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ACCOMPLISHING THE SYNTHESIS OF DIBENZALACETONE BY USING RETROSYNTHESIS APPROACH TO EXPLORE STUDENTS' PERFORMANCE

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Abstract

The university students need to have various skills in using the retrosynthetic approach to solve the problem of synthesis of organic compounds. This study aims to determine students' skill in using the organic retrosynthesis approach. Descriptive method was used in this study. The respondents were 16 students from the chemistry education department. This study used performance instrument to determine students work with the retrosynthetic approach to accomplish the synthesis Dibenzalacetone. Nonparametric differentiation statistic was applied to verify the differences in skills possessed by students between each group. The research found a difference in students' performance using the retrosynthetic ways. It was indicated by the performance of students in doing the approach of retrosynthesis. It was found that the biggest score of students' performances is in characterizing the Dibenzalacetone compound and the lowest is in making the retrosynthesis of the Dibenzalacetone. It implicated that lecturer must intensively give the learning subject of the retrosynthesis of organic compounds by using scaffolding to improve students' skill in the organic synthesis.

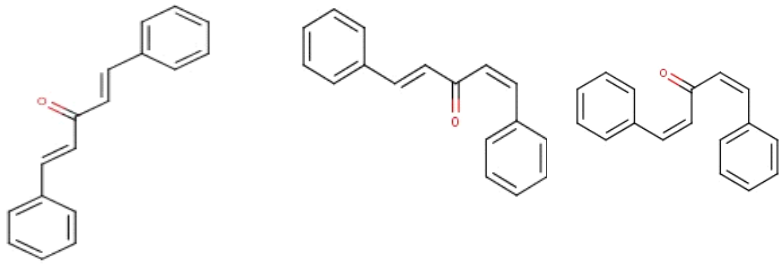
Keywords: Dibenzalacetone, Retrosynthesis approach, Students' performance.

1. Introduction

In learning the organic subject, it is found that organic synthesis problem was considered a difficult problem that has been studied by students [1]. It is also found from the previous study that students' understanding of the prerequisites concepts was closely related to their abilities in synthetic organic compounds [2]. In addition, students' skills in solving a problem in organic chemical synthesis were also very limited. In the previous study, it used problems using scaffolding to help students solve organic chemical [3]. Some studies used the problem-solving method for synthesizing organic compounds as scaffolding [4-6]. In other studies, researchers sought to find out about students' preparation in solving the case about synthesis of organic compounds through project methods in the laboratory. This method is known to be able to improve generic science and critical thinking skills [7, 8].

Mostly, the students use a laboratory project in order to synthesize the Dibenzalacetone compound. Dibenzalacetone is a compound that has been recognized as the basic ingredient for making sunblock cream that can counteract UV radiation. The presence of various UV sunblock cream products in the market will make students more familiar with the functions of Dibenzalacetone compounds. It is because the use of Dibenzalacetone compounds is very common with students' daily lives [9].

Dibenzalacetone compound is a cyclic aromatic compound that has two known phi bonds found in double bonds. The existence of the double bond makes a structural variation available [10]. The isomers structure of the Dibenzalacetone compounds can be seen as shown in Fig. 1.



(E, E)- Dibenzalacetone (Z,Z)- Dibenzalacetone (E, Z)- Dibenzalacetone

Fig. 1. The isomers structure of the Dibenzalacetone.

According to Fig. 1, there are three isomer structures of Dibenzalacetone that can be produced in the synthesis Dibenzalacetone. Each of isomers has its specific characterization and difference that can be noticed by the students in working through their synthesis activity in the laboratory.

Synthesis of Dibenzalacetone occurs via an aldol condensation reaction in Fig. 2. Figure 2 shows the reaction of an aldehyde with a ketone employing sodium hydroxide as the base which is an example of an aldol condensation mixed reaction. It is a double mixed-aldol condensation reaction between acetone and

benzaldehyde. Acetone has α-hydrogens (on both sides) and thus can be deprotonated to give a nucleophilic enolate anion. The aldehyde carbonyl is much more electrophilic than ketone, therefore it reacts rapidly with the enolate.

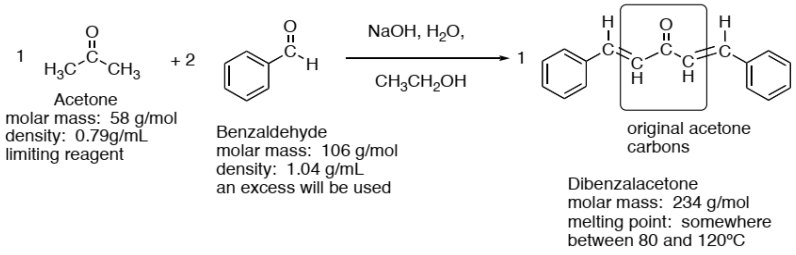


Fig. 2. Synthesis reaction of Dibenzalacetone [10].

Five steps were stated in the synthesis of Dibenzalacetone according to the synthesis reaction via aldol condensation. However, students need to understand every step to embrace the synthesis of Dibenzalacetone. This approach has often been used to answer the problem of organic synthesis. Students also need good skills of retrosynthesis through Dibenzalacetone so that they can successfully synthesize organic compounds [11].

On the other hand, the synthesis material of Dibenzalacetone compounds is one of the lab materials in the laboratory which is generally given to students. The synthesis material of Dibenzalacetone compounds is advanced organic chemical material which is related to the aldol condensation reaction. Consequently, Aldol condensation reaction is a concept that must be mastered by students in learning advanced organic chemistry [12]. The practice of synthesizing Dibenzalacetone compounds based on the reaction of Aldol condensation which is taught to students is only limited to laboratory activities that follow the procedure.

The previous research always emphasized on how students' performances in solving the problem of synthesis Dibenzalacetone but never obviously show students' performance through every stage of laboratory project to synthesis Dibenzalacetone [13, 14]. Therefore, the purpose of this study is to explore students' performance using the retrosynthesis approach to synthesis Dibenzalacetone.

2. Method

This research used the descriptive method from the results of the student's performance test. The participants were 16 fourth year students (4 males and 12 females) from one of the universities in Papua. The students were divided into 4 groups with 4 students in each group. All students had finished following the organic chemistry 1, 2 and advanced organic chemistry courses that conclude all prerequisite concepts to learn synthesis organic. The students were given the Test of Logical Thinking (TOLT) [15]. It was found that the students were at the same level in the cognitive formal operational. It means all students were able to think abstractly about what was required in solving the synthesis of organic problems.

Data were obtained from the performance of students when they were completing the worksheets given to them. They had to follow the stages of the laboratory project by using the retrosynthesis approach. The students' performance focus on five (5) stages: (1) identify the characteristics of Dibenzalacetone by using literature in media online and offline software of Student Spartan V6 and MarvinSketch ChemAxon; (2) hypothesize the retrosynthetic pathway of Dibenzalacetone; (3) make a retrosynthesis analysis of Dibenzalacetone by using media online and offline software Reactor ChemAxon; (4) propose a synthesis planning design based on retrosynthetic analysis of Dibenzalacetone; (5) make a synthesis of Dibenzalacetone in laboratory according to planning design.

In this study, the Kruskal Wallis test with a 0.05 critical point was used to determine whether there are differences in the performance of each group in changing the stages in the synthesis of Dibenzalacetone [16]. The instrument used for the synthesis of Dibenzalacetone was a performance test. The performance test given was related to the stages of retrosynthesis of organic compounds, in which the students'performance criteria can be discussed as

(1) The group of students discusses various possibilities for retrosynthetic analysis of the target molecule and the stages of retrosynthesis.

(2) Students in groups determine the hypothesis regarding the retrosynthesis analysis of the Dibenzalacetone.

(3) Design the retrosynthetic hypothesis of Dibenzalacetone using the *Marvinsketch and reactor ChemAxon software.*

Then, the score can be classified as several points:

- 0: Not answer retrosynthesis;
- 1: only do one performance criterion in stage 2;
- 2: only do two performance criteria in stage 2;
- 3: only do three/all performance criterion in stage 2

Based on above explanation, it shows an example of the assessment performance criteria. It also shows the score for performance in stage 2. All participants' score will be collected and become the group score.

3. Results and Discussion

Students need the skills in using the retrosynthetic approach in order to complete the synthesis of a molecular target compound. In addition, it is also known that the skills in conducting the retrosynthesis of organic compounds require a variety of integration of basic concepts that have been possessed by students. In this study, the stages of the retrosynthetic approach were divided into 5 stages. Then each stage of the retrosynthesis of organic compounds applied in laboratory projects was undertaken by students to synthesize the Dibenzalacetone. The results of the performance tests for each group are shown in Table 1.

Based on the data in Table 1, it can be seen that the highest score obtained from all groups is in code 1, which is greatest in identifying the characteristics of Dibenzalacetone by using various online media and offline software Students Spartan V6. It was found from code 1 that the total score from groups 1, 2, 3, and 4 respectively are 7, 7, 8, and 10. It implied that students have a good performance in conducting characteristics of Dibenzalacetone and identifying. This is understandable because this is the only stage that requires repetition for students [17].

Table 1. Results of groups' performance test in the synthesis of Dibenzalacetone.

Code	Stages of Retrosynthesis Approach	Score of Group A	Score of Group B	Score of Group C	Score of Group D
1	Identify the characteristics of the Dibenzalaceton	7	**7**	8	10
2	Hypothesize the retrosynthetic pathway of Dibenzalacetone	6	6	6	5
3	Make a retrosynthesis analysis of Dibenzalacetone	5	4	5	6
4	Propose a synthesis planning design based on retrosynthetic analysis of Dibenzalacetone	6	4	6	6
5	Make a synthesis of Dibenzalacetone in the laboratory according to planning design	5	4	6	6
	Total	29	25	31	33

From Table 1, we found that students have a lack of skill in using retrosynthesis in stage 3, 4, and 5, thus caused their performances are less. Consequently, these stages are associated with propose synthesis planning design based on proposed retrosynthetic analysis of molecular targets; make the retrosynthetic analysis of molecular targets using retrosynthetic guidance software and consider the availability of tools and materials, and proposed a hypothesis related to the retrosynthetic pathway of the molecular target compound. It is recognized that students are only skilled in identifying characteristics by using online media and offline software Students Spartan V6, but difficulties occurred when it came to associate their skill with planning design. It might be caused by students' lack of skill in using the retrosynthetic approach that requires a great deal of integration of the available organic chemical materials. It is consistent with the research that suggests that students tend to have problems in using the retrosynthesis approach of organic compounds [18]. Students cannot have critical thinking skills to predict the results of the reaction of organic compounds or precursors/starting materials of the target molecules. This will help students to be able to deal with the desired conceptual understanding in learning [19, 20].

The retrosynthesis of Dibenzalacetone that proposed by the students in groups are similar. It's according to their acknowledgment about the Aldol condensation reaction in Fig. 3.

Figure 3 shows the disconnection of Dibenzalacetone. It makes the students can easily recognize the double/ hyperconjugation that they need to make at the break of phi bond of C=C, so students in the group have the same opinion that they must make the break in the double bond so they have an aldehyde and ketone as the starting material.

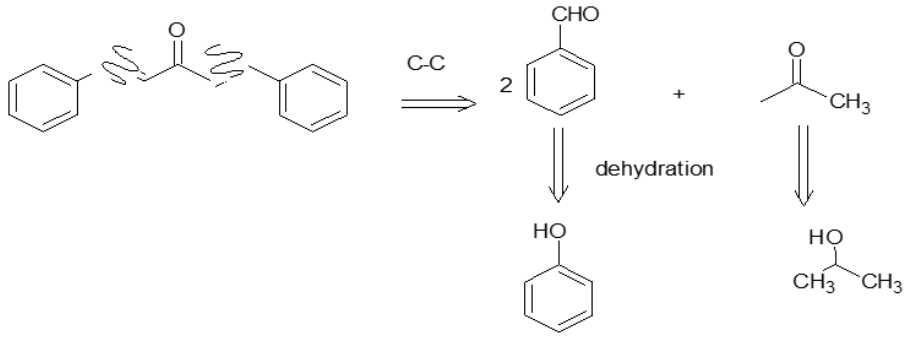


Fig. 3. The retrosynthesis of dibenzalacetone [13].

Students made the synthesis and found that the Dibenzalaceton that synthesize has a solid (crystal) with yellow colour and has a melting point at 110°C-113°C. The results of the group performance tests in the synthesis of Dibenzalacetone show that their synthesis have the result of GC-MS as shown in Fig. 4.

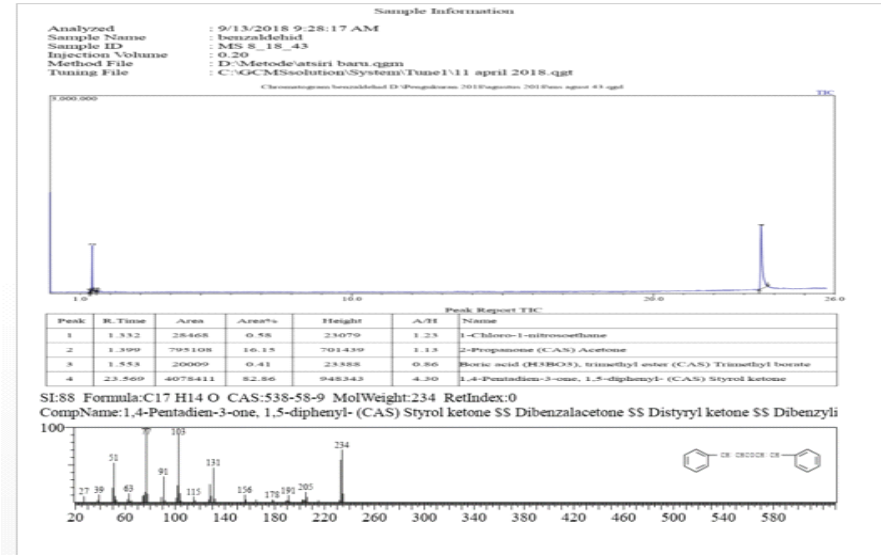


Fig. 4. The spectrum GC MS of the example of students synthesis result.

The GC-MS chromatogram in Fig. 4 shows 2 peaks with the highest peak which has a retention time of 23.569 and an abundance of 82.86 %. This peak shows that the compound is Dibenzalaseton with m/e 234. Besides, there is also fragmentation which shows the presence of m/e ion 131 which shows the release benzaldehyde ion. Then the fragmentation of m/e 103 shows the release of carbonyl ion, and there is also fragmentation with m/e showing the presence of benzene ions. All the peaks that appear in the chromatogram show that the compound is Dibenzalacetone compounds (product compounds) with the expected yield compound in m/e 234.

Until the students have finished all the steps, the N gain was calculated. It is found that the group of students who had a total of the lowest scores on each code

was group B. However, to ensure the differences between each group then the Kruskal Wallis test was used.

The differences in students' skills in groups were also determined using the retrosynthetic approach to complete a problem of organic synthesis. According to the result of statistics, it was obtained that chi-square value was 8.074 with the significant level of 5% (0.05). We found that the value of chi-square obtained on the table was 7.82 and Asymp. The sign is obtained 0.045. Result showed the chi-square test > chi-square table is 8.074 <7.82 and significant value at 5% confidence is 0.045 <0.05. It means that there are significant differences in the skills in groups using every stage of the laboratory project from students in completing the synthesis of Dibenzalacetone.

It is found from the result that there is a difference in the skills of students in the stages of retrosynthesis. The differences are related to the performance in the next stages. The stages that have affected are stages of the proposed retrosynthetic analysis of Dibenzalacetone, made an analysis of retrosynthesis Dibenzalacetone by using a retrosynthetic guiding software and considering the availability of tools and materials, and proposed a hypothesis related to the retrosynthetic pathway of the target molecular compounds. It is similar to the result of the previous study that the students' capability in completing the assignment of organic synthesis is connected with their ability to integrate the concepts [17-20].

Based on the results of the research, the students improve their ability to work in synthesis organic problems by following every stage of retrosynthesis approach. It means that the learning which is related to the synthesis of organic matter requires scaffolding. It will train students' performance on the stage of retrosynthesis approach. It is done by assistance from the use of offline software like Students Spartan V6, Marvinsketch, and Reactor ChemAxon. It can also be done through the retrosynthesis approach that leads to the synthesis of organic compounds through guided inquiry [19-21]. Through this research, the existence of retrosynthesis approach is expected to direct students toward conceptual change and improve students' performance in a synthetic organic compound in the laboratory.

4. Conclusion

Based on the research, the difference in students' performance using the retrosynthetic ways was found. Furthermore, the biggest score of students' performances is in the stage of characterizing the Dibenzalacetone compound while the lowest one is making the retrosynthesis of the Dibenzalacetone. From the result, it was obtained that the students improve their ability to work in synthesis organic problems by using a scaffolding.

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