Orbital
The electronic journal of chemistry

the electronic journal of chemistry

Paper on Education | http://dx.doi.org/10.17807/orbital.v17i4.22059

Accelerated Esterification by Domestic Microwave for the Undergraduate Organic Chemistry

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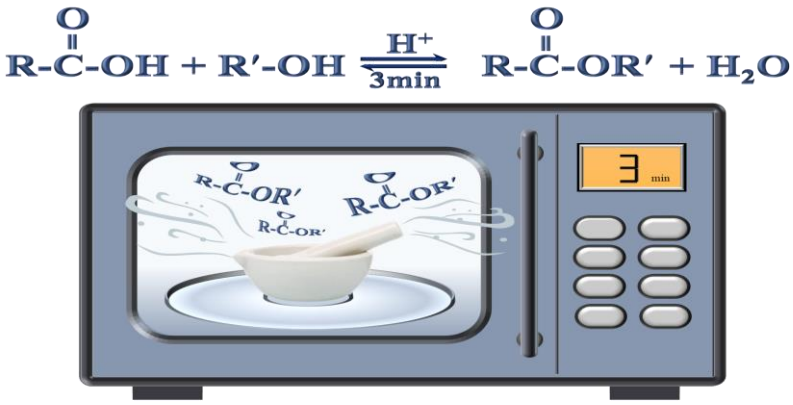
The aim of this work was to offer a green laboratory experiment for undergraduate organic chemistry course. Esterification accelerated by domestic microwave was studied. The reaction occurred within 3 minutes. The odor of the product was used as a tool to indicate the reaction. This convenient, simple and fast experiment provided ground for the lessons on esterification mechanism, nomenclature of ester, physical properties, principle of microwave and green chemistry.

Graphical abstract



Keywords

Kitchen microwave
Esterification
Green chemistry

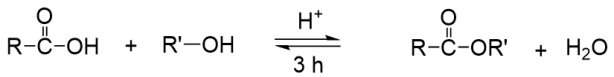
Article history

Received 11 oct 2024
Revised 21 Apr 2025
Accepted 27 Mat 2025
Available online 28 June 2025

Handling Editor: Adilson Beatriz

1. Introduction

Esters can be prepared by the reaction of carboxylic acid and alcohol in the presence of acid catalyst which is known as Fischer esterification [1, 2].



Fisher esterification

This procedure requires a long period of reaction time which is not appropriated for 3 hours undergraduate laboratory class**.** We aimed to present a new experiment laboratory for describing Fisher esterification to the students in short time**.** The experiment required only 3 minutes for each reaction by using a domestic microwave as heating source [3, 4]. The purpose of the experiment was to introduce microwave [5], green chemistry [6, 7], physical properties and nomenclature of esters, and acid catalyzed reaction mechanism of esterification to students [8].

In this study, we provided a simple and convenient experiment for learning Fisher esterification. The odor of ester product was used as a tool to indicate for the reaction.

2. Material and Methods

The experiment laboratory was performed by second year students majoring in chemistry in organic chemistry

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laboratory course. The reaction was carried out in Panasonic, Microwave Oven, NN-SM330M Model at low temperature level and 3 minutes which was the optimal condition in this study. All chemical in the experiment were analytical grade.

The optimal condition was studied by examining various condition for a simple reaction of 3 mL of ethanol, 2 mL of butanoic acid and 5 drops of sulfuric acid. All examined reactions were carried out in the microwave oven. The results were shown in Table 1.

Table 1. The smell result from various conditions.

Temperature	Time (Min)	Smell
High	3	burnt
Mid-high	3	burnt
Medium	3	strawberry (strong)
Medium	2	strawberry (weak)
Mid-low	3	odorless

There were 2 parts in this experiment. In the first part, students investigated the odors of 15 known esters from the reaction of 15 pairs of carboxylic acids and alcohols in the presence of sulfuric acid. Each student ran one reaction. After all reactions were run ester product were formed. Students then smelled the odor of the know esters and used them as references.

In second part, each student was given a pair of unknown carboxylic acids and alcohols. The reaction of these in the presence of sulfuric acid gave the corresponding unknown ester with specific odor. Students then used observations in part 1 to predict the ester product. They were also asked to write down the structure of the carboxylic acid and the alcohol that formed the ester product.

Preparation of ester

A mixture of carboxylic acid (2 mL or 0.2 g for solid), alcohol (3 mL) and 5 drops of sulfuric acid were added into a mortar. The reaction mixture was heated in a domestic microwave for 3 minutes at low temperature. After the reaction mixture was cooled to room temperature, 20 mL of saturated sodium carbonate was added. Then, each student smelled the product and identified the odor.

Pairs of carboxylic acid and alcohol used in this experiment were provided in Table 2.

Table 2. The chemical reagents for running the Fischer esterification in part 1 and part 2.

pair	carboxylic acid	alcohol	ester
1	butaric acid	methanol	methyl butanoate
2	butanoic acid	ethanol	ethyl butanoate
3	butanoic acid	pentanol	pentyl butanoate
4	butanoic acid	octanol	octyl butanoate
5	propionic acid	ethanol	ethyl propionate
6	propionic acid	benzanol	benzyl propinoate
7	acetic acid	pentanol	pentyl acetate
8	acetic acid	octanol	octyl acetate
9	acetic acid	benzanol	benzyl acetate
10	formic acid	ethanol	ethyl formate
11	formic acid	pentanol	pentyl formate
12	benzoic acid	ethanol	ethyl benzoate
13	salicylic acid	methanol	methyl salicylate
14	salicylic acid	butanol	butyl salicylate
15	cinnamic acid	butanol	butyl cinnamate

3. Results and Discussion

The goal of this experiment was to develop laboratory direction on Fisher esterification that could be performed in a domestic microwave oven, which was faster, greener and more available than the traditional method. Students in a second-semester organic chemistry laboratory were able to learn acid catalyzed esterification. Esters could be formed by the reaction between various carboxylic acids and alcohols in the presence of sulfuric acid. The reaction occurred in **3** minutes by using a domestic microwave as heating source. The odor of product was used to indicated the reaction without purification or monitoring the progress of the reaction.

In part 1, every student picked up a pair of reagents as shown in Table 3 and ran the reaction. After the reaction were completed by 15 students, they smelled all the 15 esters and identified the odor. The results were shown in Table 1 as percentage of students correctly identified the smell of each ester.

As shown in Table 3, some esters were easily recognized such as pineapple (pair 1), strawberry (pair 2) and wintergreen (pair 13). Some of them were not familiar for Thai people such as apricot (pair 3), rum (pair 10) and prunes (pair 11). In fact, poor smelling ability in human may give an error. The students could not tell exactly odor of the esters. They just sense of present smell (pair 4, 12 and 14).

In general, students had been warned to not smell things in the laboratory, however, the esters performed here had pleasant smell and low toxicity. Furthermore, carboxylic acid, alcohol and sulfuric acid were used in very small amounts and not hazardous [2, 11].

Part 2, each student was given a pair of unknown, carboxyl acid and alcohol. They ran the reaction of these two unknowns under the same condition as in part 1 to form the corresponding ester. Once their reaction was completed, the odor of the product was compared to the data they had recorded on their own and decided what ester they had prepared. Then, they analyzed the structure of the carboxylic acid and the alcohol was reacted to form the ester product. The result of their identification and analysis was shown in Table 4.

Table 3. The results of identifying the odor of ester products [9, 10].

pair	carboxylic acid	alcohol	ester	odor	Identified odor*	% correctly
1	butanoric acid	methanol	methyl butanoate	Pineapple apple	pineapple (15)	100
2	butanoic acid	ethanol	ethyl butanoate	strawberry	strawberry (14) perfume (1)	93
3	butanoic acid	pentanol	pentyl butanoate	apricots	jasmine (9) others (6)	0
4	butanoic acid	octanol	octyl butanoate	fruity	flower (10) others (5)	0
5	propionic acid	ethanol	ethyl propionate	pineapple	pineapple (3) others (12)	20
6	propionic acid	benzanol	benzyl propinoate	flower	flower (3) others (12)	20
7	acetic acid	pentanol	pentyl acetate	pear	banana (9) others (6)	60
8	acetic acid	octanol	octyl acetate	orange	orange (8) others (7)	53
9	acetic acid	benzanol	benzyl acetate	jasmine	jasmine (3) others (12)	20
10	formic acid	ethanol	ethyl formate	rum	flower (9) others (6)	0
11	formic acid	pentanol	pentyl formate	prunes	oil (12) others (3)	0
12	benzoic acid	ethanol	ethyl benzoate	perfume	oil (8) others (7)	0
13	salicylic acid	methanol	methyl salicylate	wintergreen	wintergreen (15)	100
14	salicylic acid	butanol	butyl salicylate	grape	perfume (8) others (7)	0
15	cinnamic acid	butanol	butyl cinnamate	chocolate	chocolate (8) others (7)	53

*number in ( ) as a number of student

Table 4. Prediction of esters and analysis for the starting materials.

student	unknown	results			x or /
		Identified odor	ester	Alcohol+carboxylic acid	
1	A	wintergreen	methyl salicylate	methanol+salicylic acid	x
2	B	strawberry	ethyl butanoate	ethanol+butanoic acid	/
3	C	banana	pentyl acetate	pentanol+acetic acid	x
4	D	bad smell	pentyl acetate	pentanol+acetic acid	x
5	E	wintergreen	pentyl acetate	pentanol+acetic acid	x
6	F	banana	benzyl acetate	benzanol+acetic acid	/
7	G	orange	octyl ethanoate	octanol+ethanoic acid	x
8	H	wintergreen	ethyl acetate	ethanol+acetic acid	x
9	I	wintergreen	ethyl salicylate	ethanol+salicylic acid	/
10	J	wintergreen	methyl salicylate	methanol+salicylic acid	x
11	K	apricot	pentyl butanoate	pentanol+butanoic acid	x
12	L	bad smell	pentyl formate	pentanol+formic acid	/
13	M	perfume	ethyl benzoate	ethanol+benzoic acid	x
14	N	wintergreen	methyl salicylate	methanol+salicylic acid	x
15	O	jasmine	benzyl acetate	benzanol+acetic acid	x

** / = correct, x = incorrect

In Part 2, only 4 of 15 students (27%) could identify the odor of unknown esters correctly. All of these esters had familiar and strong smells which were easy to identify. Almost all of the students lost their sense of smell because they got confused due to cloudy volatile esters in the laboratory room. Many of them gave wrong prediction of the ester by smelling sense. However, they had an opportunity to discuss and enjoy together with learning the experiment laboratory. This experiment was appropriated for learning Fischer esterification.

4. Conclusions

Domestic microwave oven could be applied to use as a heating source in organic laboratory. This experiment gave more advantages compared to the traditional method. Firstly, faster because the heating passed through the vessel walls directing into a chemistry reagent. In traditional method, the heating from hotplate heated the glass vessel and transferred to the solution slowly. Secondly, it is more convenient

because glassware was not necessary, and thirdly, this experiment was quite green as small quantities of chemicals were used and consumed less energy than traditional experiment.

Domestic microwave assisted the reaction of Fisher esterification for organic chemistry laboratory. This reaction experiment was very fast, green and easy to perform. Students had learned and discussed on microwave, green chemistry, esterification, nomenclature of ester and their odor properties.

Acknowledgments

Grateful thanks are extended to Rajamangala University of Technology Isan, Khon Kaen Campus, Khon Kaen, Thailand for Financial support. I would like to thank Surangkhana Budsombat for helpful discussion.

Author Contributions

Ruchanok Tearavarich conceived the study, designed the study, coordinated the study and composed manuscript.

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How to cite this article

Tearavarich, R. *Orbital: Electronic J. Chem*. **2025**, *17*, 393. DOI: **http://dx.doi.org/10.17807/orbital.v17i4.22059**