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Design and Validation of a Practicum Module for Gravimetric Analysis Techniques in Project-Based Learning

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ABSTRACT

Gravimetry is a simple and fast method used to determine the weight percentage of alkaloid content in natural samples when analyzing compound content. Gravimetry is a basic knowledge and skills in analytical chemistry. A student-centered learning approach is needed to acquire integrated chemical laboratory knowledge and skills. Integrated learning outcomes between knowledge and skills can be achieved, one of which is through project-based learning (PjBL). One of the things we report in this article is the development of a practical module based on PjBL. This article reported the research and development (RnD) of a project-based practical module on gravimetric analysis using the Analysis, Design, Development, Implementation, and Evaluation (ADDIE) stages to obtain a valid and reliable practical module. The module's validation was analyzed using content validity with the FGD technique through the assessment of five experts. Validity data analysis used Aiken's and Cronbach's alpha for reliability. This article also reports the results of observations of the PjBL practicum of gravimetric alkaloid analysis by 18 students regarding the clarity of the practicum objectives, content, instructions, and ease of the module in guiding the practicum in the Laboratory. By using the ADDIE development steps, we produced a PjBL practicum module with the validity of the Aiken analysis results showing an index of 0.92, indicating that the module developed was valid, had a reliability of 0.78, and had a practicality percentage of 89.77%. The developed PjBL teaching module has also increased students' motivation and basic chemistry laboratory skills when conducting gravimetric analysis practicum.

INTRODUCTION

The chemistry practical experience in the laboratory is an integrated learning activity that can enhance motivation and science expertise and improve chemistry student retention (Provost, 2022). Research experience can be gained through practicums or project-based courses in chemistry laboratories, also known as project-based learning (PjBL). Project-based learning is a form of learning that centers on students acquiring knowledge by collaboratively solving problems (Doughan & Shahmuradyan, 2022). The advantages of PjBL include encouraging student activity in learning and increasing learning motivation (Allen et al., 2021; Gouger & Mirowsky, 2022; Rio & Rodriguez, 2022). Chemistry students also reported a positive experience after learning with PjBL in the practice of the filtration of chemical analytes from water(Gouger & Mirowsky, 2022). Furthermore, research project-based learning in the laboratory can provide experiences integrating professional knowledge and skills (Watts & Rodriguez, 2023).

Project-based learning for students typically involves short-term projects where students are provided with the necessary insights to solve a problem and are allowed to use their intuition and applied work skills to solve it (Davis et al., 2017; Robinson, 2013). Project-based learning research is still predominantly used to enhance knowledge at the higher-order thinking skills

(HOTS) level, while its implementation to improve students' professional skills remains limited. Recent studies have reported that PjBL can equip students with soft and hard skills, including cognitive knowledge and professional skills (Vogler et al., 2018). Cognitive knowledge and laboratory skills are essential for chemistry students during the first two years of study at the university. One of the critical lessons learned by chemistry education program students in the second year is analytical chemistry. At the beginning of the second year, students are introduced to the fundamental principles of gravimetric chemical analysis. Gravimetry is a fundamental analytical method that uses mass as its primary signal. It is importance to teach this method to students early in the course to provide them with integrated professional knowledge and skills before introducing more complex analytical techniques. The gravimetric method involves an activity that measures the mass or mass change of the analyte (Harvey, 2004). This method remains relevant for chemistry students to learn at the beginning of their studies because it contains the fundamental learning outcomes necessary for analysis. Gravimetric learning design through an analysis project for determining alkaloids in natural materials in the laboratory is a form of integrated learning.

Gravimetric analysis of alkaloids offers learning opportunities at the HOTS level, as it involves multiple stages, including the preparation of natural material samples, extraction of secondary metabolite compounds, and the determination of alkaloid content. This makes it highly suitable for implementation in project-based learning (PjBL). Determining alkaloids in natural products using gravimetric methods has been reported in *Peperomia pellucida (L.) Kunth*, *Carica papaya* leaves, and *Rauvolfia tetraphylla* (Ahmad et al., 2017; Julianti et al., 2014; Verma, 2017). The determination of alkaloids in natural product samples, which includes technical work such as sample preparation, extraction, and determination of the percentage of alkaloids using gravimetry (Ahmad et al., 2017), is an analysis stage that provides extensive learning space for theoretical development and basic chemistry laboratory skills to support their careers in this region (Doughan & Shahmuradyan, 2022). The alkaloid analysis stage in natural ingredients begins with sample preparation and extraction using a maceration technique with an acetic acid solvent to separate alkaloids from samples of natural ingredients. The acid solvent was chosen based on the solubility of alkaloid compounds in acids (Fadhly et al., 2015; Kapondo et al., 2020; Xiao et al., 2023). Determining alkaloids involves weighing the precipitate formed in the acid solution extractant after adding a concentrated alkaline solution. The alkaloid precipitates are obtained after adding alkaline solutions to increase the pH to neutral or slightly alkaline (Teng et al., 2023). The alkaloid precipitate is formed, and then a gravimetric analysis is conducted by weighing and calculating the residue ratio from the natural samples used.

In addition to knowledge, the gravimetric method closely relates to weighing techniques and laboratory skills. The current problem is that classroom learning is not enough to build cognitive knowledge and professional skills in chemistry students, necessitating work experience in a laboratory to complete it. PjBL achieves the integration of cognitive knowledge and laboratory skills in gravimetric learning by assigning tasks based on the outcomes of the products. This article presents the results of developing a project-based gravimetric alkaloid content analysis practical module. We designed the project-based practicum module simply and systematically so students can use it as a reference when working in the laboratory. We developed the PjBL practical module using the ADDIE stages (Gagne et al., 2005), aiming to obtain a claim as a suitable practical and effective module. The term ADDIE is an acronym for the Analysis, Design, Development, Implementation, and Evaluation stages used as a development method, such as models (Nadiyah & Faaizah, 2015; Yu et al., 2021), media (Cotter et al., 2023; Putra et al., 2023; Rahman et al., 2023), and teaching modules (Bachri et al., 2024). Specifically, this article reports the process of developing a project-based practicum module as an assistant tool in PjBL gravimetric analysis material for determining alkaloid levels using samples of local natural products from North Maluku.

METHODS

Research Design

This development study follows the ADDIE model, which includes the steps of analysis, design, development, implementation, and evaluation, as outlined by Bintarti et al., (2024). We are currently developing the PjBL practicum module for gravimetric determination of alkaloids as part of the implementation stage in the ADDIE process, and the evaluation results are reported separately in other documents. The development of the PjBL practical module for gravimetric determination of alkaloids from natural materials is based on two analytical results, including the necessity for theoretical learning through collaborative learning and the requirement for learning tools to achieve desired learning outcomes. Design Stage: Based on the results of the analysis, we design student-centered learning using the PjBL practicum module. The gravimetric determination of alkaloids from natural products was chosen as a project due to the simplicity of the chemicals involved. It also includes some technical work proportional to learning, including laboratory skills for students.

Development Stage: We have developed the PjBL practicum module according to the previously proposed design. A project-based module prototype is produced and validated by experts to ensure its suitability for use in learning (Bachri et al., 2024). Implementation Stage: After receiving feasibility recommendations, we implemented the module in limited classes. During implementation, students work in groups to collect data and produce products. At this stage, we assess each student's work for its effectiveness.

Research Target

This study aims to develop a valid PjBL practicum module that is ready for implementation in learning activities. The module provides a detailed description of the PjBL stages to guide third-semester students in conducting gravimetric analysis practicums as part of analytical chemistry courses.

Research Data

We collected this research data during the module development stages, mainly focusing on the analysis stage described in the background. The design stage involves drafting the PjBL practicum module, which includes an introduction, learning objectives, design of learning, methods, and learning outcomes. In the development stage, we present validation results and expert reliability data in three aspects: material, language, and design. We present data from the trial class at the implementation stage as the results of a survey on the practicality of using the PjBL practicum module.

Research Instruments

The research instruments include expert validation questionnaires to obtain valid and reliable data. The class instrument was a student survey questionnaire in the laboratory for PjBL practicum learning.

Data Analysis

Data analysis from expert validation was analyzed using Aiken's equation to determine the validity index of each item from the three validated aspects. Reliability analysis used Cronbach's Alpha equation. Students conducted surveys in the laboratory during the gravimetric alkaloid analysis practicum utilizing the module, deriving practical analysis from the results.

RESULTS AND DISCUSSION

Analysis and the PjBL Practicum Module Design

The development of the Project-based Learning (PjBL) practicum module for gravimetric analysis of alkaloid compounds was based on the needs analysis of integrating chemistry education students' knowledge and laboratory skills, particularly in gravimetric analysis techniques. This PjBL module differs from conventional practicum modules by requiring students to create practicum reports in the form of scientific posters. The practicum involves determining the levels of alkaloid compounds in various natural samples and reporting the results as scientific posters. The PjBL module has five main sections: introduction, learning objectives and achievements, learning plans, methods, learning products/outputs, and bibliography. The module is six pages long, formatted on A4-sized paper, with Times New Roman font, size 12. It includes illustrative images to help students understand and follow the instructions during laboratory practicums. Designed as a digital module, it is easy to distribute and environmentally friendly. The graphic elements on the cover and main pages were created using the online tool Canva.

The beginning of the module is equipped with a cover and introduction, as shown in Figure 1. The cover on the first page shows the module's identity.

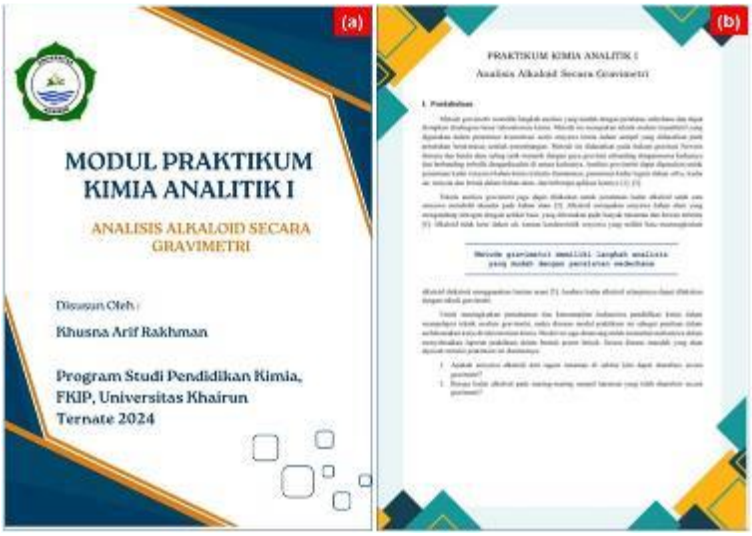

Figure 1. (a) cover, and (b) introduction

The introductory section contains the background, definitions, and applications of gravimetric analysis and identifies the problems that how to analyze alkaloids will be solved in this PjBL practicum. The introduction uses highlights in Navi font color and places a definition of gravimetric analysis in the middle of the page with borders at the top and bottom. The aim of adding these highlights is to make it easier for readers to understand a brief definition of gravimetric analysis and beautify the appearance/layout of the introductory section. The introductory paragraph also presents the initial step of the PjBL practicum by stating the results of problem identification in the form of two questions: (1) Can alkaloid compounds from various plants around us be analyzed gravimetrically? and (2) What is the alkaloid content in each plant sample that has been analyzed gravimetrically? The main problem raised in the PjBL practicum, which represents step 1 of the PjBL syntax, is shown by question sentence number 2, identification of the problem; then, by using this practicum module, students are encouraged to solve the issues and answer these questions through practicum in the laboratory. We present basic questions or main problems as step 1 of the PjBL syntax in the form of questions or descriptions of real-world issues to stimulate students' imagination, creativity, and higher-order thinking skills (Susilawati & Sahara, 2021; Wahyuni et al., 2019).

After the introduction, the next page of the PjBL practicum module design is the learning objectives and achievements in Chapter 2 (Figure 2. a). This page contains the main goals of this PjBL practicum, which includes two main objectives: (1) determining alkaloid levels in weight/volume percent gravimetrically and (2) making a report on the practicum results in the form of a scientific poster. The objectives are written simply and presented with work illustrations in the PjBL practicum module so that students can focus on two main jobs: carrying out gravimetric analysis and making a report on the study's results in the form of a scientific poster. Learning outcomes are also explained in this section to provide a concrete picture of the skills achieved after this practicum.

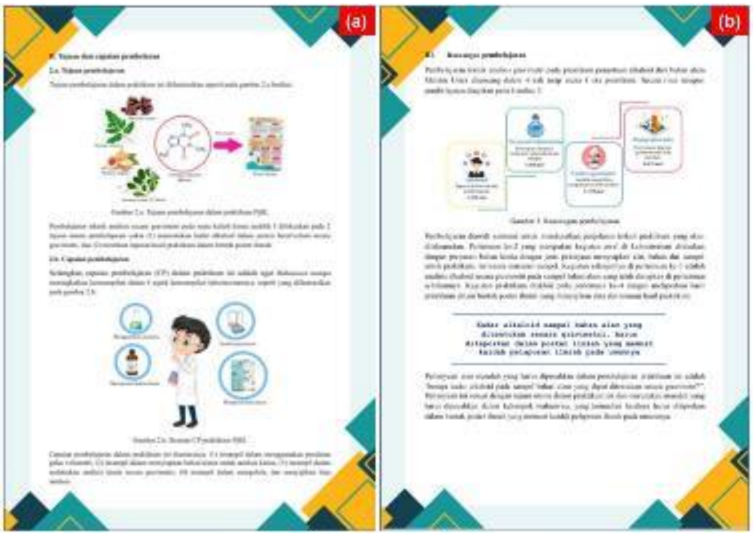

Figure 2. (a) learning objectives and outcomes, (b) learning design

This PjBL practicum aims to improve students' laboratory and chemistry skills. This PjBL practicum module outlines four learning outcomes: (1) skillful use of volumetric glass equipment, (2) preparation of chemicals for analysis, (3) conducting gravimetric chemical analysis, and (4) managing and presenting analytical data. We illustrate learning achievements with pictures and describe them in sentences to attract students' interest and make it easier for them to understand this PjBL practicum module (Setyorini et al., 2022; van der Sande et al., 2023).

In the next section, Chapter 3, we outline a learning plan (Figure 2. b) that describes the learning flow of gravimetric analysis techniques through the PjBL practicum. This section explains the plan for the number of PjBL face-to-face meetings from the first meeting in assistance, chemical preparation activities, and gravimetric analysis in practicums in the Laboratory at the 2nd and 3rd meetings, as well as data management and product preparation at the 4th meeting. We designed the learning process to include four meetings. Each meeting, as detailed in the PjBL practicum module, explains the tasks, forms, and outcomes, helping students prepare for the learning process. Several studies report that planning learning with students, in this case, conveying the design and stages of learning, can increase students' readiness in the learning process (Alkan, 2019; SeÇKen, 2008; Suciana et al., 2024). This section also presents illustrations of learning plan images and project confirmation instructions. These are highlighted separately in a box with the directive to report alkaloid levels in samples of natural materials determined gravimetrically on a scientific poster that adheres to general scientific reporting rules. Illustrations in the form of pictures and project confirmation instruction sentences guide students on how to complete this work, in addition to beautifying the layout design of the resulting PjBL practicum module. Learning instruction designs that are interesting, clear, and easy for students to understand can improve students' literacy skills and knowledge before learning begins (Aisyah & Rinjani, 2023; Samad et al., 2023). Chapters 2 and 3 outline learning objectives, achievements, and learning design integral to the second stage of PjBL, which involves designing projects. This approach offers students an overview of the learning phases and the expected deliverables.

The methods section in chapter 3 of the PjBL practicum module presents practical stages in the laboratory, which are equipped with illustrative images or work sequence charts for the gravimetric analysis of alkaloids (Figure 3. a). The method also includes four main steps for gravimetric analysis of alkaloids, including (1) chemical preparation, which explains the preparation of chemicals to be used, such as 10% acetic acid, to the selection and initial preparation of the sample before analysis. (2) The preparation of natural material samples includes explaining how to prepare natural material samples in the laboratory and the extraction stage using the maceration method. (3) Gravimetric analysis of alkaloids: This section explains the stages involved in gravimetric analysis of natural material samples that have been macerated to extract solid alkaloids. (4) Reporting alkaloid levels of natural material samples by presenting how to manage and process data from practical results to obtain alkaloid levels in percent and instructions for presenting them in scientific posters. In general, the practicum method describes instructional practicum steps to guide students in carrying out their duties (SeÇKen, 2008).

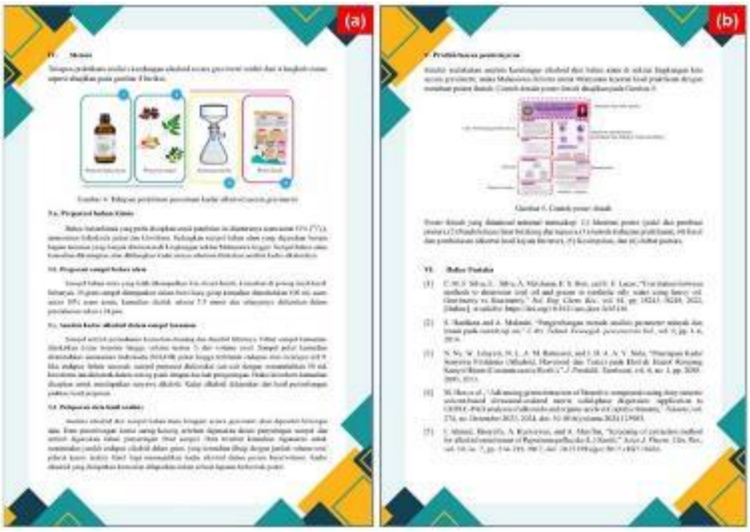

Figure 3. (a) Method and (b) Learning products/outcomes and bibliography

The next part of the PjBL practicum module is Chapter 5, learning products/outputs (Figure 3. b). This section outlines the minimum standards or components required in scientific posters resulting from student practicums. Scientific posters as a requirement for fulfilling project outputs in standard PjBL practicum in at least six parts include (1) poster identity (title and poster maker), (2) introduction (background and objectives), (3) method (practicum stages), (4) results and discussion (accompanied by the results of the literature review), (5) conclusions, and (6) bibliography. This section aims to introduce the minimum standards of scientific posters to students and emphasize the output resulting from the gravimetric alkaloid analysis practicum. The student's preparation of posters can also carry out become a bridge for integrating skills and knowledge, especially gravimetric analysis techniques and stimulate the student's creativity (Wahyuni et al., 2019). After completing the laboratory practicum, students are allocated one week (7 days) to create a scientific poster presenting the results of the PjBL practicum on gravimetric analysis. Throughout the seven days allocated for completing the project output, students are monitored and provided with opportunities to ask questions and consult with their practicum assistant regarding any difficulties or challenges encountered in producing the product or output. After seven days of project work, students must upload the PjBL practicum products implemented on Google Classroom for product assessment or assessment. The stages of providing project time, opportunities for discussion and consultation, and product assessment are by the project-based learning steps stages: (3) preparing a schedule, (4) monitoring project progress, and (5) assessing results (Sanal Kumar & Thandeeswaran, 2024; Zhao & Wang, 2022). The PjBL practicum module design for the alkaloid analysis gravimetrically presented in this article is the final form after revision based on expert input at the validation stage.

The PjBL Practicum Module Validity and Practicality

The development stage of the PjBL practicum module for gravimetric alkaloid analysis involves testing the validity of the product. The assessment of the validity of the PjBL practicum module was carried out by involving several experts to assess aspects of the material, language, and design or layout of this PjBL module. The assessment of the two aspects in the validation process is detailed in 15 question items, with 8 question items on the material aspect, four on the language aspect, and three on the design or layout aspect. The validation results of the PjBL practicum module were analyzed using Aiken's validity, detailed in Table 1.

Table 1. Analysis of the validity of the PjBL practicum module

No	Observed aspects	Aiken's index (V)	Category*
1	Conformity of substance with expert agreement	0.80	normal
2	Suitability of topic to learning outcomes	0.93	high
3	Interesting discuse	0,80	normal
4	It does not contain harmful elements	0.93	high
5	Suitability to student development	0.87	high
6	Contains project-based learning	0.93	high
7	Learning with enough scheduled time	0.93	high
8	There are no misconceptions	1.00	high
9	Suitability of language to the student's age	1.00	high
10	Grammatical correctness	0.93	high
11	Conformity of the module title with the material sections	0.87	high
12	Conformity with Indonesian grammar	1.00	high
13	Module have attractive design	1.00	high
14	Images/illustrations were artistically	1.00	high
15	Images/illustrations supported the module description	1.00	high

**) Based on the Aiken's table, the number of Raters is 5 with a choice of 4 scales of significance level $p < 0.05$ and a minimum V of 0.87*

Table 1 presents the 15 validity aspect items that were measured, with validity aspect items number 1-8 being the material aspect, 9-12 the language aspect, and 13-15 the illustration and layout aspect. Based on the analysis results, the average Aiken index was 0.92, a high-validity category (Maulana, 2022). Meanwhile, the reliability analysis results show that the module developed is reliable, with a value of 0.78. Based on the analysis results in Table 1, there are two validity statement points in the medium category: (1) suitability of the material with expert agreement and (3) attractiveness. Several articles report research on alkaloids as secondary metabolite compounds, focusing on isolating natural compounds for specific applications. Alkaloid analysis is often integrated into phytochemical screening techniques employed to analyze the content of secondary metabolite compounds in natural products (Fadhly et al., 2015; Kristiana et al., 2023; Ramayani et al., 2022). So, it is due that this topic is still less popular for chemistry practicums. It has received less than optimal assessments on material aspects by experts in the PjBL practicum module validation process. However, some validators are alleged to have understood that the separation of alkaloids can be carried out gravimetrically, thus giving high scores so that the average analysis results on the two validity points above are in the medium category. The analysis results on three aspects generally show that the module is declared valid for use.

After validation and approval by experts, the PjBL practicum module is utilized in the learning implementation stage. The implementation stage of the ADDIE learning model is conducted in the analytical chemistry practicum, which focuses on gravimetric analysis. Practical learning is carried out in 4 meetings as explained in the previous section learning design description of PjBL practicum module design Figure 2. b. Students carry out practicum using the PjBL practicum module, providing assistance, observation, and analysis of the practicality of the module through surveys. Through a survey, the practicality test was analyzed using four practical aspects of the PjBL practicum module, as presented in Figure 4.

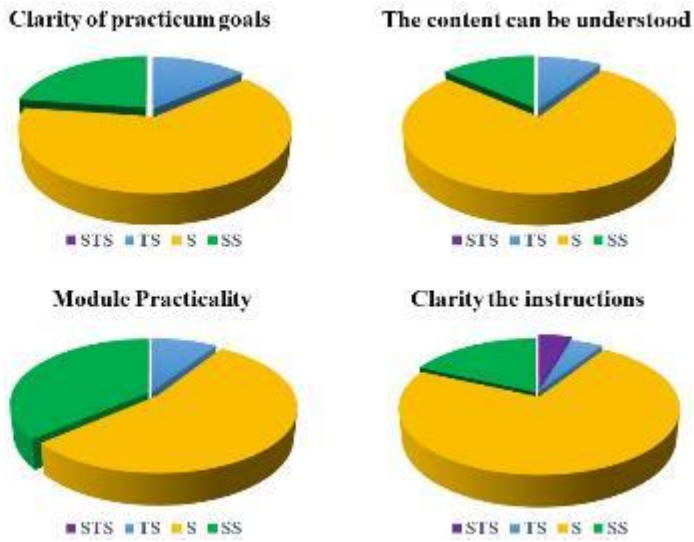

Figure 4. Diagram of student survey results on the use of the PjBL practicum module

We conducted a practicality survey of the PjBL practicum module on the aspects of (1) clarity of practicum objectives, (2) easy-to-understand material, (3) practicality of the module, and (4) clarity of work instructions. In the aspect of clarity of practical objectives, the majority of students gave positive responses, with 86.36% agreeing and strongly agreeing that the practical objectives written in the module were clear, where insert Figure 4 for STS: strongly disagree, TS: disagree, S: agree and SS: strongly agree. The ease of understanding the material shows that 90.91% of respondents agree and strongly agree, and the rest say the opposite. The same agreement was shown in terms of the practicality of the module and clarity of work instructions in practicum in the laboratory, with a percentage of 90.91%. The practicality of the PjBL practicum module, with an average percentage of 89.77% from the four aspects above, shows that the module developed is practical and suitable for learning. The practicality of learning tools, including the practicum module developed, can be analyzed based on the results of limited class trials and student responses. Several articles report that the practicality of the module developed can be evaluated in terms of clarity of objectives, ease of understanding the material, and ease of instructions written in the module (Irsalina & Dwiningsih, 2018; Samsu et al., 2020).

Analysis of Alkaloid Gravimetrically in PjBL

We conduct project-based learning for the gravimetric analysis of alkaloids in natural samples according to the syntax or stages of project-based learning. The practicum of PjBL was conducted over four sessions with a total duration of 680 minutes, resulting in the development of students' gravimetric analysis skills. The learning design focuses on student activity and collaboration in carrying out practicum analysis of alkaloid content gravimetrically and completing scientific poster products resulting from the practicum. Figure 5 shows the distribution of hours in this learning process.

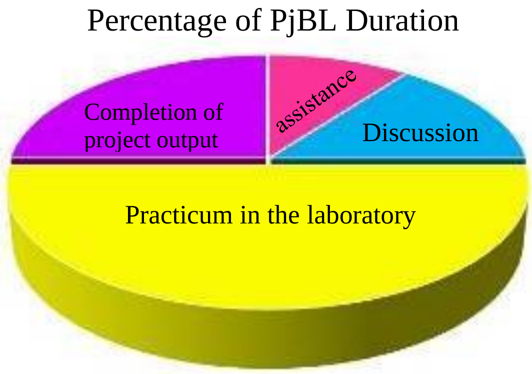

Figure 5. Diagram of PjBL activity hours distribution

Based on Figure 5, the diagram of the distribution of hours for project-based practicum learning activities contains four core activities, namely, (1) assistance in the form of explanations related to the practicum plan and group division with an allocation of 10% or 70 learning minutes, (2) discussion of the practicum plan 15% or 100 minutes. , (3) practicum in the laboratory for gravimetric analysis of alkaloids by 50% or 340 minutes, (4) completion of project outputs in the form of data management and reporting in the form of scientific posters by 25% or 170 minutes. Student-centered learning in the PjBL practicum is demonstrated by the percentage of hours of more than 75% given to students for independent study and improving skills in the laboratory.

We implemented the gravimetric analysis of PjBL alkaloids by dividing the students into four groups, each tasked with determining alkaloids from various natural samples: (1) tomi-tomi fruit (*Flacourtia inermis*), (2) nutmeg (*Myristica fragrans*), (3) Moringa leaves (*Moringa oleifera*), and (4) jambulang leaves (*Syzygium cumini (L.) Skeletal*). On the first day of practical activities in the laboratory, each group prepared and macerated natural samples with 10% acetic acid, which were left overnight. On the second day of the PjBL practical activity, each group carried out filtration of natural material extracts, then carried out alkaloid precipitation treatment by adding a concentrated amount of NH_4OH solution, and separated the resulting precipitation by filtration, which was then analyzed gravimetrically. The percentage of alkaloid content in each natural product sample is reported differently, as shown in Figure 6, a histogram of the alkaloid content of natural product samples.

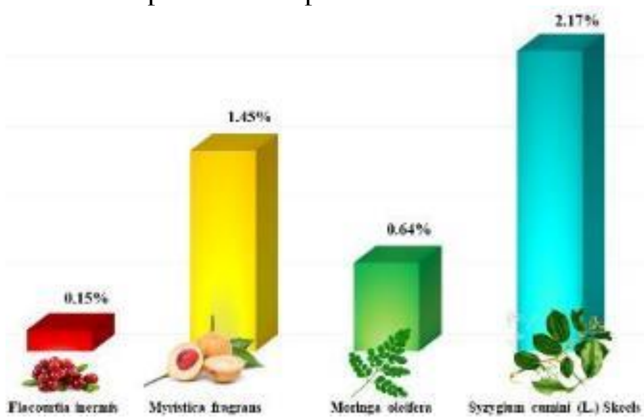

Figure 6. Histogram of percent alkaloid in natural samples

Based on Figure 6, the histogram of alkaloid levels, the results of gravimetric student analysis carried out in the PjBL practicum, shows the highest percentage of alkaloids in the *Syzygium cumini (L.) Skeels* sample was 2.17%, followed by alkaloids in *Myristica fragrans* at 1.45%, *Moringa oleifera* at 0 .64%, and *Flacourtia inermis* at 0.15% w/v.

During the PjBL practicum process, we monitor project progress, which is the 4th stage of PjBL, through several activities, including (1) practical assistance in the Laboratory, (2) assistance with data management and calculations for determining alkaloid levels of natural material samples, and (3) assistance in making a report in the form of a scientific poster. Meanwhile, we assess PjBL by observing student activities during practicums and group discussions, using indicators to measure the achievement of learning objectives within the allocated time duration. The final assessment includes evaluating the scientific poster produced and the ability to present and respond to questions related to the practicum process orally, alongside the written results on the scientific poster, as part of the learning assessment. The assessment process is a form of implementation of the 5th PjBL stage, namely the evaluation of results. Meanwhile, we implemented the 6th stage of PjBL by administering a questionnaire to assess students' learning experiences following the PjBL practicum on gravimetric determination of alkaloid content. Student experience questionnaires after implementing the PjBL practicum to determine the alkaloid content of natural material samples gravimetrically were carried out in 2 aspects: (1) Learning that motivates student activity and (2) enjoyable learning are measured, and the results of these two statements are presented in Figure 7, a diagram of student evaluation results. Based on the results of the evaluation of the student learning experience questionnaire in the PjBL practicum, gravimetric analysis of alkaloid levels of natural samples showed that 95.45% of students gave positive responses with the agreement that this learning could motivate students' activeness in learning and was fun.

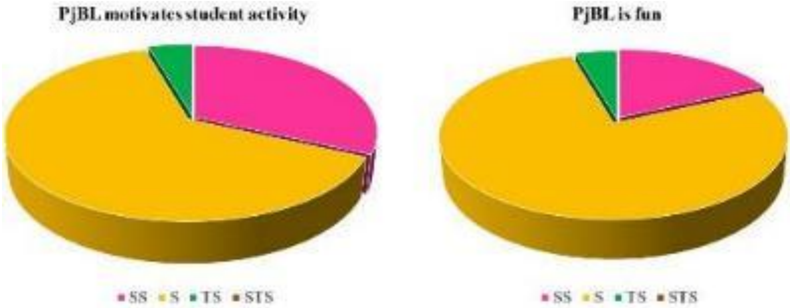

Figure 7. Diagram of student learning experiences

Based on Figure 7, the student experience diagram with SS stating that they strongly agree, S: agree, TS: disagree, and STS: strongly disagree, in the PjBL practicum analysis of alkaloid levels gravimetrically assisted by the PjBL practicum module, shows positive results that the learning carried out is capable motivate students' active learning and have fun learning characteristics.

CONCLUSION AND RECOMMENDATIONS

The PjBL practicum module for analyzing alkaloid levels from natural samples gravimetrically has been successfully developed by following the ADDIE steps. The development process is motivated by the need for practicum modules that can direct activity in student-centered learning and encourage students to collaborate and produce scientific products. Experts and students in limited class trials affirmed the PjBL practicum module's validity and practicality for use. Implementing the PjBL practicum using the PjBL practicum module has also been successfully implemented and received a positive response from students, as learning can increase student activity in learning and is enjoyable learning. The recommendation from this research is to adapt project-based learning in modules according to the local context, student needs, and the latest developments in chemistry.

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