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Laboratory Experiment

Direct Catalytic Conversion of Lignocellulosic Biomass Wastes to Ethylene Glycol: Practical Learning for Chemical Engineering Students

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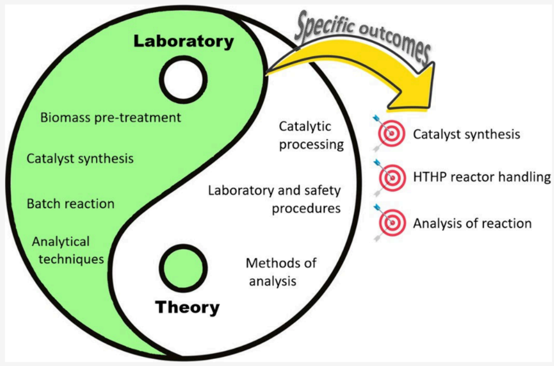
ABSTRACT: This experiment is specifically designed for Chemical Engineering students to comprehend the catalytic process of biomass residue direct conversion to ethylene glycol through practical procedures and enhance their knowledge of hydrogenation reactions and catalysts preparation. More importantly, this laboratory experiment can enable Chemical Engineering students to understand the catalytic reaction mechanism of lignocellulosic biomass conversion and learn various techniques, for example, carbon nanotube (CNT) supported Ni−W catalyst preparation, substrate pretreatment, and product analysis. As an extension to this work, students could additionally characterize the prepared catalyst using different methods such as N_2 adsorption, temperature-programmed reduction, inductively coupled plasma analysis, and thermogravimetry to discuss the experimental catalytic results. Then, the students can be assessed based on their participation, experimental performance (e.g., catalyst preparation, catalytic conversion, product analysis), and discussion of results. This experiment aims to encourage students to link their understanding of chemical engineering with practical operation, giving extra motivation to carry out more research in the area of biomass selective catalytic hydrogenation.



KEYWORDS: *Upper-Division Undergraduate, Graduate Education/Research, Chemical Engineering, Interdisciplinary/Multidisciplinary, Laboratory Instruction, Safety/Hazards, Hands-on Learning, Catalysis, Instrumental Methods, Reactions*

■ INTRODUCTION

Heterogeneous catalysis and biomass valorization are two main pillars of today's society, which are clearly translated to academic research; for example, searching for "heterogeneous catalysis" and "biomass conversion" in Web of Science generates more than 51,000 and 64,000 papers, respectively. Thus, these areas are of great interest to Chemical Engineers. In fact, over the past years, many works have been published studying the catalytic conversion of biomass to valuable chemicals, focusing on the development of efficient and stable catalysts, study of reaction mechanisms, and optimization of reaction conditions.[1−8] However, from all those more than 115,000 papers above-mentioned, only 92 (61 for "heterogeneous catalysis" and 31 for "biomass conversion") are categorized as educational ("Education Educational Research" or "Education Scientific Disciplines"),[9−24] which indicates that research and education are generally divided as two different matters. According to these numbers, there is very limited research on educational practices considering biomass conversion and heterogeneous catalysis. Nevertheless, many Chemical Engineering students study these subjects and might work in this field later in their careers. Besides, many of the experiments related to the catalytic conversion of biomass to chemicals are relatively simple and suitable for Chemical Engineering students to perform. In fact, the opportunity to conduct such experiments would be of great significance for students, through hands-on experiments, analyzing data and drawing conclusions. This allows one to acquire procedural knowledge (knowledge of how to do a determined task or "know-how"), which is of paramount importance for a Chemical Engineer. Some works have already been suggested considering catalyst synthesis, characterization, and evaluation in a pedagogical point of view,[9−11] while others reported the valorization of biomass and wastes for diverse applications.[12−14] For example, Guo et al. presented an undergraduate laboratory experiment focusing on the synthesis of Ce/TiO_2 catalysts for selective catalytic reduction of NO,[9] while Li et al. reported a laboratory experiment based on the synthesis, characterization, and activity evaluation of Pt/Al_2O_3 catalysts in soot oxidation.[10] On the other hand, Mascal and Scown

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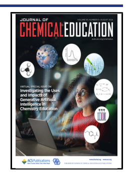

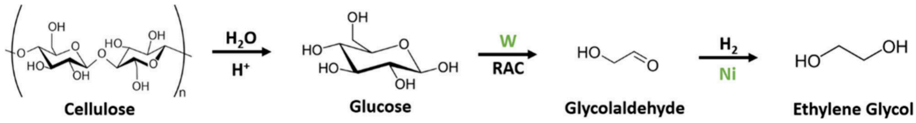

Figure 1. Reaction process for catalytic conversion of cellulose to EG.

reported a laboratory experiment for converting newspaper into ethanol,[12] while de Dios-Pérez et al. focused on the valorization of olive leaves for the extraction of polyphenols.[13] Finally, Zhou et al. developed a laboratory experiment to introduce students to integrated biorefinery concepts, through an integrated conversion of corncob holocellulose and lignin into biodiesel, bioethanol, and sunscreen.[14] Although advancements have continuously been made to prioritize novel education methods and shift the emphasis toward the laboratory, instructional experiments involving biomass catalytic conversion have not yet been discussed, as far as we are concerned. Therefore, a laboratory experiment combining multidisciplinary knowledge is herein presented, with the goal of stimulating laboratory proficiency and Chemical Engineering students' curiosity and passion for research.

■ CONTEXTUALIZATION

The rapid increase of environmental pollution and fossil resources consumption has enforced the development of new strategies for exploring renewable sources for the production of valuable chemicals and fuels.[3] The International Energy Agency defines "Biorefining" as "the sustainable processing of biomass into a spectrum of biobased products (food, feed, chemicals, materials) and bioenergy (biofuels, power and/or heat)".[25] In fact, applying biomass as biorefinery feedstocks has several benefits besides producing biofuels and valuable chemicals, such as reducing the negative environmental impacts of these residues when disposed of and generating greenhouse gases.[26] In this sense, the biorefinery concept shows up as an alternative to environmental pollution and fast fossil fuels depletion caused by petroleum-based refineries, aiming to attain major goals like: (i) pollution minimization through biomass waste valorization, (ii) meeting the energy needs of the increasing population through a constant fuel supply, and (iii) creating local, national, and international employment opportunities.[26] So, lignocellulosic biomass is one of the renewable sources that has been attracting a great deal of attention worldwide.[27,28] However, despite its potential as a feedstock, lignocellulosic resources are still underutilized. Nevertheless, lignocellulosic biomass is expected to play a large role in carbon emission mitigation,[4] and its valorization into value added products such as ethylene glycol (EG) is one of the most desired processes, since EG is widely used as a monomer in polymer synthesis and as antifreeze in automotive industries and has an extensive market demand for pharmaceuticals and cosmetics development.[29−33] Cellulose is the main component of lignocellulosic biomass, and its direct conversion to EG consists in three subsequent steps: (i) hydrolysis of cellulose to glucose, promoted by protons from hot water or from an acidic environment; (ii) retro-aldol condensation (RAC) of glucose to glycolaldehyde (GA), promoted by tungsten species; and (iii) hydrogenation of GA to EG, promoted by hydrogenation active sites (Figure 1).[33−35] Through this laboratory experiment, students will be able to learn this process and hopefully raise their interest toward this current research topic.

■ PEDAGOGICAL SIGNIFICANCE

This experiment can help assimilate the fundamentals of two different courses: the catalyst evaluation as part of the Reaction Engineering course and the identification and quantification of the products obtained during catalyst screening as part of Instrumental Methods of Analysis. At this level, multidisciplinary integration of previously acquired knowledge is essential to conduct this process successfully. This work can also be an opportunity to highlight the interconnectedness of distinct areas of Chemical Engineering, illustrating that they are intricately linked and cannot be effectively addressed separately in interpreting real-world processes. Accordingly, the learning objectives associated with this laboratory experiment are: (i) reflect on and apply the correct safety procedures for working with high-temperature and high-pressure (HTHP) batch reactor at laboratory scale; (ii) operate a hydrogenation lab unit and identify its components; (iii) identify the necessary product analyses and integrate multiple analysis techniques; (iv) analyze, qualitatively and quantitatively, the conversion and products yields; (v) make informed decisions regarding materials and sample preparation; (vi) plan laboratory work and engage in teamwork. The final goal of this project is to interpret the qualitative and quantitative results obtained, calculate substrate conversion and product yields, and present all of the results in a final report. As a consequence, additional learning objectives should be attained, like catalysts' role and characteristics, understanding diverse analysis techniques, etc.

■ EXPERIMENTAL SECTION

Pretreatment of Substrates

Besides cellulose, three residues were selected as substrates for this work (i.e., urban, agricultural, and forestry) to provide the students with a more diverse experience. Accordingly, cotton wool, corncob, and eucalyptus wood were the selected urban, agricultural, and forestry wastes, respectively, since these materials had previously attained a higher yield of EG compared to other residues of the same kind.[1] Cellulose and cotton wool were used as purchased. Eucalyptus wood and corncob were initially cut and crushed in the appropriate apparatus and subsequently dried overnight in a furnace at 100 °C.

According to our previous study, to reduce the substrate crystallinity and facilitate the initial hydrolysis step, each substrate was ball-milled for 4 h at a frequency of 20 s^{-1} prior to reaction in a Retsch Mixer Mill MM200 equipped with two ceramic pots of 10 mL, each one loaded with two ZrO_2 balls (12 mm of diameter) (Figure 2).[36] If ball-milling equipment is unavailable, the students could alternatively use a mortar and pestle to pretreat cellulose. To perform the catalyst evaluation, the students should select one of these three ball-milled residues (cotton, corncob, or eucalyptus) to pretreat and convert.

Figure 2. Ball-milling equipment and ceramic pot with ZrO_2 balls.

Catalyst Preparation

Considering our previous study, the Ni−W/carbon nanotube (CNT) catalyst (nominal Ni and W metal loadings of 20 wt %) used in this work was prepared by incipient wetness impregnation of commercial CNT with a solution of both Ni and W precursors.[37] Herein, we suggest this catalyst, since it is one of the carbon-supported metal catalysts that reaches higher yields of EG among those reported in the literature.[37] Furthermore, this catalyst has been shown to be relatively stable after repeated use for at least six successive runs. Yet, other less expensive alternatives (e.g., activated carbon) could be considered for learning purposes. The synthesis procedure is detailed in Supporting Information (cf. Catalyst Preparation Section).

After impregnation, the material is thermally treated for 3 h under a N_2 flow (100 cm^3 min^{-1}, STP) and reduced for 3 h under a H_2 flow (100 cm^3 min^{-1}, STP) in a vertical oven (Figure 3). The reduction temperature of 500 °C was selected based on temperature-programmed reduction (TPR) results (Figure S4), and the thermal treatment is carried out at the same temperature. Due to the high temperatures involved, this process must be periodically checked and not left unattended. Also, the students must be accompanied by a supervisor.

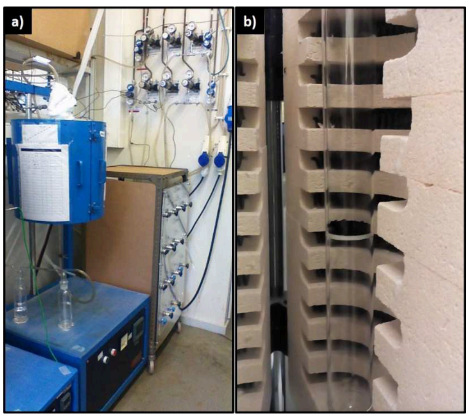

Figure 3. (a) Oven used for thermal treatment and reduction of the catalyst and (b) quartz reactor with catalyst placed in the center of the oven.

Catalytic Tests and Product Analysis

The catalytic experiments were performed in a 1000 mL stainless-steel Parr reactor (USA Mod. 5120) (Figure 4), equipped with a gas supply system, a manometer, a temperature sensor, and a filtered sample outlet, preventing the catalyst particles from passing through it. A schematic representation of the experimental setup and the detailed experimental procedure can be found in the Supporting Information (Figure S1 and Catalytic Tests Section).

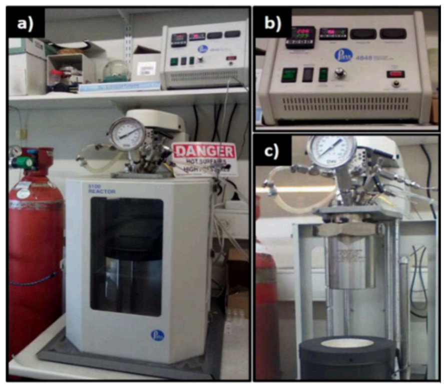

Figure 4. (a) Experimental setup used for catalytic evaluation, (b) temperature and stirring rate controller, and (c) stainless-steel reactor.

The liquid products were analyzed by high performance liquid chromatography (HPLC) using a refractive index detector. The different products were separated using an Alltech OA-1000 ion exclusion column (300 × 6.5 mm) with a 5 mmol L^{-1} H_2SO_4 mobile phase at 0.5 mL min^{-1}. An example of a chromatogram that can be obtained and the product retention times can be found in Figure S2 and Table S2, respectively.

The yield (Y_i) of each product i can be calculated according to eq 1, and the conversion (X) of nonsoluble cellulose can be determined on the basis of total organic carbon (TOC) data (Shimadzu TOC-L) using eq 2:

$$Y_i\,(\%) = [\text{moles of product } i \text{ formed (measured by HPLC)} \times \text{C atoms in product } i]/[\text{moles of C in the substrate initially present}] \times 100\% \tag{1}$$

$$X\,(\%) = [\text{moles of total organic carbon in the resultant liquid}]/[\text{moles of carbon in the substrate charged into the reactor}] \times 100\% \tag{2}$$

For the calculations, consider that EG can only be produced from cellulose and hemicellulose (holocellulose), the composition being previously determined (Table 1),[1] and cotton is practically only composed of cellulose. If a TOC analyzer is unavailable, the students could alternatively estimate the substrate conversion by weight difference from the beginning and the end of the reaction.

Table 1. Dry Basis Component Analysis Results for the Lignocellulosic Materials and the Corresponding Standard Deviations

	Component (wt %)		
Sample	Holocellulose	Lignin	Extractives
Eucalyptus wood	61.9 ± 0.7	25.7 ± 1.2	6.4 ± 2.8
Corncob	59.3 ± 1.8	27.6 ± 4.0	5.8 ± 5.3

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■ HAZARDS AND SAFETY

Some chemicals used in this work can be potentially hazardous and should be handled with caution (detailed information in the Supporting Information).

To ensure safety, students must use protective gear during the entire experiment, including a lab coat, safety gloves, goggles, and mask. In the case of inappropriate exposure to chemicals, it is fundamental to clean with plenty of water. At least one graduate instructor or teacher must be in the laboratory, while the student is conducting the experiment.

■ EXAMPLE OF EXPERIMENTAL RESULTS AND DISCUSSION

Figure 5 depicts the results obtained for the conversion of cellulose, cotton wool, corncob, and eucalyptus wood, as well as the respective yield of produced EG.[37] Reproducibility tests were performed in triplicate, and the results showed standard deviations of less than 6% and 2% for conversion and yield, respectively.[37] In Figure 5a, it is possible to notice that cellulose can be completely converted after 3 h of the reaction.

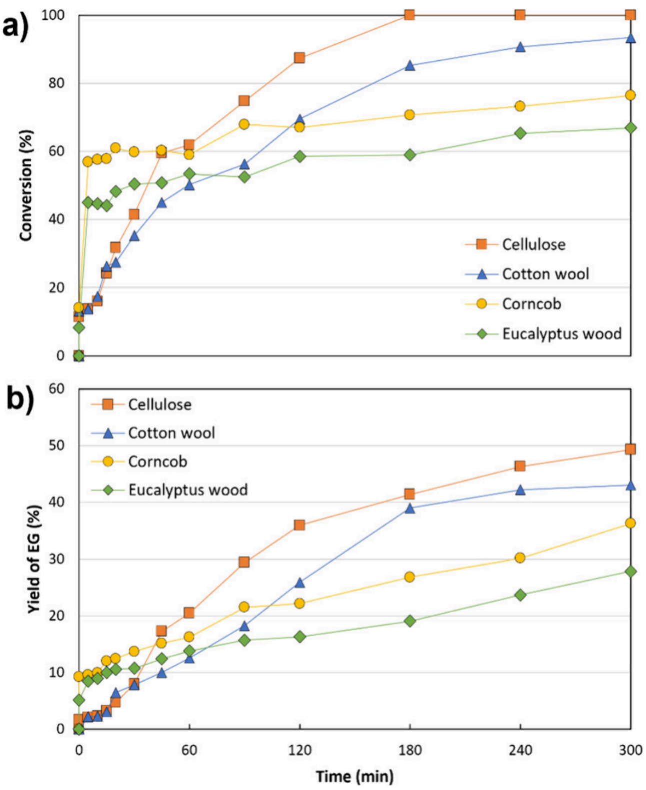

Figure 5. (a) Biomass conversion and (b) EG yield evolution with the reaction time.

On the other hand, the three biowastes do not get fully converted within 5 h of the reaction, cotton wool being the one that gets closer to a complete conversion since it is mainly composed of cellulose, unlike eucalyptus wood and corncob that contain a substantial amount of lignin (Table 1). In addition, concerning EG production, the behavior is similar. A yield of EG of around 50% could be attained from cellulose, followed by cotton wool that produced more than 40% of EG. Despite their lignocellulosic composition, eucalyptus wood and corncob could also be transformed to EG with yields of 28% and 36%, respectively. Besides EG, the students should also be able to identify and quantify the remaining reaction products (Table S3).

■ EXTENSION

To enrich the project for larger programs, characterization of the prepared catalyst could be carried out. The respective experimental procedures and characterization results of this section are presented in the Supporting Information (cf. Extension Section). Additionally, the students could also explore the impact of catalyst recyclability by further testing the recovered catalyst sample.

■ LEARNING OUTCOMES AND ASSESSMENT

Undergraduate students from different education levels (38.5% BSc, 30.8% MSc, and 30.8% PhD) have already been enrolled in this laboratory experiment since 2013, of which 23.1% were international students. The work has not been divided among the students, but each student worked individually and learned to perform each of the proposed tasks on his/her own. During the experiments, the students received direction and help from colleagues and supervisors. Moreover, the students were encouraged to raise awareness about the safety operation of HTHP reactors and chemical handling and became familiar with the process of biomass pretreatment, catalyst synthesis, and biomass conversion. Furthermore, no safety issues arose in this experiment. At the end of the work, the students were questioned about their satisfaction of the work and whether it met, exceeded, or failed their expectations. In general, the students considered the experiment intensive but valuable for their formation and learning process.

The students' assessment was based on their preparation for the work (preliminary study of the topic), laboratory performance (e.g., safety aspects; do they understand the experimental setup such as valve positions/role? do they consider leak tests?), and final report (Table 2). The table of contents of the final report is presented in Figure S8. Laboratory participation, such as punctuality, attitude, and interest, was also considered. The PhD students have a qualitative classification: do not pass or pass. On the other

Table 2. Main Learning Outcomes and Demonstration of Students' Achievement

	Learning Outcome	Achievement Demonstration
General	Search and analyze existing literature	Perform a literature review to understand the topic of biomass catalytic conversion to EG
	Follow proper procedures for safe handling of chemicals and equipment	Use appropriate personal protective equipment, appropriate handling of chemicals, gas leak detection, etc.
	Effectively and clearly communicate technical information	Write a final report presenting the experiment background and results obtained
Specific	Catalyst synthesis	Prepare a carbon-supported metal catalyst
	HTHP reactor handling	Identify the reactor components and safety issues
	Reaction analysis	Identify and quantify reaction products by HPLC and calculate conversions and yields

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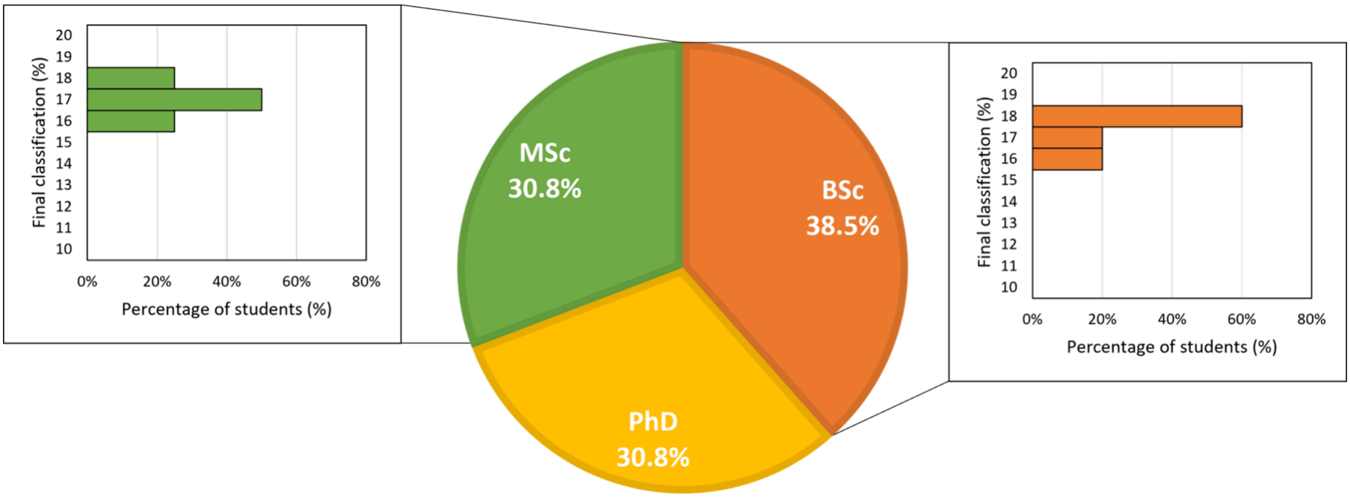

Figure 6. Final score of the students from different graduation levels.

hand, BSc and MSc students are scored from 0 to 20. The results obtained to date are presented in Figure 6.

Since the students conducted an initial literature review to understand the topic of biomass catalytic conversion to EG, they were asked to compare their results with the existing literature in the final report. Besides that, another suggestion would be to extend it into a discussion of differences between the laboratory and industrial performances.

Furthermore, this experiment is regularly shown to high-school students who visit our laboratory facilities. In fact, our last visit was from the program "Erasmus+ − Learn From the Past to Build a Greener Future", and some of the comments collected on this last visit (May 2023) were the following:

- From the accompanying teachers: "Laboratory about biomass: The best according to our students. The researcher presenting it was so enthusiastic and with her whole heart into her research. She was both emotional and professional at the same time. She made an impression that her research was something she strongly believed in."
- From the students: "We loved the activities the university representatives prepared and carried out for us. Personally, I think they were the activities which most related to the theme to our project, at least the part with green energy. We particularly loved the explanations about biomass and its uses."

CONCLUSIONS

This multidisciplinary laboratory experiment is tailored for Chemical Engineering students and focuses on the hydrolytic hydrogenation of biowastes into EG. Throughout this experiment, students can learn the basic methods for the preparation of a supported heterogeneous catalyst and biomass substrates, understand how to operate a HTHP batch reactor, and obtain knowledge of how to apply instrumental techniques, which lays a foundation for their future career. It is noteworthy to clarify that the aim of this work is not to grade students but to foster their experimental abilities, provide hands-on experience, encourage them to critically analyze their data, and ultimately prepare them for future research in the area of biomass catalytic conversion and industry work.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.4c00527.

Additional experimental details, materials, and methods, including photographs of experimental setup, procedures, and results (PDF; DOCX)

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Notes

The authors declare no competing financial interest.

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