

Isolation of artelasticine for student practice in lowresource laboratory settings

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Isolation of artelasticine for student practice in low-resource laboratory settings

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Abstract. The isolation of secondary metabolites can be used by students for laboratory activities in the natural product chemistry course. The laboratory experience of isolating secondary metabolites will be useful for students to isolate secondary metabolites in other plant species whose chemical content has never been reported. In this laboratory the student isolated artelasticine from *Artocarpus scortechinii*. Students learn about extraction, fractionation, purification, and structural elucidation through the natural product chemistry laboratory activities. Various chromatography techniques are used to obtain pure artelasticine compounds. The artelasticine structure was determined based on UV, IR, ¹H-NMR and ¹³C-NMR spectroscopic data. The laboratory method allowed students to readily understand the process of isolating artelasticine.

1. Introduction

Secondary metabolites are metabolite compounds that are not essential for the growth of organisms and are found in unique or different forms from one species to another [1]. Each organism usually produces different secondary metabolites. The function of secondary metabolites is to defend themselves from unfavorable environmental conditions, for example to overcome pests and diseases. One class of secondary metabolite compounds, namely flavonoids. In this natural product chemistry laboratory, students isolated one of the flavonoid compounds, namely artelasticine compounds from *Artocarpus scortechinii*. Students could learn essential skills required to perform the extraction, fractionation, purification, and structural elucidation of a flavonoid.

The isolation of secondary metabolites, including the isolation of flavonoid compounds, can be a core activity in the study of the chemistry of natural materials [2]. Practical experience in isolating the flavonoid, artelasticine, will be useful for students to isolate secondary metabolites from other plant species whose chemical content has never been reported. Other important reasons why laboratory activity needed in natural product chemistry that can generate learning motivation, develop generic science skills, and improve concept understanding [3]. The natural product chemistry laboratory had been previously described in several studies [4-11].

2. Materials And Methods

Participants in this study consisted of 26 third-year students (teacher preparation) from chemistry education department at one of the state universities in West Nusa Tenggara, Indonesia. These laboratory activities took place during the second semester of the 2019–2020 academic years. Participants were divided into 8 groups with 3-4 participants per group. All of the groups worked on the same plant sample, heart wood of *A. scortechinii*. This study uses quasi-experimental research with one



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group pretest–posttest design. The instrument containing 20 questions was used to assess the ability of students in isolation processes. The validity of the test was determined by using content validity experts. Qualitative analysis was done by interpretive descriptive analysis. Quantitative analysis of data was done by calculating the percentage of normalized gain scores (%g) using the formula in eq 1.¹⁶

$$\%g = \frac{S_{\text{post}} - S_{\text{pre}}}{S_{\text{max}} - S_{\text{pre}}} \times 100\% \quad (1)$$

Where S_{post} and S_{pre} are the posttest and pretest scores, respectively, and S_{max} is the maximum possible score. Values of %g were then characterized as high for %g > 70%, medium for 30% ≤ %g ≤ 70%, and poor for %g < 30%.

Laboratory reports were also used at the end of the laboratory activity that was arranged in groups. Student surveys were used to demonstrate student engagement with the experiment.

3. Results And Discussion

The learning begins with the provision of a laboratory project, "the isolation process of artelasticine from heart wood *Artocarpus scortechinii*". The isolated artelasticine from heart wood *A. scortechinii* has been determined by the team based on the results of the isolation conducted by the research team. Students were asked to conduct a literature study on the artelastisine and *A. scortechinii* which became their student practice subject. From the results of their literature study, students designed their isolation laboratory project. In designing the laboratory activities, students find out for themselves the secondary metabolite isolation procedure used. These procedures can come from journals or research reports. The lecturer guides and informs the students what to do in their practicum project proposal. Students present their practicum proposal in front of the class in groups. Lecturers and students from other groups responded to the proposals presented. After the proposal is revised based on the lecturer suggestion, students can implement the practical design. Students implement the design in groups and develop procedures that have been designed based on the results of the implementation during the implementation process in the laboratory. At this stage, students will be able to better understand the concepts that exist in the secondary metabolite isolation process. After the implementation stage, students make reports and present the results to the class.

3.1. Students Result. Of the eight groups who participated in this laboratory, only two groups succeeded artelasticine from heart wood *Artocarpus scortechinii*. However, all groups were still given the UV, IR, ¹H-NMR, and ¹³C-NMR spectra provided by the instructor for interpretation by all groups. From the reports collected, all groups could describe the presence or absence of conjugated bond bonds from UV data and could determine all functional groups found in the artelasticine compounds based on IR data. They had difficulty interpreting the ¹H-NMR, and ¹³C-NMR spectrum. Two groups could show an almost-correct interpretation of the NMR data. The other groups demonstrated poor NMR interpretation abilities. In the quantitative analysis of pretest and posttest scores, the average of students' n-gain percentage was 61% (medium category).

3.2. Isolation of Artelasticine from *Artocarpus scortechinii*. In general, the procedure for isolating artelasticine from *Artocarpus scortechinii* is as follows. The groups prepared air-dried heart wood of *A. scortechinii* by macerating these using methanol. The methanol extract was filtered using cellulose filter paper. The solvent was evaporated by a rotary evaporator to dryness to obtain a crude extract. The crude extract was fractionated using gravity column chromatography with eluent n-hexane:chloroform (7.5 : 2.5). A single compound was characterized by the presence of a single spot in a TLC chromatogram.

3.3. Structure Elucidation Studies. The UV spectrum in methanol shows the absorption at λ_{max} (log ϵ) (0.8629); 340 (shoulder; 0.55); and 368 (0.67) nm. This data indicates the presence of a conjugated double bond. The IR (KBr) spectrum of ν_{max} (cm⁻¹) showed the presence of a conjugated carbonyl group (C=O), typical for flavone at an uptake of ν_{max} 1645 cm⁻¹ and C=C aromatic groups at an uptake 1620, 1556, 1440, 1355 cm⁻¹. The IR spectrum also showed the presence of absorption for the hydroxyl

(OH) group at V_{\max} 3369 cm^{-1} and aliphatic C–H at V_{\max} 2914 cm^{-1} . The IR data of a single compound showed that all functional groups were found in the artelastine compounds, as seen in Figure 1.

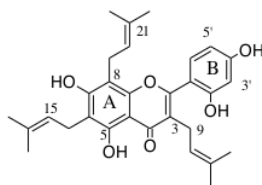


Figure 1. Structure of artelastine.

Further determination of the structure of a single compound was obtained from the ^{13}C -NMR, which indicated 30 signals of carbon atoms. There was a chemical shift at δ_c 183.32 ppm, which is a typical sign of carbonyl (C=O), and a sign of carbon oxyaryl at δ_c (154.37–162.09) ppm. In addition, there were also signs of quaternary carbon at δ_c 105.19–132.26 ppm. ^1H -NMR (500MHz, CDCl_3) showed a typical sign in the region of 13.14 ppm for a chelated –OH, which was evidence of flavonoid derivative. Typical signals for the isoprenyl group are chemical shift at (δ ppm) 1.41; 1.55; 1.56; 1.57; 1.63; and 1.76 (each brs) for six methyls. Furthermore, the presence of vinyl protons is shown in the chemical shift (δ ppm) 5.12 (2H, m) and 5.21 (1H, m) which are neighbors with the methylene protons respectively on the chemical shift (δ ppm) 3.13 (2H, d, $J = 6.7\text{Hz}$) and 3.42 (4H, d, $J = 4.9\text{ Hz}$). The existence of the ABX system in the isolated compound is indicated by the presence of a signal in the area of 6.50 (1H, dd, $J = 2.4$; 8.5 Hz); 6.56 (1H, d, $J = 2.4$ 8.5, Hz) and 7.20 (1H, d, $J = 8.5\text{ Hz}$).

4. Conclusions

The isolation activity of secondary metabolites can be a laboratory activity for natural product chemistry. This experiment, allows students to isolate and characterize artelastine, an *Artocarpus scortechinii* flavonoid compound. Students work in small groups. If one group fails to isolate pure material, that group can join a successful group for subsequent characterization and analysis. Flavonoid compounds can be replaced by another secondary metabolites like alkaloid or terpenoid. This laboratory activities can provide awareness to students about the importance of preserving various types of plants that contain secondary metabolites for improvement of human life. This awareness can provide sustainable environmental preservation.

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