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Nessler Method Verification for Determining Ammonia in Shrimp Pond Wastewater and Its Application in the Ammonia Adsorption Test with Lampung Natural Zeolite

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ABSTRACT. Ammonia levels exceeding 0.50 mg/L can threaten organisms in aquatic environments. The Nessler method is one of the ammonia analysis methods based on the reaction between ammonia in a basic solution and Nessler reagent (K_2HgI_4), forming a colloidal dispersion with a brownish-yellow colour. The colour intensity is determined by spectrophotometry. This research aims to verify the Nessler method for determining ammonia levels in shrimp pond wastewater. The research results indicate that the Nessler method shows good linearity in the range of ammonia concentrations from 1 to 5 mg/L, with a correlation coefficient (R^2) value of 0.9962. The precision value was determined from repeatability, expressed as %RSD (Relative Standard Deviation), i.e., 1.92%, and it meets acceptance criteria, which should be less than 0.5 of the Horwitz RSD. The accuracy obtained from the standard addition method provides a percentage recovery value of 99.25%, meeting the AOAC acceptance criteria. The detection limit and quantification limit of the technique are 0.3883 mg/L and 1.2943 mg/L, respectively. The verified method is then applied to analyze shrimp pond wastewater samples from Sriminosari Village, East Lampung, resulting in an ammonia concentration of 1.52 mg/L. The ammonia levels were then reduced by adsorption with natural zeolite Lampung, decreasing ammonia levels by 20.30%. Meanwhile, adsorption with an activated zeolite reduced the ammonia levels by 45.30%.

INTRODUCTION

Shrimp are the main export commodity for Indonesian fishery products (Yusuf *et al.*, 2021). The rapid development of the pond industry is accompanied by negative impacts, including pollution and environmental damage (Romadhona *et al.*, 2016). Organic waste is the biggest polluter of the aquatic environment. Leftover food that settles to the bottom of the aquatic environments will undergo a decomposition process, producing nitrates, nitrites, ammonia, carbon dioxide, and hydrogen sulphide (Suwoyo *et al.*, 2015). Ammonia (NH_3) will form ammonium ions (NH_4^+) in water at low pH conditions. Ammonia can cause toxic conditions for life in the aquatic environment. Based on PP no. 22 of 2021, the permitted ammonia level in river water is 0.5 mg/L, while an ammonia level of 1 mg/L can cause death to aquatic organisms (Marsidi and Herlambang, 2011). Reducing ammonia levels in wastewater can be achieved by utilizing the adsorption method. Ammonia adsorption can be carried out by using the absorption capacity of zeolite. The results of studies related to ammonia reduction using zeolite adsorbents were carried out by Nuryoto *et al.* (2020), with a percentage of ammonia absorbed at 68%.

Ammonia can be detected using several tests, including the phenate method (Alkindi *et al.*, 2023; Kurniawan *et al.*, 2021; Berliana and Wijayanti, 2022), rapid test method analysis (Nuraini and Yanti, 2020) and Nessler's reagent. The determination of ammonia levels in water with the Nessler method using a UV-Vis spectrophotometer is referred to as SNI 06-2479-1991. The Nessler method was discovered by J. Nessler in 1856. The principle of the Nessler method is based on the Nessler reagent (K_2HgI_4), which reacts with ammonia in an alkaline solution to form a yellow-brown colloidal dispersion. The intensity of the colour in the sample is then determined

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spectrophotometrically. The higher the colour intensity produced, the higher the ammonia concentration in the solution (Rahman, 2019).

Analysis using the Nessler reagent is based on the reaction between potassium tetraiodomercurate (II) (K_2HgI_4) and ammonia in an alkaline solution, which will form a yellow-brown colloidal dispersion. Determining ammonia using the Nessler method is the simplest, fastest, and most commonly used method. Ammonia analysis using Nessler's reagent was carried out by Ngibad (2019), who detected ammonia content in Pelayaran Ngelom river water at several points; the ammonia levels detected were 1.61 – 14.68 mg/L. Absorbance values were measured using UV-Vis Spectrophotometry (Alkindi *et al*., 2023; Berliana and Wijayanti, 2022).

Nessler method verification is used to ensure that the ammonia test method meets the requirements. Method verification is a set of standard experimental tests that produce data to ensure that the methods used in research meet the requirements so that it can be stated that the data obtained during the study are good and trustworthy results (Riyanto, 2014). Despite the Nessler method being a standard method, before this method is applied in a laboratory to determine the ammonia level in the shrimp pond wastewater, verification of the method needs to be carried out because the sample matrix of shrimp pond wastewater may be different from the sample matrix in surface water and other industrial wastewater. The parameters for verifying the Nessler method include linearity, precision, accuracy, limit of detection (LoD), and limit of quantification (LoQ).

Linearity is the ability of an analytical method to show test results directly or by mathematical transformation precisely and proportionally to the analyte concentration within a certain range. Linearity is a measure that shows the relationship between the analyte response (y) and the concentration (x) in the calibration curve, which is determined by measuring the absorbance of various concentrations of standard solutions at the maximum wavelength. The absorbance results were analyzed using a linear regression equation, and the correlation coefficient was obtained (Harmita, 2012; Juliasih *et al*., 2021).

Precision is a measure of the closeness of analytical results obtained from a series of repeated measurements of the same size. Precision can be expressed as repeatability or reproducibility. Precision criteria are given if the method provides a certain relative standard deviation (RSD) or coefficient of variation (CV), which depends on the concentration of the analyte being examined, the number of samples, and laboratory conditions (Riyanto, 2014).

Accuracy is the closeness of test results to actual values. There are three methods for determining accuracy, namely the comparison method (comparing analyte levels with a comparison standard such as Certified Reference Material (CRM), the simulation method (spiked placebo recovery method), and the standard addition method (Ravisankar *et al*., 2015). The standard addition method is often selected, especially when the placebo of the sample is unknown (Ramadhan and Musfiroh, 2021). The Limit of Detection (LoD) is the smallest amount of analyte in a sample that can be detected, which still provides a significant response compared to a blank, while the Limit of Quantification (LoQ) is the smallest quantity of analyte in a sample that can still meet the careful and thorough criteria. LoD and LoQ determination can be done in three ways: signal-to-noise method, blank determination, and calibration curve method (Riyanto, 2014).

This research will focus on verifying the Nessler method for analyzing ammonia in shrimp pond wastewater and applying it to samples from Sriminosari Village, Labuhan Maringgai District, East Lampung Regency. Adsorption tests to reduce ammonia levels were also carried out on natural zeolite from Lampung and activated zeolite using the verified Nessler method.

RESEARCH METHODS

The equipment used in this research was a set of glassware, spatulas, Whatman filter paper 0.45 µm, Marmert UN 55 oven, Wiggen Hauser JD 300-3 analytical balance, Scanning Electron Microscopy-Energy Dispersive X-Ray (SEM-EDX) Zeiss Evo MA 10 and UV-Vis Spectrophotometer Cary 100. The materials used in this research were shrimp pond wastewater, natural zeolite obtained from the Katibung area, Campang Tiga District, Lampung Regency, made in 100 mesh size, distilled water, hydrochloric acid (HCl p.a.), aluminum chloride hexahydrate ($AlCl_3.6H_2O$ p.a.) Merck, ammonium chloride (NH_4Cl p.a.) Merck, methylene blue (Merck, CAS No. 1.15943.0025) and Nessler reagent.

Procedures

Method verification

The determination of the wavelength that provides maximum absorbance was carried out by inserting a standard solution of NH_4Cl 5 ppm into a test tube to which Nessler reagent was added, and then the solution was stirred and allowed to react. The absorbance value was measured using a UV-Vis Spectrophotometer at a wavelength of 300 to 600 nm. The determination of linearity was carried out by making a calibration curve for NH_4Cl standard solutions with varying concentrations of 1, 2, 3, 4, and 5 ppm.

The accuracy was determined by adding 0.1 mL of 100 ppm standard solution into a 10 mL measuring flask, adding the sample solution to the limit mark, then repeating 8 times and determining the recovery percentage. The recovery percentage was determined using calculations as in Equation (1). Repeatability precision measurements were carried out using UV-Vis Spectrophotometry with eight repetitions. The absorbance value obtained was then determined by the concentration value, standard deviation (SD), relative standard deviation (RSD) value as in Equations (2 and 3), as well as the Horwitz %RSD value. The determination of the detection limit (Limit of Detection, LoD) and limit of quantification (LoQ) for ammonium was obtained using Equations (4, 5, and 6) (Miller and Miller, 1991; Riyanto, 2014).

$$\% \text{ Recovery} = \frac{(C1-C2)}{C3} \times 100 \quad (1)$$

C1 represents the concentration of the sample added to the target standard analyte; C2 is the concentration of sample; and C3 is the concentration of the target standard analyte.

$$\text{SD} = \sqrt{\frac{\Sigma(x-\bar{x})^2}{n-1}} \quad (2)$$

$$\text{RSD} = \frac{\text{SD}}{\bar{X}} \text{ x } 100\% \quad (3)$$

$$\text{LoD} = \frac{3 \times S_{y/x}}{\text{Slope}} \quad (4)$$

$$S_{y/x} = \left(\frac{\Sigma(y-yi)^2}{n-2}\right)^{1/2} \quad (5)$$

$$\text{LoQ} = \frac{10 \times S_{y/x}}{\text{Slope}} \quad (6)$$

SD represents the standard deviation; RSD is the relative standard deviation; x is the analyte concentration; $\bar{x}$ is the average concentration value of the analyte; y is the instrument response; n is the repetition; LoD is the limit of detection; Sy/x is the standard deviation, linear equation y = bx + a; and the slope is b in the line equation y = bx + a.

Analysis of ammonia levels in shrimp pond wastewater

Analysis of ammonia levels was carried out by taking a 5 mL sample of shrimp pond wastewater and placing it in a test tube to which Nessler reagent was added. The solution was stirred and allowed to react for 10 minutes. Next, the absorbance of the solution was measured at the maximum wavelength (425 nm).

Zeolite treatment

The natural zeolite used in this research was obtained from the Katibung area, Campang Tiga District, South Lampung Regency, Lampung Province. Natural zeolite was obtained in powder form with a size of 100 mesh. The natural zeolite was washed using distilled water and dried. The zeolite treatment in this research followed the method of Yafeng and Liu (2018). Natural zeolite was soaked in 3 M HCl solution for 10 hours, and then the zeolite was washed and dried at 110 °C. The zeolite obtained was soaked in $AlCl_3.6H_2O$ solution for 7 hours and then washed and dried at 110 °C. The resulting zeolites were then characterized using SEM-EDX, which was intended to compare natural and activated zeolites.

Surface area analysis of methylene blue adsorption method

Determination of the surface area of zeolite was done by making a 16 ppm methylene blue solution, then taking 10 mL and adding 0.05 g of natural and activated zeolites. The solution was homogenized with a shaker for

60 minutes. Then, the mixture was filtered, and the filtrate was measured with a UV-Vis Spectrophotometer at a wavelength of 664 nm.

Ammonium adsorption test in shrimp pond wastewater

This test was carried out using samples of shrimp pond wastewater originating from Sriminosari Village, Labuhan Maringgai subdistrict, East Lampung district. The adsorption test began by weighing samples of natural zeolite and activated zeolite as much as 0.5, 1, 1.5, 2, and 2.5 g and were then added to the wastewater and shaken using a shaker for 24 hours. The precipitate and filtrate formed were separated using filter paper. The ammonia remaining in the filtrate was then measured using the verified Nessler method using a UV-Vis spectrophotometer.

RESULTS AND DISCUSSION

Method Verification

Linearity

The calibration curve was measured over a 1 – 5 mg/L concentration range. This study's selection of concentration range was based on the commonly used ammonia concentration range. The results of the linearity test obtained a linear regression equation y = 0.1391x + 0.237 with a correlation coefficient (R^2) = 0.9962. This value meets the acceptance requirements based on SNI, with a correlation coefficient value ≥0.97 (SNI 06.6989.30-2005). The slope value for ammonia obtained was 0.1391. This value shows that a change in each unit of ammonia concentration results in a change as significant as the slope value. This slope value shows the sensitivity of the method. Sensitivity is the ability to differentiate between two different concentrations. The intercept value (a) obtained was 0.237. The calibration curve can be seen in Figure 1.

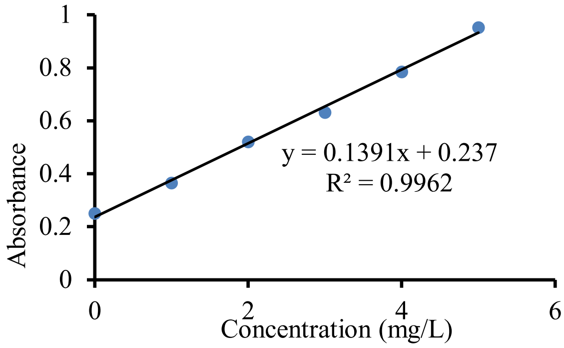

Figure 1. Calibration curve of ammonium standard solution and Nessler's reagent.

Accuracy

Accuracy is a value that shows the closeness of the measurement results obtained to the actual results. Accuracy in this study was measured by the spiking method, which adds a standard solution to the sample and is expressed as a percentage of the recovery of the added analyte, the calculation using Equation 1. The results of measuring the accuracy of the Nessler method for analyzing ammonium in shrimp pond wastewater can be seen in Table 1. The recovery percentage values range from 92.1% to 104.8%. Several error factors can affect the accuracy value, including personal errors such as pipetting and systematic errors such as the equipment or reagents used.

Table 1. Method accuracy results.

No.	C1 (mg/L)	C2 (mg/L)	C3 (mg/L)	Recovery (%)	Average Recovery (%)
1.	2.573	1.525	1.00	104.8	99.25
2.	2.567	1.528	1.00	103.9	
3.	2.559	1.531	1.00	102.8	
4.	2.541	1.519	1.00	102.2	
5.	2.491	1.520	1.00	97.1	
6.	2.489	1.533	1.00	95.6	
7.	2.479	1.528	1.00	95.1	
8.	2.448	1.527	1.00	92.1	

The standard requirements added to the sample (spike) must also have specific properties, namely having high purity, almost the same matrix as the sample, and having nearly the same solubility as the sample (Riyanto, 2014). The average % recovery value in this study was 99.25%. These results have met the requirements for accuracy acceptance, with the % recovery value requirement for analyte concentrations of 1 – 10 ppm being 80 – 110% (AOAC, 2012). The % recovery results state that the method for analyzing ammonium levels in shrimp pond wastewater has good accuracy, so the analytical method in this research can be used to test ammonia levels in the laboratory.

Precision

The results of precision measurements of ammonia using repeatability in shrimp pond wastewater samples can be seen in Table 2. The precision measurements obtained a %RSD value of 1.92%. These results have met the precision requirements, with the required % RSD value for analyte concentrations of 1–10 ppm being <7.3% (AOAC, 2012).

Table 2. Method precision results.

Repitition	Absorbance	Measurable concentration (mg/L)
1	0.5954	2.57
2	0.5944	2.56
3	0.5922	2.55
4	0.5915	2.54
5	0.5835	2.49
6	0.5829	2.48
7	0.5815	2.47
8	0.5776	2.44
Average		2.51
SD		0.04
% RSD		1.92%
0,5% RSD Horwitz		6.91%

Acceptance values for precision parameters can also be expressed by % Horwitz, indicated by a %RSD value smaller than 0.5% RSD Horwitz. Horwitz's % RSD value for shrimp pond wastewater samples was 6.91%. The results of measuring ammonia levels in shrimp pond wastewater met the acceptance requirements and showed that the method used had good repeatability and precision (Horwitz, 1995). The % RSD value is greatly influenced by the concentration of the analyte in the sample matrix, so the % RSD value obtained from a method also needs to be compared with the precision requirements.

Limit of Detection (limit of detection = LoD) and Limit of Quantification (limit of quantitation = LoQ)

The research results showed that the LoD value obtained was 0.3883 mg/L, and the LoQ value was 1.2943 mg/L. The LoD value in this study indicates that the method can only determine samples with a minimum level of 0.3883 mg/L. Sample levels with concentrations smaller than the LoD value will be reported as undetectable levels. The LoQ value in this study shows that the smallest level that can be detected with accuracy and precision is 1.2943 mg/L. Both values are the smallest limits of analytes that can still be detected by a UV-Vis Spectrophotometer (Riyanto, 2014).

Ammonia concentration in shrimp pond wastewater

The ammonia concentration in shrimp pond wastewater was obtained at 1.52 mg/L. The determination of ammonia concentration in shrimp pond wastewater was based on the calibration curve in Figure 1, with the regression equation $y = 0.1391x + 0.237$. Based on PP No. 22 of 2021, the permitted ammonia level in river water is 0.5 mg/L, while an ammonia level of 1 mg/L can cause death to aquatic organisms (Marsidi and Herlambang, 2011), so the ammonia concentration in shrimp pond wastewater needs to be lowered first before disposal into the aquatic environment. Therefore, in this research, efforts were made to reduce ammonia levels in shrimp pond wastewater using the zeolite adsorption method.

Characterization of natural zeolite and activated natural zeolite

The zeolite was characterized using Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX). The results of SEM-EDX analysis of natural zeolite and activated zeolite can be seen in Figure 2 and Table 3.

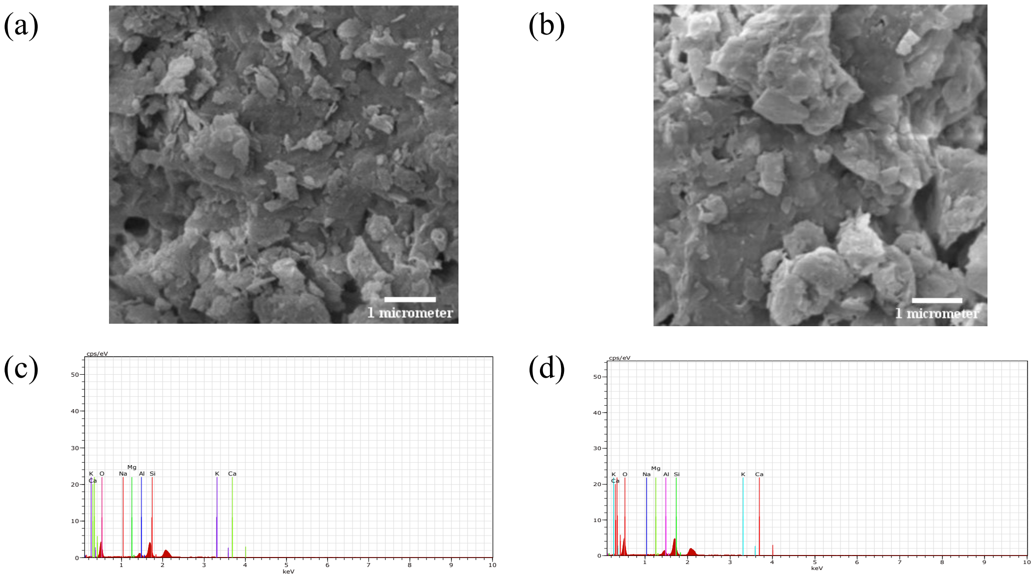

Figure 2. SEM-EDX analysis of natural zeolites (a and c) and activated zeolites (b and d).

Based on the results of SEM analysis, the surface morphology of the natural zeolite in Figure 2(a) and the activated zeolite in Figure 2(b) do not show any fundamental differences. Both natural zeolite and activated zeolite show a heterogeneous surface. However, the results of EDX analysis show that the natural zeolite without activation contains the elements K, Ca, and Mg, apart from the elements O, Si, and Al as constituents of the zeolite framework in Figure 2(c). In contrast, the activated zeolite no longer contains the elements K, Ca, and Mg Figure 2(d), caused by the activation of the zeolite. Immersing the zeolite in a 3 N HCl solution causes a de-cationization process or removal of metal cations (Prasetyo *et al.*, 2012). This metal de-cationization process causes the cations covering the zeolite surface to decrease or disappear, so the zeolite's adsorption ability increases.

Table 3. Elemental content of natural and activated zeolites.

	Elements	Atomic Number	Series	Weight %	Atomic %
Natural zeolite	O	8	K-series	52.04	65.64
	Si	14	K-series	40.06	28.79
	Al	13	K-series	6.31	4.72
	Ca	20	K-series	0.74	0.37
	K	19	K-series	0.70	0.36
	Mg	12	K-series	0.14	0.12
Total				100.00	100.00
Activated zeolite	O	8	K-series	51.31	64.75
	Si	14	K-series	41.58	29.89
	Al	13	K-series	6.84	5.12
	K	19	K-series	0.00	0.00
	Mg	12	K-series	0.00	0.00
	Ca	20	K-series	0.00	0.00
Total				100.00	100.00

Surface area analysis of methylene blue adsorption method

Surface area is an important parameter in zeolite because it is related to its adsorption capacity. The greater the surface area of the adsorbent, the more substance will be adsorbed. The surface area of the zeolite in this study was determined based on its adsorption on methylene blue. The surface area of natural zeolite obtained in this study was 3.81 m^2/g, and activated zeolite was 4.51 m^2/g. The activation process in zeolite, in this case, is the de-cationization process, causing the cations covering the surface of the zeolite to be reduced or lost; thus, the adsorption ability of the zeolite is increased (Prasetyo *et al*., 2012).

The surface area of the zeolite in this study is still lower when compared to research by Pamungkas *et al.* (2015), who carried out surface area analysis using methylene blue on Malang natural zeolite, which was modified with $SnCl_2.2H_2O$ and had been calcined at a temperature of 500 °C; The surface area of Malang natural zeolite without treatment was 11.40 m^2/g and the modified one was 11.69 m^2/g. The source of raw materials and the zeolite activation process are likely to influence the properties of the resulting zeolite product. Malang's natural zeolite is generally the modernite type, while Lampung's natural zeolite is generally the clinoptilolite type (Kalista *et al.*, 2017). Apart from that, the calcination process can also affect the surface area of the zeolite. The study conducted by Menad *et al*. (2016) showed that the calcination process up to 700 °C did not affect the surface area, micro-meso-pore volume, and final amount of zeolite. However, calcination at higher temperatures could destroy the original framework and porosity of the zeolite.

Ammonia adsorption test in shrimp pond wastewater

The results of ammonia adsorption in shrimp pond wastewater using natural zeolite and activated zeolite obtained data as in Figure 3 and Table 4.

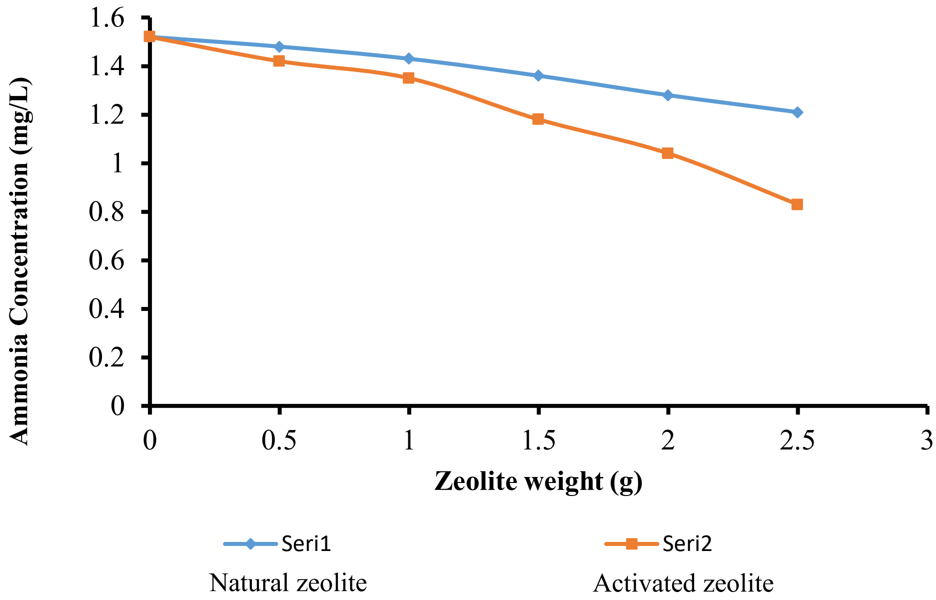

Figure 3. Adsorption curve of natural zeolite and activated natural zeolite.

Figure 3 is the adsorption curve of natural zeolite and activated zeolite. Zeolite was added to reduce the ammonia levels in shrimp pond wastewater so that it could be discharged into public waters. As previously mentioned, based on PP no. 22 of 2021, the permitted ammonia level in river water is 0.5 mg/L because 1 mg/L can cause death to aquatic organisms (Marsidi and Herlambang, 2011). The initial ammonia level in shrimp pond wastewater is 1.52 mg/L, above the ammonia level permitted to be discharged into the waters. Therefore, efforts are needed to reduce ammonia levels. The data in Table 4 shows that natural zeolite can reduce ammonia levels to 1.21 ± 0.01 mg/L or an ammonia reduction percentage of 20.30 ± 0.06%, while activated zeolite can reduce ammonia levels from 1.52 ± 0.03 mg/L to 0.83 ± 0.01 mg/L or a reduction percentage of 45.30 ± 0.02%. This is plausible because the de-cationization process occurring in activated zeolite, as shown by the analysis results with EDX, increases adsorption ability (Prasetyo *et al*., 2012).

Table 4. The decrease of ammonia concentrations.

No	Zeolite weight (g)	Ammonia concentration (mg/L)		Absorption percentage (%)	
		Natural zeolite	Activated zeolite	Natural zeolite	Activated zeolite
1.	0	1.52 ± 0.03	1.52 ± 0.02	0	0
2.	0.5	1.48 ± 0.01	1.42 ± 0.007	2.6 ± 0.04	6.5 ± 0.09
3.	1	1.43 ± 0.03	1.35 ± 0.01	5.9 ± 0.07	11.1 ± 0.09
4.	1.5	1.36 ± 0.06	1.18 ± 0.01	10.5 ± 0.07	24.3 ± 0.02
5.	2	1.28 ± 0.01	1.04 ± 0.03	15.7 ± 0.04	34.8 ± 0.08
6.	2.5	1.21 ± 0.01	0.83 ± 0.01	20.3 ± 0.06	45.3 ± 0.02

The results of this research show that the addition of 2.5 g of activated zeolite is able to reduce ammonia levels in shrimp pond wastewater two times better than natural zeolite so that the ammonia levels in shrimp pond waste become safe for aquatic organisms (ammonium levels are below 1 mg/L; (Marsidi and Herlambang, 2011); however, a greater mass of activated natural zeolite is needed to increase the ammonia absorption process so that the ammonia level can reach a value below the quality standard based on PP No. 22 of 2021. The more activated zeolite is added, the more ammonia will be absorbed. As done by Nuryoto *et al.* (2020), using 30 g/L of Bayah natural zeolite of 100 mesh without activation, with an adsorption time of 75 minutes, gave an ammonia adsorption percentage of 68%; however, the ammonia analysis was carried out using the titration method.

CONCLUSION

Nessler method verification for determining ammonia in shrimp pond wastewater has met the acceptance requirements based on SNI (Indonesian National Standard) and Associations of Analytical Communities (AOAC) references. The correlation coefficient (R^2) value in the concentration range 1 – 5 mg/L was obtained at 0.9962; the precision value expressed as %RSD was 1.92% and was smaller than 0.5 Horwitz RSD; the accuracy value obtained from the %recovery is 99.25%; limit of detection and limit of quantification of the method were 0.3883 mg/L and 1.2943 mg/L, respectively. The concentration of ammonia in the sample of shrimp pond wastewater is 1.52 mg/L; this concentration is still above the quality standard for ammonia levels in river water (PP No. 22 of 2021), with the allowable ammonia level in river water being 0.5 mg/L. Ammonia levels decreased by 45.30% after adding 2.5 g of activated zeolite, while natural zeolite only reduced ammonia levels by 20.30%. After adding 2.5 g of activated zeolite, the ammonia level is still above the quality standard. Therefore, more activated zeolite is needed to absorb ammonia in the wastewater before its disposal into public waters. Optimization of activated zeolite is also necessary to provide a better adsorption percentage.

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CONFLICT OF INTEREST

The author declares there are no conflicts of interest in this article.

AUTHOR CONTRIBUTIONS

NLGRJ: Conceptualization, Methodology, Manuscript Drafting, Manuscript Review and Editing, Supervision; AS: Conceptualization, Methodology, Manuscript Drafting, Manuscript Review and Editing, Supervision; LMR: Formal Analysis, Manuscript Drafting; MR: Investigation, Manuscript Drafting, Manuscript Review and Editing, Supervision; RR: Investigation, Supervision; FK: Investigation, Manuscript Review and Editing; AAK: Investigation, Manuscript Review and Editing, Supervision.

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