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Ethanol Production from Food Waste at High Solids Content with Vacuum Recovery Technology

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ABSTRACT: Ethanol production from food wastes does not only solve environmental issues but also provides renewable biofuels. This study investigated the feasibility of producing ethanol from food wastes at high solids content (35%, w/w). A vacuum recovery system was developed and applied to remove ethanol from fermentation broth to reduce yeast ethanol inhibition. A high concentration of ethanol (144 g/L) was produced by the conventional fermentation of food waste without a vacuum recovery system. When the vacuum recovery is applied to the fermentation process, the ethanol concentration in the fermentation broth was controlled below 100 g/L, thus reducing yeast ethanol inhibition. At the end of the conventional fermentation, the residual glucose in the fermentation broth was 5.7 g/L, indicating incomplete utilization of glucose, while the vacuum fermentation allowed for complete utilization of glucose. The ethanol yield for the vacuum fermentation was found to be 358 g/kg of food waste (dry basis), higher than that for the conventional fermentation at 327 g/kg of food waste (dry basis).

KEYWORDS: *food waste, ethanol, fermentation, vacuum recovery, high solids content*

INTRODUCTION

Over the past few decades, the objectives to establish national energy independence and to reduce the greenhouse gas emissions have led to the development of renewable biofuel technologies based on agricultural materials. Ethanol is by far the most significant biofuel in the United States, accounting for 94% of all biofuel production in 2012.[1] Ethanol is mainly produced from corn in the U.S. and from sugar cane in Brazil.[2] However, corn and sugar cane are also used as food; overuse of corn or sugar cane as feedstock for ethanol production would create "food versus fuel" competition. Furthermore, increasing prices of corn and sugar cane are the main drivers of the high cost of ethanol production. According to the previous studies, corn and sugar cane feedstock costs contributed to 70−90% of the total ethanol production costs.[3−5] Researchers have investigated the production of ethanol from low-cost agricultural residues, such as corn stover,[6,7] wheat straw,[8] sugar cane bagasse,[9] and rice straw.[10] Efficiently releasing sugars from cellulose and hemicellulose is one of the main challenges of using cellulosic biomass.[11] To break cellulose and hemicellulose into monosaccharides, the biomass materials have to be processed with a harsh pretreatment process, followed by hydrolysis with the addition of a high dosage of enzymes, which significantly increases the capital and processing costs of the ethanol production.[11,12]

Food waste is a complex biomass discharged from households, restaurants, cafeterias, and retail stores and accounts for a considerable portion of municipal solid waste.[13] In the U.S., more than 36 million tons of food waste were generated in 2012 alone.[14] Food waste management raises significant environmental concerns. Disposal of food waste in a landfill is not only costly but also causing potential environmental problems, with direct and indirect emissions of greenhouse gases (CH_4 and CO_2).[15,16] Incineration is another way to manage food waste but is banned in different countries because of environmental concerns. Also, energy recovery through incineration may not be feasible, because of the energy loss to evaporate the large water content in food waste.[16] Food waste can be diverted from landfills and incinerators by turning it into compost to improve the soil fertility, but it may cause severe pollution to surface and underground water.[17] On the other hand, food waste contains abundant nutrition (starch, glucose, protein, etc.), making it a good raw material for biofuel production. Until now, most of the research has been focusing on the usage of food waste to produce biogas through anaerobic digestion.[18−20] Food waste can also be used as a low-cost feedstock for producing ethanol,[15,21,22] which is a more valuable fuel compared to biogas.

Besides using the low-cost materials, fermentation at higher solids contents can lower the ethanol production costs, because it can reduce the energy and water consumptions as well as the volumes of the processing equipment.[23] However, higher solids fermentation results in a higher ethanol concentration, which inhibits yeast activity, thereby causing reduced ethanol yield and fermentation efficiency.[24] Vacuum stripping, one of the *in situ* ethanol removal technologies, has been reported to improve the ethanol or butanol fermentations at high solids contents.[23,25−27] In a vacuum fermentation system, the produced ethanol is removed by maintaining the bioreactor under vacuum conditions, so that ethanol boils off at the fermentation temperature and is subsequently recovered by the

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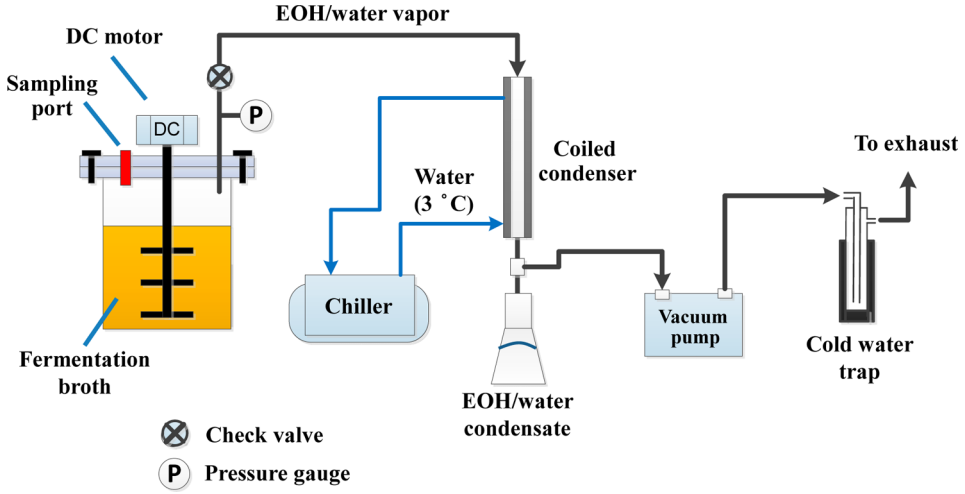

Figure 1. Experimental setup for the food waste fermentation with the vacuum stripping system. The arrows in the figure show flow direction. The vacuum fermentation system consisted of a 3 L water-jacked fermenter, a vacuum pump, a condensation system with a chiller and a coiled condenser, and a cold water trap. During the vacuum application, ethanol and water vapors evaporated from the fermentation broth and condensed in the condensation system. The escaped (uncondensed) ethanol and water vapors were collected in the cold-water trap.

following condensation system with cooling or chilling water. In the vacuum fermentation system, a low ethanol concentration can be maintained in the fermenter during fermentation, thereby eliminating or minimizing yeast ethanol inhibition.[23,27]

The objective of this study was to produce ethanol from food waste at high solids content with vacuum recovery technology. Fermentation of food waste at high solids content results in a very viscous mash and a high glucose concentration (which also inhibits yeast). To overcome this problem, granular starch hydrolyzing enzyme (GSHE) was used to directly digest raw starch in food waste at low temperatures (<48 °C) during simultaneous saccharification and fermentation. GSHE decreases the viscosity of the fermentation broth and reduces the yeast glucose inhibition by gradually breaking down starch to glucose during fermentation.[23,28]

MATERIALS AND METHODS

Design and Characterization of the Vacuum Recovery Process. The vacuum fermentation system was modified on the basis of the system reported by Shihadeh et al.[23] (Figure 1). It consisted of a 3 L jacketed fermenter (Biostat MD, Sartorius BBI Systems, Edgewood, NY), which was sealed and modified for accommodating thermocouples and venting and sampling ports. Slurry (fermentation mixture) was agitated by paddle-type blades driven by an alternating current (AC)/direct current (DC) motor drive (KD P/N 3402-008, Hurst, Princeton, NJ). The vacuum recovery system was connected to the fermenter venting port through a check valve (Figure 1). The evaporated ethanol and water vapor was condensed by passing through a coiled condenser (5977-19, Ace Glass, Vineland, NJ), which was circulated with chilled water at 3 °C from a refrigerated water bath (PolyScience 9106, Cole-Parmer, Vernon Hills, IL). The condensate was collected in a 250 mL conical flask. Vacuum was generated with a dry vacuum pump at 6.7 kPa (28 in Hg gauge) (DryFast model 2044, Welch, Niles, IL). Vacuum pressure was monitored at the fermentation vessel with a pressure gauge (45W1000, Ashcroft, Straford, CT). Vacuum outlet was connected to a cold-water trap (LG-11025, Wilmad Lab Glass, Buena, NJ) chilled with iced water, to collect the escaping ethanol vapors.

To evaluate the vacuum process for ethanol recovery from a dilute solution, the vacuum recovery experiment was conducted at 32 °C using a model ethanol solution in the fermenter. The initial ethanol concentrations in the model solution were 40, 80, and 160 g/L, respectively. Vacuum pressure at 6.7 kPa (28 in Hg gauge) was applied to the ethanol model solution for 1 h, during which the model solution boiled and the vapors were condensed in a 250 mL conical flask. At the completion of each vacuum application, the remaining solution in the fermenter, the condensate in the conical flask, and the solution in the cold water trap were collected and analyzed for volumes and ethanol concentrations.

Experimental Materials and Reagents. The food waste was obtained from a local retail store in Urbana, IL, and mainly contained mashed potatoes, sweet corn, and white bread and was used as a model food waste. The moisture content of the food waste was 64.0%. The composition of the food waste (dry matter basis) was 63.5% starch, 4.3% glucose, 13.9% protein, 4.1% oil, 5.2% neutral detergent fiber, and 3.4% ash. The procedures of the composition measurements are provided in Analytical Procedures and Calculations. The high starch content in the food waste sample was very similar to the sample reported in the previous study.[29] Received food waste was pulverized and mixed using a fruit/vegetable mixer for 3 min, analyzed for moisture content, and stored at 4 °C for the following experiments. For the fermentation slurry preparation, 10 N sulfuric acid (Ricca Chemical, Arlington, TX), solid urea (Fisher Scientific, Waltham, MA), and active dry yeast (Ethanol Red, Fermentis, Lesaffre Yeast, Milwaukee, WI) were used. Enzymes used in the fermentation processes were GHSE (Stargen 002) and protease (GC 212) obtained from Dupont Industrial Biosciences (Palo Alto, CA). Stargen 002 contained *Aspergillus kawachi* α-amylase expressed in *Trichoderma reesei* and a glucoamylase from *Trichoderma reesei* that work synergistically to hydrolyze granular starch substrate to glucose, and it had an activity of ≥570 GSHU/g (where GSHU = granular starch hydrolyzing units). Protease enzyme, GC 212, was obtained by fermentation of a selected strain of *Aspergillus niger* and was able to hydrolyze peptide bonds along a protein chain. It had an activity of 2000 SAPU/g (where SAPU = spectrophotometer acid protease units).

Conventional Fermentation in a Batch Reactor. Fermentation slurry at 35% solid contents (w/w) was prepared by mixing 500 g of dry weight of mixed food waste with a calculated amount of deionized water. The fermentation slurry was then adjusted to pH 4.0 using 10 N sulfuric acid. Experiments were performed in a 3 L fermenter. The slurry was added with 3 mL of GSHE enzyme (Stargen 002), 0.2 mL of protease enzyme (GC 212), 2 mL of urea solution (50%, w/v), and 2 mL of prepared yeast culture. Yeast culture was prepared by dispersing 5 g of active dry yeast and 25 g of deionized water and agitated at 90 rpm at 32 °C for 20 min in an incubator shaker (C24 incubator shaker, New Brunswick, NJ). Fermentation was conducted for 72 h at 32 °C with continuous agitation at 30 rpm. The enzymatic

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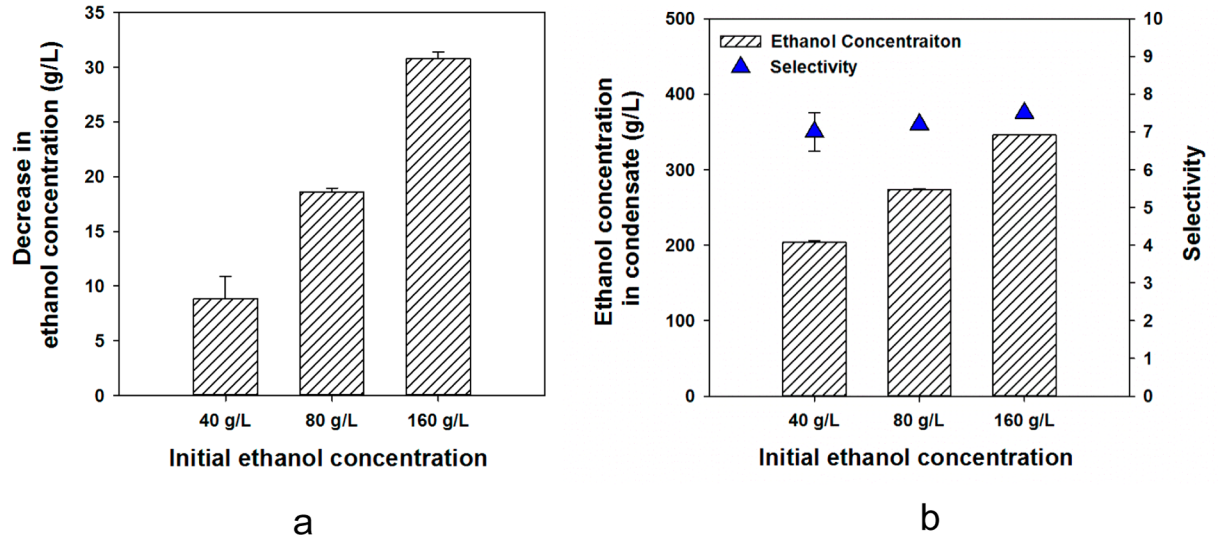

Figure 2. (a) Decrease in ethanol concentrations in the fermenter and (b) ethanol concentrations in the condensate and the vacuum selectivity after 1 h, with vacuum application of the model solution at different initial concentrations.

hydrolysis gradually reduced the slurry viscosity, thus causing an increase in revolutions per minute (rpm) of the agitator higher than 30. Therefore, the agitation rate was checked and manually reset to 30 rpm after each sampling. Fermentation was monitored by taking 1 mL of slurry sample at 4, 8, 12, 24, 36, 48, and 72 h and measuring sugars, ethanol, glycerol, and acid concentrations using high-performance liquid chromatography (HPLC) with at least two determinations. Fermentation was conducted in triplicates.

Vacuum Fermentation. The slurry used for the vacuum fermentation was the same as that used for the conventional fermentation experiments. The developed vacuum recovery system was used (Figure 1). The fermentation was allowed to proceed for 24 h, during which the ethanol concentration approached around 100 g/L and was followed by ethanol recovery by the vacuum stripping (6.7 ± 0.2 kPa), during which broth in the bioreactor boiled at the fermentation temperature, generating ethanol and water vapors. Ethanol and water vapors were then condensed in the coiled condenser at 3 °C and collected in the conical flask. The outlet from the vacuum pump was connected to the cold-water trap, to collect the uncondensed ethanol vapors. Foaming, caused by CO_2 bubbling during the initial vacuum application, was controlled by increasing the agitation rate to 60 rpm and gradually increasing vacuum to the set point. At the completion of each vacuum application, vacuum was stopped and filtered air (filter = 0.2 μm) was introduced gradually to the system until pressure equilibrated to atmosphere. Samples were taken before and after each vacuum application to monitor the immediate effect of vacuum stripping on the ethanol concentration in the fermentation broth. Also, the condensate in the conical flask and the solution in the cold-water trap were collected and analyzed for volumes and ethanol concentrations, to calculate the ethanol yield at each time period. Fermentation was conducted in triplicates.

Analytical Procedures and Calculations. The moisture content of the food waste was determined by drying samples in a forced-draft oven at 135 °C for 2 h.[30] The glucose and starch concentrations in the food waste was measured by the modified dilute acid method.[31] The crude protein, oil, and ash concentrations in the food waste were determined according the AOCS Official and Tentative Methods Ba 4e-93, Am 5-04, and Ba 5a-49, respectively. The neutral detergent fiber concentration in the food waste was determined using the ANKOM$^{200/220}$ fiber analyzer (ANKOM Technology, Macedon, NY).[32] Fermentation samples were analyzed with HPLC (column, Aminex HPX-87H organic acid, Bio-Rad, Hercules, CA; system, Breeze, Waters Corporation, Milford, MA) for glucose, ethanol, glycerol, and acetic acid concentrations. Broth samples were centrifuged for 3 min at 11000*g* (model 5425, Eppendorf, Westbury, NY). Supernatants were filtered through a 0.2 μm filter into a 0.2 mL HPLC vial insert. Ethanol productivity or ethanol production rate was calculated as the produced ethanol (g/L in broth) divided by the fermentation time period. The ethanol yield was defined as total grams of ethanol produced per kilograms of dry food waste.[11,12,33] Ethanol conversion efficiency was calculated by the ratio of actual ethanol yield over theoretical ethanol yield, which was based on the starch and sugar contents in the food waste.[34] Because both ethanol and water in the fermentation broth evaporated during the vacuum application, ethanol selectivity was calculated to measure the preferential removal of ethanol over other components (water in this case). The ethanol selectivity was calculated as

$$\alpha = \frac{y/(1-y)}{x/(1-x)}$$

where x and y are weight fractions of ethanol in fermentation broth and condensate, respectively.

RESULTS AND DISCUSSION

Characterization of Vacuum Technology for Ethanol Recovery. To study the ethanol recovery characteristics under the vacuum application, ethanol recovery experiments were conducted using model ethanol solution at different initial ethanol concentrations (40, 80, and 160 g/L), which covered the possible ethanol concentration range in the food waste fermentation. After 1 h of vacuum application, the ethanol concentration in the model solution decreased by 9−31 g/L, depending upon the initial ethanol concentrations in the model solution. As the initial ethanol concentration in the fermenter increased from 40 to 160 g/L, a greater decrease in the ethanol concentration was observed after the vacuum stripping (Figure 2a). This trend agrees well with the previous studies,[35] which used vacuum technology to remove butanol during fermentation. For a successful application of vacuum stripping to ethanol recovery, the ethanol removal rate should be greater than the ethanol production rate by fermentation, to avoid the ethanol accumulation in the fermentation broth. The ethanol removal rates (9−31 g L^{-1} h^{-1}) in this study were higher than the ethanol production rates (<5 g L^{-1} h^{-1}), as shown in Figure 4. Therefore, the developed vacuum recovery system could be applied to maintain ethanol concentrations in broth at low levels and reduce yeast ethanol inhibition.

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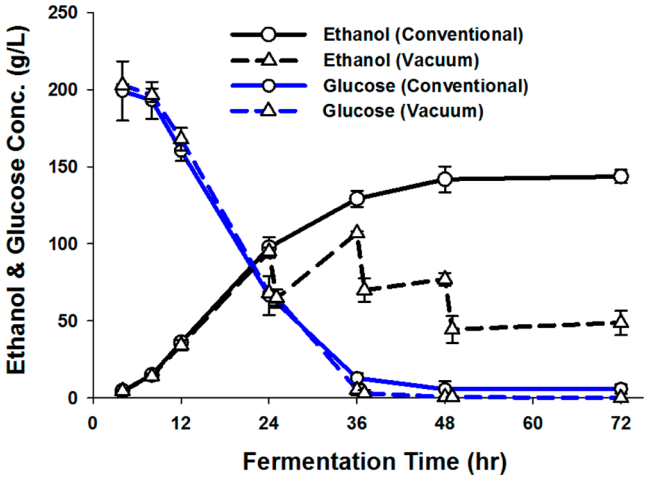

Figure 3. Ethanol and glucose concentrations during SLSF in both conventional and vacuum fermentations.

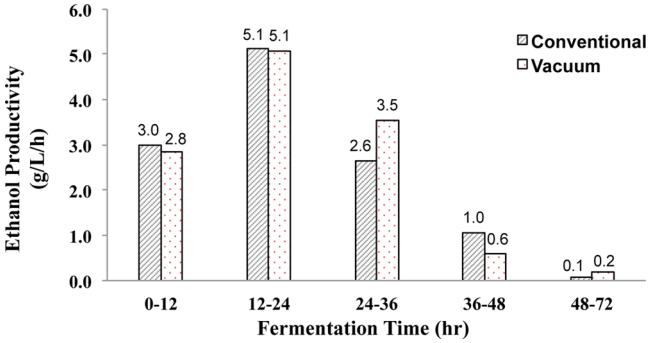

Figure 4. Ethanol productivity in the conventional and vacuum fermentation.

The ethanol concentration in the condensate was found to be much higher than that in the model solution in the fermenter (Figure 2b). The ethanol concentrations in the condensates were 204, 274, and 346 g/L, when the initial ethanol concentrations in the fermenter were 40, 80, and 160 g/L, respectively. This indicates that the vacuum recovery system not only removes ethanol from the fermentation broth but also concentrates it. Selectivity is a variable commonly used to evaluate the ability of a product recovery technology to separate a target product (ethanol in this case) from a mixture. Figure 2b shows that the initial ethanol concentrations in the model solution did not have large effects on the ethanol selectivity. The selectivity was found to be close to 7 for the model solution with all three ethanol concentrations.

Conventional Ethanol Fermentation. The conventional fermentation without vacuum application was conducted in a batch mode. Figure 3 shows the ethanol and glucose concentrations during 72 h of fermentation. The glucose concentration was 199 g/L at 4 h and decreased rapidly during the fermentation process. The ethanol concentration increased to 144 g/L at the end of the fermentation. The residual glucose concentration was 5.7 g/L at 72 h (Table 1), indicating an incomplete fermentation in the batch fermenter. The major reason for fermentation cessation before the exhaustion of glucose was probably due to the high ethanol concentration, which can inhibit yeast.[23,26] Nevertheless, the final ethanol concentration of the batch fermentation was higher than that from the previous studies (Table 2), where the final ethanol concentrations in the broth were between 9 and 82 g/L. The high ethanol concentrations obtained in this study were mainly attributed to the high solids content slurry used for the fermentation and the high starch concentration in the food waste. Higher ethanol concentrations in the fermentation broth can decrease the distillation energy costs, thereby reducing the related capital and operation costs for the conversion of food waste to ethanol.

Table 1. Comparison of Ethanol Fermentation of Food Waste with and without Vacuum Stripping

	residual glucose (g/L)	ethanol yield (g/kg of food waste)	conversion efficiency (%)
conventional fermentation	5.7 ± 2.0	326.5 ± 5.4	85.4 ± 1.4
vacuum fermentation	0.0 ± 0.0	357.5 ± 13.0	93.6 ± 3.4

Table 2. Production of Ethanol from Food Wastes from Different Sources

source of food waste	solid content for fermentation	final ethanol concentration (g/L)	reference
cafeteria	12.1% (w/w)	29.1	15
cafeteria	16.3−19.0% (w/v)	45.0	22
cafeteria	12.9% (w/w)	8.9−55.7	39
dinning room	<20.5% (w/w)	75.9−81.5	29
household	35 and 45% (w/v)	34.9 and 42.8	11
retail store	35% (w/w)	144	this study

Ethanol Fermentation with Vacuum Stripping. To assess the performance of simultaneous ethanol fermentation and recovery by vacuum stripping, the vacuum recovery system for the food waste fermentation was applied. During the 72 h fermentation, vacuum stripping was applied for 1 h at 24, 36, and 48 h, respectively. Figure 3 shows the ethanol and glucose concentrations during fermentation. In the first 24 h, the ethanol concentration increased rapidly to 95 g/L because of sufficient glucose in the fermentation broth. After 1 h of vacuum application, the ethanol concentration decreased to 64 g/L, because of the evaporation of ethanol under the vacuum condition. The ethanol concentration in the fermentation broth decreased by 31, 37, and 33 g/L, when vacuum stripping was applied for 1 h at 24, 36, and 48 h, respectively. Overall, the vacuum stripping successfully removed ethanol and controlled ethanol concentrations below 100 g/L in the fermentation broth, thus reducing yeast ethanol inhibition.

The glucose concentration was 203 g/L in the fermentation broth at 4 h and decreased rapidly to a negligible amount at 36 h (Figure 5). At the end of fermentation (72 h), there was no residual glucose left (Table 1), indicating a complete fermentation in this study. This was mainly due to the reduced ethanol inhibition by controlling the ethanol concentrations at low levels in the broth with the vacuum application. To note, even at 0 g/L glucose concentration, the ethanol concentration was shown to increase slightly between 48 and 72 h. This may be due to glucose being slowly released during the simultaneous liquefaction, saccharification, and fermentation (SLSF) with GSHE enzyme, and yeast used glucose faster than it was released by hydrolysis of the food waste. This phenomenon was also reported in the previous studies when corn or wheat straw was used as the substrate for the simultaneous saccharification and fermentation to produce ethanol or butanol.[23,28,36]

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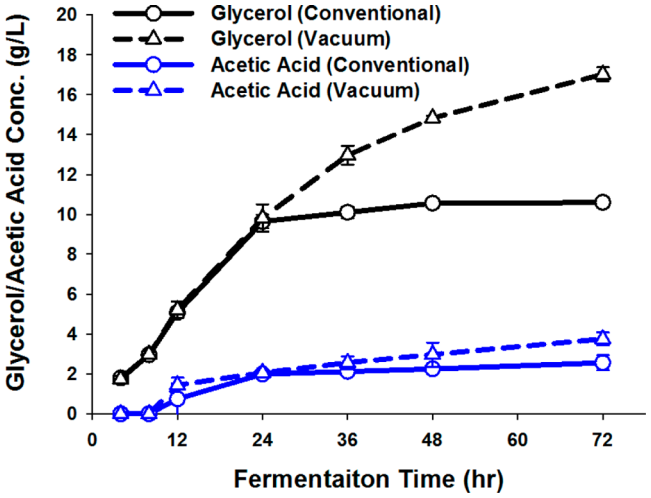

Figure 5. Byproduct (glycerol and acetic acid) concentrations during 72 h of fermentation of the food waste.

Comparison between Ethanol Fermentation with and without Vacuum Stripping. The residual glucose concentration, ethanol yield, and conversion efficiency for both the conventional and vacuum fermentations are presented in Table 1. The ethanol yield for the conventional fermentation was found to be 327 g/kg of food waste (dry matter), while the ethanol yield for the vacuum fermentation was 9% higher at 358 g/kg of food waste. This finding was in agreement with the residual glucose concentrations and the conversion efficiency in Table 1. As discussed above, the residual glucose was zero in the vacuum fermentation but was 5.7 g/L in the conventional fermentation because of yeast ethanol inhibition, which lowered the ethanol yield. The ethanol conversion efficiency for the vacuum fermentation was 93.6%, higher than that for the conventional fermentation efficiency at 85.4%, showing the advantages of applying the vacuum recovery system to the fermentation process. Considering that some sugar was used for yeast growth and that there were other metabolites produced (glycerol, succinic and acetic acids, etc.),[37] the ethanol conversion by vacuum application at 93.6% was quite satisfactory. Overall, the vacuum fermentation showed some advantages over the conventional fermentation for food waste, in terms of the ethanol yield and conversion efficiency.

For the vacuum recovery system, the fermentation design is more complex compared to the conventional fermentation. The energy consumption for vacuum pumps and chilling required for condensation of evaporated ethanol may be high. However, the increase in the ethanol yield and productivity could potentially offset the increased cost of energy for vacuum pumps and chilling. For example, a 9% increase in the ethanol yield in a 50 million gallon capacity ethanol plant (typical size in the U.S.) means an additional 4.5 million gallons of ethanol, which can generate multimillion dollars for the plant. A detailed techno-economic evaluation of vacuum recovery fermentation is warranted to determine its economic viability.

Ethanol productivity for both the conventional and vacuum fermentations at different time periods are shown in Figure 4. The ethanol productivities at 0−12 and 12−24 h time periods were very close between the conventional and vacuum fermentation, because the vacuum stripping was not applied until 24 h. During the time period from 24 to 36 h, the ethanol productivity for the vacuum fermentation (3.5 g L^{-1} h^{-1}) was higher than that for the conventional fermentation (2.6 g L^{-1} h^{-1}), which again was because of the relieved ethanol inhibition by applying the vacuum stripping. During the time period from 36 to 48 h, the ethanol productivity for the vacuum fermentation was 0.6 g L^{-1} h^{-1}, while the ethanol productivity for the conventional fermentation was higher at 1.0 g L^{-1} h^{-1}. This was probably due to the higher amount of glucose left in the conventional fermentation during this time period (Figures 2 and 3). Matsakas et al.[11] summarized various studies on food waste fermentations and showed that the ethanol productivities were between 0.49 and 4.11 g L^{-1} h^{-1}, which was very close to the values showed in Figure 4 in this study.

Glycerol is a byproduct of yeast metabolism, and its production amount was less than ethanol and carbon dioxide during yeast ethanol fermentation. A higher amount of glycerol production usually indicates increased yeast stress.[37] In this study, the glycerol concentrations in the fermentation broth were higher during the vacuum fermentation (17 g/L) than the conventional fermentation (10 g/L) (Figure 5). It was because the removal of ethanol and water from the fermentation broth by vacuum stripping concentrated the glycerol in fermentation broth. A higher glycerol concentration could inhibit yeast metabolism; however, a review of the literature shows that the glycerol inhibition is not significant until it reached 100 g/L.[38] Therefore, the accumulated glycerol by the vacuum recovery system was not a factor in our study. Again, because of the concentration effect, the organic acid (acetic acid) concentrations were higher during the vacuum fermentation than the conventional fermentation (Figure 5). It is noteworthy that no glycerol or acetic acid was found in the condensate.

In conclusion, food waste at high solids content (35%, w/w) was used to produce ethanol with and without the vacuum recovery technology. After 72 h of fermentation, the ethanol concentration in the fermentation broth was 144 g/L for the conventional fermentation. The residual glucose concentration at 72 h was 5.7 g/L, indicating an incomplete fermentation. The conventional fermentation produced 327 g of ethanol from each kilogram of food waste. The vacuum fermentation process integrated with product recovery successfully controlled the ethanol concentration in the fermentation broth below 100 g/L, thereby reducing yeast ethanol inhibition. The vacuum fermentation allowed for complete utilization of glucose in the fermentation broth and produced 358 g of ethanol from each kilogram of food waste. The ethanol conversion efficiency for the vacuum fermentation was 93.6%, higher than that for the conventional fermentation at 85.4%.

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Notes

The authors declare no competing financial interest.

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■ ABBREVIATIONS USED

GSHE, granular starch hydrolyzing enzyme; SLSF, simultaneous liquefaction, saccharification, and fermentation

■ REFERENCES

(1) United States Department of Agriculture (USDA). *Bioenergy Statistics*; USDA: Washington, D.C., 2013; www.ers.usda.gov/data-products/us-bioenergy-statitics.aspx#.VCK0tNdFOXd (accessed Sept 22, 2014).

(2) Walker, K.; Vadlani, P.; Madl, R.; Ugorowski, P.; Hohn, K. L. Ethanol fermentation from food processing waste. *Environ. Prog. Sustainable Energy* **2013**, *32*, 1280−1283.

(3) McAloon, A.; Taylor, F.; Yee, W.; Ibsen, K.; Wooley, R. *Determining the Cost of Producing Ethanol from Corn Starch and Lignocellulosic Feedstocks*; National Renewable Energy Laboratory (NREL): Golden, CO, 2000; NREL/TP-580-28893, http://www.nrel.gov/docs/fy01osti/28893.pdf (accessed Sept 22, 2014).

(4) Kwiatkowski, J. R.; McAloon, A. J.; Taylor, F.; Johnston, D. B. Modeling the process and costs of fuel ethanol production by the corn dry-grind process. *Ind. Crops Prod.* **2006**, *23*, 288−296.

(5) Quintero, J.; Montoya, M.; Sánchez, O.; Giraldo, O.; Cardona, C. Fuel ethanol production from sugarcane and corn: Comparative analysis for a Colombian case. *Energy* **2008**, *33*, 385−399.

(6) Lau, M. W.; Dale, B. E. Cellulosic ethanol production from AFEX-treated corn stover using *Saccharomyces cerevisiae* 424A (LNH-ST). *Proc. Natl. Acad. Sci. U. S. A.* **2009**, *106*, 1368−1373.

(7) Kazi, F. K.; Fortman, J. A.; Anex, R. P.; Hsu, D. D.; Aden, A.; Dutta, A.; Kothandaraman, G. Techno-economic comparison of process technologies for biochemical ethanol production from corn stover. *Fuel* **2010**, *89*, S20−S28.

(8) Saha, B. C.; Iten, L. B.; Cotta, M. A.; Wu, Y. V. Dilute acid pretreatment, enzymatic saccharification and fermentation of wheat straw to ethanol. *Process Biochem.* **2005**, *40*, 3693−3700.

(9) Martín, C.; Galbe, M.; Wahlbom, C. F.; Hahn-Hägerdal, B.; Jönsson, L. J. Ethanol production from enzymatic hydrolysates of sugarcane bagasse using recombinant xylose-utilising *Saccharomyces cerevisiae*. *Enzyme Microb. Technol.* **2002**, *31*, 274−282.

(10) Binod, P.; Sindhu, R.; Singhania, R. R.; Vikram, S.; Devi, L.; Nagalakshmi, S.; Kurien, N.; Sukumaran, R. K.; Pandey, A. Bioethanol production from rice straw: An overview. *Bioresour. Technol.* **2010**, *101*, 4767−4774.

(11) Matsakas, L.; Kekos, D.; Loizidou, M.; Christakopoulos, P. Utilization of household food waste for the production of ethanol at high dry material content. *Biotechnol. Biofuels* **2014**, *7*, 1−9.

(12) Humbird, D.; Davis, R.; Tao, L.; Kinchin, C.; Hsu, D.; Aden, A.; Schoen, P.; Lukas, J.; Olthof, B.; Worley, M. *Process Design and Economics for Biochemical Conversion of Lignocellulosic Biomass to Ethanol*; National Renewable Energy Laboratory (NREL): Golden, CO, 2011; NREL/TP-5100-47764, http://www.nrel.gov/docs/fy11osti/47764.pdf (accessed Sept 22, 2014).

(13) Yan, S.; Chen, X.; Wu, J.; Wang, P. Ethanol production from concentrated food waste hydrolysates with yeast cells immobilized on corn stalk. *Appl. Microbiol. Biotechnol.* **2012**, *94*, 829−838.

(14) United States Environmental Protection Agency (U.S. EPA). *Food Waste Basics*; U.S. EPA: Washington, D.C., 2014; www.epa.gov/foodrecovery/ (accessed Sept 22, 2014).

(15) Moon, H. C.; Song, I. S.; Kim, J. C.; Shirai, Y.; Lee, D. H.; Kim, J. K.; Chung, S. O.; Kim, D. H.; Oh, K. K.; Cho, Y. S. Enzymatic hydrolysis of food waste and ethanol fermentation. *Int. J. Energy Res.* **2009**, *33*, 164−172.

(16) Lin, C. S. K.; Pfaltzgraff, L. A.; Herrero-Davila, L.; Mubofu, E. B.; Abderrahim, S.; Clark, J. H.; Koutinas, A. A.; Kopsahelis, N.; Stamatelatou, K.; Dickson, F. Food waste as a valuable resource for the production of chemicals, materials and fuels. Current situation and global perspective. *Energy Environ. Sci.* **2013**, *6*, 426−464.

(17) Uncu, O. N.; Cekmecelioglu, D. Cost-effective approach to ethanol production and optimization by response surface methodology. *Waste Manage.* **2011**, *31*, 636−643.

(18) Kim, J. K.; Oh, B. R.; Chun, Y. N.; Kim, S. W. Effects of temperature and hydraulic retention time on anaerobic digestion of food waste. *J. Biosci. Bioeng.* **2006**, *102*, 328−332.

(19) Lee, D.-Y.; Ebie, Y.; Xu, K.-Q.; Li, Y.-Y.; Inamori, Y. Continuous H_2 and CH_4 production from high-solid food waste in the two-stage thermophilic fermentation process with the recirculation of digester sludge. *Bioresour. Technol.* **2010**, *101*, S42−S47.

(20) Ma, J.; Duong, T. H.; Smits, M.; Verstraete, W.; Carballa, M. Enhanced biomethanation of kitchen waste by different pre-treatments. *Bioresour. Technol.* **2011**, *102*, 592−599.

(21) Nakamura, Y.; Sawada, T. Ethanol production from artificial domestic household waste solubilized by steam explosion. *Biotechnol. Bioprocess Eng.* **2003**, *8*, 205−209.

(22) Kim, J. H.; Lee, J. C.; Pak, D. Feasibility of producing ethanol from food waste. *Waste Manage.* **2011**, *31*, 2121−2125.

(23) Shihadeh, J. K.; Huang, H.; Rausch, K. D.; Tumbleson, M. E.; Singh, V. Design of a vacuum flashing system for high-solids fermentation of corn. *Trans. ASABE* **2013**, *56*, 1441−1447.

(24) Wang, S.; Ingledew, W.; Thomas, K.; Sosulski, K.; Sosulski, F. Optimization of fermentation temperature and mash specific gravity for fuel alcohol production. *Cereal Chem.* **1999**, *76*, 82−86.

(25) da Silva, F. L. H.; Rodrigues, M. I.; Maugeri, F. Dynamic modelling, simulation and optimization of an extractive continuous alcoholic fermentation process. *J. Chem. Technol. Biotechnol.* **1999**, *74*, 176−182.

(26) Shihadeh, J. K.; Huang, H.; Rausch, K. D.; Tumbleson, M. E.; Singh, V. Vacuum stripping of ethanol during high solids fermentation of corn. *Appl. Biochem. Biotechnol.* **2014**, *173*, 486−500.

(27) Mariano, A. P.; Qureshi, N.; Ezeji, T. C. Bioproduction of butanol in bioreactors: New insights from simultaneous in situ butanol recovery to eliminate product toxicity. *Biotechnol. Bioeng.* **2011**, *108*, 1757−1765.

(28) Wang, P.; Singh, V.; Xue, H.; Johnston, D. B.; Rausch, K. D.; Tumbleson, M. Comparison of raw starch hydrolyzing enzyme with conventional liquefaction and saccharification enzymes in dry-grind corn processing. *Cereal Chem.* **2007**, *84*, 10−14.

(29) Yan, S.; Li, J.; Chen, X.; Wu, J.; Wang, P.; Ye, J.; Yao, J. Enzymatical hydrolysis of food waste and ethanol production from the hydrolysate. *Renewable Energy* **2011**, *36*, 1259−1265.

(30) AACC International. Methods 44-19. *Approved Methods of the American Association of Cereal Chemists*, 10th ed.; AACC International: St. Paul, MN, 2000.

(31) Vidal, B. C.; Rausch, K. D.; Tumbleson, M. E.; Singh, V. Determining corn germ and pericarp residual starch by acid hydrolysis. *Cereal Chem.* **2009**, *86*, 133−135.

(32) John Morris Scientific. *Neutral Detergent Fiber in Feeds—Filter Bag Technique*; John Morris Scientific: Sydney, New South Wales, Australia, 2003; http://www.johnmorris.com.au/files/product/attachments/4469/68518_opt2.pdf (accessed Oct 2014).

(33) Dien, B. S.; Sarath, G.; Pedersen, J. F.; Sattler, S. E.; Chen, H.; Funnell-Harris, D. L.; Nichols, N. N.; Cotta, M. A. Improved sugar conversion and ethanol yield for forage sorghum (*Sorghum bicolor* L. Moench) lines with reduced lignin contents. *BioEnergy Res.* **2009**, *2*, 153−164.

(34) Huang, H.; Liu, W.; Singh, V.; Danao, M. C.; Eckhoff, S. R. Effect of corn maturity on dry-grind fermentation characteristics and DDGS composition. *Cereal Chem.* **2012**, *89*, 217−221.

(35) Mariano, A. P.; Qureshi, N.; Filho, R. M.; Ezeji, T. C. Assessment of in situ butanol recovery by vacuum during acetone butanol ethanol (ABE) fermentation. *J. Chem. Technol. Biotechnol.* **2012**, *87*, 334−340.

(36) Qureshi, N.; Saha, B. C.; Hector, R. E.; Hughes, S.; Cotta, M. A. Butanol production from wheat straw by simultaneous saccharification and fermentation using *Clostridium beijerinckii*: Part 1—Batch fermentation. *Biomass Bioenergy* **2008**, *32*, 168−175.

DOI: 10.1021/jf5054029
J. Agric. Food Chem. 2015, 63, 2760−2766

(37) Russell, I. Understand yeast fundamentals. In *Alcohol Textbook: A Reference for the Beverage, Fuel and Industrial Alcohol Industries*, 4th ed.; Jacques, K. A., Lyons, T. P., Kelsall, D. R., Eds.; Nottingham University Press: Nottingham, U.K., 2003; Vol. 9, pp 85−120.
(38) Maiorella, B.; Blanch, H. W.; Wilke, C. R. By-product inhibition effects on ethanolic fermentation by *Saccharomyces cerevisiae*. *Biotechnol. Bioeng.* **1983**, *25*, 103−121.
(39) Kim, J. K.; Oh, B. R.; Shin, H.-J.; Eom, C.-Y.; Kim, S. W. Statistical optimization of enzymatic saccharification and ethanol fermentation using food waste. *Process Biochem.* **2008**, *43*, 1308−1312.

DOI: 10.1021/jf5054029
J. Agric. Food Chem. 2015, 63, 2760−2766