The 4th International Seminar of Science and Technology
ISST 2024 Vol 4 (2025) 025
Innovations in Science and Technology to Realize Sustainable Development Goals
Faculty of Science and Technology
Universitas Terbuka

Characterization and Application of Distillation By-product from Kjeldahl Protein Testing as A Liquid Medium for Air Pollution Control

Aliefi Mutiara Syafitri[1*], Mona Dwi Fenska[2], Anisa Nuraeni[2], Bambang Herwanto[2], Adittya Dwi Apriadi[2], Ilham Fajar Subakti[2]

[1]Universitas Terbuka, Food Technology Study Program, South Tangerang, Indonesia, 15437
[2]PT. Sanghiang Perkasa, Karawang, West Java, Indonesia, 41373

Abstract - The challenge of managing industrial waste and controlling air pollution necessitates innovative solutions. Wet scrubbers effectively remove pollutants from industrial emissions; however, the conventional liquid media used in these systems can be costly and environmentally detrimental. This study explores the feasibility of repurposing by-products from distillation processes, specifically those derived from protein analysis using the Kjeldahl method, as an alternative liquid medium for wet scrubbers. The research evaluates the characteristic and pollutant absorption efficiency of these distillation by-products. The findings demonstrate that the distillation by-products, with proper adjustments to volume and concentration, can effectively neutralize pollutants. The results indicate significant reductions in NaOH concentration over a 10-day testing period, showcasing the solution's viability for long-term use. This approach not only offers a cost-effective solution but also aligns with sustainability goals, particularly in reducing waste and promoting responsible resource consumption. The study supports Sustainable Development Goals (SDGs) 12 (Responsible Consumption and Production) and 13 (Climate Action) by proposing a method to enhance air quality and minimize waste production.

Keywords: Kjeldahl method, air pollution control, distillation by-product, wet scrubber, sustainability

1 Introduction

Food is a vital part of daily life, providing essential nutrition for health and well-being. Ensuring food safety is critical, as any compromise in quality can lead to serious health risks. Consequently, the entire food production chain must undergo stringent monitoring and testing, primarily conducted by the food industry. Laboratories play a crucial role in assessing food safety and quality, mitigating potential health hazards through comprehensive testing for adulterants, contaminants, and nutritional value [1]. They also enforce labeling regulations and contribute to research in food safety. As foundational elements of national health programs and public health frameworks, laboratories serve as the backbone of effective food safety control systems [2].

* Corresponding author: aliefi.syafitri@kalbenutritionals.com

By providing independent and accurate testing, they deliver reliable data to consumers, regulators, researchers, and the food industry, ensuring compliance with safety standards and reinforcing public trust in the food supply chain. One of the most frequently tested parameters in food products is protein content. Protein is a vital nutrient necessary to maintain overall health, and insufficient protein intake can lead to serious health disorders such as marasmus, kwashiorkor, organ failure, and a weakened immune system [3].

Therefore, the protein content in food is a critical factor in determining both the quality and safety of food products [4]. Reliable analytical methods are essential, and the Kjeldahl method is the most commonly used technique for determining protein content. The total protein nitrogen content is determined in 1 gram of dried samples using the micro-Kjeldahl technique, based on method 981.10 of the AOAC International [5], [6], [7]. The Kjeldahl method consists of three main stages:

1. Digestion: using sulfuric acid along with various catalysts and salts to convert organically bound nitrogen in the sample into ammonium bisulfate. sulfuric acid breaks down all organic material into carbon dioxide, water, and ammonium bisulfate. [8]

   $$NH_2\text{-}CH_2\text{-}COOH + 4H_2SO_4 \rightarrow 2CO_2 + 3SO_2 + 4H_2O + (NH_4)HSO_4$$

2. Distillation: free ammonia (NH_3) is liberated from the ammonium sulfate solution by steam distillation in the presence of excess sodium hydroxide (NaOH), according to the reaction:

   $$(NH_4)HSO_4 + 2NaOH \rightarrow NH_3 + Na_2SO_4 + 2H_2O$$

   The evolved ammonia is then collected in excess boric acid, forming an ammonium-borate complex:

   $$NH_3 + H_3BO_3 \rightarrow + H_2BO-$$

3. Titration: the solution is titrated with hydrochloric acid (HCl) to estimate the total nitrogen content, as shown by the reaction:

   $$NH_4^+ + H_2BO_3^- + H_2SO_4 \rightarrow (NH_4)HSO_4 + H_3BO_3$$

The Kjeldahl method produces three main by-products: carbon dioxide (CO_2) gas during digestion, a solution containing sulfate salts and a base from distillation, and a mixture of ammonium chloride salts and boric acid from titration which is straightly discarded to waste treatment due to its hazardous characteristic. While laboratories play an essential role in ensuring food quality and safety, their operations are not without drawbacks. Conventional laboratory practices often generate substantial environmental pollutants, posing health risks to those exposed [9]. Additionally, these methods are associated with high energy consumption, excessive waste production, and significant resource depletion, contributing to environmental challenges as shown below in figure 1 [10].

To address these challenges, businesses, laboratories, and governments have implemented various efforts to mitigate the environmental impacts of industrial and laboratory activities [11]. These initiatives aim to promote more sustainable practices and reduce the ecological footprint associated with such operations. For instance, PT Sanghiang Perkasa has established a waste management system to handle both domestic and hazardous waste generated from its activities, along with air quality control measures using air pollution control devices, specifically wet scrubbers.

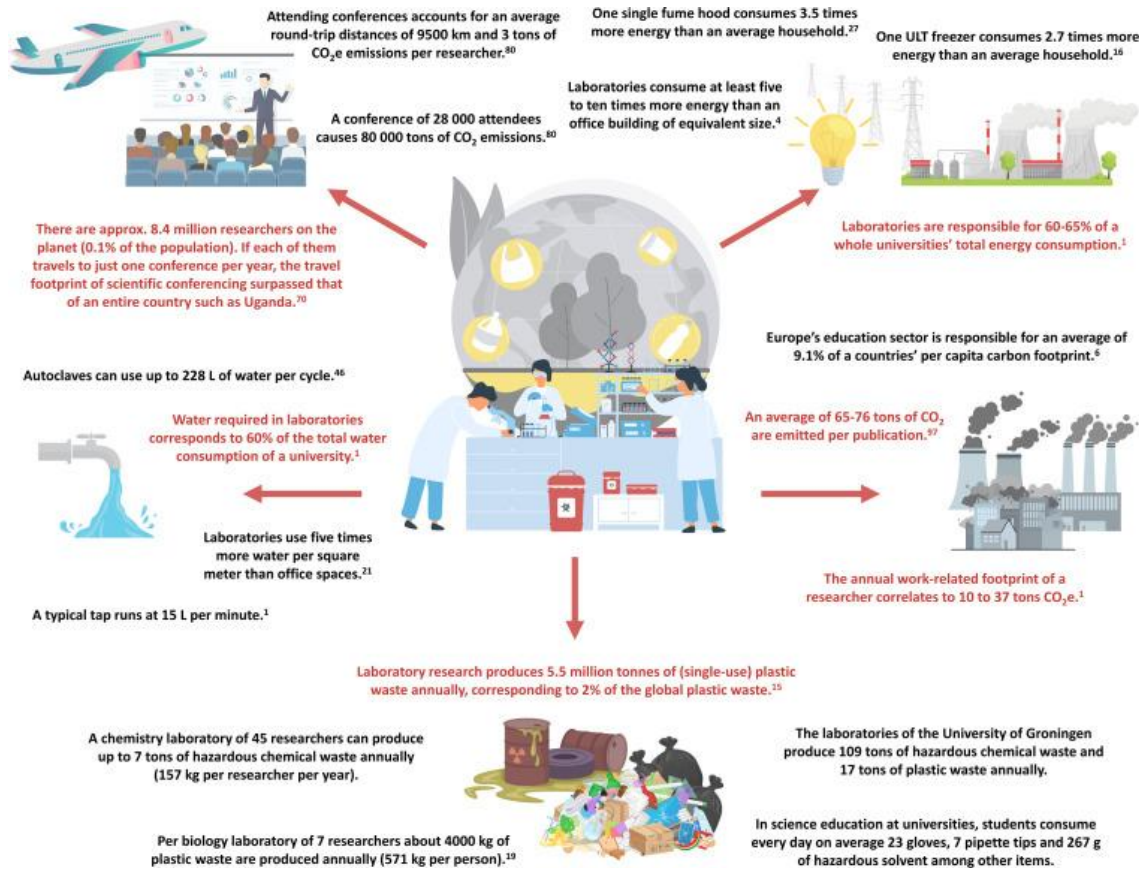

Fig 1. The overlooked environmental impact of laboratories and scientific research.

Wet scrubbers are effective devices for air pollution control, designed to remove particulate matter and gases from industrial exhaust streams. They operate by introducing a scrubbing liquid into the contaminated gas stream, allowing pollutants to be absorbed or captured. Wet scrubbers are effective for applications requiring simultaneous removal of both particulates and gases [12]. Various systems are tailored to specific process requirements, with key operational factors influencing the selection of size and type [13]. In the treatment process, gases are continuously scrubbed within a column, and the aqueous phase is circulated in either a counter-current or co-current mode. This involves the absorption of gaseous reactants into the liquid phase, enhanced by rapid chemical reactions with oxidizing agents in the solution. The efficiency of gas absorption is further improved by using high-surface-area materials with large pore sizes packed into the column, which maximizes the gas-liquid interfacial area for better mass transfer [14].

In the present study, a variety of commonly used substances, including seawater, sodium hydroxide (NaOH), sodium hypochlorite (NaClO), sodium chlorite ($NaClO_2$), hydrogen peroxide (H_2O_2), and potassium permanganate ($KMnO_4$), were systematically compared for their effectiveness in removing SOx and NOx [14]. Findings from Chu et al. (2009) [15] indicated that SO_2 absorption is primarily controlled by the gas film when NaOH concentrations exceed 0.1 M or $KMnO_4$ concentrations exceed 0.05 M. Similarly, a study by Premkumar et al. [16] demonstrated that using NaOH as a wet scrubber medium effectively reduces levels of NO, hydrocarbons (HC), carbon monoxide (CO), and smoke in emissions. These findings highlight NaOH's significant capacity to reduce air pollutants.

Wet scrubbers have emerged as highly effective technologies for controlling air pollution by using liquid media to capture pollutants from industrial exhaust gases. However, conventional scrubbing solutions, such as sodium hydroxide (NaOH), can be costly and present significant environmental challenges due to their hazardous nature. Typical wet scrubber operations require around 117 kg of NaOH and 480 liters of distilled water annually to maintain the scrubbing solution. At the same time, industries, particularly through Kjeldahl protein testing, generate up to 3 tons of distillation by-products annually, which contain NaOH. These figures are derived from internal documentation and direct observation of laboratory practices at PT Sanghiang Perkasa.

This research aims to explore the potential of repurposing this distillation by-product as an alternative scrubbing medium. Currently, distillation by-product is classified as hazardous material, requiring third-party treatment, which incurs additional costs for disposal. By repurposing the by-product for use in wet scrubbers, the reliance on NaOH as a hazardous material could be reduced, while also mitigating the costs associated with third-party waste management.

The novelty of this study lies in transforming a waste product that would otherwise require expensive treatment into a functional, environmentally friendly solution for air pollution control. This approach could promote more sustainable industrial practices by reducing hazardous waste and supporting cost-effective, environmentally responsible air pollution management. The research findings are expected to contribute significantly to both waste management and air pollution control sectors.

2 Materials and methods

2.1 Time and Place of Research

The research was conducted from July to October 2023 at PT Sanghiang Perkasa's Analytical Center, an ISO 17025:2017 accredited testing laboratory, located in Cikampek, West Java.

2.2 Materials and Equipment

2.2.1 Materials

The materials used in this research include sodium hydroxide (NaOH) solution, prepared by dissolving technical grade NaOH in distilled water for comparison. Phenolphthalein is utilized as a pH indicator during the titration process, while distilled water (aquadest) is employed for solution preparation and as a reference liquid. Additional materials, such as reagents and chemicals, are included as required by the reference method. The other material used is the distillation by-product from Kjeldahl protein testing, which serves as the primary alternative scrubbing medium. Figure 2 depicts the process flow from which the by-products are collected.

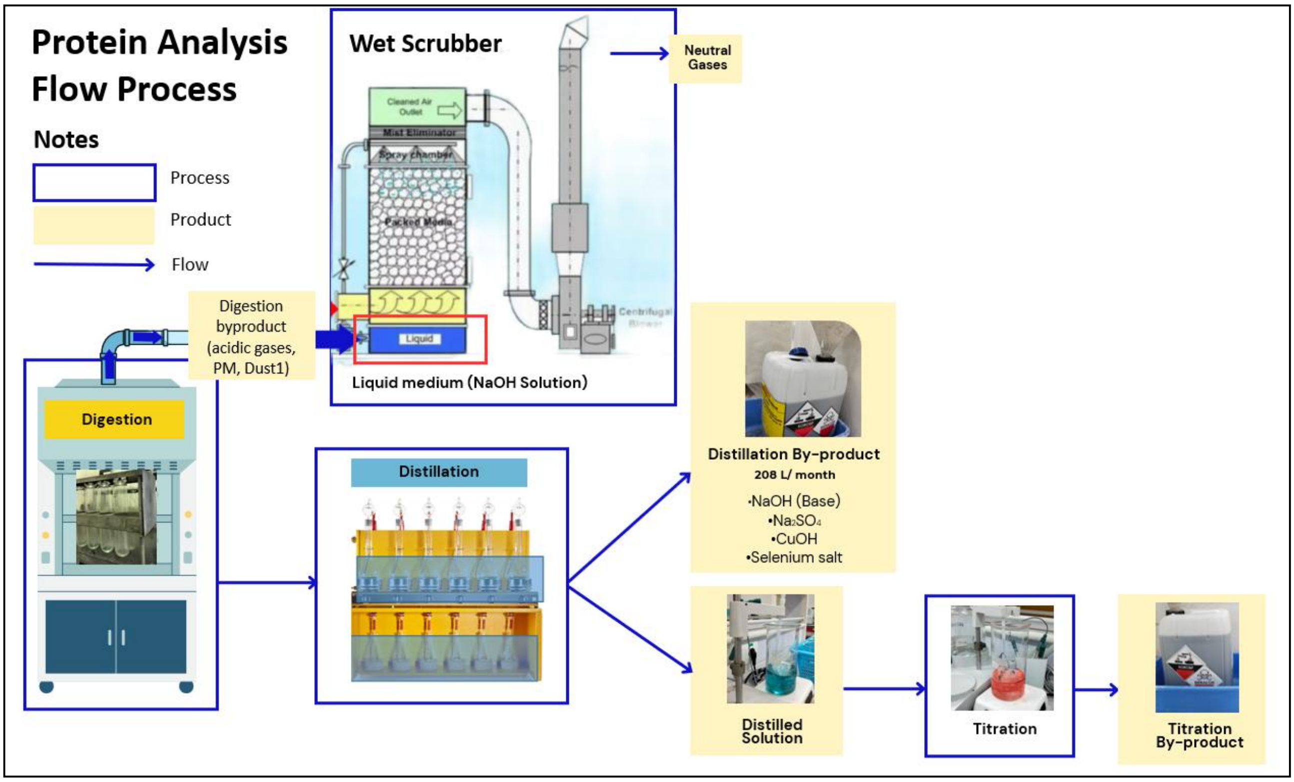

Fig 2. Flow process of protein content analysis using the Kjeldahl method [8]

2.2.2 Equipment

The equipment used in this study includes a sampling kit for collecting liquid samples from the wet scrubber output. Laboratory glassware, such as beakers, flasks, and burettes, are sourced from Iwaki (Japan). Two types of wet scrubbers are utilized: a mini-size wet scrubber with a 35-liter capacity and a regular-size wet scrubber with a 105-liter capacity, both custom-built by Rolland. For pH measurement, a universal pH indicator from Merck is used for quick assessments, while a Mettler Toledo pH meter provides more accurate readings. A conductivity meter and Total Dissolved Solids (TDS) meter from Hach are employed to determine ion concentration and the level of dissolved solids in the scrubbing solutions, respectively. Additional instruments, such as spectrophotometers, are included as needed, depending on the reference method, though the specific model is not detailed in this study.

2.3 Research Design

2.3.1 Characterization of Distillation By-product

In the initial stage of characterization, the concentration of a conventional NaOH wet scrubber solution was compared to that of the distillation by-product by determining the molarity of NaOH in each solution through acid-base titration. Each type of solution was sampled and analyzed 30 times to ensure accuracy and repeatability. Hydrochloric acid (HCl) was used as the titrant, and phenolphthalein served as the pH indicator. The endpoint of the titration was identified by the appearance of a light pink color in the solution. The concentration of NaOH was then calculated using the standard titration equation.

$$M\ sample = \frac{M\ HCl\ x\ V\ HCl}{V\ sample}$$

The next step involved analyzing the physical and chemical properties of both the NaOH solution and the distillation by-product. The parameters tested included pH, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), and Methylene Blue Active Substances (MBAS) to have a better understanding of distillation byproduct potential to be repurposed as a liquid medium of a wet scrubber. The reference methods for these analyses are listed in Table 1, which provides the standard procedures followed for each parameter.

2.3.2 Effectivity Testing

The effectiveness testing was based on comparing molar equivalency and observing reduction in NaOH content over time through acid-base titration, following procedures similar to those described in Vogel's Textbook of Quantitative Chemical Analysis [17] .Effectivity testing begins with adjusting or calculating the amount of distillation by-product to be used based on the initial characterization results. The aim is to ensure that the content of NaOH in the by-product (expressed in millimoles, mmol) is equivalent to the NaOH content in the conventional NaOH solution so that both solutions have the same level of alkalinity and can perform similarly as scrubbing media.

Table 1. Analyses Reference Method

Testing Parameter	Unit	Reference Method
pH	-	SNI 6989.11-2019 [20]
TDS (Total Dissolved Solid)	mg/L	SNI 6989.27:2019 [21]
TSS (Total Suspended Solid)	mg/L	SNI 6989.3:2019 [22]
COD (Chemical Oxygen Demand)	mg/L	SNI 6989.2-2019 [23]
BOD (Biological Oxygen Demand)	mg/L	SNI 6989.72.2009 [24]
MBAS (Methylene Blue Active Substances)	mg/L	SNI 06-6989.51 – 2005 [25]

This adjustment can be made using the molar concept, where the molarity (M) is calculated using the equation:

$$M\ (molarity) = \frac{mmol}{mL}$$

By using this formula, the volume of the solution can be adjusted based on the sample molarity and the desired NaOH content (in mmol). This ensures that the by-product solution matches the performance of the conventional NaOH solution in terms of neutralizing pollutants.

After calculating and adjusting the volume of the distillation by-product to match the molar concentration of conventional NaOH solution, wet scrubbers were operated using the adjusted distillation by-product as the liquid medium. Three test replications were conducted, each monitored over a 10-day period. The concentration of NaOH, expressed in millimoles (mmol), was measured on days 1, 3, 5, 7, and 10 to assess the effectiveness of the solution in capturing pollutants. This effectiveness was determined by observing the reduction in NaOH concentration, which occurred due to the neutralization process.

To measure NaOH concentration, a 50 mL sample was collected from the wet scrubber, and an acid-base titration was performed using hydrochloric acid (HCl) as the titrant and phenolphthalein as the indicator. This allowed for a precise evaluation of how well the distillation by-product performed as a scrubbing medium over time.

3 Results and discussion

Table 2. Characterization comparison result of conventional wet scrubber liquid medium (NaOH solution) and distillation by-product

Testing Parameter	Unit	NaOH Solution	Distillation By-Product
NaOH Concentration	M	1.9200	0.8102
pH	-	14.4	11.1
TDS	mg/L	9612	4799
TSS	mg/L	118	78
COD	mg/L	332	205
BOD	mg/L	100	62
MBAS	mg/L	0.24	0.22

3.1 Characterization Result

The NaOH concentration is higher in the NaOH solution (1.9200 M) compared to the distillation by-product (0.8102 M), resulting in stronger alkalinity and a higher pH (14.4 vs. 11.1). However, the alkalinity of the distillation by-product can be adjusted by increasing its volume to achieve the desired concentration, thereby ensuring it functions similarly to the NaOH solution in absorbing acidic gases like SO_2 and NOx.

The Total Dissolved Solids (TDS) and Total Suspended Solids (TSS) are lower in the distillation by-product (TDS: 4799 mg/L, TSS: 78 mg/L) compared to the NaOH solution (TDS: 9612 mg/L, TSS: 118 mg/L). A lower TDS means the distillation by-product has fewer dissolved substances, which could result in less scaling and fouling within the scrubber system. Lower TSS indicates that the solution contains fewer particulate matter or solid residues, reducing the likelihood of clogging or blockages during operation. This suggests that the distillation by-product can maintain a cleaner and more efficient scrubbing process, with reduced maintenance needs and longer operational periods between cleanings [18].

The Chemical Oxygen Demand (COD) and Biological Oxygen Demand (BOD) values are notably higher in the NaOH solution (COD: 332 mg/L, BOD: 100 mg/L) compared to the distillation by-product (COD: 205 mg/L19, BOD: 62 mg/L). These higher values indicate that the NaOH solution contains more organic contaminants, making it more environmentally harmful when disposed of. In contrast, the lower COD and BOD values of the distillation by-product reflect a lower organic load, making it less detrimental to the environment and easier to manage in terms of waste treatment and disposal.

The MBAS (Methylene Blue Active Substances) levels are slightly higher in the NaOH solution (0.24 mg/L) compared to the distillation by-product (0.22 mg/L). MBAS is an indicator of surfactants, which can cause foaming during the scrubbing process. Excessive foam can hinder the efficiency of gas absorption, decreasing the overall performance of the scrubber. The lower MBAS value in the distillation by-product suggests a reduced risk of foam formation, contributing to a more stable and efficient scrubbing process [19].

3.2 Effectivity Testing Result

3.2.1 Formula adjustment

Table 3 outlines the necessary adjustments in the formula for using distillation by-products to match the alkalinity properties of conventional NaOH solutions in both mini-size and regular-size wet scrubber machines.

Table 3. Formula adjustment for distillation by-product to match alkalinity property of NaOH Solution

Machine Type	Alkaline Source	mmol NaOH per Batch	Volume per batch (mL)
Mini size (35L)	NaOH Solution	9600	5000
	Distillation By-product	9600	12000
Regular size (105L)	NaOH Solution	36000	5000
	Distillation By-product	36000	45000

In the mini-size scrubber (35L), the amount of NaOH solution used per batch is 9600 mmol, corresponding to a volume of 5000 mL. To achieve the same alkalinity using the distillation by-product, a higher volume of 12000 mL is required to match the same mmol of NaOH. This indicates that while the distillation by-product can effectively provide the necessary alkalinity, it requires a larger volume due to its lower NaOH concentration compared to the conventional solution. This is consistent with the principle of stoichiometry in neutralization reactions, which states that a lower concentration base requires a proportionally larger volume to neutralize a given amount of acid (eCampusOntario, 2020).

For the regular-size scrubber (105L), the NaOH solution provides 36000 mmol per batch at a volume of 5000 mL. However, when using the distillation by-product, a volume of 45000 mL is needed to achieve the same alkalinity level. This underlines the need for a significant increase in the volume of distillation by-products to maintain effective scrubbing performance.

3.2.2 Effectivity testing

The following graph illustrates the changes in NaOH concentration over a 10-day testing period across three replicates, showcasing the performance of the wet scrubber system using distillation by-products as the scrubbing medium. Each data point reflects the concentration of NaOH, measured in millimoles.

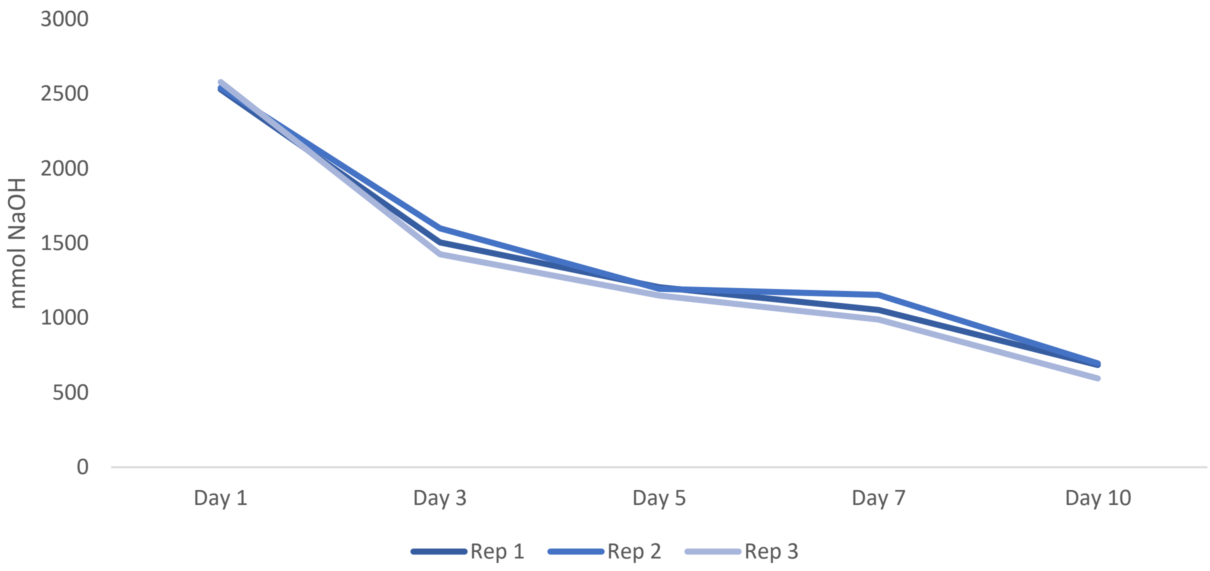

Fig 3. Graph of decreasing NaOH concentration in distillation by-product

The data indicates a clear trend of decreasing NaOH concentration, demonstrating the ability of the wet scrubber system to effectively absorb pollutants from the gas stream over time. The initial sharp drop in concentration from Day 1 to Day 3 reflects the system's rapid response to pollutant capture, likely due to the high alkalinity of the NaOH solution, which facilitates the neutralization of acidic gases.

The gradual decline in concentrations from Day 3 to Day 10 further highlights the system's efficiency, although the decrease rate appears to slow as time progresses. This could suggest that as pollutants are captured, the remaining concentration of reactive NaOH becomes more limited, necessitating periodic adjustments or replacements of the scrubbing solution to maintain optimal performance.

This data shows the wet scrubber's performance in substantial reduction of NaOH concentrations, indicating its efficacy in capturing and neutralizing gaseous pollutants over the 10-day operational period. These findings underline the importance of ongoing monitoring and adjustments to the scrubbing medium to ensure sustained effectiveness in air pollution control.

4 Conclusion

The findings of this research strongly support the potential of distillation by-products as a highly effective and sustainable alternative to conventional NaOH solutions in air pollution control. Despite the initial lower NaOH concentration of the distillation by-product (0.8102 M compared to 1.9200 M in the traditional solution), careful adjustments to volume and alkalinity allow it to achieve comparable pollutant absorption efficiency. This demonstrates that industrial waste, when appropriately managed, can serve as a valuable resource rather than a liability. The environmental advantages, such as lower TSS, TDS, COD, BOD, and MBAS values, further emphasize the by-product's superior environmental profile, improving the operational stability and efficiency of wet scrubbers.

The effectivity testing results clearly show a significant decline in NaOH concentration over a 10-day period, affirming the by-product's capability in pollutant capture and neutralization. This not only confirms the hypothesis that distillation by-products can perform similarly to traditional NaOH solutions but also highlights the importance of precise adjustments to ensure optimal scrubbing efficiency. The consistent reduction in NaOH concentration underscores the practicality of using distillation by-products in industrial air pollution control systems.

However, the study's initial success opens the realm of possibility for further research. Investigating the long-term stability of the by-product under varying operational conditions and conducting more extensive air quality assessments will be crucial for validating its broader industrial applicability. Moreover, research should explore how variations in waste composition and specific pollutants impact the by-product’s effectiveness, ensuring its utility across diverse industrial environments.

This research has far-reaching implications for sustainable industrial practices. By repurposing waste into functional solutions, this study directly supports SDG 12 (Responsible Consumption and Production) by enhancing resource efficiency and minimizing waste and and 13 (Climate Action) by proposing a method to enhance air quality and minimize waste production. Furthermore, it aligns with SDG 9 (Industry, Innovation, and Infrastructure), advocating for innovative, environmentally responsible industrial practices that can foster a more resilient and sustainable future. Advancing this approach has the potential to revolutionize industrial waste management, reduce reliance on hazardous chemicals, and contribute significantly to global efforts toward sustainable development.

5 Acknowledgements

The authors would like to express their gratitude to PT Sanghiang Perkasa for providing essential research facilities, resources, and funding that were vital for the successful completion of this study. Special appreciation is extended to the Quality Control Circle and The SIRIUS Team for their collaborative efforts in developing the project. The invaluable support from Fida Farhana and the management team at PT Sanghiang Perkasa is also acknowledged. Furthermore, the expert guidance of Andri Awaludin, the supervisor and advisor, was crucial throughout the research process. The contributions of Athiefah Fauziyyah, S.T.P., M.Si., as the academic advisor, provided essential insights that significantly enhanced the research quality. Finally, heartfelt thanks are given to Salma Billah Attaqy for her meticulous proofreading and insightful suggestions, which greatly improved the overall quality of this article The collective support and collaboration of all parties involved have been instrumental in achieving the objectives of this research.

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