Application of Inquiry-Based Learning in Summer Engineering Programs: Acid Hydrolysis of Sugars from Biomass

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Introduction

Inquiry-based learning is a teaching method that emphasizes the use and development of experimental and analytical skills over rote memorization of facts. This teaching method is well-suited to the sciences in general and particularly to engineering, where the application of theoretical knowledge to the optimization of desired results through investigation is a requirement for success in the field. The different levels of inquiry-based learning can be used to tailor an experiment within a selected engineering field to differing student ages and needs.[1]

One step in the process of the production of fuel alcohols from biomass, the hydrolysis of complex carbohydrates to obtain sugars for fermentation, provides a good introduction for students interested in chemical engineering and lends itself to an inquiry-based learning approach. Sugar recovery from starch, hemicellulose and cellulose is rather difficult because each of these carbohydrates hydrolyzes at different rates and the resulting sugars may degrade to furans under acidic conditions. This experiment has been tailored to provide a learning experience for students participating in 10^{th}-12^{th} grade high-school engineering summer workshops at the University of Arkansas and has also been incorporated into the chemical engineering department's Lab I curriculum. This paper focuses on the experiment as performed in the summer workshops.

Students were exposed to background information on the need for alternative energies, the economic issues involved in biofuel development, and the relevant portions of the process of conversion of cellulosic feedstocks into biofuels. The experiment was based on guided inquiry, in which only a question is provided and the students are allowed to develop their own experimental procedures and find a range of possible solutions.[1] The question provided was, "What is the best way to hydrolyze the given feedstock in order to extract the greatest quantities of glucose for later fermentation into fuel alcohols?" The students were then divided into groups, and allotted time to perform literature searches and develop experimental procedures for approval.

Experiment Development

Examples of student methods for algae conversion and switchgrass conversion are provided in Table 1. Reducing sugars were measured using a 3,5-dinitrosalicylic acid reduction, which caused a color change in each sample commensurate with its total amount of reducing sugars.

Table 1. Engineering Student Academy hydrolysis methods by group

2012 – Switchgrass			
Group	**Physical Pretreatment**	**Treatment**	**Yield (g sugar/g switchgrass)**
1	Blender grinding Mortar and pestle	Boiling 40 min, 0.5% H_2SO_4	0.006
2	Blender grinding Mortar and pestle	80 °C 10 min, 60 °C 10 min, 3% NaOH	0.006
3	Scissors Blender grinding	Boiling 10 min, 0.75% H_2SO_4	0.007
4	Blender grinding Mortar and pestle	Boiling 40 min, 0.5% H_2SO_4	0.013
5	Blender grinding Mortar and pestle	Boiling 1 hr, 4% H_2SO_4	0.034

Results

Hydrolysis of algal cell walls for sugar recovery requires grinding and the application of heat and pressure in an acidic solution, in order to depolymerize the available cellulose. The procedure developed at the University of Arkansas for research purposes is to grind the algae, then soak it in a 2% sulfuric acid solution in an autoclave at 121 °C for 30 min for a yield of 1.4 g/L, or approximately 0.02 g glucose/g algae.[2] Switchgrass contains lignin in addition to cellulose and hemicellulose, which significantly increases the processing required to recover sugars. Keshwani and Cheng[3] recommend a dilute acid and ammonia percolation pretreatment to break down the lignin bonds in switchgrass and increase sugar recovery; using enzymatic treatment methods, they report a 70%-93% conversion of cellulose.

Student Feedback

Students in the workshops were in agreement that the experiment was helpful in furthering their understanding of the process of producing fuel alcohols from biomass, as indicated by survey results. Additionally, their responses on the surveys showed that they better understood the need for alternative energies and the economic considerations involved. Most students noted an increased interest in chemical engineering and energy development, even as many of the recorded comments in the surveys showed that students were surprised by the difficulty level and amount of work required to successfully conduct the experiment.

Future Development

Future designs of this experiment would incorporate an increased emphasis on the economic consequences of their choices for their experimental procedure. Different methods and chemicals would have monetary values assigned; the students would need to consider trade-offs between the most effective method for sugar extraction versus the most cost-effective method. At the university level, the students could perform multiple experiments, changing their methodology to optimize their results.

Bibliography

1. Banchi, H.; Bell, R. The Many Levels of Inquiry. *Science and Children*, Oct 2008, pp 26-29.
2. Potts, T.; Du, J.; Paul, M.; May, P.; Beitle, R.; Hestekin, J. The production of butanol from Jamaica bay macro algae. *Environ. Prog. Sustainable Energy*, **2012**, *31(1)*, 29-36.
3. Keshwani, D.; Cheng, J. Switchgrass for bioethanol and other value-added applications: a review. *Bioresour. Technol.*, **2009**, *100*, 1515-1523.

Biography

Amy McGraw is an undergraduate senior chemical engineering student at the University of Arkansas. Her interests include research into alternative fuels, environmental protection, and the encouragement of student interest in STEM fields.
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Dr. Ed Clausen serves as Professor, Associate Department Head and the Ray C. Adam Endowed Chair in Chemical Engineering at the University of Arkansas. His research interests include bioprocess engineering, the production of energy and chemicals from biomass and waste, and enhancement of the K-12 educational experience. Email: eclause@uark.edu

Dr. Jamie Hestekin is an Associate Professor at the University of Arkansas. His research groups are focused on applications of charged separations, mainly using wafer-enhanced electrodeionization; and the extraction of oils and carbohydrates from algae for eventual production into biodiesel and butanol, and related applications. Email: jhesteki@uark.edu

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Introduction

Inquiry-based learning is a teaching method that emphasizes the use and development of experimental and analytical skills over rote memorization of facts.

	Inquiry Level	Question	Procedure	Solution
1	Confirmation Inquiry *Students confirm a principle through an activity when the results are known in advance*	x	x	x
2	Structured Inquiry *Students investigate a teacher-presented question through a prescribed procedure*	x	x	
3	Guided Inquiry *Students investigate a teacher-presented question using student designed/ selected procedures*	x		
4	Open Inquiry *Students investigate questions that are student formulated through student designed/selected procedures*			

Figure 1. The four levels of inquiry and the information provided to the student in each level.[1]

The production of fuel alcohols such as ethanol and butanol from cellulosic feedstocks is a multi-step process.

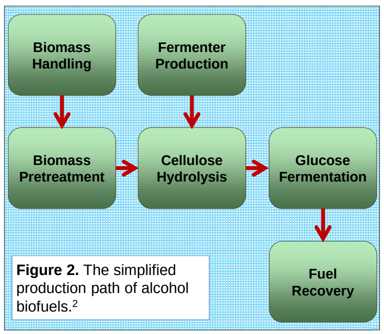

Figure 2. The simplified production path of alcohol biofuels.[2]

One step in the process is the recovery of sugars from starch, hemicellulose and cellulose. Each of these carbohydrates hydrolyzes at different rates, and the resulting sugars may degrade to furans under acidic conditions.

Figure 3. Sample of switchgrass hydrolyzing in a heated acidic solution.

The hydrolysis step provides a good introduction to alternative fuel production and the chemical engineering experience, when presented as a guided inquiry experiment.

Guided Inquiry

This experiment was tailored for 10th-12th grade students participating in the 2010-2012 Engineering Summer Academy workshops at the University of Arkansas. Students were exposed to background information on the need for alternative energy sources, the step-by-step process of producing them, the economic and environmental issues involved. They were then asked the question:

What is the best way to hydrolyze the feedstock provided in order to extract the greatest quantities of glucose for later fermentation into fuel alcohols?

Divided into groups, the students were given time to perform literature searches for information and develop experimental procedures for approval. It was suggested to the students that they explore physical, thermodynamic and chemical means of depolymerizing the starches, cellulose and hemicellulose. When their methods were approved, they were given 20 g of *Ulva lactuca* algae (2010, 2011) or 20 g of switchgrass (2012).

Figure 4. 20 g sample of switchgrass for student experimentation.

Experiment Development

Student methods for algal and switchgrass hydrolysis and their results are provided in Table 1. Reducing sugars were measured using a 3,5-dinitrosalicylic acid reduction, which caused a color change in each sample commensurate with its total amount of reducing sugars. These color changes were compared to a set of glucose solutions of standard concentration.

Table 1. Engineering Student Academy hydrolysis methods by group

2011 – *Ulva lactuca* algae			
Group	Physical Pretreatment	Treatment	Yield (g sugar/g algae)
1	Mortar and pestle	80 °C 30 min, agitation, 2% H_2SO_4	0.066
2	Mortar and pestle Blender grinding	Boiling 5 min, cooling to 23 °C, 2% H_2SO_4	0.023
3	Mortar and pestle	60 °C 30 min, 1% H_2SO_4	0.013
4	Mortar and pestle	70 °C 45 min, 0.75% H_2SO_4	0.014
5	Blender grinding Sonication 5 min	50 °C 10 min, 1% H_2SO_4	0.109
6	Mortar and pestle	60 °C 30 min, 2% HCl	0.060
2012 – Switchgrass			
Group	Physical Pretreatment	Treatment	Yield (g sugar/g switchgrass)
1	Blender grinding Mortar and pestle	Boiling 40 min, 0.5% H_2SO_4	0.006
2	Blender grinding Mortar and pestle	80 °C 10 min, 60 °C 10 min, 3% NaOH	0.006
3	Scissors Blender grinding	Boiling 10 min, 0.75% H_2SO_4	0.007
4	Blender grinding Mortar and pestle	Boiling 40 min, 0.5% H_2SO_4	0.013
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Figure 5. Physical pretreatment of switchgrass using blender grinding.

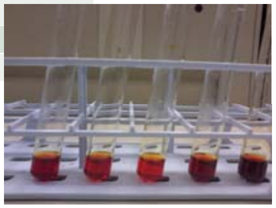

Figure 6. Samples treated with 3,5-dinitrosalicylic acid. Note the differing color changes.

Results

Algal hydrolysis at the University of Arkansas involves grinding the algae, then soaking it in a 2% H2SO4 solution in a 121 ° C autoclave for 30 minutes, for a yield of 1.4 g/L or 0.02 g sugar/g algae.[3] Keshwani and Cheng[4] recommend a dilute acid and ammonia percolation pretreatment, followed by an enzymatic treatment, and report cellulose-to-sugar conversions of 70%-93%.

Student Feedback

Students were provided with the survey questions shown in Table 2, and their responses recorded.

Table 2. ESA survey responses for 2010 and 2012.

Questions	SA	A	N	D	SD	Total
The presentation on energy and chemical engineering was useful and informative	16	18				34
I understand the need to develop alternative energy sources and what will be required to develop them	26	6	1			33
The biomass to energy exercise was useful and informative	21	13				34
I understand the need to include economic considerations when developing alternative energy sources	23	11				34
Energy as a part of the chemical engineering profession is of interest to me	10	16	6	1	1	34

Comments on the surveys indicated an increased interest in chemical engineering and energy development, even as the students were surprised by the difficulty level and amount of work required to conduct the experiment.

Future Development

Future versions of this experiment would incorporate an increased emphasis on the economic consequences for each treatment choice. Different methods would have monetary values assigned, and students would need to consider effectiveness versus cost effectiveness of their chosen method.

Bibliography

1. Banchi, H.; Bell, R. The Many Levels of Inquiry. *Science and Children*, Oct 2008, pp 26-29.
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