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Laboratory Experiment

A Simplified Haber-Bosch Experiment for the Chemistry Laboratory: A Low-Cost Approach to Teaching Ammonia Synthesis and Green Hydrogen Concepts

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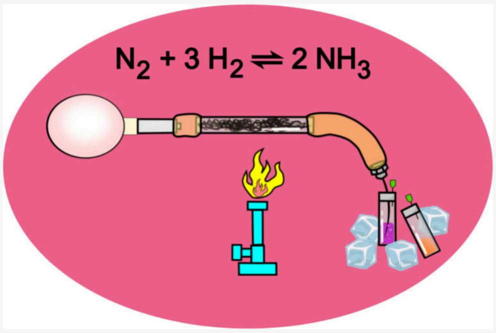
ABSTRACT: Reducing greenhouse gas emissions is one of the major global challenges and involves rethinking established industrial processes. Ammonia synthesis using the Haber-Bosch process is fundamental to modern agriculture, but it is also one of the largest sources of CO_2 emissions. This is mainly due to one of the steps in the process in which hydrogen is obtained from fossil fuels. A promising alternative is to replace this step with the use of green hydrogen, obtained by water splitting with renewable energy sources. However, accessible experimental practices addressing this topic are still limited. Here, we describe a low-cost experiment in which students produce hydrogen by water splitting and use it, in combination with nitrogen, to simulate ammonia synthesis by a simplified Haber-Bosch process. This approach enables students to explore the concept of green hydrogen as a more sustainable alternative for industrial hydrogen production. The reaction is conducted in a simple heated reactor, and the product is identified using a pH indicator. The experiment was developed for general chemistry courses and promotes an integrated discussion of fundamental concepts of thermodynamics, chemical equilibrium, kinetics, and catalysis, alongside contemporary issues related to sustainability, including the role of green hydrogen in reducing the carbon footprint of ammonia production.



KEYWORDS: *Undergraduate, Inorganic Chemistry, Laboratory Instruction, Ammonia Synthesis, Water Electrolysis, Catalysis, Sustainability, Green Chemistry.*

INTRODUCTION

Global warming has caused serious climatic and environmental imbalances. Extreme events, such as heat waves, prolonged droughts, and floods, have impacted food security and ecosystems. Reducing carbon dioxide (CO_2) emissions has become a global priority, requiring cleaner energy carriers and sustainable solutions for the energy transition.[1−4]

Among these solutions, hydrogen (H_2) stands out as a promising energy carrier due to its high gravimetric energy density (approximately three times higher than that of gasoline (143.0 kJ g^{-1} for H_2 versus 52.3 kJ g^{-1} for gasoline).[5] Moreover, its combustion produces only water.[6] However, the use of hydrogen as a fuel is still very limited. Nevertheless, almost 50% of the hydrogen produced worldwide is used to produce ammonia (NH_3) via the Haber-Bosch process.[7]

Ammonia is essential for synthesizing fertilizers and feeding the planet.[8] This simple molecule is so important that without industrial NH_3 we would produce food for only half the world's population, see the discussion in Supporting Information I (section 2) and Supporting Information II (section 1). However, there is still challenge regarding hydrogen production processes: 95% of produced H_2 comes from fossil fuels and is obtained from industrial processes that emit large amounts of CO_2, with steam methane reforming (SMR) accounting for 70–80% of H_2 production.[7,9] As a result, for each ton of H_2 generated, about 9–10 tons of CO_2 are emitted, representing roughly 1.5% of global greenhouse gas emissions.[10−12] On the other hand, green H_2 can be produced from water using renewable electricity, without direct CO_2 emission, see a discussion in the Supporting Information I (section 2) and Supporting Information II (section 1).[7]

Considering this context, the Haber-Bosch process can be considered one of the most polluting industrial processes on the planet.[8,9,13] However, it is important to understand that the success of the Haber-Bosch process, developed in the early 20th century, was its ability to increase agricultural production

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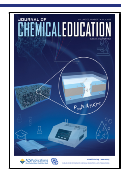

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by enabling the fixation of atmospheric nitrogen on an industrial scale.[14] In this context, the expression "fixation" refers to the conversion of molecular nitrogen (N_2) into ammonia, which is used as a fertilizer and as a feedstock for urea synthesis.[11,15] Microorganisms naturally present in the soil convert nitrogen compounds into plant-available form, particularly nitrate (NO_3^-), which is readily absorbed by plants. Therefore, the Haber-Bosch process attempts to mimic the nitrogen fixation carried out by nitrogen-fixing bacteria.[16,17]

Modern discussions around sustainability have reignited interest in integrating renewable technologies into this century-old process. In particular, the combination of water-splitting technologies for green hydrogen generation with the Haber–Bosch process could drastically reduce CO_2 emissions.[9,18] Beyond its technological relevance, this theme offers fertile ground for teaching core concepts in general chemistry such as kinetics, thermodynamics, chemical equilibrium, and catalysis. Therefore, ammonia synthesis can be approached as a Generating Theme (GT), from the pedagogical perspective of Paulo Freire (1921–1997), to explore academic content connected to social and technological issues.[19,20]

Most of the studies found in the literature about ammonia synthesis and Haber-Bosch process are theoretical, use virtual simulators or involve other experiments with ammonia. Among the few experimental initiatives related to the synthesis of NH_3 by the Haber-Bosch process, only few studies[21,22] provide practical work through complex instrumentation involving N_2 and H_2 gases. While these initiatives offer a quantitative and precise approach suitable for physical chemistry laboratories, our proposal prioritizes qualitative experiments that are more accessible, using alternative materials for the synthesis and identification of ammonia.

Therefore, the objective of this work is to present a low-cost and accessible laboratory experiment for undergraduate general chemistry courses. This laboratory experiment has been designed to support students connect core chemical concepts to contemporary sustainability challenges, particularly the role of chemistry in reducing greenhouse gas emissions associated with fertilizer production. Comprehensive pedagogical guidance and detailed chemical explanations are provided in two accompanying Supporting Information documents—one for instructors and another for students—facilitating classroom implementation and fostering active learning.

■ PEDAGOGICAL FRAMEWORK: GENERATIVE THEMES

The Generative Themes approach is grounded in the pedagogical framework of Paulo Freire, a Brazilian educator and philosopher whose work emphasized critical, dialogical, and contextualized learning. In contrast to traditional models centered on content transmission, Freire proposed a problem-posing education in which knowledge is constructed through the critical analysis of situations that are meaningful to students. Within this perspective, GT emerge from real-world contexts and function as starting points for inquiry, enabling students to engage with scientific concepts while reflecting on their social and environmental implications.[19,23,24]

In this work, ammonia synthesis is adopted as a GT to structure the learning sequence. The topic is introduced through its relevance to food production and its environmental impact, particularly the dependence on fossil-based hydrogen. From this starting point, students engage in a sequence of activities that allows fundamental general chemistry concepts to be explored in connection with contemporary challenges related to sustainability and energy transition (see a discussion in the Supporting Information I (section 1)). In this way, the GT serves as an organizing axis that integrates experimental practice with conceptual understanding and active learning.

■ EDUCATIONAL CONTEXT

The Integrated Chemistry Course

The Integrated Chemistry course, QG464 (Q = química (chemistry) G = geral (general)), is part of the undergraduate Chemistry curriculum at the State University of Campinas (Universidade Estadual de Campinas - UNICAMP), in Brazil. It is a general chemistry laboratory course with a weekly workload of 4 h, totaling 60 h throughout the semester. The course program focuses on interdisciplinary experiments that involve methods for preparing, characterizing, and analyzing the products of chemical reactions, and it is divided into four modules: (i) inorganic chemistry, (ii) organic chemistry; (iii) physical chemistry and (iv) analytical chemistry. Topics explored include, for example, the extraction of natural products such as fragrances and aromas, pharmaceutical and cosmetic formulations, agrochemical applications, biodiesel production, and the production of polyurethane foams, silicones, and hydrogen. The content may vary according to the professors responsible for each module or the partner company supporting the module theme.

Each course module is conducted by a professor specializing in one of the four areas of Chemistry. Students are divided into four groups (A, B, C, and D), which rotate through the modules over the semester—each group attends to classes of each module for three consecutive sessions and then moves on to the next module, until all four modules have been completed. Figure 1 illustrates the organization of this course.

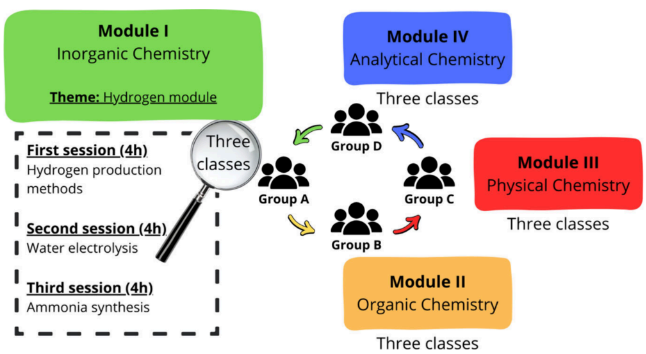

Figure 1. Schematic organization of course QG464. The students attend all modules during the semester.

Each module integrates theoretical study, prelaboratory activities, experimental practices, and postlaboratory tasks. Through the active learning methodology of the flipped classroom, students study theoretical content at home, reserving class time for discussions and practical applications.[25] Initially, they complete readings and prelaboratory exercises to consolidate theoretical knowledge. In the laboratory, practical activities are conducted collaboratively, and the results are discussed at the end of each session. After class, students complete postlaboratory tasks, analyzing the collected data and reflecting on the experimental conditions.

The inorganic chemistry module on hydrogen explores the properties, production methods, and applications. Before the

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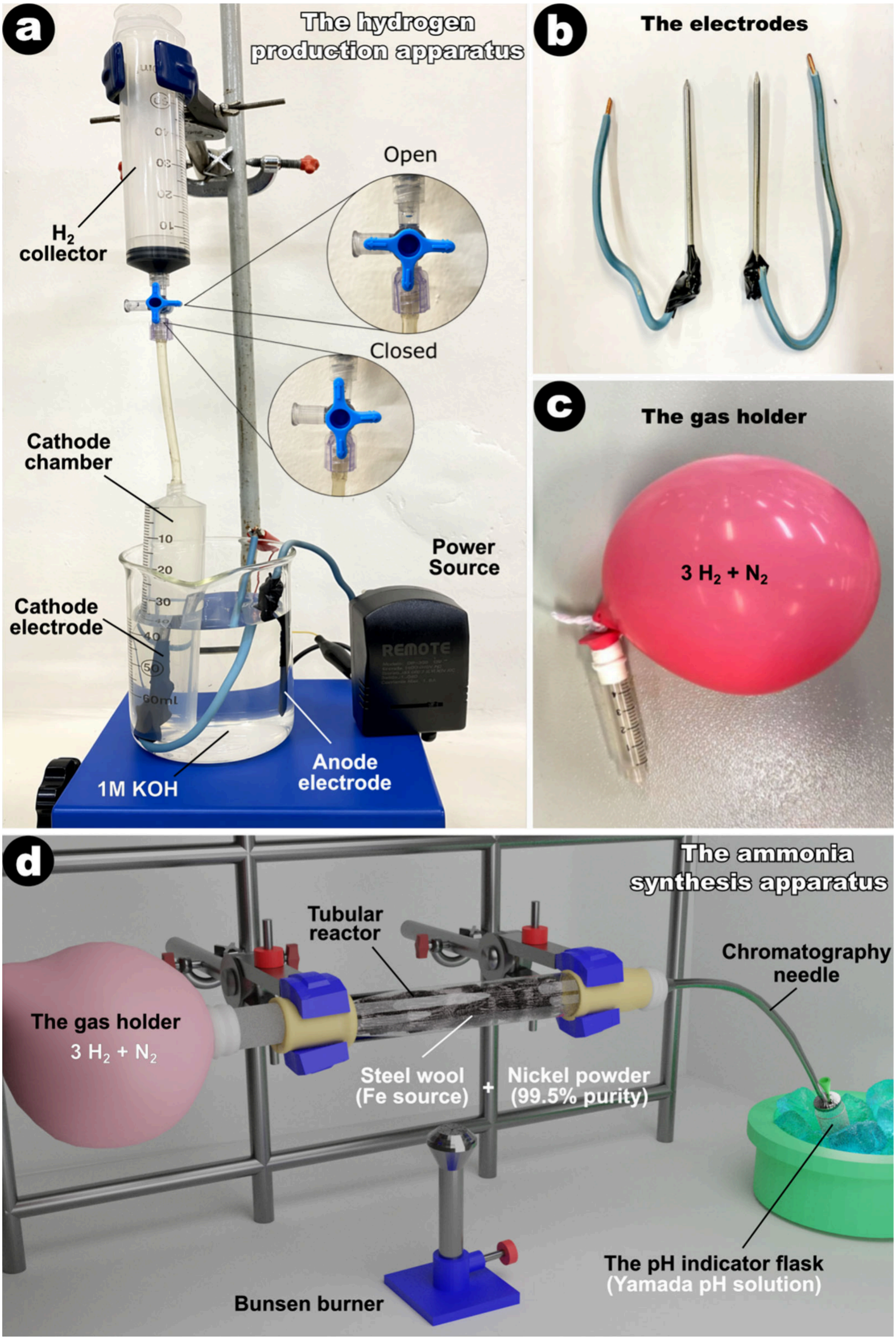

Figure 2. Experimental setup for hydrogen production and ammonia synthesis.

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experimental sessions, students receive introductory materials that provide context for the activities, covering topics such as the environmental impact of fossil fuels, hydrogen as an energy alternative, challenges in hydrogen production through water splitting, and its applications, including ammonia synthesis by the Haber-Bosch process.

In the laboratory, each session lasts 4 h, and students are organized into groups of 3 or 4 members. In the first session, students are oriented to investigate hydrogen production methods through spontaneous reactions with different metals. The objective is to develop critical thinking skills to evaluate efficiency, cost, and sustainability among different methods. The approach follows methodologies previously described in the literature.[5,26] In the second session, students perform water electrolysis in different chemical environments, analyzing the most efficient conditions for hydrogen production. This stage introduces the concept of hydrogen generation through electrically driven processes and provides a basis for discussing more sustainable hydrogen production pathways. The experimental approach is based on literature.[27] In the third session, students simulate ammonia synthesis using a simplified Haber−Bosch process. They use N_2 from a nitrogen cylinder and hydrogen is produced according to the optimized procedure in the previous session. This progression allows students to connect hydrogen production pathways with their environmental impact, including the concept of green hydrogen as a more sustainable alternative to fossil-based hydrogen. The experimental procedure is described in the next section. In this article, we will focus on the third session of the module, in which we explore the Haber−Bosch process in connection with hydrogen production by alkaline water electrolysis.

Learning Outcomes

The educational goals of the proposed experiment are related to understanding how hydrogen can be produced and the impact of different production pathways, as well as understanding the overall principles of the Haber-Bosch industrial process and the role of catalysts in this and other processes.

After completing this laboratory experiment, students are expected to be able to

- Explain the role of hydrogen production pathways in the carbon footprint of ammonia synthesis, including the concept of green hydrogen as a more sustainable alternative
- Relate electrolysis and catalysis to sustainability challenges in industrial chemical processes
- Qualitatively analyze the influence of temperature, pressure, and catalysts on the Haber-Bosch process
- Interpret qualitative experimental evidence (pH changes) as an indication of ammonia formation
- Critically discuss the limitations of lab-scale models of industrial processes

Experimental Part

The ammonia synthesis using a simplified Haber-Bosch process is divided into three stages:

1. Hydrogen production: Students produce 480 mL of H_2 through water splitting using a simple electrolytic cell constructed with accessible materials (Figure 2a). The setup employs metallic electrodes (iron nails, Figure 2b) immersed in an aqueous electrolyte, allowing hydrogen generation via water splitting. The experimental conditions used in this stage are based on prior investigations conducted by the students in earlier sessions of the module, in which different hydrogen production methods and electrolysis conditions are explored. In the present activity, these optimized conditions are applied to generate H_2 for subsequent ammonia synthesis. The use of water electrolysis as the hydrogen source is intentional, as it introduces students to hydrogen production through electrically driven processes and its relevance to sustainable chemical technologies. In contrast to conventional metal-acid reactions, this approach enables discussion of hydrogen as an energy carrier and its role in low-carbon chemical processes, often referred to as "green hydrogen". The generated gas is collected using disposable syringes and transferred to a latex balloon (Figure 2c), where 160 mL of N_2 is subsequently added to obtain the desired stoichiometric mixture. Details of the experimental setup, construction of the H_2 production apparatus, and operation setup are described in Supporting Information I (sections 3, 4, and 8). The experimental script for students is detailed in Part A of Supporting Information II (section 2).
2. Ammonia synthesis: Students connect the latex balloon containing H_2 and N_2 in stoichiometric proportions to the tubular reactor (Figure 2d), which is preprepared by the instructor before the session begins. The reactor consists of a packed catalytic bed containing steel wool (Fe) and nickel powder (Ni 99.5% purity), assembled to maximize gas−solid contact within the reaction zone. Students manually control the gas flow from the balloon into the reactor, while it is heated using a Bunsen burner. Under these simplified conditions—ambient pressure and external heating—the system does not aim to reproduce industrial efficiency, but rather to qualitatively demonstrate the key principles underlying the Haber-Bosch process, including the roles of temperature, catalysis, and gas−solid interactions. Details of the NH_3 production apparatus its operation, and catalyst preparation are described in Supporting Information I (sections 3, 5, 8, and 9). The experimental script for students is provided in Part B of Supporting Information II (section 3).
3. Ammonia identification: Students confirm the formation of ammonia using a pH indicator. The gaseous products exiting the reactor are bubbled through an aqueous indicator solution, where ammonia dissolution leads to an increase in pH and a corresponding color change. This approach provides a qualitative and visually accessible method for detecting NH_3 formation under laboratory-scale conditions. Instructions for preparing the indicator solution are provided in Supporting Information I (sections 3 and 7).

■ HAZARDS AND SAFETY PRECAUTIONS

All experiments should be conducted in a well-ventilated laboratory or under a fume hood. Physical protection between the experimental apparatus and the operator, such as a properly positioned fume hood sash, is strongly recommended. Appropriate personal protective equipment (PPE), including safety glasses, a lab coat, and gloves, must be worn at all times. KOH solutions are corrosive and may cause skin and eye burns

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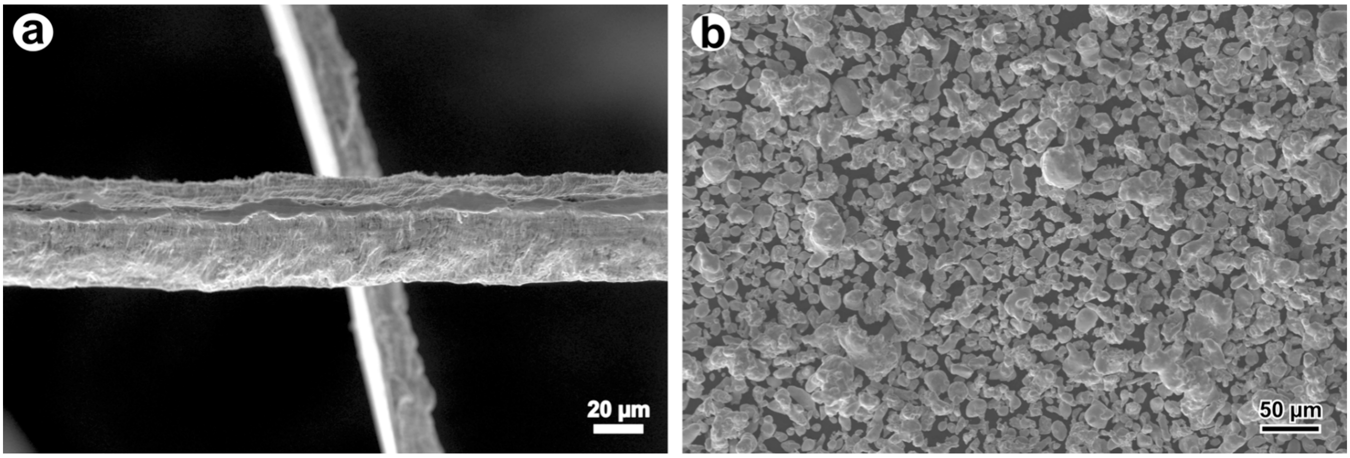

Figure 3. SEM images of the catalysts: (a) steel wool at 1,000× magnification; (b) Ni powder at 500× magnification.

if handled improperly. NH_3 is a noxious gas and prolonged exposure may cause irritation and chemical burns. H_2 is a highly flammable gas and mixtures of H_2 and oxygen gas (O_2) are explosive, therefore the gases must be generated in separate compartments. All chemical waste, including alkaline solutions and catalyst materials, must be neutralized and disposed of according to local regulations. The significant hazards associated with flammable gases, corrosive alkaline solutions, and open flames were mitigated through the use of physical protection, controlled gas handling, appropriate PPE, and instructor supervision.

The electrolysis system uses a 12 V DC power supply, presenting a minor electrical hazard. During ammonia synthesis, heating with a Bunsen burner requires special attention to open flames and hot glass surfaces. Latex balloons used as gas reservoirs may rupture if overfilled and should therefore be operated within the recommended gas volumes.

The "ammonia synthesis" step involves the passage of H_2 through a glass tube heated with a Bunsen burner flame, creating a potential fire hazard. The experiment should therefore be performed under a fume hood with physical protection between the apparatus and the operator. The glass tube used as a reactor must be resistant to high temperatures and direct flame contact. Before initiating H_2 flow, the reactor should be purged with nitrogen or another inert gas to remove atmospheric oxygen. Operators should be familiar with safely interrupting gas flow to the burner and ensure that the gas valve remains easily accessible during operation. The flame from the Bunsen burner must be carefully controlled to avoid excessively high flames. To minimize thermal stress, the Bunsen burner flame should be continuously moved along the reactor tube rather than concentrated at a single point.

Additional safety precautions, such as the use of flame arrestors and/or nonreturn valves, are recommended and should not interfere with the development of the experiment.

■ ETHICS STATEMENT

This work involved routine instructional activities conducted as part of a regular undergraduate laboratory course rather than a separately organized human-subject research protocol. Observations related to student learning were derived from standard classroom activities, discussions, and written reflections conducted as part of normal instruction. No identifiable student data were collected, retained, or formally analyzed, and no interventions beyond standard educational practice were performed. Therefore, the study did not constitute human subjects research requiring formal institutional ethics review or written informed consent.

■ RESULTS AND DISCUSSION

Materials and Catalyst Characteristics

To ensure reproducibility of the experiment, the materials used as the catalytic bed were characterized using complementary techniques (see Supporting Information I (section 6) for full details). Figure 3a shows scanning electron microscopy (SEM) images revealing that the steel wool consists of a highly fibrous structure, providing a large surface area and facilitating gas–solid contact. Energy-dispersive X-ray spectroscopy (EDS) and X-ray fluorescence (XRF) analyses confirmed that iron is the predominant element with minor contributions from other elements depending on the commercial source.

Figure 3b shows that the nickel powder presents a heterogeneous particulate morphology with relatively uniform composition (89.7 ± 6.8% by weight), as confirmed by EDS analysis. These materials were selected due to their commercial availability and ease of use in a teaching laboratory context.

A comparative analysis of different commercially available steel wool samples (Supporting Information I) indicates that, despite minor variations in morphology and composition, their general characteristics are similar. Therefore, comparable qualitative results are expected, although minor adjustments in catalyst packing and experimental conditions may be required depending on the specific materials used.

Laboratory Experiment Overview

The class has a maximum duration of 4 h. The session begins with a brief discussion of the topic and the experimental procedures. The experiments are then conducted over 2–3 h, followed by a wrap-up discussion in which students share their impressions, questions, and interpretations of the results. This final stage typically takes 20–30 min.

The instructional approach is inspired by Paulo Freire's concept of "cultural circles", where small groups of three to five students engage in dialogue and share their prior experiences.[20,28] In this context, the academic environment immerses students in a cultural setting where discussions on emerging sustainability issues are routine. Prelaboratory activities introduce real-world challenges related to industrial ammonia production, its environmental impact, and the necessity for sustainable alternatives. These activities enable students to

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grasp essential concepts related to the problems posed by laboratory experiments, such as understanding the stages of NH_3 synthesis and the execution of these processes. Furthermore, the achievement of ammonia synthesis through the Haber-Bosch process encompasses various areas of general chemistry knowledge, making it suitable as a GT because it stems from the students' real-world experiences and serves as a foundation for constructing new knowledge.[20] Consequently, students engage with knowledge of electrochemistry, chemical bonds, chemical equilibria, kinetics, thermodynamics, and more, in the adaptations of H_2 and NH_3 production apparatus, as well as in identifying the final product.

Another relevant aspect is the challenge presented by experimental adaptations in H_2 synthesis technologies via water splitting and ammonia synthesis through the Haber-Bosch process. Students are instructed to produce gases following the experimental procedure and to contemplate the similarities and differences between the processes, such as using iron nails as electrodes in water electrolysis and steel wool in ammonia synthesis. Ammonia formation is confirmed by observing the color change of a universal pH indicator from orange or yellow (pH 5−7) to yellow-green, blue-green, purple, or blue (pH 8−14), indicating the alkalinization of the solution, as illustrated in Figure 4.

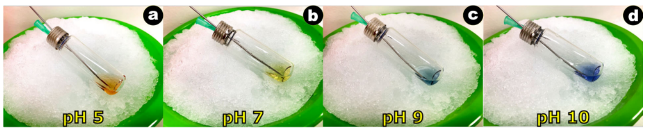

Figure 4. Progress of ammonia synthesis via the Haber−Bosch process can be monitored by observing the color change of the Yamada indicator.

Finally, postlaboratory activities promote reflection, data analysis, and information synthesis. This sequence was tested over three academic semesters with a total of 140 students involved and promoted active student participation.

Evidence of Student Learning

This laboratory experiment was designed to support student learning at the interface of general chemistry concepts and sustainability challenges. Evidence of learning was collected through multiple instructional components. To provide a more systematic framework for evaluating student understanding, an assessment rubric aligned with the learning objectives of the activity is included in Supporting Information I (section 11). This rubric defines levels of achievement across key conceptual domains and supports the interpretation of how students attained the intended learning outcomes.

During prelaboratory activities and in-lab discussions, students were prompted to compare fossil-based hydrogen production with water electrolysis and to articulate how these routes influence CO_2 emissions associated with fertilizer production. These discussions indicated that most student groups were able to correctly identify hydrogen production as the primary contributor to the environmental impact of the Haber-Bosch process and to recognize green hydrogen as a potential strategy for mitigating these emissions.

In the experimental sessions, students qualitatively evaluated the influence of temperature, pressure, and catalysts by contrasting industrial operating conditions with the simplified laboratory setup. Guided questions encouraged students to explain why elevated temperatures favor reaction kinetics but disfavor equilibrium yield, as well as why catalysts are essential for nitrogen activation. These questions are intentionally designed to connect the simplified experimental conditions to industrial ammonia synthesis, prompting students to reflect on the limitations of the laboratory model (e.g., ambient pressure, absence of gas recycling, and qualitative detection) and their implications for reaction equilibrium and efficiency. For example, students were asked: "Why does ammonia synthesis require high temperature and pressure?" and "What modifications could be made to the experimental apparatus to improve its performance, and how would these changes affect ammonia yield?" Student understanding of these concepts was assessed through oral explanations during laboratory work and through written postlaboratory reflections (see Supporting Information II (sections 4 and 5)).

The identification of ammonia using a mixed pH indicator provided a visual and accessible means for students to connect chemical equilibria and acid−base concepts to observable reaction outcomes. Successful interpretation of the color changes served as qualitative evidence that students could relate experimental observations to ammonia formation in aqueous solution.

Finally, postlaboratory assignments required students to reflect on the limitations of the laboratory-scale model, including the absence of high pressure, quantitative gas measurements, and gas recycling. These reflections demonstrated that students were able to distinguish between didactic representations and industrial-scale processes, reinforcing a critical understanding of how chemical principles operate across different scales.

Taken together, these observations indicate that the experiment effectively supported student learning by explicitly connecting fundamental chemical principles to sustainability-oriented discussions, particularly regarding the role of chemistry in reducing greenhouse gas emissions associated with fertilizer production.

Limitations

The production of H_2 through the procedure described in Supporting Information II (sections 2 and 3) must be carefully executed. In some cases, the high concentration of KOH, combined with improper handling by students, led to corrosion and damage to components such as the three-way stopcock and balloons of the gas holders, requiring the repetition of experimental steps. Moreover, alkaline solutions can cause burns to the skin and eyes, therefore the use of PPE is always emphasized to students.

During the hydrogen production phase for ammonia synthesis, the volume of H_2 produced can sometimes be insufficient. To address this, groups can combine their resources to ensure an adequate supply of gas, or they can use industrial gas. Of the 140 students participating, organized into teams, approximately 65% successfully performed the synthesis using the gas produced by their own group. All N_2 used in the experiment is collected from a cylinder. The gases are mixed in a balloon. Despite these challenges, observing the pH change as evidence of ammonia formation significantly enhanced student engagement and deepened their understanding of the process. It is important to note that measuring gas volume with disposable syringes was only qualitative, as these instruments are not designed for gas handling. Additionally, during the transfer steps, gas losses or contamination

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from atmospheric contact may occur and low pH change can be observed.

Given these limitations, conducting the laboratory experiment in a larger class can be problematic if the logistics of reorganizing the groups are not properly managed. In our experience, the strategy of regrouping students who were unable to produce the required amount of H_2 with those who were successful in the same procedure proved effective and may serve as an alternative for other instructors. Additionally, a solution guide for the most common problems is provided in Supporting Information I (section 10).

In addition to these practical considerations, the limitations of the experimental setup are intentionally used as pedagogical tools to promote critical reflection. Students are encouraged to compare the simplified laboratory setup with industrial ammonia synthesis, particularly regarding the effects of pressure, temperature, and catalysis on reaction performance.

■ CONCLUSION

This experiment introduces students to emerging topics in sustainability, such as integrating green hydrogen into the Haber-Bosch process. Through a low-cost and easily implemented practice, students produce hydrogen via water splitting and use it in a didactic simulation of ammonia synthesis. The activity contextualizes key chemical principles of industrial relevance while fostering critical discussions on environmental impacts and sustainable alternatives. Its versatility allows for the integrated exploration of thermodynamics, chemical equilibrium, kinetics, and catalysis, bridging general chemistry concepts with contemporary global issues. The use of simple, readily available materials makes the experiment feasible across diverse teaching laboratories, thereby broadening its pedagogical reach. In this way, the activity promotes active and reflective learning—an essential characteristic in the education of chemists today.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.6c00111.

Instructor's guide containing a more detailed discussion of Paulo Freire's approach, as well as the chemistry involved in ammonia synthesis and water electrolysis, additional guidelines for preparing the experiment, notes on catalyst selection, preparation, and characterization, a troubleshooting guide for common issues, and a student assessment rubric with criteria to support evaluation (PDF)

Student handbook containing an introductory text on the importance of ammonia, environmental impacts, and sustainable alternatives, experimental script for hydrogen production and ammonia synthesis, and pre- and postlab activities (PDF)

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Notes

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