EXPERIENCING PROCESS PLANT CONDITIONS THROUGH A PILOT PLANT-BASED LABORATORY CLASS

Colin A. Scholes and Guoping Hu
University of Melbourne • Parkville VIC 3010, Australia

INTRODUCTION

In the chemical engineering curriculum many laboratory classes focus on a particular learning objective, such as demonstrating the ability to apply theory through closed-ended problems;[1,2] that is, having the students follow a prescribed recipe consisting of rigid instructions that produce a determined result. This is essentially a passive learning activity, as there is little scope for innovation or creativity on behalf of the students, limiting transference of the learning objective.[1,2] Furthermore, many laboratory experiences have little correlation with real engineering environments, as the equipment is small scale and the interactions are confined to student – demonstrator and/or amongst small peer groups.[3,4] This approach is limited in enabling conceptual understanding of engineering topics and inadequate for preparing students for post-graduate roles in process plant environments.[5,6] To address these shortfalls, a laboratory class has been developed that successfully simulates the working environment and interactions engineers will experience within a chemical process plant. This is achieved by having chemical engineering students in groups operate a large-scale pilot plant for carbon dioxide capture, with the students taking on several key roles essential for process plant operation. This laboratory class is focused on developing initiative and problem-solving skills within students based on their engineering knowledge, as well as ensuring students gain an understanding of structured teamwork.

The large-scale pilot plant is based on a solvent absorption process for carbon dioxide capture from a feed gas utilizing a potassium carbonate-based solvent.[7] The reaction between CO_2 and the solvent is:

$$CO_2 + K_2CO_3 + H_2O \leftrightarrow 2KHCO_3 \tag{1}$$

The pilot plant consists of two columns, one undertaking the absorption of CO_2 from the feed gas, while the second column is a desorber regenerating the solvent and producing a purified CO_2 product stream (see Figure 1). The students' specific objective is to ensure the pilot plant achieves set CO_2 recovery and purity targets at steady-state operating conditions, while also having to successfully start up and shut down the process. The pilot plant is designed to capture 10 kg/hr of CO_2 from the feed gas into a potassium carbonate solvent with the concentration ranging from 20 to 40 wt%. The size of the process and the physical presence of the equipment provide experience to the students in operating and working around large process equipment.

Colin A. Scholes *CChem FRACI CEng MIChemE is a Senior Lecturer in the Department of Chemical Engineering at the University of Melbourne. He is an expert in clean energy processing and membrane science, particularly developing strategies to assist the transition to a low carbon future. He is also a passionate engineering educator to people from disadvantage backgrounds. He has worked with disadvantaged communities in Australia and the Pacific on sustainable water and energy projects.*

Guoping Hu *is a Research Fellow in the Department of Chemical Engineering at the University of Melbourne. He focuses on the deployment of research outcomes into industry in the separation field, particularly gas separations using solvent absorption and solid adsorption. He is also keen on developing application-oriented teaching methods such as video teaching materials and industry case analysis.*

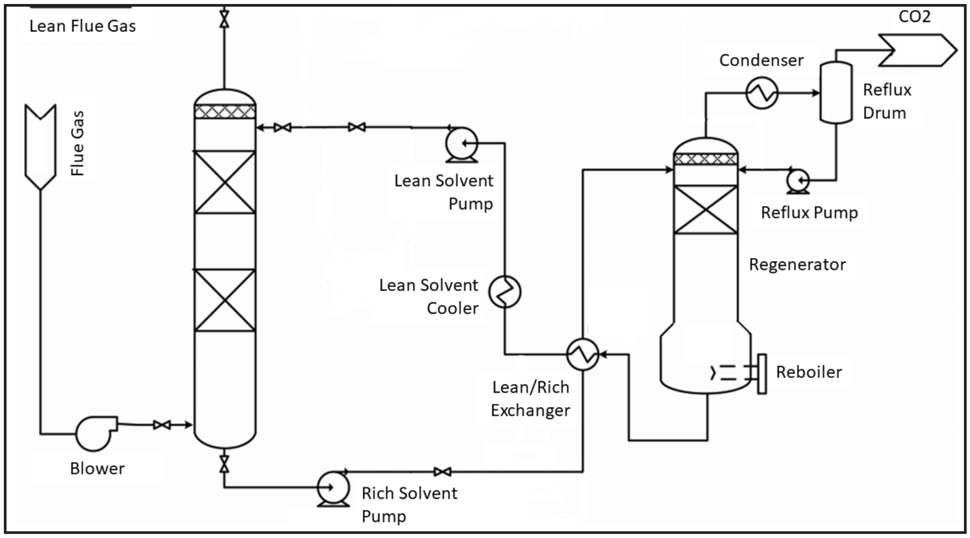

***Figure 1**. Schematic overview of the CO_2 solvent absorption capture pilot plant, based on absorber and desorber columns with reboiler, operated by the students.*

The laboratory class is associated with a chemical engineering elective in energy and carbon capture strategies and is designed for engineering students in their corresponding third or fourth year of study. The class assumes prior knowledge in mass and heat transfer, process control and engineering, thermodynamics, and mass and energy balances. This knowledge is necessary for the students to successfully operate the pilot plant and achieve the performance targets set, as they must make a range of engineering-based decisions to operate the pilot plant successfully. Importantly, teamwork amongst the students is promoted and students interact with each other in a manner that correlates to a process plant environment.[8] Individual students take on specific roles and have an established chain-of-command when operating the plant. This ensures students are exposed to leadership structures as well as roles and responsibilities designations, since successful operation of the pilot plant depends on good coordination and communication between the students and understanding of their respective roles. This also provides insight to the students about how they will function in post-graduate roles in process plant operations.

PEDAGOGICAL OBJECTIVES

The main technical objective is to familiarize the students with operation of large process units[9] for better understanding of the theoretical material, as well as to allow students to experience the engineering and personnel dynamics involved in process plant operations. The engineering focus of the laboratory is mainly on the hydrodynamics of operating packed columns. There are also several other engineering aspects that the students experience during the class: coupling of different operating variables and the impact on the plant control scheme, time lag in operation and product quality data, the importance of correct startup and shutdown procedures, and intrinsic safety in design. This enables the acquisition of procedural knowledge, as well as experiencing chemical engineering phenomena, such as flooding, entrainment, temperature and pressure fluctuations, through hands-on experience. This requires students to review key chemical engineering concepts, which reinforces and improves their cognitive background.

The pedagogical objective is for the students to gain experience in real-time problem solving, as well as the translation of their theoretical understanding into practical outcomes based on how the interaction of variables impacts the pilot plant performance. The secondary objective is for students to experience the group dynamics and logistics of process plant operations, particularly the various roles, responsibilities, and leadership structure involved in successfully operating large process equipment and plants. This is a complex objective that varies for every laboratory class; it is a function of the student personalities, existing student relationships, and the real-time conditions of the pilot plant process. Importantly, the average engineering student is an introverted thinker,[10] so expressiveness and cooperation are not necessarily comfortable modes for their learning, especially as the laboratory class requires adapting to situations based on discussions and/or equipment limitations.

The final aim is to achieve a more effective inductive learning approach, balancing deductive engineering instructions with hands-on experience, while gaining an understanding and experience of team dynamics. The laboratory experience will assist students in acquiring additional transferable skills, such as time management and communication, that both strengthen the technical knowledge and interpersonal skills necessary for successful chemical engineering graduates.

To pass the laboratory class, all students must submit a detailed individual laboratory report on the operation of the pilot plant, including analysis of the control performance of the absorber and desorber columns and a discussion of their ability to achieve the CO_2 recovery and purity targets. The students do not need to achieve the CO_2 targets to pass the class, but failure to meet this objective is generally reflected in the grades awarded. However, the grading rubric does reward students who identify process conditions and trouble-shooting of plant operations that limit their ability to meet the targets, if these are linked with chemical engineering concepts.

EXPERIMENTAL DESIGN

The first component of the laboratory class is safety, with the students having to demonstrate a detailed understanding of the safety aspects of the pilot plant, along with personal protective equipment (PPE) and chemical hazards, before they can begin the class. This is facilitated by an online introduction video and associated documentation that the students must view and read before the class. To ensure they have acquired the necessary information, an online quiz is linked with the video that, upon successful completion, provides notification to the student as well as the demonstrators/lecturer that the student is approved for the class. Safety is further reinforced at the beginning of the class with the students having to complete a thorough risk assessment, with assistance from the demonstrators, for the steady-state operation as well as shutdown of the pilot plant. Students are provided with an already completed risk assessment for startup of the plant, which acts as a template and guide to the students. As an example of good engineering practice, students are also provided with the process flow diagram (PFD) and piping and instrumentation diagram (P&ID) of the pilot plant and instructed to identify the various unit operations on the plant with the respective diagrams, as well as follow the various streams to familiarize themselves with the layout of the plant. This information becomes vital to the students during operation. Finally, demonstrators are present throughout the class, providing assistance and instruction when needed.

Students then self-assign their respective roles in operating the pilot plant. The group size is six students, with some fluctuations depending on the number enrolled in the class. Each group runs the pilot plant between four to nine hours, depending on their success in achieving the CO_2 recovery and purity targets. The make-up of the student groups is also self-assigned as well as the respective roles during the practical, as the laboratory class is available to the students over a number of days, with students choosing the most appropriate date that avoids clashes with other commitments. This is recognized as not ideal,[11] as groups are created based on their timetable availability rather the personal attributes and, as a result, issues with group cohesion and dysfunctionality have been a problem.

The students then self-allocate into the respective roles to operate the pilot plant: plant operator, absorber column controller, desorber column controller, gas streams monitor and solvent loading analytical chemists. The *plant operator* is responsible for the entire process to ensure startup, steady state, and shutdown operations are successful and the CO_2 recovery and purity targets are achieved. The *absorber column controller* is responsible for ensuring the absorber is operating within optimal range and avoids adverse hydrodynamic conditions such as flooding from occurring. The *desorber column controller* is responsible for ensuring the desorber is operating within optimal range. This team member also ensures that the temperature profile in the column is adequate to regenerate the solvent and produce the CO_2 product stream and that the reboiler has sufficient solvent level. The *gas streams monitor* ensures that the simulated flue gas, absorber exit gas, and CO_2 product streams are within operating parameters and measures the CO_2 concentration within the respective gas streams. The *solvent analytical chemists* are responsible for measuring the CO_2 loading in the lean and rich solvent streams; this role is normally undertaken by two people given the number of samples taken and the time commitment involved in the analysis titrations. The leadership structure of the team is set out in Figure 2, with the plant operator having overall responsibility for the plant operation.

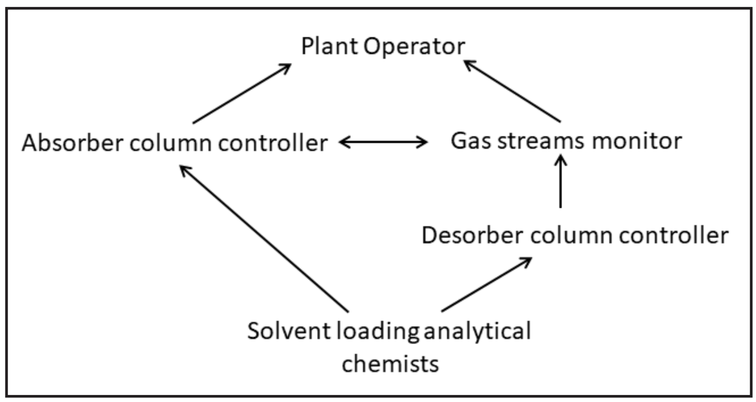

Figure 2*. Leadership structure of the pilot plant operational team.*

PILOT PLANT SET-UP

The feed gas to the pilot plant contains 10 – 25 vol% CO_2 (Coregas), with the flow rate controlled via Bronkhorst EL-FLOW mass controllers, followed by a heated water bath that increased the feed gas temperature to ~50 °C before the gas entered the absorber packed column. A gas saturator is used to obtain feed gas with 80 – 95% relative humidity, with the temperature and humidity measured by a probe located near the entry to the absorber column. The potassium carbonate solution is made-up on site to the desired concentration and pumped into the desorber - reboiler. The absorber column is made of borosilicate glass with a diameter of 100 mm and a total height of 4.25 m. The absorber column has three packed bed sections, each a height of 0.8 m. These sections are filled with stainless-steel pall rings with a diameter of 10 mm. A rich solvent tank at the base of the absorber is used for solvent storage and contains a heating element for use during the pilot plant startup procedures. The rich solvent is sent from the absorber column to the regenerator column via the rich solvent tank, rich solvent pump, and the lean-rich heat exchanger. The exchanger is a spiral heat exchanger that heats the rich solvent stream via the lean solvent from the reboiler. The gas leaving the top of the absorber column passes through a condenser (glass – process water flowrate < 2 L/min) to remove most of the moisture in the gas before passing through a rotameter to measure flowrate and a Horiba VA-3000 gas analyzer to determine CO_2 concentration.

The desorber consists of a borosilicate glass column with a diameter of 100 mm and a stainless steel reboiler tank attached at the base of the column. The total height of the desorber is 4.6 m, inclusive of the reboiler. The desorber consists of three packed bed sections, each up to 1 m in height filled with stainless-steel pall rings with a diameter of 10 mm. The reboiler is heated by an electrical two-stage element bundle that provides a heating duty of 12, 18 or 30 kW. The temperature of the solvent can be controlled up to 150 °C. Gas leaving the top of the regenerator is fed to an overhead condenser (process water flowrate < 10 L/min), followed by a separator/reflux drum. Condensed water is returned to the desorber column or sent to the drain to maintain the water balance of the process. The CO_2 product gas is sent to an exhaust via a back-pressure control valve that can control the pressure of the regenerator up to 150 kPa. The lean solvent from the reboiler is fed back to the top of the absorber via the spiral heat exchanger, lean solvent pump, and lean solvent cooler (process water flowrate < 1 L/min). The pilot plant programmable logic controller (PLC) and operator interface are by LabView software, with the plant data recorded into a text file at time intervals dictated by the operator.

Numerous pressure and temperature indicators and transmitters are located throughout the plant and generally recorded every 10 sec on the plant's PLC. The CO_2 loading in the potassium carbonate solvent samples are determined through acid titrations using a Metrohm 905 Titrando autotitrator or by colorimetry titration using a UIC Inc. CM5015 Coulometer.[12] These methods enable the concentration of carbonate and bicarbonate to be determined, and correspondingly the CO_2 loading of the solvent. More details on the pilot plant are provided in the literature.[7,13]

The startup procedure establishes the solvent circuit first through both columns and reboiler, as well as achieving the desired temperature profile in the desorber column that will enable solvent regenerator. The initiation of the solvent circuit to process conditions can take an hour, after which time the CO_2-rich feed gas enters the absorber column. The following period involves ensuring the correct hydrodynamics within the absorber column that correspond to steady-state operation, which includes solvent flowrate rate control, pressure regulation within the absorber, and monitoring the rich solvent temperature. Students identify steady-state conditions by monitoring the absorber column pressure and temperature profiles. Once CO_2 absorption has been established, the desorber column is monitored to ensure solvent regeneration was occurring and CO_2 product gas is being produced. A period of up to six hours can pass until the pilot plant achieves steady-state operations, with continual adjustment to operating parameters required to achieve the target CO_2 recovery and purity. Students identify steady-state operations by monitoring the desorber pressure and temperature profiles. The shutdown procedure involves switching off the feed gas supply to the absorber and continual running of the solvent circuit through both circuits until the CO_2 loading in the lean solvent reduces to an acceptable level. At that point the reboiler is shut down, and the desorber column continues to operate until the temperature profile approaches ambient. Finally, the solvent circuit is stopped, isolation valves engaged, and the PLC shut down. Photos of the pilot plant in operation are provided in Figure 3.

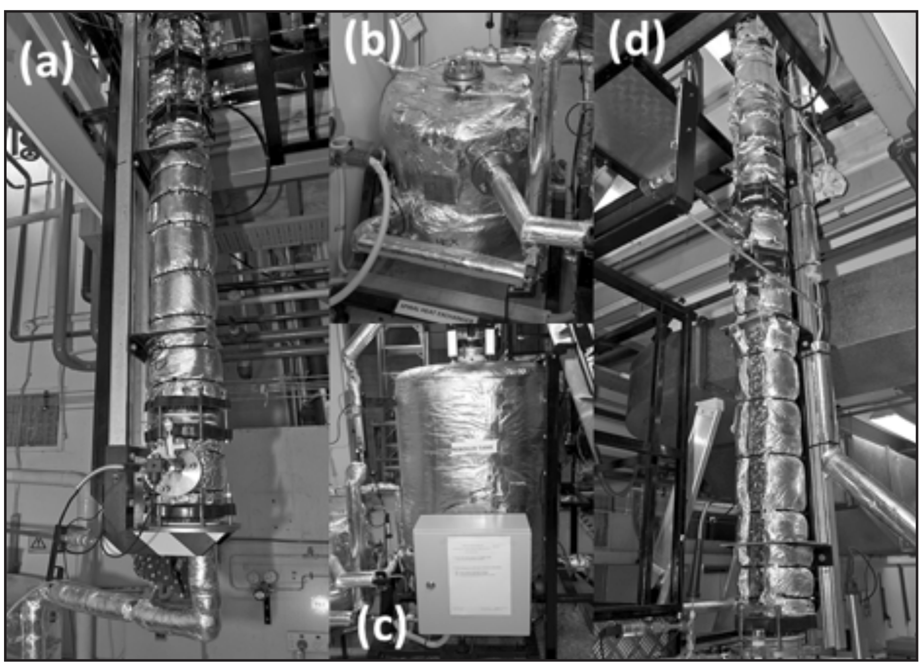

Figure 3. *Photos of the pilot plant during operation: (a) absorber column, (b) spiral heat-exchanger, (c) reboiler and (d) desorber column.*

OBSERVATIONS AND DISCUSSION

A critical component of operating the pilot plant is determining the operating lines of the absorber and desorber columns, as this establishes whether the process conditions can achieve the 90% CO_2 recovery target. The students generally have considerable success in establishing the steady-state operating lines of the absorber column, as well as extrapolating the recovery amount based on equilibrium data for the CO_2 - potassium carbonate system. The determination of these lines is critical to calculate if their L/G ratio is adequate to achieve the CO_2 recovery target.[14] The students also use the equilibrium line and column operating performance targets to determine the minimum L/G ratio, which enables them to calculate the lower boundary of the flowrate condition. Almost always, the first L/G determination by the students is not high enough to achieve the recovery target, with the students then calculating the necessary change (increase) required for the solvent flowrate. There is then a time lag of up to an hour before the pilot plant reestablishes at steady-state conditions, and the students can redetermine their operating line to verify performance. Most student groups require three or four attempts to adjust the solvent flowrate, reestablishing steady state and then confirming the absorber operating line to achieve operating conditions that remove 90% CO_2 from the feed gas and meet the recovery target. A key problem for the students is setting the solvent flowrate too high, leading to flooding of the column, and hence understanding the behavior of the solvent in the absorber column is a learning outcome. This process is strongly associated with the technical objectives of the practical understanding of the column operations and hydrodynamics.

The desorber operation is critical to the CO_2 loading of the lean solvent and hence operation of the absorber, which most student groups initially attempt to reduce to almost zero. This places too great a demand on the desorber/reboiler and is the main reason why student groups find the pilot plant takes extended time to approach steady-state conditions. Good students identify from their prior reading that the lean solvent loading should be around 0.2 and therefore operate the desorber column accordingly. Instructions on how to achieve the 95% CO_2 purity target are not provided to students. As a result, there is considerable problem solving undertaken by the students using the reflux ratio and condenser duty, which provides an excellent hands-on experience on column operation. This is strongly associated with the solvent flowrate in the desorber as well as the pressure gradient present. Many groups struggle to produce a CO_2 product of 95% purity or greater, as it requires a good temperature gradient to be established within the desorber column and a large condenser cooling water duty, which students are generally cautious about employing. This component of the practical is also linked with the technical objectives, along with problem solving and translating theoretical understanding into beneficial outcomes. In addition, communication, leadership structure, and coordination amongst the students play an important role in achieving positive outcomes, which all align strongly with the pedagogical objectives.

There are several common issues that student groups regularly struggle with in operating the pilot plant, which produce predictable outcomes:

Startup

- *Exposing the absorber to feed gas before the solvent circuit has been established*. This results in considerable solvent entrainment in the exiting gas and channeling within the column. This forces the students to consider the hydrodynamics of the process. The CO_2 recovery is minor, forcing the students to shut down the pilot plant and begin again.
- *Reboiler initiation temperature is too high*. This results in significant solvent boiling and a dramatic decrease in the reboiler solvent level as well as fluctuations to the pressure profile of the column. Students need to quickly identify the cause for the solvent loss before the reboiler safety trips the plant to turn off.

Steady-state

- *The absorber column solvent level is not adequately controlled*. This normally results in the solvent level at the base of the column decreasing enough that the feed gas can pass under the underflow baffle, entering the rich solvent stream piping, and feed gas begins to accumulate in the top of the rich solvent storage tank. If the gas flowrate is considerably high, pressure relief on this tank exposes the surrounding area to concentrated CO_2, setting off the laboratory gas alarm. Alternatively, the solvent flowrate is unrestricted, and the column beings to flood. This eventually releases solvent into the exiting gas stream, overflowing the knock-out pot and flooding the laboratory. Hence, students generally establish the upper and lower operating bounds of the solvent flowrate in the pilot plant.
- *Desorber reflux ratio is not adequately controlled*. This results in the reflux drum overfilling and solvent backfilling the CO_2 product gas line. If this occurs for a considerable period, the solvent significantly damages the CO_2 analyzer. Avoiding this scenario is a critical task of the gas stream monitor.
- *Solvent circuit is not equal between the two columns, due to the lean and rich solvent pumps flowrates being out of sync*. This results in a buildup or depletion of solvent in sections of the pilot plant, disrupting steady-state operation, and CO_2 capture performance is lost.

Shutdown

- *CO_2 product gas line is isolated too soon, while solvent regeneration is still occurring.* This results in an increase in pressure within the desorber column. If students do not respond to the alarm in adequate time, the pressure relief valve activates, resulting in solvent/steam and CO_2 entering the laboratory air extraction system.

The complexity of the pilot plant and variability in student groups means that every laboratory class is slightly different with regard to the challenges faced and the performance outcome the plant achieves. This complexity and variability mean it is difficult for students to collude based on their colleagues' prior experience. This also has the added benefit of keeping the laboratory class interesting for the demonstrators/instructors, as it alleviates the monotony that can be experienced in other more routine laboratory classes.[15]

EVALUATION OF THE LEARNING APPROACH

This laboratory class has been successfully run for eight years, with detailed metrics on the performance obtained over the last five years through the official student survey run by the University of Melbourne. The survey is generic to all degree courses and teaching topics, with the laboratory class associated with the specific question: "Focusing on my own learning in this subject, I learnt to apply knowledge to practice"; with the scoring system ranging from 1 (strongly disagree) to 5 (strongly agree). The average scores for this question over the past five years are provided in Figure 4, along with the standard deviation in the student responses.

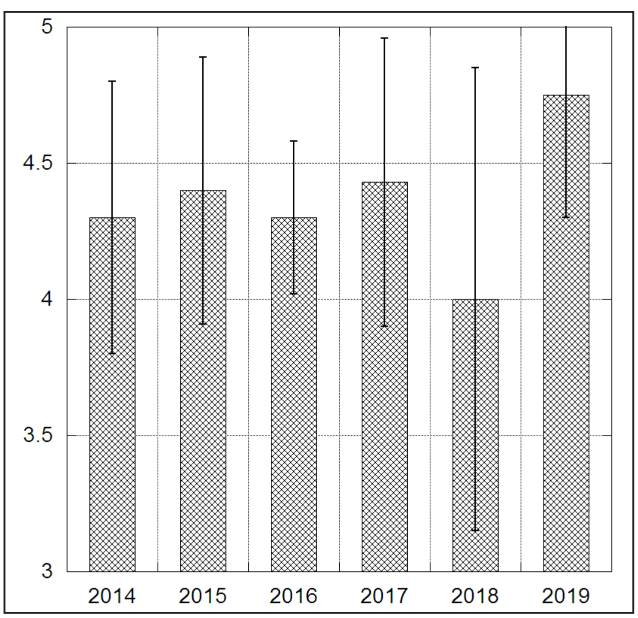

***Figure 4**. Analysis of the laboratory class performance based on student feedback.*

The average class size per year is between 20 to 25 students. The answer rate to this question has been above 80% of students undertaking the subject for all years, except 2014 when it was 65%. Importantly, for the past five years the average response has ranged from 4 to 5, which demonstrates the students have a very positive opinion of the learning outcomes of the laboratory class. The 2018 result is lower than the average, and this is associated with one of the groups not coordinating effectively, leading to flooding of the absorber column and major solvent spillage in the laboratory. Consequently, this student group had to mop these spills up, which they did not look favorably upon during the class. We believe this is reflected in the survey score, given the very large standard deviation for 2018 compared to other years.

Most of the student comments about the laboratory class in the official survey were very positive. Some student comments follow:

- *The prac was awesome! I enjoyed the strong links made to real-life examples.*
- *The practical on the pilot plant was something new and interesting.*
- *Experiencing how a plant operates was good, but challenging.*
- *I enjoyed the laboratory class.*
- *Operation of the pilot plant was one of the best pracs in the whole course.*

However, several student comments reflect their concern about their grades being based on a team activity.

- *It is not fair that the prac outcome is so dependent on getting a good group, others made mistakes and my marks suffered.*

This reflects the emphasis students place on their individual grades relative to the learning experience and is a challenge for all team-dependent assessments. Importantly, the assessment is an individual laboratory report, and so issues with team functionality and the ability to reach the CO_2 recovery and purity targets can be supplanted somewhat by a well-constructed, thoughtful report that clearly displays the individual student's understanding and competency. Furthermore, it is made clear to students that demonstration of their knowledge, particularly about what went wrong during plant operations to prevent them from achieving the performance targets, will be rewarded in the grading rubric.

CONCLUSIONS

A laboratory class based on operating a CO_2 solvent absorption pilot plant has been developed to enable students to operate large process equipment as well as experience the interpersonal dynamics involved in operating process plants. This class enables students in groups of six to take on defined roles necessary to operate the large-scale plant and attempt to achieve prescribed CO_2 recovery and purity targets. Importantly, this laboratory class is focused on developing initiative and problem-solving skills within students, based on their engineering knowledge given the complexity of operating the plant successfully. In addition, the practical provides the students with the opportunity to develop teamwork and communication skills to prepare them for post-graduate employment. The laboratory class is well received by students as a learning experience, with formal evaluation of the class strongly positive.

ACKNOWLEDGMENTS

The authors acknowledge the support of Australian National Low Emissions Coal Research and Development, who initially funded the pilot plant construction, as well as the Peter Cook Centre for Carbon Capture and Storage Research and the Melbourne School of Engineering at the University of Melbourne for contribute to the maintenance of the pilot plant.

REFERENCES

1. Young BR, Yarranton HW, Bellehumeur CT, and Svrcek WY (2006) An experimental design approach to chemical engineering unit operations laboratories. *Educ. Chem. Eng.* 1(1):16-22.
2. Woods DR, Felder RM, Rugarcia A, and Stice JE (2000) The future of engineering education III. Developing critical skills. *Chem. Eng. Ed.* 34(2):108-117.
3. Lamancusa JS, Zayas JL, Soyster AL, Morell L, and Jorgensen J (2006) Bernard M. Gordon prize lecture: The learning factory: Industry-partnered active learning. *J. Eng. Educ.* 97(1):5-11.
4. Pirola C (2019) Learning distillation by a combined experimental and simulated approach in a three steps laboratory: Vapor pressure, vapor-liquid equilibria and distillation column. *Educ. Chem. Eng.* 28:54-65.
5. Felder RM, Brent R, and Prince JJ (2011) Engineering instructional development: programs, best practices, and recommendations. *J. Eng. Educ.* 100(1):89-122.
6. Doskocil EJ (2003) Incorporating experimental design into the unit operations laboratory. *Chem. Eng. Ed.* 37(3):196-201.
7. Smith K et al. (2014) Demonstration of a concentrated potassium carbonate process for CO_2 capture. *Energy Fuels.* 28:299-306.
8. Chen W, Shah UV, and Brechtelsbauer C (2019) A framework for hands-on learning in chemical engineering education -- Training students with the end goal in mind. *Educ. Chem. Eng.* 28:25-29.
9. Ortiz FJG (2019) A pilot-scale laboratory experience for an inductive learning of hydrodynamics in a sieve-tray tower. *Educ. Chem. Eng.* 29:42-55.
10. McCaulley MH (1990) The Myers-Briggs Type Indicator: A Measure for Individuals and Groups. *Measurement and Evaluation in Counseling and Development.* 22(4):181-95.
11. Mills JE and Treagust D (2003) Engineering education, Is problem-based or project-based learning the answer. *Australasian J. Engng. Educ.* 3:2-16.
12. Horowitz W (1974) Official methods of analysis of the Association of Official Analytical Chemists. 12th ed. Washington Association of Official Analytical Chemists. Washington, DC.
13. Quyn D, Rayer AV, Gouw J, Indrawan I, Mumford KA, Anderson CJ, Hooper B, and Stevens GW (2013) Results from a pilot plant using un-promoted potassium carbonate for carbon capture. *Energy Procedia.* 37:448-54.
14. Coulson JM, Richardson JF, Backhurst JR, and Harker JH (2002) *Coulson & Richardson's Chemical Engineering Volume 2 -- Particle Technology & Separation Processes.* 5th ed. Butterworth Heinemann, Oxford.
15. Hofstein A (2004) The laboratory in chemistry education: Thirty years of experience with developments, implementation, and research. *Chem. Educ. Res. Pract.* 5:247-64. ❒