DESIGNING AND CONDUCTING AN OPEN-ENDED EXPERIMENT AS AN ORGANIC CHEMISTRY LABORATORY PRACTICAL

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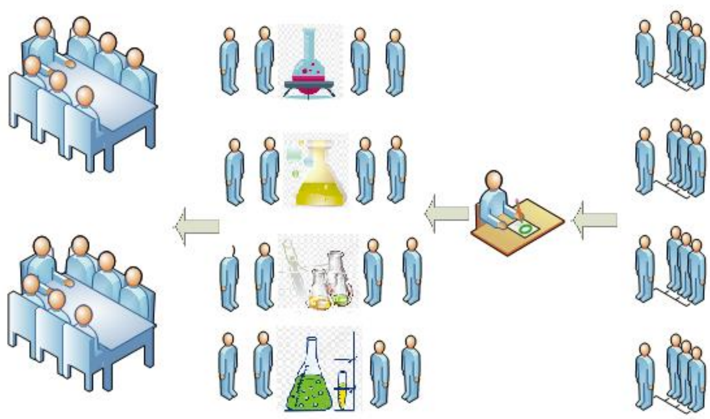
ABSTRACT

An open-ended laboratory practical has been developed that challenges students to evaluate the synthesis of aspirin, which is a typical experiment of organic synthesis. In contrast to a traditional textbook laboratory practical, the overall grade includes an evaluation of the experimental preparation, process, report, and PowerPoint presentation of the student. This practical improves undergraduate students' ability to think innovatively and the skills pertaining for team cooperation, which would exhibit importance in the organic chemistry teaching experiment's course. *[African Journal of Chemical Education—AJCE 9(2), July 2019]*

GRAPHICAL ABSTRACT



INTRODUCTION

Organic chemistry is a practical subject. It is closely related to the development of materials, energy, environment, biology, agriculture, pharmaceuticals, and other related science and technology. Experiment is not only the cornerstone of this discipline but also the source of innovation of chemistry knowledge, which highlights the important role of basic chemistry experiment teaching in cultivating innovative talents. During the teaching of chemical experiments, students install instruments and devices by participating in the entire experiment. Observing the experimental phenomenon and analyzing the results can enrich their perceptual knowledge and deepen the understanding of basic chemical theory to obtain and master basic knowledge, then ultimately improve the thinking ability, practical ability, and innovation ability.

Traditional experiments involve listing the principle, steps, reagent dosage, and even each step can be as one experiment. Due to the limitation of teaching mode, students can only learn from the textbook in "mechanical" operation practice involving the same steps and schedules. The synthesis of aspirin from salicylic acid and acetic anhydride is the most basic experiment in the experimental teaching of basic organic chemistry in university [1,2]. The experiment with salicylic acid and acetic anhydride as raw materials, involving the alcoholysis reaction of acetic anhydride to prepare acetyl salicylic acid, is aimed at using the alcoholysis reaction of carboxylic acid derivatives in the preparation of ester compounds. The synthesis of ester compounds is a typical reaction [3-5]. These experiments can motivate the students to gain theoretical knowledge and understand the alcoholysis reaction of carboxylic acid derivatives, making them familiar with basic experiment skills such as ordinary distillation, extraction and recrystallization, helping them create a solid foundation for subsequent related courses.

In the light of the widely used product and the importance of experimental contents, the experiment courses of basic organic chemistry in university teaching and practice mainly focus on the experimental skills of students and improvement of their comprehension ability.

An open-ended experiment in basic organic chemistry is reported. This experiment is comprehensive in terms of teaching students how to find relevant documents and screen experiment schemes. The experiment course that is determined based on the acquired knowledge and the obtained product in the form of written and PowerPoint documents with the results is discussed. This method has been applied in practice for many years to optimize teaching staff and laboratory resources.

EXPERIMENTAL DESIGN

Goals and objectives

The four hours' period in laboratory was used to inspect the improvement of experimental skills and comprehension abilities of sophomores. Specifically, the goal of this experiment was to train students in the following:

- Find literature using Scifinder
- Evaluate relevant literature
- Prepare an experiment based on the published results
- Achieve a common goal within the team
- Present the results in a professional manner
- Share research results with classmates
- Analyze the reasons for different experimental results

Firstly, the synthesis of aspirin may seem like a simple reaction, but a variety of catalysts should be used. This provides opportunity for students to look up a lot of available literature because there are many different catalysts to be chosen, such as (L)-ascorbic acid [11], AcONa [12], H_3PO_4 [13], H_2SO_4 [14], C_5H_5N [15], $NaHSO_4$ [16], KH_2PO_4 [17], citric acid [18], $Zn(OAc)_2$ [19], $NiSO_4$ [20], p-$MeC_6H_4SO_3H$ [21], and CaO [22]. Secondly, the characterization and purity analysis of aspirin are relatively simple. Simple infrared spectroscopy and liquid chromatography can be used to determine whether aspirin is pure or not and analyze the purity level. This is more suitable for the relatively ordinary Chinese universities. Thirdly, the goal of this project is to highlight the following point: let students choose their own experimental scheme, rather than simply follow the textbook process. Problem-solving lessons would be learned during the execution of experiment. Finally, the goal is to arouse the interest of students in the experiment, then make them truly understand the experiment they want to do, think deeply about it and carry out the experiment with questions. They should also be able to master the basic steps involved in the chemical synthesis of drugs and simple separation and purification processes.

Because the assigned work exceeds what one student could reasonably expect, the project was considered a cooperative group task. Generally, there are two or three students in a group. In addition, there is evidence that cooperative group learning can improve students' attitudes, understanding of concepts, on-the-job performance, and what's more, the meta-cognitive ability to solve problems that are open or not clearly defined. Finally, group work imitates research in a professional environment, helping to narrow the gap between undergraduate learning experience and the laboratory work.

Preparation

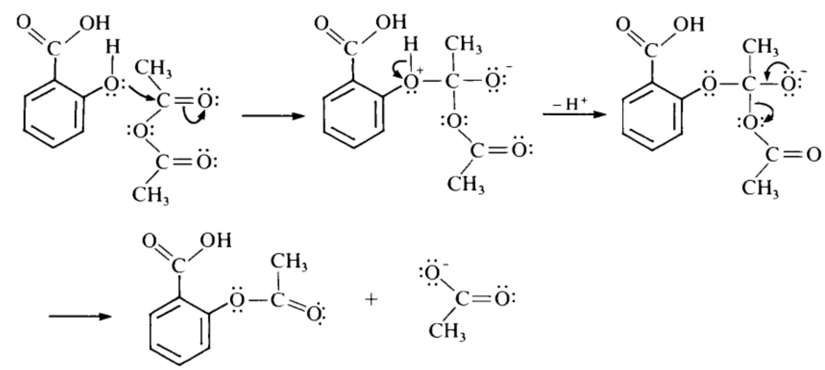
Before coming to the lab, the students had two weeks to prepare their projects and put forward detailed plans for the selection of catalysts and the design of experimental schemes. Two or three students in each group discussed the experimental method in detail with the instructor before performing the experiment and gave the reasons for choosing their particular scheme. Students were also asked to attend a training course in which science librarians explicated how to search and retrieve documents in terms of how to use SciFinder and Reaxys to search structures and keywords. Finally, the students were given a tutorial on how to download and use ChemDraw.

All of the above preparations were not covered in previous courses. Once literature retrieval was completed, the students selected a catalyst and designed the experimental scheme according to the literature. In the experimental design, the starting material was limited to 1.38 g (0.01 mol). The design also included the size of reaction flask and other glass instruments, reaction conditions, and methods of purifying the reactant and characterizing the product. Students should submit in advance their reports in the written form and can only conduct experiments with the permission of instructor. This is a practical question to make sure that all the students in a group are assigned to discuss what type of equipment to prepare, how much laboratory reagent is needed, ratio of reactants, and how to manage the time properly. Here, experiments involving high temperature, long reaction time, high toxicity, or sensitivity to water or oxygen were removed under the guidance of instructors.

During the experiment, two students worked as a group to complete the task independently. Students of the same subject should be grouped together as much as possible and conduct experiment at the same time, so as to compare the experimental phenomena with each other, discuss the problems in time and try to solve them. When students carry out experiments with questions, they will carefully observe the experimental phenomena, pay attention to the changes in the reaction system at any time, carefully grasp every step, and truthfully record every observation. Teachers should keep checking in the laboratory in order to correct the operational errors and deal with emergencies. In addition, they should supervise the students to faithfully record the dosage of catalyst used, the experimental phenomena observed and the results obtained, leading to the final calculation of the production rate of acetyl salicylic acid.

Experimental execution

Understanding the fundamental mechanism is very important in organic synthesis. The mechanism for the acetylation of salicylic acid [2] is as follows:



Acetylsalicylic acid

According to the reaction mechanism, one way is to activate acetic anhydride and increase the electronegativity of the carbonyl groups. Another way to activate salicylic acid is through an alkaline catalyst. Therefore, students can design this experiment using either an acid or a base as the catalyst. The students had a week to prepare the plan for the experiment. However, the entire experiment took four hours to complete. The amount of salicylic acid, acetic anhydride and catalyst used was 0.01 mol, 0.02 mol and 0.30 g, respectively.

Based on the twelve different catalysts selected, twelve different experimental subjects were set up. Students were divided into different groups, with each group given a topic. Students should refer to their subject literature for the relevant information, summarize and discuss within the group, develop an experimental scheme, specify the experimental principle, instruments, reagents and the experimental steps required. According to the existing laboratory conditions and the practical operating abilities of students, the teachers should guide the experiments for each group, make detailed modifications, point out the deficiencies, and finally determine the appropriate experimental scheme for each group. The reaction conditions and experimental results of the synthesis of acetyl salicylic acid using 12 different catalysts are shown in Table 1.

Table 1. Experimental results of each group

Group	Catalyst	Yield (%)
1	(L)-Ascorbic acid	54.5
2	AcONa	60.1
3	H_3PO_4	87.9
4	H_2SO_4	38.1
5	C_5H_5N	68.9
6	$NaHSO_4$	61.5
7	KH_2PO_4	60.1
8	Citric acid	25.1
9	$Zn(OAc)_2$	32.5
10	$NiSO_4$	58.4
11	p-$MeC_6H_4SO_3H$	40.5
12	CaO	27.8
13	None	8.7

Because the students carried out this experiment for the first time, they didn't have to obtain the highest yields. Table 1 showed the results of final yield, with most student groups obtaining lower yields than that published in the literature, though, in certain groups, the yields were more than that reported in the literature. With respect to the students' results for the 2018 spring semester, 84% of the students completed all the experiments, including separation and purification.

Only a small number of research groups gained the product yields that were consistent with or exceed the literature values. Although the actual output is not the basis for evaluating the success of the experiments, these results show that students can solve problems by themselves. However, here are some questions worth discussing. What effect would (L)-ascorbic acid or citric acid have? How might H_3PO_4, H_2SO_4 or p-$MeC_6H_4SO_3H$ affect the nucleophilic attack of the phenolic oxygen on acetic anhydride? How might AcONa, $NaHSO_4$, KH_2PO_4, $Zn(OAc)_2$, and $NiSO_4$ affect the yield of the reaction? With what compound might C_5H_5N, CaO, and the base associate? Through such questioning, although each student only performed one experiment, over the course of the joint discussions, the student will understand the conditions and catalytic effects of the reaction for 12 different catalysts. This is helpful in broadening their horizons and triggering their enthusiasm to participate in the experiment. The experimental skills of the students can also be improved.

RESULTS, DISCUSSION, AND PROBLEM SOLVING

After the experiment, each team member firstly compared his/her result with that of others in the group during the discussion, and tried to solve the problems by thinking, instead of approaching the teacher for help at any time; teachers should be involved with the students to discuss and guide them in solving problems. After the group discussion, each group presented its own experimental scheme, procedures, results and the problems encountered during the experiment in the form of a PowerPoint presentation to the class. In this manner, although each student only did one experiment, over the course of the joint discussion, he/she would understand the conditions and catalytic effects of the reaction under twelve different catalysts. For many students, this was their first attempt to write a research report and make a PowerPoint presentation. Students used PowerPoint to report their research results, which helped them develop a deeper understanding of the experiment’s significance.

The final results were based on the experimental preparation, process, report, and presentation. The experimental report was written as a group and the students were required to complete it together. The criterion for the report was to complete the process under review, discuss the data accurately, evaluate the results that included production and spectral characterizations, show the experimental details, and provide suggestions for improving the experiment.

Hazards

Some individuals may be allergic to aspirin. Acetic anhydride is toxic and can be irritated. Some of the catalysts used in the experiment (H_2SO_4, H_3PO_4, C_5H_5N), which can be highly corrosive, may also cause severe skin or eye burns. Most of the reagents used in the experiment otherwise are safe.

FEEDBACK AND ASSESSMENT

College students performed innovative experiments with the aim of producing a good communication platform for teachers and students. This is one of the important strategies for research universities to cultivate innovative talents. Here, the authors tried to introduce innovative teaching contents and methods, so that the students could be active in the experimental class. Teachers provided experimental subjects, divided the students into groups, and made full use of the preview time to check the text by themselves, in order to present the data, design the experimental plan, conduct the experiment, analyze the experimental results of the groups independently, and compare advantages and disadvantages of the experimental plan of each group through communication and discussion both within and among the groups. The authors of this paper put forward the reform scheme for four years as a part of teaching practice, and the students involved participated a survey based on a questionnaire that sampled a total of 240 students. the survey results were shown in Table 2.

Table 2. Results of survey based on questionnaire involving 240 students

Entry	Question	Scores				
		5	**4**	**3**	**2**	**1**
1	Did the experiments help you understand the purpose of the course?	124	101	15	0	0
2	Did the experiments teach you useful lab techniques?	221	19	0	0	0
3	Did the experiments teach you to search the literature?	235	5	0	0	0
4	Does the experiment help you better understand the organic synthesis presented in the textbook?	189	25	26	0	0
5	Do you want organic synthesis experiments to be designed in this way?	218	21	1	0	0

Practice and survey over the past three years have shown that overwhelming majority of the students give a positive answer. The results illustrate that the students are willing to consider the suitability of the project and demonstrate their willingness to be involved in such experiments. When asked “Does the experiment help you better understand the organic synthesis presented in the textbook?”, only 78% of the students scored five points. This may have something to do with the simplicity of the experiment. More than half of the students thought that doing the experiment helped them understand its purpose. In 2018, students were asked “Did the experiment teach you to search the literature?”, and 98% of the responses were obtained in the affirmative. The feedback showed that students valued the project and considered it a worthwhile exercise. All of the above results indicated that the goal of the project had been achieved.

CONCLUSION

In this open experiment, students were exposed to the abovementioned professional skills, which are key to the overall development of college students. Students have mastered the ability to use online databases to obtain specific journal articles, select appropriate methods based on the literature, discuss with the instructor, and finally, determine the experimental scheme. Through cooperative work, students designed experimental methods, set up research goals, obtained experimental data, and completed experiments in groups. Each team came up with a solution to achieve the project goals.

Students were required to write a comprehensive experimental report, including research background, experimental details, results and conclusions. The students made PowerPoint presentations summarizing the findings of their projects. These skills are not required in normal organic chemistry experiments, in which the instruments, processes, discussion and analysis are written for the students to follow. Although the progress of the students varied from semester to semester, the project aimed to promote these skills. The introduction of innovative teaching contents and methods greatly stimulated the thirst for knowledge among the students, and made them acquire knowledge and skills through both passive and active learning, which could not only improve their experimental operation capabilities, but also their ability to think innovatively and cooperate as a team. Both of the abilities have been given significance in the organic chemistry teaching experiment courses.

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