 3.7 by automatic addition of 10% sodium hydroxide. Samples were taken automatically every

2 h using a timer-controlled pump and fraction collector. A few drops of a quaternary ammonium detergent (Clearbath; Spectrum, Houston, TX) were added to each sample with a timer-controlled pump to stop the fermentation at the time of sampling. The time for the fermentation to finish was taken as the time from inoculation until the time (estimated to the nearest hour) when the remaining glucose was 1 g/L.

The glucose and ethanol concentrations in the samples after clarification by centrifugation were measured with a biochemistry analyzer (YSI, Yellow Springs, OH). The yeast cell concentration was measured by counting cells in a hemocytometer under the microscope. The initial glucose concentration was the average of the first two or three samples. These were analyzed at least 16 h after collection, when little or no starch detectable by iodine test remained in them. (The quaternary ammonium detergent in the samples prevented yeast growth but did not inhibit the starch-hydrolyzing enzymes.) The initial glucose concentration was used to calculate the glucose conversion, and the theoretical maximum ethanol concentration ($0.51 \times$ initial glucose concentration). Ethanol yield was calculated as the percentage of theoretical maximum concentration. From the liquefied mash density, total solids, suspended solids, and the initial glucose concentration, the starch content of the starting material was calculated.

After fermentation, the dissolved and suspended solids were measured by centrifugation and drying overnight in a 70°C vacuum oven. It was assumed that the initial suspended solids were unaffected by fermentation, and the initial suspended solids were calculated as the final suspended solids minus the final yeast concentration. The whole beer was boiled by supplying steam to the fermentor jacket until the ethanol concentration was 1–3 g/L. The dissolved and suspended solids were measured again, and the stillage was separated in a Sharples P660 decanter centrifuge (Alfa-Laval, Warminster, PA) to yield thin stillage and wet distiller's grains. The thin stillage was refrigerated and shipped overnight to UIUC for evaporator fouling studies. The wet grains were frozen for future drying and shipment to the University of Missouri at Columbia for nutritional studies. The results of the fouling and nutritional studies will be reported in future publications.

To estimate the operating cost for a hypothetical Quick-Germ process producing 57,000,000 L (15,000,000 gal) of ethanol/yr, a computer model *(5)* previously created using the Aspen Plus process simulator (Aspen Technology, Cambridge, MA) was modified. This base-case process is a state-of-the-art dry-grind process using continuous cascade fermentors and molecular sieves for ethanol dehydration. Germ yield and oil content were estimated at 6% of corn and 44.5% of germ, respectively, and additional costs for Quick-Germ equipment *(6)* were escalated to year 2000 prices at 1%/yr. The value of germ was calculated as the value of the oil in the germ plus the value of the protein in the germ ($0.04/kg of germ) minus the cost of extracting the oil ($0.02/kg of germ). Because the total operating cost may vary greatly depending on various factors and assumptions, the difference between the Quick-Germ and base case was calculated, and the results were expressed as savings per liter or gallon of ethanol produced with the Quick-Germ process. The rate of feedstock (corn) consumption was the same for both processes. Costs for the Quick-Fiber process were not included because no estimate of the selling price for corn fiber was available.

Results and Discussion

The amount of 10% NaOH required to adjust the pH for liquefaction varied from 0 (for most control runs) to approx 450 mL for runs with added lactic acid. The amount of 7.5 *N* sulfuric acid required to adjust the pH for saccharification was approx 140 mL. As the initial glucose concentration increased from 165 to 250 g/L, the amount of 10% NaOH required to control the pH during fermentation decreased from approx 600 to 150 mL, and the final yeast concentration decreased from approx 8 to 6 g/L. The final dissolved solids concentration of the beer (before boiling) varied from 2.4 to 3.6 wt%. The total solids of the thin stillage varied from 3.4 to 7.9 wt%. There was no excessive foaming, and no addition of antifoam was required in any of the 3- or 50-L fermentations. It was concluded that sufficient oil remained in the Quick-Germ fermentations to control foaming.

Typical results obtained by analysis of the automatically collected samples are shown in Fig. 1. The final ethanol yield varied from 88 to 105% of theoretical (average 98%) and was not significantly different among the different treatments. Some sucrose present in corn is converted to ethanol. Because this fermentable sugar was not measured, or included with the initial glucose determination, the calculated theoretical yield is skewed toward the high side. (Typical industrial batch fermentation yields are approx 95–96% of theoretical.) The time to finish the fermentation increased from 23 to 60 h as the initial glucose concentration increased from 165 to 250 g/L. There was no significant difference among the different treatments. Data for 12 fermentations, including 5 controls, 5 Quick-Germ, and 2 Quick-Fiber, are shown in Fig. 2. The data were fit by nonlinear least squares to the empirical mathematical model:

$$y = \frac{4449}{321.55 - x} - 3.5$$

in which y is the fermentation duration (hours) and x is the initial glucose concentration (grams/liter).

Because no difference was observed in the fermentation characteristics of whole-corn controls and degermed corn, the process and cost models for continuous-cascade fermentation in the base case were also used for cost analysis of the Quick-Germ process. The solids concentration was adjusted to give the same concentration of ethanol leaving the fermentors as in the base case (94 g/L of ethanol in the liquid phase). Although not needed to estimate savings with Quick-Germ, the previous mathematical

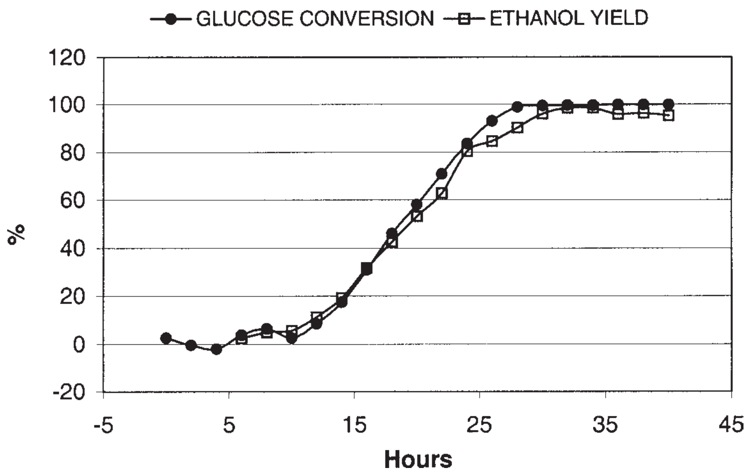

Fig. 1. Typical fermentation data; automatic sampling every 2 h. The time to finish the fermentation was estimated from these data.

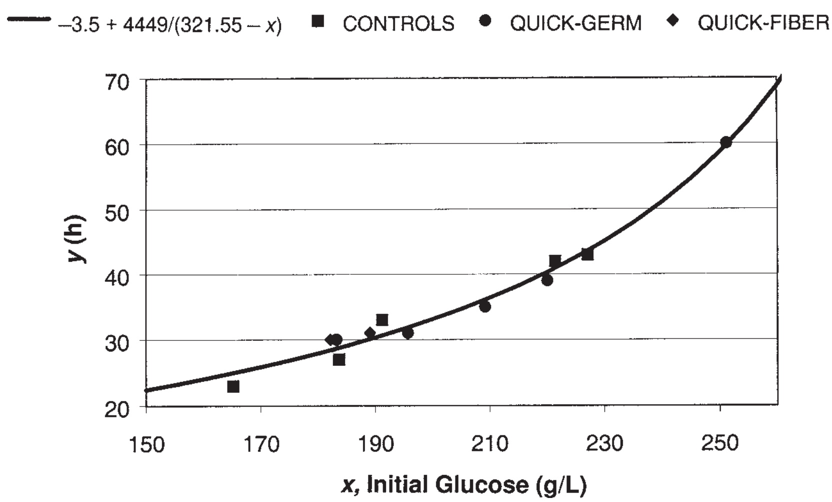

Fig. 2. The time to finish the fermentation increased with concentration but did not vary among the treatments. Data were fit to an empirical model by nonlinear least squares.

model agrees well with current industrial practice at some fuel ethanol facilities operating batch fermentors. It permits estimation of the size and cost of batch fermentors at different solids concentrations. Preliminary results indicate that at 200 g/L of initial glucose there is little difference in size or cost between continuous-cascade fermentors and batch fermentors. Larger fermentors are required for high-gravity (high initial specific gravity) batch fermentation (approx 250 g/L of initial glucose), but savings of approx $0.005/L of ethanol ($0.02/gal) on overall process costs are predicted.

The results of process operating cost estimation indicate that the savings with Quick-Germ are very sensitive to the price of crude corn oil. At \$0.44/kg of crude corn oil (\$0.20/lb), revenue from the sale of germ amounts to \$0.032/L of ethanol (\$0.122/gal), but at \$0.55/kg of crude corn oil (\$0.25/lb), revenue from the sale of germ increases to \$0.038/L of ethanol (\$0.145/gal). Revenue from the sale of germ is offset by estimated additional utility costs of \$0.002/L of ethanol (\$0.006/gal) and estimated additional capital-related costs (including depreciation, maintenance, labor, administration, taxes and insurance—each calculated as a percentage of total capital investment) amounting to \$0.013/L of ethanol (\$0.051/gal) for equipment to separate the germ in the Quick-Germ process.

Although the estimated selling price of DDGS from the Quick-Germ process based on its protein content (28%) is approximately the same as in the base case, the amount of DDGS sold is reduced by the amount of germ recovered. Revenue from the sale of DDGS at \$0.088/kg (\$80/t) is reduced by \$0.013/L of ethanol (\$0.050/gal) compared with the base case. The sum of estimated increased process operating costs and reduced DDGS revenue is \$0.028/L of ethanol (\$0.107/gal). By subtracting this from the germ revenue, the savings at the lower price of crude corn oil are estimated to be only \$0.004/L of ethanol (\$0.015/gal), but the estimated savings increase to \$0.010/L (\$0.039/gal) at the higher crude corn oil price. Details of the process and cost models are available from the authors on request.

These estimates of Quick-Germ process operating costs include savings owing to smaller sizes for some of the base-case equipment. These arise because the flow of process streams after germ separation is less by the volume of the germ removed. For example, the estimated size of each of the four continuous-cascade fermentors decreases from 887,000 to 873,000 L. These savings are modest, on the order of only 1% of the capital cost of base-case equipment, except for the DDGS dryer, which is significantly smaller because approx 20% less DDGS is produced. In the Quick-Germ process, the germ is washed to recover almost all the starch. Losses of starch with the germ are estimated to be approx 0.5%, reducing the ethanol yield from 0.407 L/kg (2.73 gal of denatured ethanol/bushel) in the base case, to 0.405 L/kg (2.72 gal/bushel) with Quick-Germ.

Lower suspended solids with Quick-Germ offer advantages such as higher heat transfer coefficients in heat exchangers and lower utility consumption of agitators and pumps. Also, removing most of the oil from the stillage will reduce fouling in the reboiler and evaporator. However, the impact of these improvements on the process operating cost could not be estimated and was not included. An opportunity for greater cost savings is created when Quick-Germ is combined with an advanced fermentation design such as continuous high-gravity fermentation with stripping *(5)*. Although estimated savings with the continuously stripped fermentor alone are comparable with those for Quick-Germ alone, approx \$0.005–\$0.01/L (2–4 cents/gal), the high-gravity stripping process is limited by high suspended solids. The Quick-Germ process lowers the suspended solids, allowing higher total solids high-gravity fermentation. Savings on the order of \$0.02–\$0.03/L (6–10 cents/gal) are indicated for a combined process. Integration of Quick-Germ and high-gravity continuous fermentation into the fuel ethanol industry is anticipated to increase profits and expand the use of ethanol in gasoline. This, in turn, would benefit corn growers and transportation fuel consumers.

Conclusion

Unique methods for standard inoculation and automatic sampling provide accurate estimates of the duration of batch fermentations of corn mash to ethanol. Data agree well with a new empirical mathematical model that permits estimation of the size and cost of batch fermentors. Except for lower suspended solids in Quick-Germ fermentations, there is no difference between Quick-Germ and whole-corn fermentations. Process simulation and cost estimation of the Quick-Germ process reveal that the savings with Quick-Germ are sensitive to the price of crude corn oil. At \$0.55/kg of crude corn oil (\$0.25/lb), savings are estimated to be 1 cent/L of ethanol (4 cents/gal). However, additional savings that may be realized from the lower suspended solids after germ recovery were not included in the analysis.

References

1. Singh, V. and Eckhoff, S. R. (1996), *Cereal Chem.* **73,** 716–720.
2. Doner, L. W., Chau, H. K., Fishman, M. L., and Hicks, K. B. (1998), *Cereal Chem.* **75,** 408–411.
3. Moreau, R. A., Hicks, K. B., Norton, R., and Nicolosi, R. (1997), US patent 5,843,488, filed Oct. 8, 1997, issued Dec. 1, 1998.
4. American Association of Cereal Chemists (1983), *Approved Methods of the AACC*, 8th ed., Method 44-15A, The Association, St. Paul, MN.
5. Taylor, F., Kurantz, M. J., Goldberg, N., McAloon, A. J., and Craig, J. C., Jr. (2000), *Biotechnol. Prog.* **16(4),** 541–547.
6. Singh, V. and Eckhoff, S. R. (1997), *Cereal Chem.* **74,** 462–466.