Utilizing Recycled Evaporated Organic Solvent Waste for Green Chemistry in Chemical Laboratories to Reduce Liquid Waste

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ABSTRACT

The use of solvents, especially organic solvents in chemical laboratories, particularly in extraction processes, cannot be separated from laboratory activities. Since extracted organic solvents are not seen as having desirable qualities, they are not recycled and end up as liquid waste. The evaporation method was used to handle organic solvent waste from evaporation, and the evaporator exhibited the same physical and chemical characteristics as technical solvents. This study aimed to determine whether organic solvent waste from the evaporation process could be recycled and reused following the concept of green chemistry in laboratory waste treatment. The research method used in processing the recycled organic solvent waste (ethanol & methanol) from the evaporation was the experimental method through the evaporation process by evaporation and characterized based on the FT-IR and GC-MS test parameters on organic solvents pro-analysis and technical. Based on the results, the correlation coefficient analysis for testing the second recycled methanol- pro-analysis methanol, the second recycled methanol - technical methanol, and the second recycled ethanol- pro-analysis ethanol was quite strong. according to the results of the solvent characterization test of methanol and ethanol from the second recycling solvents by evaporation through the FT-IR test. Technical correlation values of 0.9982, 0.9983 and 0.9902. Meanwhile, the second recycled ethanol- pro-analysis ethanol had correlation coefficient value, 0.0079, was quite low. However, the second recycled solvent did not significantly differ from the technical solvent in the GC-MS test ($p>0.05$).

Keywords: *Evaporation, laboratory, Liquid waste, Organic solvent*

1. INTRODUCTION

The Chemistry Laboratory is located in buildings C5 and C6, Faculty of Mathematics and Natural Sciences at Unesa, which serves instructors and research students. The amount of laboratory waste containing harmful and hazardous pollutants may rise as a result of lab activity. The majority of the waste produced is liquid waste from the remaining sample, practicum, or research reagents [1].

One of the sources of liquid waste from organic solvents created by the research and development process based on natural materials comes from the organic chemistry lab [2]. Secondary metabolites were frequently extracted from plants using organic solvents. Depending on the type of polarity group, organic solvents could be either polar or non-polar. Alcohol, ether, ester, ketone, and other substances consist of organic solvents [3].

The filtrate of the extraction was evaporated with an evaporator to obtained a thick extract. This process produced organic solvents left over from evaporation that were not utilized and could eventually turn into liquid waste that could damage living things and affect the environment [2]. This was a big problem in the laboratory because waste treatment was expensive. Efforts to handle organic solvent waste were oriented towards the concept and approach of *green chemistry.*

A concept knew as "green chemistry" sought to solved environmental issues by using chemicals generated, procedures, or reaction steps. This concept was connected to a technique that minimized the usage and production of poisonous chemicals through both process and design considerations [4]. According to research conducted in 2021 [5], the results of recycling organic solvent waste through evaporation exhibited the same properties as technical solvents when measured using the parameters of FT-IR, and HPLC testing. This result indicated that organic solvents from recycled waste solvents from evaporation could be reused, particularly in the maceration process of natural materials.

Based on this background, the authors were interested in continuing to studied the recycling method by evaporation and examined the stability of the quality of organic solvents from the second recycled waste solvent based on characterization by FTIR and GC-MS tests. The recycling of the evaporated solvent waste could be carried out continuously and following the orientation of *green chemistry* to reduce liquid waste in chemical laboratories.

2. METHOD

2.1. Tools and Materials

This research used the following tools: beaker, measuring cup, dropper, glass funnel, glass bottle, thermometer, Buchner funnel, vacuum pump (Comecta), analytical balance (Adventure), vacuum rotatory (Buchi R-300), FT-IR Spectrophotometer (Perkin Elmer), GC-MS (Perkin Elmer).

This research used the following materials: temulawak, recycled organic solvent waste from the first evaporation (methanol and ethanol), methanol pa, technical methanol, ethanol pa, technical ethanol, and aquades

2.2. Procedure of Research

This research was carried out for the processing of waste from the second evaporation of research students using methanol and ethanol organic solvents, obtained from the recycling of the waste from the first evaporation that was recycled and used for the maceration process [5]. The polar solvents methanol and ethanol used for maceration due to their great capacity to attract secondary metabolites in the sample and their affordable and economical were chosen.

The sample taken was the second evaporation waste which was previously carried out by the maceration process with the recycled solvent from the first evaporation waste. The resulting filtrate from the maceration process was added to a rotary evaporator. A thick extract, as well as the solvent from the rotary evaporator, would be created. We'll utilize this solvent as a sample. These samples will also be evaluated using FT-IR and GC-MS tests to compare the qualities of the second evaporation solvent waste recycling to pure solvents (pro analysis) and technical solvents.

1.3. Data Analysis

The recycling of the second evaporated solvent waste that had been processed by the evaporation method was compared with technical and pro-analysis (pa) solvents to determine the characterization of waste treatment results. Analysis was carried out using a comparative descriptive method for results of the FT-IR test and the t-test for the result of GS-MS test.

3. RESULT AND DISCUSSION

Organic solvents were chemicals often used in organic chemistry laboratories to extract natural materials. Vacuum rotary evaporator was used to separate the acquired extract from the solvent. According to research [5], processing organic solvent waste from evaporation was done using an evaporator that had similar physical and chemical properties to technical solvents.

The purpose of this study was to determine whether the results of recycling organic solvent waste from the second evaporation process could be reused so that it was following the concept of green chemistry in laboratory waste treatment. The solvent waste tested in this study was methanol and ethanol. So that organic solvent waste from the second evaporation process was recycled, the solvent waste was re-evaporated with an evaporator. The second recycled organic solvent was compared with pro-analysis and technical solvents through characterization with FT-IR and GC-MS tests.

3.1. FT-IR Testing

FT-IR (Fourier Transform Infrared) was a technique used to identify functional groups in materials (gas, liquid, and solid) using infrared radiation beams. IR spectroscopy applied to a wide variety of materials and conditions and could be utilized for qualitative and quantitative analyses. Infrared spectroscopy measures the absorption of IR radiation created by each bond in a molecule and as a result, reached a spectrum generally expressed as % transmittance versus wavenumber (cm^{-1}). Spectrum analysis was effective for identifying organic compounds, because of it was highly functional spectrum that absorbs infrared light at a unique frequency [6-7].

IR spectral data were collected from observations in the intact absorption region (4000-550 cm-1). Figure 1-2 presented the infrared spectral pattern as a result of comparing the second recycled methanol solvent with pro-analyst and technical methanol gave almost the same absorption pattern. The quantitative magnitude of absorption in each wavelength was just marginally different. The spectral data in Figure 4 also presented the infrared spectrum pattern as a result of comparing the second ethanol solvent with technical ethanol. These spectral patterns gave rise to similar absorption patterns, with the only difference being the quantitative value of the absorbance of each spectrum. Meanwhile, Figure 3 showed the infrared spectrum pattern as a result of comparing the second recycled ethanol solvent with ethanol p.a, additional peaks indicated that the tested solvent contained contaminants.

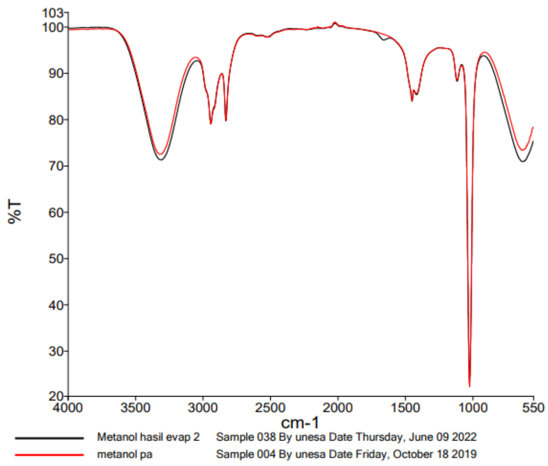

Figure 1 Spectrum IR of compering the second recycled methanol solvents with methanol p.a

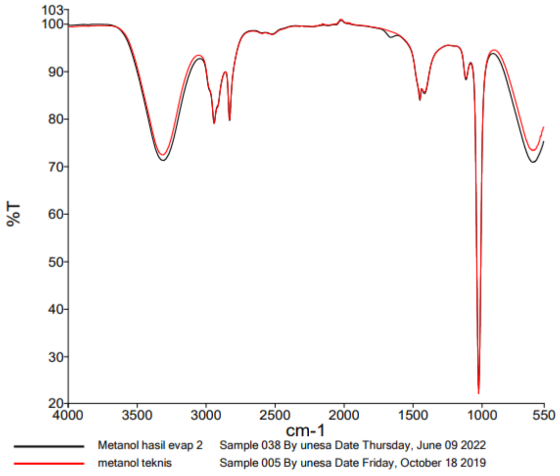

Figure 2 Spectrum IR of compering the second recycled methanol solvents with technical methanol

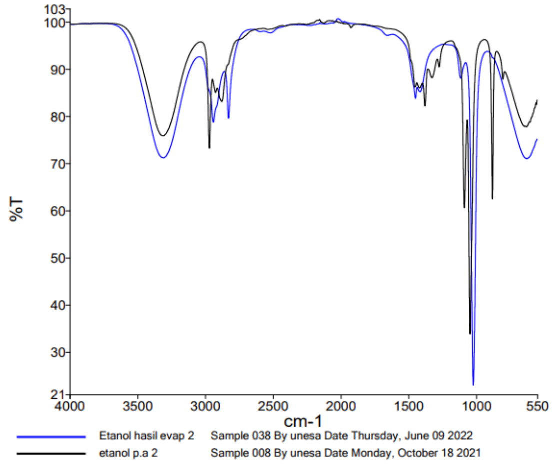

Figure 3 Spectrum IR of compering the second recycled ethanol solvents with ethanol p.a

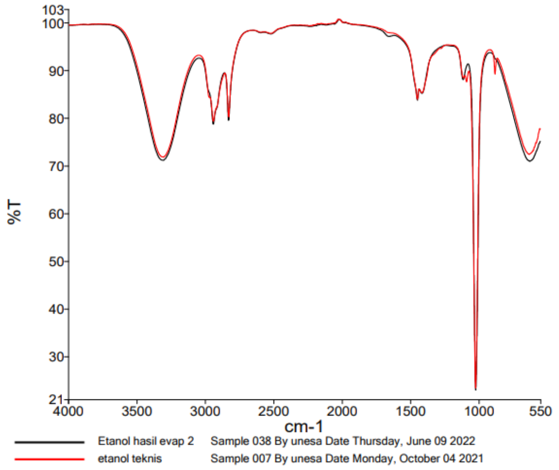

Figure 4 Spectrum IR of compering the second recycled ethanol solvents with technical ethanols.

The second recycled solvents from evaporation wastes were analyzed by FT-IR to saw the correlation coefficient of the functional groups based on the intensity of infrared light absorbed compared to the FT-IR spectrum of pro analyses and technical solvents. Correlation analysis was carried out to studied the closeness or strength of the relationship between the second recycled solvent, pro-analysis, and technical solvents expressed by the correlation coefficient based on the IR spectrum. The correlation coefficient (r) was a value that indicated whether or not there was a strong linear relationship between two variables with values in the range -1 to 1. Correlation coefficient (r): strong r= ± 0.7 to ± 1, moderate r = ± 0.3 to ± 0.699, weak r = 0 to ± 0.299 [8]. The following correlation figures are presented in table 1.

Table 1. The results of the correlation coefficient analysis of the FT-IR test.

Type of Solvent	Sample	Correlation
Methanol	The second recycled methanol-methanol p.a	0,9982
	The second recycled methanol- technical methanol	0,9983
Ethanol	The second recycled ethanol-ethanol p.a	0,0079
	The second recycled ethanol- technical ethanol	0,9902

Whether it was a correlation to pro-analysis or technical methanol, it could be showed from table 1 that the second recycled methanol solvent had a strong correlation coefficient value closed to number one. These findings suggested that the qualities and purity of the second recycled methanol were essentially identical with pro-analysis and technical methanol. Similarly, the correlation coefficient between the second recycled ethanol and technical ethanol had a substantial value closed to number 1. These findings suggested that the second recycled ethanol qualities and purity were essentially identical with technical ethanol.

Meanwhile, the correlation coefficient value of the second recycled ethanol with pro-analysis ethanol was categorized as wea, because correlation coefficient value closed to number 0, different findings were displayed. These findings suggested that the second recycled ethanol qualities and purity were substandard or different from pro-analysis ethanol.

3.2. GC-MS Testing

Chromatography was a term used to described a separation technique in which a mobile phase carrying a mixture was caused to move in contact with a selectively absorbing stationary phase. The principle of gas chromatography was adsorption and partition. Gas chromatography was used in the separation and analysis of multi-component mixtures such as essential oils, hydrocarbons, and solvents. GC-MS had became a highly recommended tool for monitoring and tracking organic solvents in samples [9]. The following was the percentage of solvent content in the second recycled solvent by GC-MS testing.

Table 2. Percentage of methanol content

Sample	% Methanol content ± SD
The second recycled methanol solvents	99,56 ± 0,35
The technical methanol	99,47 ± 0.55
	p = 0,450

Table 3. Percentage of ethanol content

Sample	% Ethanol content ± SD
The second recycled ethanol solvents	0.52 ± 0,01
The technical ethanol	0,66 ± 0,01
	p = 1,000

Based on table 2, there was no significant difference in the percentage of methanol content between the second recycled methanol and technical methanol using the independent sample t-test (p>0.05). It could be argued that the second recycled methanol had a methanol content that was not significantly different from pro-analysis and technical methanol due to the high percentage of methanol content in each sample, which was close to 100%.

Based on table 3, there was no significant difference in the percentage of ethanol content between second recycled ethanol and technical ethanol using the t-test sample independent (p>0.05). It could be argued that the second recycled ethanol had an ethanol content that was not significantly different from technical ethanol. The percentage of ethanol content in each sample was very low, below 1%. However, a low proportion of ethanol content revealed that both had low ethanol purity.

The low correlation coefficient value of the FT-IR test findings between the second recycled ethanol and pro-analysis ethanol of 0.00786803, which was near 0, supported the low ethanol content in both the second recycled ethanol and technical ethanol. This suggests that mixtures or impurities in the technical solvent employed in the first extraction procedure impaired the technical purity of ethanol. The ethanol concentration was impacted by contaminants like water that were present in ethanol. The fraud committed by ethanol sellers was selling 96% technical ethanol added with water so that the concentration of technical ethanol traded to consumers decreases. This fraud was supported by the difficulty of distinguishing between technical ethanol with high or low concentrations because the density of ethanol (0.8 gr/cm3) [10] was close to water (1 g/cm3) [11], so it was impossible to distinguish only by eye observation.

The processing of liquid waste from used solvent evaporation was one of the efforts to approached the concept of green chemistry to realized safe and environmentally friendly laboratory activities by reducing the use and production of hazardous substances, preventing the formation of waste, and using renewable raw materials [12-13].

4. CONCLUSION

Based on the results of the solvent characterization test of methanol and ethanol from the second recycling by evaporation through the FT-IR test, it showed that the correlation coefficient analysis was quite strong for testing the second recycled methanol- pro-analysis methanol, the second recycled methanol-technical methanol and the second recycled ethanol-technical ethanol with a correlation value close to 1, namely 0.9982, 0.9983 and 0.9902. While the correlation coefficient value of the second recycled ethanol- pro-analysis ethanol was relatively weak, namely 0.0079. Meanwhile, the GC-MS test did not showed a significant difference *($p>0.05$)* between the second recycled solvent by evaporation compared to the technical solvent. The GC-MS test did not showed a significant difference *($p>0.05$)* between the second recycled solvent by evaporation compared to the technical solvent. The purity of the second recycled ethanol solvent and technical ethanol was very low based on the percent ethanol content. The second recycled solvent using the evaporation method could be reused because it had the same characteristics as the technical solvent and was expected to reduced liquid waste from chemistry laboratories.

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