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Potential Use of Expired Starch for Practicum Materials as an Effort to Reduce Chemical Laboratory Waste

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ABSTRACT

The laboratory is a facilitator of scientific activities. The use of chemicals is commonplace but is a factor in increasing waste, mainly unused and expired chemicals. This research aims to determine the potential for reusing starch as a practical material to reduce laboratory waste. The research method used was experimental by comparing expired starch with pro-analysis starch (new condition) and technical starch, including ash content, starch content and infrared spectrum (IR), which were then analyzed using one-way ANOVA. The results showed that the ash content and starch content of expired starch with pro-analysis starch did not have a significant difference (p-value > 0.05) but had a significant difference with technical starch (p-value < 0.05). The IR spectrum of the third starch also does not provide much difference from the correlation value close to 1. So, it can be seen that expired starch still has the potential to be reused as a practical material to reduce laboratory waste. Further research can be conducted to determine further characteristics so that chemicals can be reused at a higher level.

INTRODUCTION

The Chemistry Laboratory is located in the C5 and C6 buildings of the Faculty of Mathematics and Natural Sciences at Unesa serving practicum and research. Laboratory activities can cause an increase in the amount of laboratory waste containing pollutants that are toxic and dangerous (Setiawati, 2019). Waste can also be generated from expired chemicals and damaged packaging. Based on PP No. 101 of 2014 articles 31 paragraph 2 states that expired chemicals and damaged packaging are a type of waste (Peaturan Pemerintah RI, 2014). B3 waste must be managed or handed over to a B3 chemical processing institution such as the Industrial Waste Management Center (PPLI) Cileungsi Bandung West Java. The costs of processing B3 waste are pretty significant, depending on the pollution emissions that will be produced. The higher the level of toxins which can cause air emissions pollution if burned, the greater the costs. Management of B3 material waste must follow established procedures. Reusing expired chemicals can be done as long as the chemicals have not undergone degradation as proven through further testing.

One of the materials often used in laboratories with a high potential for expiration is starch. Starch is a reagent that is generally used in iodo-iodimetric titrations with a colored complex formation mechanism. This reagent is only needed in small amounts because the concentration needed is only around 1-2% (Meyiwa, 2020). Starch, a natural polysaccharide composed of glucose units linked by α-1,4-glycosidic bonds, is frequently employed in laboratories for various purposes, such as preparing agar plates, stabilizing solutions, or acting as a matrix in biochemical assays (Marichelvam, et al., 2022). However, like many other laboratory reagents, starch can reach its expiration date and may be considered unusable. This disposal of expired starch contributes to the accumulation of chemical waste in laboratories worldwide. Starch is a polysaccharide composed of linear and helical amylose and branched amylopectin (Santana, et al., 2018), the structures of amylose and amylopectin are presented in Figure 1. Starch is often employed in laboratory experiments to investigate various biochemical phenomena, such as the hydrolysis of glycosidic bonds, the determination of amylase activity, and the measurement of viscosity in solutions. Moreover, starch is a crucial component in techniques like gel electrophoresis, where it is used as a matrix for separating macromolecules based on size and charge (Indriani, Sumarlan, & Munawaroh, 2019; Ramirez-Cortes, et al. 2016; Abdullah, et al., 2018).

Based on this background, the author is interested in studying the characterization of the quality stability of expired chemicals in starch by chemical testing. This research provides an innovation in the use of expired chemicals (starch) as supporting materials for experiments in laboratories to reduce the potential for waste disposal. This test is to determine whether the quality of expired starch is still classified as good or not, provided that it is not contaminated or has experienced quality degradation, so that the expired chemical can be reused in practical work. This is an effort to reduce solid waste in chemical laboratories.

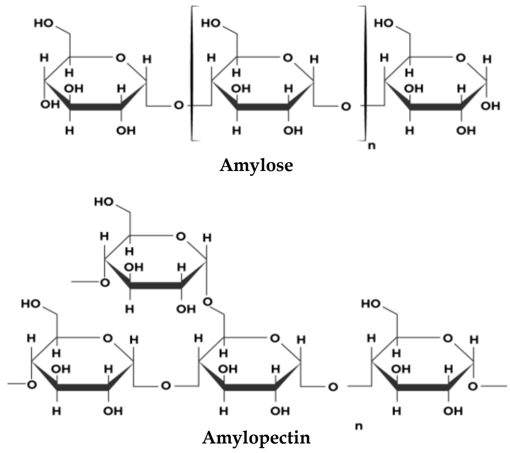

Figure 1. Structur of Amylose and Amylopectin as Amylum Constituent.

METHODS

Sample Collection

Expired starch samples were obtained from laboratories that did not need them and the expiration date was identified. Pro-analytical starch samples (MERCK) were obtained from Sigmaaldrich. Technical starch samples were obtained from flour shops.

Ash Content Determination

Ash content testing was carried out by the SNI 3751: 2018 method. The cup preparation was carried out by igniting it in a furnace with a temperature of 550 C, then cooling it in a desiccator and weighing it as (W1). Each starch sample weighed 3 grams (W) and was then heated in a bath. Next, ashing is carried out at a temperature of 550 until white or grey for 5 to 8 hours. Next, it is cooled in a desiccator and weighed as W2. The following equation calculates ash content:

$$\%Ash=\frac{W_2-W_1}{W-W_1} \quad (1)$$

Starch Content Determination

Determination of starch content was carried out through titration using the Somogyi method. 10 gram sample was put into a centrifuge tube and 50 mL of 8% alcoholic KOH was added. Then heated at 90-95 C for 30-40 minutes. Next, it was handed over and centrifuged at 2000 rpm for minutes. The filtrate was taken and put into a volumetric flask and 200 mL of 2.5% HCl was added. The sample was boiled for 2.5 hours in a water bath, the pH was made 6.5 - 7.0 with the addition of 15% NaOH, then a volume of 500 mL was made with aquademineral. Pipette 10 mL aliquots into an Erlenmeyer flask and add 20 mL of Somogyi A reagent (sodium potassium tartrate + trisodium phosphate + copper (ii) sulfate + potassium iodate) and then dry it in a water bath covered with aluminum foil for 25 minutes. After that, resolution with ice water and added Somogyi B reagent (potassium oxalate + potassium iodide) and 10 mL sulfuric acid 2 N then waited 2 minutes. After that, the sample was titrated with 0.05 N sodium thiosulfate with starch indicator. Blank samples were prepared without using 10 mL of aquademineral (Matsui, Agravante, & Kitagawa, 1990). Starch content is calculated using the following equation:

$$\%Starch=0.001499(B\text{-}A)F\times\frac{500}{10}\times0.9\times\frac{1}{S}\times100 \quad (2)$$

FT-IR Analysis

FT-IR analysis was carried out to determine the infrared spectrum of each starch sample. The FTIR instrument used with PerkinElmer specifications uses a wave number with a frequency of 4000-500 cm^{-1} (Tkachenko & Niedzielski, 2022). The IR spectrum obtained was then analyzed and the correlation value (similarity) of expired starch to pro-analysis starch and technical starch was obtained.

Statistical Analysis

Statistical analysis was performed using SPSS to determine whether there were significant differences between sample groups through a one-way ANOVA test. Interpretation is done by looking at the significance value (p-value) of expired starch to pa and technical starch.

Result and Discussion

The laboratory is a scientific training facilitator used for various science and engineering activities. Laboratory activities require chemical materials that provide potential waste, especially if the chemicals are expired. This research aims to determine the potential of the expired chemical "starch" as a material that can be reused in practical activities. The potential for starch as waste appears when the starch has expired, so this research aims to identify the potential for using expired starch as a material that can be used in making practical materials in the laboratory, including analysis of ash content, starch content, and FTIR analysis using a starch comparison (new condition), and technical starch.

The ash and starch content were analyzed to determine the composition of ash and starch in expired starch and its similarity to PA starch (new condition) and technical starch. Ash content is the content of a mixture of inorganic or mineral components contained in a sample (mainly metals) (Matsui, Agravante, & Kitagawa, 1990). The ash content was determined gravimetrically by heating the sample in a furnace for 5 hours at 550 °C. At the same time, the starch content was determined titrimetrically through iodimetric titration of hydrolyzed glucose in the sample. The starch sample is added with alcoholic KOH and aims to hydrolyze the sugar molecule bonds in starch to obtain glucose. Glucose was then determined through iodimetric titration with the Somogyi method (Matsui, Agravante, & Kitagawa, 1990). The two tests were carried out in triplicate at the Baristan Laboratory in Surabaya. The analysis of the ash and starch content presented in Table 1 shows that the starch content in expired starch is still close to the starch content in pa starch (new condition), namely 97.82% and a low ash content of 0.19%. This provides a qualitative representation that expired starch is still suitable and similar to new-condition starch.

Table 1. Results of Analysis of Ash Content and Starch Content of Starch Samples.

Sample	Ash content (%)	Starch content (%)
Pro-analysis starch (new)	0.17	98.10
Technical starch	0.28	75.30
Expired starch	0.19	97.82

The data obtained was then analyzed statistically to determine whether there were significant differences with expired starch. The analysis used is one-way ANOVA analysis, but before testing parametric study, a prerequisite test is required: normality analysis. The results of the normality analysis are presented in Table 2; based on the test results, it was found that all data had a normality p-value of more than 0.05. So, it was found that all test data were normally distributed, and one-way ANOVA analysis could be carried out (Connolly, 2007).

Table 2. Results of Normality Analysis.

Sample	*p-value*	
	Ash content	Starch content
Pro-analysis starch (new)	1.000	1.000
Technical starch	1.000	0.942
Expired starch	1.000	0.736

Furthermore, statistical analysis was carried out using a one-way ANOVA test using SPSS to determine the significance value (p-value) of differences between expired and new pa and technical starch. The test results are presented in Table 3, obtained data where the significance value (p-value) of ash content and starch content of expired starch was not significantly different from pa starch but significantly different when compared to technical starch. This is obtained from the p-value. If it is more than 0.05, there is no significant difference between variables, whereas if the p-value is less than 0.05, there is a significant difference between variables (Connolly, 2007). Therefore, based on the results obtained, expired starch's ash and starch content are still the same or not significantly different from the new starch.

Table 3. Results of One-Way ANOVA Analysis.

Expired Starch Sample	*p-value*	
	Starch pro-analysis	Technical starch
Ash content	0.269	0,001
Starch content	0.060	0,000

The following analysis is FTIR analysis, which aims to identify the functional groups of a sample. This analysis seeks to compare the IR spectra of expired starch samples with new starch and technical starch to obtain spectral similarities and correlation values. The spectrum similarity is the basis for analysis to support the stability of expired starch and whether it is still suitable for use in practice. The sample is prepared, and the number of waves is read at a distance of 4500-500 cm^{-1}. The results of FTIR spectrum analysis are presented in Figures 2 each starch sample. Meanwhile, Figures 2 show the differences in expired starch. The known similarity values are presented in Table 4, where, based on the IR spectrum, passed starch is more similar to technical starch because it has a higher correlation value of 0.976504. Nevertheless, this value has a low difference with the correlation value of pa starch, which is 0.975026, so based on the IR spectrum of expired starch and technical starch, there is no significant difference (Tkachenko & Niedzielski, 2022). Based on the FTIR spectrum, it is known that there is a decrease in the %transmittance of expired starch compared to technical starch as a whole; this can be caused by starch undergoing degradation so that monomers are formed, which causes an increase in functional groups [19]. The X-axis in the spectrum shows the wave number (1/wavelength) while the Y-axis shows the transmittance percentage. The transmittance percentage indicates the richness of functional groups. This addition causes a decrease in the percentage transmittance value. In addition, when observed in the technical starch spectrum in Figure 2, some peaks do not exist in the pa and expired starch spectrum, which identifies impurities such as metallic minerals.

Table 4. Correlation of FT-IR Spectrum of Expired Starch to pro-analysis Starch and Technical Starch.

Sample	Correlation Value of Expired Starch
Pro-analysis starch	0.975026
Technical starchg	0.976504

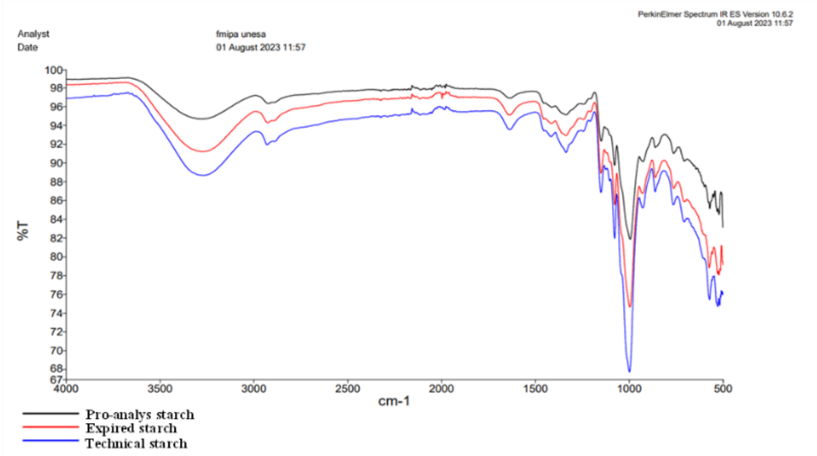

Figure 2. FTIR Spectrum of Starch Samples.

Conclusion

The results of the analysis showed that expired starch had no significant difference with the pro-analysis starch in terms of ash and starch content (p-value > 0.05). In addition, the FTIR spectrum did not differ much from the starch spectrum in new conditions (correlation value close to 1). So, expired starch is still suitable for use as a practical material to reduce potential waste. Further research can be carried out to provide more support in supporting the use of expired chemicals as supporting materials for experiments in laboratories in an effort to reduce chemical waste.

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