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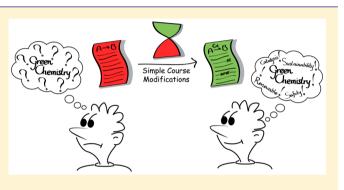
Simple and Effective Integration of Green Chemistry and Sustainability Education into an Existing Organic Chemistry Course

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Supporting Information

ABSTRACT: Green chemistry and sustainable development have become increasingly important topics for the education of future chemists. The cross-disciplinary nature of green chemistry and sustainable development often means these subjects are taught in conjunction with other subjects, such as organic chemistry and chemical engineering. Herein, a straightforward and efficient approach for vertical integration of green chemistry concepts within existing undergraduate organic chemistry courses is shown. The gradual selfevaluation, "greenification", and reassessment of an organic chemistry course at KTH Royal Institute of Technology from 2013 to 2017 is described, with particular focus on the



laboratory course and a novel green chemistry project designed to promote sustainability thinking and reasoning. The laboratory project, which can also be conducted as an independent organic chemistry laboratory exercise, required students to critically evaluate variations of the same Pechmann condensation experiment according to the twelve principles of green chemistry. The course evaluation shows that, after the modifications, students feel more comfortable with the topics "green chemistry" and "sustainability" and consider these topics more important for their future careers. Furthermore, the ability of students to discuss and critically evaluate green chemistry parameters improved considerably as determined from the laboratory project reports.

KEYWORDS: Second-Year Undergraduate, Organic Chemistry, Problem Solving/Decision Making, Testing/Assessment, Green Chemistry, Reactions

BACKGROUND

Recently, the United Nations declared the 2030 Agenda for Sustainable Development as their plan of action for addressing the people, the planet, and prosperity.¹ In the same manner, academic universities, such as KTH Royal Institute of Technology, started developing policies to drive education and research toward more sustainable development.² The increasing awareness of environmental issues emerging from overutilization of natural resources has made sustainable development of utmost importance for industrial chemical processes. The concept of green chemistry has in part been developed to aid sustainable development, and is based on integrating a sustainability perspective already from the early development stages of chemical process development. Integration of green chemistry and sustainability in the education of future chemists and chemical engineers is thus critical in order to ensure a good position for students on the labor market.4,5

To support this new educational demand, different approaches have already been implemented to introduce students to the well-established "Twelve Principles of Green Chemistry".³ Selected examples include discussion of case studies,^{6,7} green chemistry laboratory exercises,^{8–10} full courses in sustainability or green chemistry,^{11–14} educational tools to measure how "green" a reaction is,^{15,16} and introduction of green chemistry undergraduate research into the curriculum.¹⁷ Green chemistry and sustainability are often taught as overarching mindsets rather than singular concepts.¹⁸ Previously, it has been shown that this can be accomplished by requiring students to apply acquired knowledge, for example, by critically assessing literature procedures.^{19–21}

In order to improve green chemistry education, it is desirable to develop methods of teaching these concepts without major changes to existing course structures. In this work, a simple approach to integrate green chemistry concepts into an organic chemistry course is described, with a large focus on the practical organic chemistry laboratory exercises that comprise a major part of the course. Substantial progress to promote critical thinking on topics such as "green chemistry" has been made through the development of a novel laboratory project, which is described in detail in the present work. Additionally, how other course components support achievement of the desired pedagogic goal, training of students in the critical assessment on sustainability of chemical processes, is also described.

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COURSE DESIGN

The course central to the current study is "Organic Chemistry, Basic Concepts and Practice 2" (KD1270). This course is given in the second or third year of engineering programs in Biotechnology and Technical Chemistry at KTH Royal Institute of Technology. The full course is spread over three different modules: traditional lectures and exercises with a theoretical exam, a laboratory course, and a literature searching project. The modules have been designed to intertwine (Figure 1) to provide students with an effective learning environment

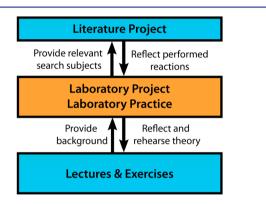


Figure 1. Global construction of the course "Organic Chemistry, Basic Concepts and Practice 2".

where they apply acquired theoretical knowledge in a laboratory environment, which in turn provides the basis for the literature searching project.

The learning outcomes of this course (Supporting Information, page S2) include development of the ability to "analyze and evaluate organic chemical reactions and processes from a sustainable development perspective based on the principles and methods of the green chemistry concept." Initially, this ability was intended to be developed via short examples during lectures and a full page in the laboratory compendium describing the concept and 12 principles of green chemistry. The topic was also treated during a 6 h laboratory exercise session, which contained a short introductory discussion regarding environmental concerns associated with organic chemistry. However, from an initial self-assessment of the course, it was evident that the 2013 course did not appropriately introduce the desired knowledge of green chemistry and sustainability to students (*vide infra*).

COURSE MODIFICATIONS

At this stage of the curriculum, the undergraduate students at KTH had encountered some scarce examples of sustainable development in engineering, while the concept of green chemistry had not been introduced in detail. Thus, the concepts were introduced from the basics, assuming only superficial previous knowledge levels for the students. Given the disappointing outcome of the initial self-assessment of the course, as described later, we aimed to implement green chemistry by modifying the existing organic chemistry course according to three requirements:

- The modifications should be applied in a manner that allows instructors to maintain the original course structure and content.
- The modifications should provide continuous recitations of the concepts of green chemistry and sustainability.
- The modifications into the existing course should require (relatively) low effort, primarily to encourage adaptation of the approach at other universities.

The changes were gradually introduced over two years to minimize any potentially negative impact on students' development. Changes to the course lectures and literature project were of a relatively conventional nature and are thus detailed in the Supporting Information (S17–S19). As previously stated, the laboratory course did however undergo the most significant changes (Supporting Information S3), including the design of the new practical project. Overall, the described approach combines simple introduction and repetition of green chemistry aspects with a novel laboratory project that can be easily generated for any undergraduate laboratory experiment. The following section outlines the structure of the laboratory course and the approach toward green chemistry integration that was implemented therein.

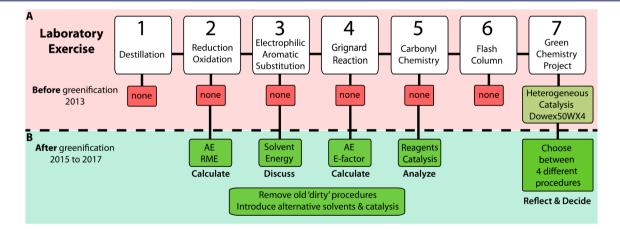


Figure 2. Overview of the changes implemented in the laboratory exercises. (A) Different laboratory exercises before greenification and the extent to which green chemistry and sustainability were treated during each session. (B) Additions/adjustments to the course, where in the last laboratory exercise students use the acquired knowledge to decide on the "greenest" procedure. Laboratory exercises 2–5 included calculating green chemistry metrics such as atom economy (AE), reaction mass efficiency (RME), and the E-factor, and discussing important aspects regarding solvent usage, energy consumption, and catalysis.

Laboratory Course

The laboratory part of KD1270 consists of seven mandatory laboratory exercises of 6 h, during which students become familiar with practical aspects of reactions encountered during lectures (Supporting Information S4–S5). The exercises (both before and after modifications) covered purification by distillation, oxidation/reduction reactions, electrophilic aromatic substitution, Grignard reactions, carbonyl chemistry (e.g., aldol reactions), flash column chromatography, and a green chemistry project. An overview of the laboratory course and the degree of reflection on green chemical aspects during each laboratory session before and after the course improvements is found in Figure 2.

To support increased demand of theory, the single page on green chemistry in the laboratory compendium was expanded to provide written introductions to topics such as green metrics, green solvents, and renewable feedstocks (Supporting Information S6–S10). Furthermore, green-chemistry-related questions were incorporated at the end of each synthesis to provide opportunity for continuous reflection (Table 1). This

Table 1. Green Chemistry Questions and Discussion Points Raised after the Various Experiments

Synthesis Topic	Green Chemistry Questions and Discussion Points
Oxidation and reduction	Calculate the atom economy and RME-value for the synthesis.
Electrophilic aromatic substitutions	Analyze the synthesis with respect to the solvent and energy consumption during the reaction. What are the benefits and drawbacks of the solvent?
Grignard reactions	Calculate the atom economy and the approximate E-factor for the synthesis.
Carbonyl chemistry	Analyze the synthesis with respect to the reagents used. How environmentally friendly are these reagents and your generated product? Is the reaction catalytic?

training, ranging from calculation of green metrics to discussions on alternative solvents and energy consumption, required students to continuously think about their practical work from an environmental perspective. The questions had to be answered in written format in the laboratory journal, with the answers being graded (pass/fail) by the associated teaching assistant. Additionally, some laboratory exercises were updated to more relevant modern examples that introduced students to alternative solvents, such as PEG-400²² and ionic liquids.²³

Laboratory Project

To further promote green-chemistry-based critical thinking, a practical problem-solving project was developed for the laboratory course (cf. Supporting Information S11–S14 for full details on implementation). The project was based on synthesis of the drug 4-methylumbelliferon via a catalytic Pechmann condensation (Figure 3). Before course modifications, students used the immobilized acid catalyst Dowex S0WX4 and solvent-free conditions to conduct the transformation. The environmental aspects of this laboratory exercise were also discussed in small groups ahead of the experiment. The students then realized why this procedure was a good example of green chemistry (e.g., tandem reaction, benign conditions, reusable catalyst).

However, as environmental concerns in organic synthesis are seldom clear-cut, a better way to train students in green chemistry is to expose them to a problem. Thus, four different protocols for the same transformation were extracted from the literature, chosen to highlight different aspects of green chemistry and sustainability in synthesis. The protocols varied in the choice of catalyst, which consequently led to different reaction conditions and workup procedures. From the variety of possibilities for this Pechmann condensation, protocols using copper(II) bromide,²⁴ Amberlyst 15,²⁵ sulfuric acid,²⁶ and iodine²⁷ were adapted (cf. Supporting Information S12-S14). These catalysts were chosen to ensure no alternative was significantly more or less "green" than the other. For example, many transition metal catalysts such as CuBr₂ are inherently toxic and environmentally unfriendly, but the catalyst remains active even at low loadings. Sulfuric acid, which is cheap and readily available, was the only option that could promote the reaction at room temperature, but a large excess of this highly corrosive mineral acid was instead required. In this manner, each of the four protocols had its own advantages and drawbacks (Figure 3), and students had to draw upon their recently acquired knowledge in green chemistry to pick the protocol they identified as the optimal regarding sustainability. The subtle differences in the procedures were intended to make

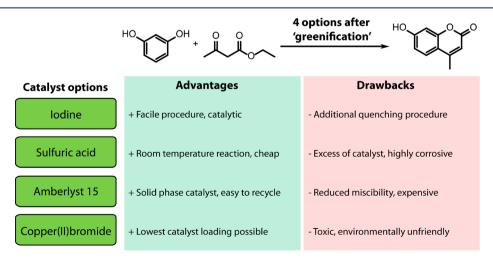


Figure 3. Pechmann condensation of resorcinol and ethyl acetoacetate to form 4-methylumbelliferone. Before modification, the reaction was performed with immobilized catalyst Dowex 50WX4. After modification students were provided with a choice of four options, each with distinct advantages and drawbacks.

each student analyze hazards, waste development, practicality on industrial scale, energy consumption, and other aspects of the synthesis which are normally not highlighted during classical laboratory practice. An interesting aspect of incorporating a small laboratory project as the one described above is the absence of a clear "right" or "wrong" choice in green chemistry experienced by the students. This initiates discussion among peers and helps in realizing that green chemistry and sustainability are not binary in nature. Additionally, the project design benefits from marginal differences in the experimental procedures, promoting the need for critical evaluation by the students, and minimizing the effort required of course administrators to generate similar projects from existing laboratory exercises.

Execution of the laboratory project was done by allowing students to analyze the procedures and select the "greenest" procedure before the start of the laboratory session. The students then carried out their chosen synthesis in the laboratory, leading them to experience practical consequences of their choice. The laboratory project session still opened with a short discussion regarding green chemistry, followed by a quick open survey to quantify the choices made by the students. Following the practical exercise, the acquired knowledge was assessed through a written project report (cf. Supporting Information S14). The laboratory report was supposed to be brief (3–4 pages) and include the following aspects:

- Introduction
- Detailed motivation of why the procedure in question was chosen and the others disregarded
- Advantages and drawbacks of performing the procedure on industrial scale
- Description of the procedure along with observations and results
- Analytical data including yield, thin layer chromatography results, melting point analysis, and calculations of atom economy and E-value
- Discussion about the environmental friendliness of their procedure
- Detailed mechanism, including discussion

The project report was graded by the teaching assistant (pass/fail, cf. Supporting Information S15–S16 for further details), and provided the primary tool for assessing how well the learning outcomes regarding green chemistry had been fulfilled (*vide infra*).

EVALUATION

As previously stated, this study was initiated after investigating how well the old structure of the course "Organic Chemistry, Basic Concepts and Practice 2" fulfilled the green chemistry learning objectives (Supporting Information S2). Before the modifications, no examination on green chemistry was performed. Instead, a survey was conducted among the students (76 individuals) to self-assess their knowledge of green chemistry and sustainability. In the survey, the students were allowed to take a position for eight statements (Table 2). The survey ended with an open question, where students could express which laboratory exercise provided the best understanding of green chemistry, and why this exercise contributed the most (cf. Supporting Information S21).

Table 2. Statements in the Survey Provided to Students

Number	Statements for Student Response ^a
1	I believe that the laboratory course has contributed to my understanding of what green chemistry means.
2	I know more about green chemistry after the laboratory course than that I did before.
3	I can, after having taken this laboratory course, assess how well a synthesis follows the green chemistry principles.
4	I think I will have use of my knowledge about green chemistry as an engineer.
5	I believe that the laboratory course has contributed to my understanding of what sustainable development means.
6	I know more about sustainable development after the laboratory course than that I did before.
7	I think I will have use of my knowledge about sustainable development as an engineer.
8	I can, after having taken this laboratory course, give examples of

8 I can, after having taken this laboratory course, give examples of connections between green chemistry and sustainable development.

"The options to answer the statement were provided as "Fully Agree", "Partly Agree", "Partly Disagree", and "Fully Disagree".

Survey Results

The survey conducted in 2013 (Supporting Information S22) provided a "reference point" assessment aligned with a course with only a low amount of reflection on green chemistry and sustainability. Re-evaluation of the course after implementation of the modifications was performed with students participating in the 2015 curriculum (67 individuals, Supporting Information S23), and the observed results were validated with students participating in the 2016 and 2017 curriculum (74 and 78 individuals, Supporting Information S24–S25) (Figure 4).

Before the course modifications, statements including the term "green chemistry" were on average answered with partial agreement by students (Table 2, statements 1-4). However, the more complex term "sustainable development" scored

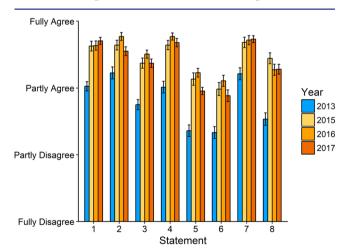


Figure 4. Survey results reflecting the students' perceptions on their green chemistry/sustainability knowledge obtained during the course and their perceptions toward usability of this knowledge now and in their futures. The bars represent average scores on the statements provided in Table 2, prior to "greenification" (blue, spring term 2013, n = 76) and post "greenification" (different shades of orange, spring term 2015–2017, n = 67, 74, and 78, respectively). The bars represent the mean of the results obtained from the survey answers by attaching each option to a numerical value (1 = Fully Disagree to 4 = Fully Agree) and are represented with their respective standard error of the mean.

lower, and even though there seemed to be partial agreement that knowledge on this subject was useful (Table 2, statement 7), the survey revealed that there was a tendency to disagree with the other statements including this term (Table 2, statements 5–6 and 8). After the course modifications were implemented, the survey showed an increase regarding the agreement of students with the provided statements. This not only indicates that students felt a better understanding of the topics of green chemistry and sustainability was obtained, but also that these topics are considered more important for future career development (Table 2, statements 4 and 7). The survey conducted in 2016 and 2017 revealed similar results.

From the open questions in the end of the survey it could be seen that, in the 2013 curriculum, 96% of the students had, as expected, chosen the laboratory exercise with the main topic of "green chemistry" as most rewarding. Representative comments read: "because it was about green chemistry", "because we used Dowex instead of acid", and even "the other laboratory exercises did not contribute at all". These explanations contributed to the impression that, after the course, the students had heard of sustainable development and green chemistry, but could not apply the acquired knowledge. After "greenification" of the course, an average of 84% over 2015-2017 still chose the same laboratory exercise as most rewarding, though the exercise was now transformed into a full project. However, the motivations were more satisfactory and ranged from "we had to think for ourselves" and "it required more preparation" to "we had to think through all we went through before". It is apparent that the independent choice in the project required students to apply their acquired knowledge, rewarding them with a deeper understanding of the subject.

Examination

Achievement of the required learning objectives by students was assessed primarily through grading of written answers to green chemistry questions after each laboratory exercise and grading of the discussion in the project report (cf. Supporting Information S15-S16). All teaching assistants noted clear progression in understanding of green chemistry concepts as students provided better answers to the questions provided after each practical laboratory exercise. Discussion with representative students during course evaluations confirmed that students considered the progressive green chemistry questions associated with practical laboratory exercises to be beneficial for translating the theory behind green chemistry into practice. Subsequently, the attainment of the green chemistry learning objectives was clear from the written project report, as the green-chemistry-related discussions were generally at a high level and indicated a fundamental to advanced understanding of the concepts for >90% of the students.

CONCLUSION

A simple and time-efficient method for vertical integration of the topics "green chemistry" and "sustainability" into an existing organic chemistry course was demonstrated. This method combined a conventional approach to introduce students to key terms with a novel laboratory project design allowing continuous rehearsal and reflection of the acquired knowledge. The project design is easily implemented within existing laboratory exercises, and the currently described Pechmann condensation project can easily be conducted as a stand-alone laboratory exercise in green chemistry as well. This design provides students with a good basis in the topics "green chemistry" and "sustainable development" for their future careers, while also being simple to implement into existing courses and applicable to disciplines other than organic chemistry, as well.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.7b00720.

Intended learning outcomes of the course, summary of modifications to laboratory course, laboratory course outline after modification, green chemistry chapter from compendium, detailed green chemistry project description, student report evaluation, literature project after modification, survey design, and survey results (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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