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Utilizing Natural Zeolites in Sapric Soils for Mitigating Nitrous Oxide Emissions and Improving MD2 Pineapple Productivity

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ABSTRACT

Draining of organic soils and fertilization activities increases nitrogen mineralization, causing higher nitrous oxide (N_2O) emission and raising concerns on managing pineapple cultivation sustainably in drained peat soils. Natural clinoptilolite zeolite was utilized in the pineapple fertilization regime to assess this mineral's ability to mitigate N_2O emission and improve MD2 yields planted on sapric soils. Soil N_2O emissions were monitored for two pineapple seasons utilizing the static closed chamber according to these treatments: (a) four ratios of clinoptilolite at 25, 60, 75, and 100% incorporated with compound NPK (30:1:32) fertilizer, (b) conventional pineapple fertilization (NPK 30:1:32), and (c) non-fertilized soil. Clinoptilolite fertilization on sapric soils minimized N_2O emissions (1.41 to 1.85 kg N_2O ha^{-1} $year^{-1}$) relative to conventional pineapple fertilization (2.05 to 2.44 kg N_2O ha^{-1} $year^{-1}$) throughout the planting seasons. The adsorption of ammonium and nitrate ions onto the clinoptilolite framework via ion exchange and mass transport inhibited microbial nitrification and denitrification processes. Amending NPK fertilizers with clinoptilolite also reduced peat acidity (pH:4.35–4.81) and increased soil ammonium and nitrate availability because of the sorption of these macronutrients onto clinoptilolite's lattices, thereby improving pineapple N use efficiency and increased MD2 yield (92.0 to 101.1 t ha^{-1}) and fruit weight (2.42 to 2.66 kg/fruit) compared with conventional agronomic practices (57.0 to 66.9 t ha^{-1} or 1.5 to 1.76 kg/fruit). The recommended clinoptilolite fertilization (25% zeolite + NPK 30:1:32) at 3-, 6-, and 9-months pineapple age provides agronomic options for pineapple growers to mitigate N_2O emissions besides enhancing pineapple productivity.

Keywords: Clinoptilolite zeolite, greenhouse gases, histosols, pineapple, soil amendment

INTRODUCTION

In their innate ecosystems, tropical peat soils are a negligible source of N_2O but draining these organic soils for agriculture increases the transformation of organic nitrogen (N) to ammonium (NH_4^+) and nitrate (NO_3^-) ions, causing the emission of N_2O into the atmosphere (Lee *et al.,* 2017; Guo *et al.,* 2019). The United Nations Framework Convention on Climate Change (UNFCCC) requires signatory countries under the 2015 Paris Agreement and 1997 Kyoto Protocol to report annual N_2O emission from their agriculture sector because of the ozone depleting properties of N_2O and its hazardous effect on global warming. Microbial nitrification and denitrification processes are the main drivers of N_2O emission from drained peat soils and these N_2O emissions are regulated by fertilization, nitrogen (N) availability, soil pH, water content, temperature, and microbial community structure (Saggar *et al.,* 2013; Oktarita *et al.,* 2017). Synthetic fertilizer use has increased annually to meet the growing demands for food required by a rapidly expanding population. In 2025, Malaysia's N_2O emission from fertilizer usage (urea) was estimated at 394.47 Gg CO_2 eq., whereas direct and indirect N_2O emissions from managed soils was 2,744.06 Gg CO_2 eq. (NRES, 2025). Pineapples grown on drained peat soils in Malaysia emit 15.7 t N_2O ha^{-1} annually (Choo *et al.,* 2021) and these N_2O

emissions from the pineapple industry is expected to increase with the expansion of MD2, Sarawak Gold and MS16 cultivated areas for export and local consumption. Pineapples in Malaysia is predominantly planted on peat soils with an estimated acreage of 16,450 ha in 2024 (DOA, 2024). Compared with the extensive greenhouse gas (GHG) emission monitoring on oil palm as the primary plantation crop, minimal effort had been made to measure N_2O emissions from the pineapple industry. Moreover, there is insubstantial mitigation measures to reduce GHG emission and clarify the effects of fertilization on N_2O emission from pineapple planted on histosols. Substantial amount of N, P, and K fertilizers are needed for the optimal growth of pineapples from the vegetative phase, flowering, and fruit development until harvest (Malézieux and Bartholomew, 2003; Khuong *et al.,* 2024). Therefore, it is vital to comprehend the contribution of N_2O emissions from the pineapple industry to sustainably manage agricultural peatlands for ensuring agricultural productivity and quality as outlined in Malaysia's National Agrifood Policy (2021–2030).

Natural zeolite of the clinoptilolite species is applied as a soil amendment for improving soil physico-chemical properties and enhancing crop productivity (Cataldo *et al.,* 2021; Javaid *et al.,* 2024), particularly on problematic soils. Clinoptilolite is a sedimentary deposit which occur at volcanic areas and is composed of aluminosilicates frameworks with open cavities that are interlinked and attached with exchangeable cations namely Na, K, Ca, Ba, Sr, Fe, and Mg ions (Wahono *et al.,* 2020). These porous cavities of the clinoptilolite permits the exchange and adsorption of nutrients and molecules within its framework. The types of nutrients or molecules adsorb by clinoptilolite is regulated by its framework size and this zeolite species is thermally stable to above 600 °C and do not easily disintegrate in the soil system (Mansouri *et al.,* 2013; Nur Aainaa *et al.,* 2018; Wahono *et al.,* 2020), thus allowing this mineral to serve as a soil amendment. However, there are inadequate details on utilizing clinoptilolite as a GHG mitigation amendment to minimize N_2O emitted from acidic peat soils. Because clinoptilolite is alkaline, its application may increase N_2O emission by reducing soil acidity and improving peat organic matter decomposition. Conversely, clinoptilolite may decrease N_2O emission via the adsorption of N_2O molecules, ammonium ions or organic nitrogen onto its framework. To this end, the aim of the present study was to determine the potential of incorporating clinoptilolite in the pineapple fertilization regime for minimizing N_2O emissions and enhancing pineapple productivity on acidic peat soils cultivated with MD2 pineapples. For this, field studies with two pineapple seasons employing the static closed chamber method was utilized to ascertain the impact of clinoptilolite fertilization on N_2O emission and pineapple yield on sapric peat soils. Results from the study may provide mitigation strategies to reduce N_2O emissions on drained cultivated organic soils.

MATERIALS AND METHODS

Physiography of the study area

The assessment of clinoptilolite to mitigate N_2O emission in pineapple cultivation on drained sapric soils was conducted on drained histosols located at Saratok in the region of Sarawak, Malaysia (latitude 1° 55' 05" N and longitude 111° 14'09" E). The site receives high annual rainfall (3923 mm) with a recorded annual mean temperature of 27.7 °C. The histosols at Saratok is identified as highly degraded and deep sapric soils (2.5 to 3 m depth and von Post class: H7 to H9) with indiscernible plant structure. The sapric soils is acidic (mean pH of 3.9), contained high cation exchange capacity (CEC: 143.2 $cmol_{(+)}\ kg^{-1}$), total organic carbon (41.8%), and total nitrogen (1.39%), whereas available phosphorus (PO_4^{3-}: mean 22.5 $mg\ kg^{-1}$), and exchangeable potassium (K^+: mean 85.7 $mg\ kg^{-1}$) were reported to be low.

Specific traits of clinoptilolite zeolite

Clinoptilolite zeolite in powder form (Figure 1a) was utilized in this study and the mineral was commercially imported from West Java, Indonesia. The clinoptilolite is alkaline (pH 8.77) and has a high CEC (103.8 $cmol_{(+)}$ kg^{-1}). The clinoptilolite is composed of Si (78.08%), Al (7.03%), K (1.29%), Ca (1.27%), Mg (1.10%), and Na (0.53%) oxides. This mineral's Si:Al ratio is 11.1 whereas its surface area is approximately 23.19 m^2 g^{-1}. From the Scanning Electron Microscope (SEM) micrographs in Figures 1b and c, the clinoptilolite zeolite is crystalline with irregular shaped particles.

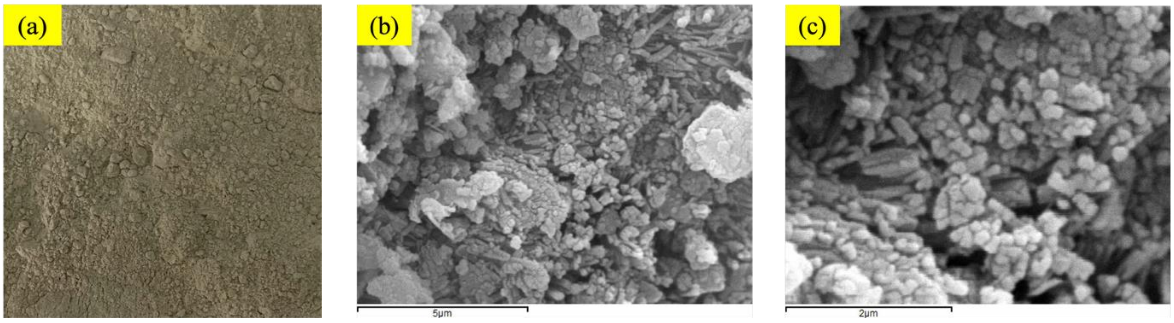


Figure 1. Clinoptilolite zeolite in powder form **(a)** and Scanning Electron Microscope (SEM) micrographs of clinoptilolite at different magnification **(b and c)**

Pineapple planting and clinoptilolite fertilization treatments

MD2 pineapple was cultivated for two seasons at Saratok (first cycle: January 2023 to January 2024 and second cycle: December 2023 to December 2024). The MD2 pineapple planting utilized the 6 x 3 x 3 factorial experiment in a randomized complete block design with four replications comprising (i) six recommended pineapple fertilization ratios including controls (**Table 1**); (ii) soil N_2O monitoring period (30 days after pineapple fertilization at three-, six-, and nine-months following pineapple planting); and (iii) soil N_2O flux sampling period (morning: 9 a.m., noon: 12 p.m., and afternoon: 3 p.m.). MD2 pineapple suckers (mean height of 30 cm) were planted on sapric soils in two rows and the spacing between pineapple plants were 30 cm, whereas spacing between rows were 60 cm. For each pineapple season, there were a total of 2400 MD2 pineapple plants with each treatment consisting of 400 plants. The size of each experimental block was 15.7 m (length) x 8.6 m (width). To prevent inter-plot fertilizer residual contamination and to remove excessive rainwater during heavy rainfall, narrow trenches (width: 0.3 m and depth: 0.5 m) were erected between treatments blocks. The agronomic management for the MD2 pineapples follows the standard procedures for pineapple cultivation on tropical peat soils (Mohammed Selamat, 1996). Treatments (**Table 1**) were applied to the pineapple plants via surface application, approximately 5 cm from the base of the pineapple stems. For each pineapple season, clinoptilolite zeolite and NPK fertilizers were applied thrice every three months after planting (three-, sixth-, and ninth-months age). Throughout the pineapple planting season, water table at the study area fluctuated between 41 and 56 cm.

Table 1. Clinoptilolite fertilization treatments for MD2 pineapple cultivated on drained organic sapric soils

Treatment code	Description (application per plant)
Z1	25% clinoptilolite (5 g) + 20 g NPK 30:1:32
Z2	60% clinoptilolite (12 g) + 20 g NPK 30:1:32
Z3	75% clinoptilolite (15 g) + 20 g NPK 30:1:32
Z4	100% clinoptilolite (20 g) + 20 g NPK 30:1:32
NP	Conventional pineapple fertilization on drained peat soils (20 g NPK 30:1:32) – positive control
NC	Non-treated soil (negative control)

Note: Clinoptilolite treatments per plant were calculated according to the macronutrient requirement of MD2 pineapple plants planted on drained peat soils

Soil nitrous oxide monitoring

Sapric soil N_2O emitted from MD2 cultivation were monitored using a cylinder-shaped static closed chamber (IAEA, 1992; Widén and Lindroth, 2003), which was inserted between rows of MD2 plants. These gas chambers were made from stainless steel with a dimension of 0.2 m (height) x 0.1 m (diameter), taking into consideration the MD2 pineapple planting distance. The gas chambers were fitted with thermometers (to record chamber temperature), cooling fan (to ensure a balance gas pressure within the chamber), and silicone septas (to collect gas samples). Prior to N_2O sampling, 24 cylinder-shaped soil collars were inserted into the soil (up to 6 cm depth because organic matter degradation processes transpire actively at this horizon) (Jauhiainen *et al.,* 2012), after which gas chambers were position onto the soil collars. Gas samples (20 mL) from the chambers were obtained every minute for six minutes using a polypropylene syringe and transferred into vacuumed glass vials. Thereafter, N_2O flux were analysed using an Agilent 7890A gas chromatography, which uses stainless column and fitted with flame ionization and thermal conductivity detectors. Soil N_2O samples were collected at three-hours interval in the morning (9 a.m.), at noon (12 p.m.) and in the afternoon (3 p.m.) and measured at thirty days after fertilization. For each pineapple season, N_2O fluxes were monitored at the third-, sixth-, and ninth months of pineapple growth. Soil N_2O emitted from the gas chambers were quantified based on the accumulation of N_2O fluxes with time, chamber volume, and soil area enclosed by the gas chambers (IAEA, 1992). Linear regression analysis (N_2O concentration versus time) was used to compute soil N_2O emitted from MD2 pineapple cultivation and N_2O fluxes were expressed as kg ha^{-1} $year^{-1}$. Soil temperature and water table were measured, respectively using a soil temperature sensor (Eijkelkamp 1P68) and portable water level meter (Eijkelkamp) at the same time of the N_2O flux measurement.

Sapric soil sampling, MD2 pineapple yield attributes, and crop efficiency of applied clinoptilolite zeolite

For each pineapple season, soil samples were collected thirty days after MD2 pineapple fertilization at the vegetative (three and sixth months of plant growth) and flower induction (nine months growth) phases. Soil samples in each treatment blocks were sampled at 10 points utilizing the zigzag method at two depths (0-20 cm and 20-40 cm), taking into consideration the pineapple root zones above the water table. There was a total of 120 samples for each experimental block. Soil samples were determined for pH (1:5 soil to water ratio using a pH meter: Eutech Cyberscan pH 1500) and exchangeable ammonium (NH_4^+–N) and available nitrate (NO_3^-–N), which were analysed using the steam distillation method (Bremner and Keeney, 1966).

For every planting season, MD2 pineapples were harvested after 12 months of growth (maturity index 1) in January and December 2024, respectively for the first and second planting season. At harvest, the MD2 plants were partitioned into stems, roots, and leaves for the determination of total N (Miller, 1998) and the uptake of N by MD2 pineapples was determined by multiplying the total N concentrations with pineapple dry matter. The N use efficiency of MD2 pineapples were calculated according to the method outlined by Dobermann (2005). Fresh fruit weight of the MD2 pineapple was recorded using an electronic weighing scale (ACS-30-JC21G Camry). MD2 pineapple fruit yield was computed according to the average fresh fruit weight and planting density of the pineapples (Khuong *et al.,* 2024). MD2 pineapple fruits with a maturity index of 5, indicating 50% fruit ripeness were selected and used for the determination of total soluble solids and fruit juice pH (Siti Rashima *et al.,* 2019). A refractometer (Atago PAL-1) was utilized to measure total soluble solids (TSS) of the pineapple fruits, whereas sodium hydroxide solution (0.1 N) was used to determine titratable acidity of the MD2 pineapple fruits (Hajar *et al.,* 2012).

Statistical data analysis

Data obtained from the field N_2O monitoring throughout the two pineapple seasons were analysed using the Statistical Analysis Version 9.1 software. The effects of the addition of clinoptilolite zeolite in MD2 pineapple fertilization regime on soil N_2O emission, soil macronutrients availability, MD2 pineapple yield and fruit quality were determined using analysis of variance (ANOVA). Tukey's New Multiple Range Test procedure at $P \leq 0.05$ was used to acquire treatments means diverging from the control treatments. The evaluation of soil N_2O emission throughout the two planting seasons were executed using repeated measures ANOVA utilizing the linear mixed effect model procedure by taking into consideration sampling time and N_2O flux monitoring as random effects, whereas ratios of clinoptilolite fertilization as fixed effects. The relationship between soil N_2O emission, soil temperature, and water table were analysed using Pearson correlation analysis.

RESULTS AND DISCUSSION

Influence of clinoptilolite fertilization on peat soil nitrous oxide emission in MD2 pineapple cultivation

Throughout the MD2 pineapple growth and development, the addition of clinoptilolite in the pineapple fertilization regime at three, six, and nine months of plant growth influenced soil N_2O emissions. Regardless of pineapple growth phases, acidic sapric soils amended with clinoptilolite zeolite (Z1 to Z4) minimized N_2O emission relative to the control (NP: standard compound NPK 30:1:32 fertilization for pineapple on peat soils) (**Figure 2**). However, N_2O emissions were similar irrespective of the amount of clinoptilolite applied (**Figure 2**). The significant N_2O emission reduction for zeolite treated sapric soils were consistent for all two MD2 pineapple planting cycles from January 2023 to December 2024. Similarly, the average N_2O emissions were lower in all the treatments with clinoptilolite zeolite (Z1 to Z4: 1.41 to 1.85 kg N_2O ha year^{-1}) compared with NPK fertilization (NP) (2.05 to 2.44 kg N_2O ha year^{-1}) for both pineapple planting cycles (**Figure 3**). Nevertheless, soil N_2O emissions from unfertilized sapric soils (negative control: NC) were similar to those of zeolite amended soils (Z1 to Z4) but N_2O emissions remained lower compared with NPK fertilization (NP). This observation occurred throughout the MD2 pineapple growth period (**Figures 2** and **3**) for two planting seasons. The effectiveness of all the clinoptilolite zeolite treatments (Z1 to Z4) in minimizing N_2O emissions after fertilization is attributed to the temporary adsorption of NH_4^+ ion within the lattices of the clinoptilolite zeolite via ion exchange (Jakkula and Wani, 2017), which protected NH_4^+ ions from microbial nitrification. This relates to clinoptilolite zeolite's high CEC (103.8 cmol$_{(+)}$ kg^{-1}), which permitted the mobilization and exchange of NH_4^+ ions within the clinoptilolite's framework. Furthermore, these observations concurred with the higher soil exchangeable NH_4^+–N content with the clinoptilolite zeolite treatments (Z1 to Z4) compared with the controls throughout the pineapple growth phases (**Table 2**). In addition, the lower N_2O emissions from clinoptilolite amended sapric soils (Z1 to Z4) could also be ascribed to their higher soil pH (ranging from pH 4.35 to 4.86) (Table 2) compared with NPK fertilization (pH 3.47 to 3.99). This is because N_2O:N_2 product ratio of denitrification processes decreases with high pH (Saggar *et al.,* 2013; Liimatainen *et al.,* 2014). Nonetheless, the reason N_2O emissions were similar irrespective of clinoptilolite treatments (Z1 to Z4) throughout the plant growth phases for two planting cycles is unclear but it is possible that these anomalies could be associated with the optimal and equilibrium sorption of NH_4^+ and NO_3^- ions, N_2O molecules, and nutrients onto and within the clinoptilolite's framework. On the contrary, the higher N_2O emissions in the control treatment with NPK fertilization (NP) could be associated with the adequate availability of NH_4^+–N and NO_3^-–N for nitrifying microorganisms that may have affected nitrification and denitrification processes in the rhizosphere (Saggar *et al.,* 2013). Compared with NPK fertilization (NP), the N_2O emissions from unfertilized sapric soils (NC) were lower because denitrification processes transpire slowly under acidic peat conditions (Saggar *et al.,* 2013). Moreover, this finding is agreement with the acidic pH of the control treatment (NC: unfertilized peat soils) that remained acidic (pH 3.43 to 3.84) throughout the growth and development of MD2 pineapples (**Table 2**).

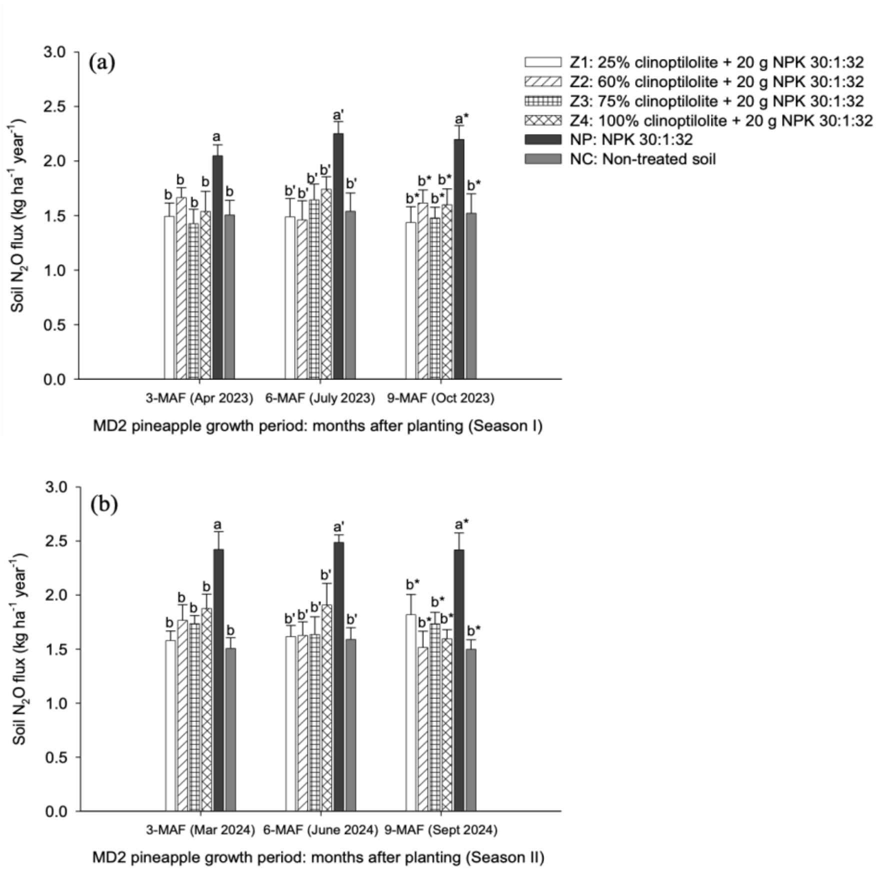


Figure 2. Soil N_2O emissions in MD2 pineapple cultivation on sapric soils amended with clinoptilolite zeolite and compound NPK fertilizer at different growth phases for two planting seasons: **(a)** First season: January 2023 to January 2024 and **(b)** Second season: December 2023 to December 2024. Error bars indicate standard error of the mean and means with different alphabets are significantly different using Tukey's test with $P \leq 0.05$. MAF: months after pineapple planting.

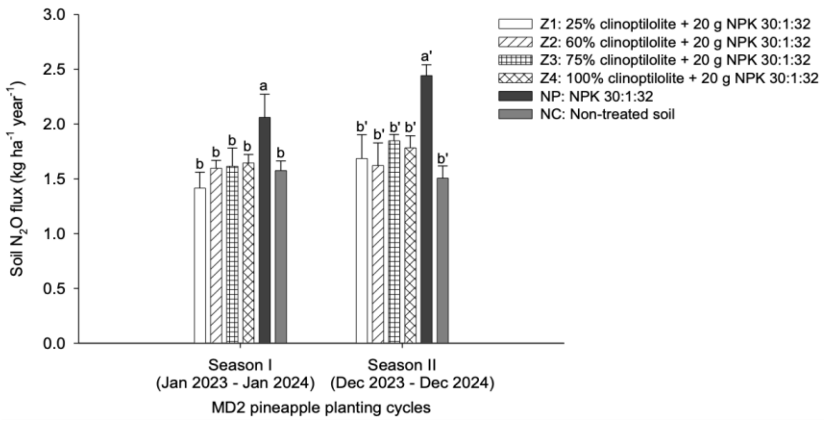


Figure 3. Mean soil N_2O emissions throughout the MD2 planting seasons in drained sapric soils amended with clinoptilolite zeolite and compound NPK 30:1:32 fertilizer. Error bars indicate standard error of the mean and means with different alphabets are significantly different using Tukey's test with P ≤ 0.05.

Table 2. Mean soil ammonium-N, nitrate-N, and pH of MD2 pineapples planted on drained sapric soils treated with clinoptilolite zeolite and NPK fertilizers for two planting seasons

Treatment code	Season I (Jan 2023 – Jan 2024)			Season II (Dec 2023 – Dec 2024)		
	Soil nutrients (mg kg^{-1})		Soil pH	Soil nutrients (mg kg^{-1})		Soil pH
	NH_4^+–N	NO_3^-–N		NH_4^+–N	NO_3^-–N	
Z1	476.32^b ± 7.07	208.55^b ± 4.86	4.51^a ± 0.10	285.10^b ± 8.67	168.66^a ± 3.99	4.65^a ± 0.10
Z2	727.42^a ± 8.81	271.51^a ± 9.46	4.35^a ± 0.04	255.34^b ± 5.25	179.41^a ± 5.74	4.81^a ± 0.26
Z3	497.40^b ± 8.97	243.15^{ab} ± 5.73	4.86^a ± 0.05	295.41^b ± 7.27	156.76^a ± 4.08	4.66^a ± 0.12
Z4	497.63^b ± 7.73	200.47^b ± 5.69	4.52^a ± 0.01	395.24^a ± 7.38	161.63^a ± 3.13	4.68^a ± 0.11
NP	355.70^c ± 9.51	101.31^c ± 3.08	3.47^b ± 0.01	129.96^c ± 5.30	70.62^b ± 3.78	3.99^b ± 0.06
NC	150.82^d ± 5.02	89.10^c ± 2.36	3.43^b ± 0.02	103.77^c ± 6.08	54.11b ± 2.89	3.84^b ± 0.02

Note: Mean values (± standard error) with different alphabets within the same column are significantly different by Tukey's test at P ≤ 0.05.

Soil N_2O emission was not affected by time of flux sampling (morning, noon, and afternoon) irrespective of treatments throughout the MD2 planting cycles and growth phases (**Figure 4**). Soil N_2O emissions with the clinoptilolite zeolite treatments (Z1 to Z4) and unfertilized soils (NC) remained lower compared with NPK fertilization (NP). Likewise, averaged soil N_2O emissions (inclusive of all treatments: Z1 to Z4 and controls: NP and NC) were similar regardless of flux measurement time and MD2 pineapple growth phases (**Figure 5**). These findings were unexpected although these abnormalities suggested that N_2O emission is regulated by microbial nitrification and denitrification processes (Oktarita *et al.,* 2017; Saggar *et al.,* 2013), diversity and structure of microorganisms at the root zone of the pineapple plants (Mäkiranta *et al.,* 2009), and mobilization of soil NH_4^+–N and NO_3^-–N ions for plant uptake, which is influenced by peat preferential flow (Liu and Lennartz, 2019) in acidic sapric soils.

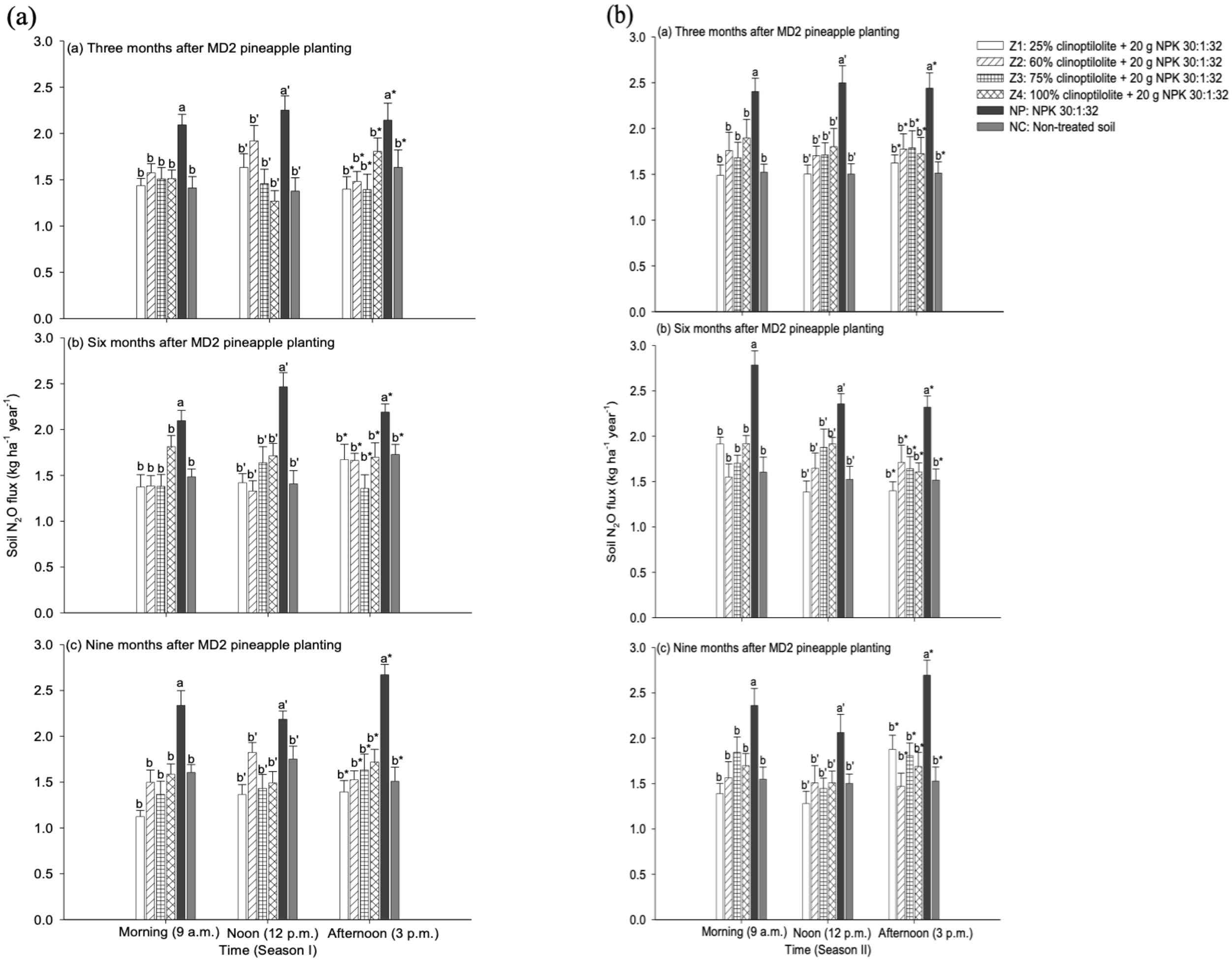


Figure 4. Soil N_2O emissions in MD2 pineapple cultivation on drained sapric soils amended with clinoptilolite zeolite and NPK fertilizer at differing times throughout the two planting cycles: **(a)** first season: January 2023 to January 2024 and **(b)** second season: December 2023 to December 2024. Error bars indicate standard error of the mean and means with different alphabets are significantly different using Tukey's test with $P \leq 0.05$.

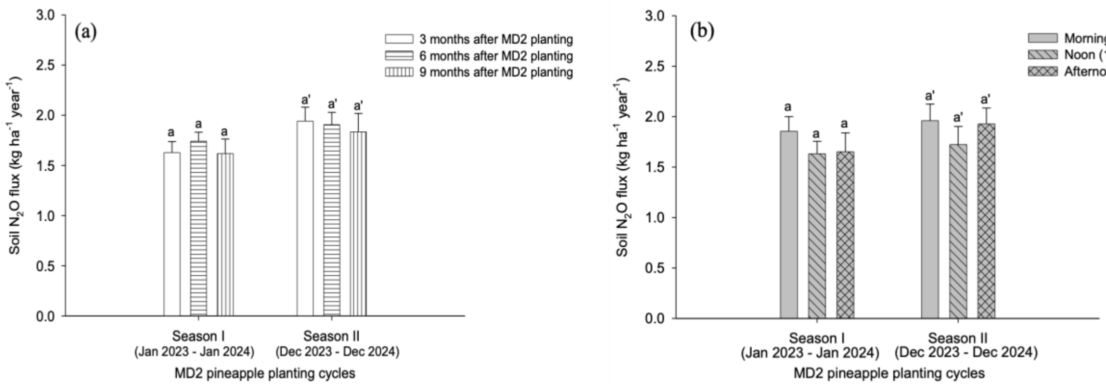


Figure 5. Average soil N_2O emissions (inclusive of all clinoptilolite treatments including controls) at **(a)** different growth phases and **(b)** differing times from drained sapric soils throughout the MD2 pineapple planting seasons. Error bars indicate standard error of the mean and means with the same alphabets are not significantly different using Tukey's test with $P \leq 0.05$.

Throughout the growth and development of the MD2 planting seasons (January 2023 to December 2024), mean soil temperature varied with the time of N_2O sampling, but the opposite was true for water table (**Table 3**). In this present study, no significant correlation was observed between peat soil N_2O emission, soil temperature, and water table (**Figure 6**). Although N_2O emission is regulated by soil temperature because peat soil organic matter decomposition increases with increasing temperature (Jauhiainen *et al.,* 2014; Girkin *et al.,* 2020), the findings from this present study demonstrates that soil temperature had minimal effect on N_2O emission from drained tropical peat soils because of the moderate temperature fluctuations in the tropics (Jauhiainen *et al,* 2012; Oktarita *et al.,* 2017). The drainage system for the sapric farmlands at the research plot in Saratok was designed to provide proper water table control at the farm level. The water table was controlled to fluctuate between 40 and 60 cm from the soil surface to minimize peat subsidence. This likely explains the insignificant correlation between N_2O emission and water table because of the extent of the aerobic zone in the soil top horizon, which decreased anaerobic soil conditions that generally favours N_2O emission through nitrifying bacteria which utilize nitrate ions for their metabolic processes (Oktarita *et al.,* 2017).

Table 3. Mean soil temperature and water table during N_2O flux sampling for MD2 pineapple cultivation on drained sapric soils (two planting seasons)

Time of N_2O flux sampling	Soil temperature (°C)		Water table depth (cm)	
	Season I	Season II	Season I	Season II
Morning (9 a.m.)	$29.0^{b} \pm 0.06$	$29.0^{b} \pm 0.23$	$49.2^{a} \pm 0.20$	$51.2^{a} \pm 0.24$
Noon (12 p.m.)	$31.3^{ab} \pm 0.11$	$30.7^{ab} \pm 0.13$	$49.6^{a} \pm 0.10$	$52.0^{a} \pm 0.34$
Afternoon (3 p.m.)	$32.1^{a} \pm 0.12$	$32.0^{a} \pm 0.15$	$49.0^{a} \pm 0.23$	$51.7^{a} \pm 0.25$

Note: Different alphabets within the same column indicate significant differences between means using Tukey's test with P ≤ 0.05.

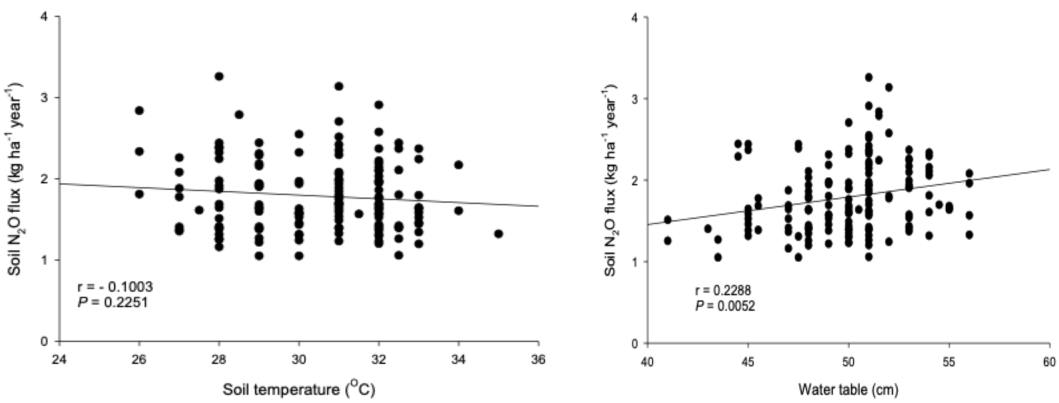


Figure 6. Relationship between soil N_2O emission, soil temperature and water table for MD2 pineapples planted on drained sapric soils throughout two planting seasons (January 2023 to December 2024). The *r* values indicate Pearson's correlation coefficient whereas the *P* values denote the probability level at 0.05.

Effect of clinoptilolite zeolite on sapric peat soil properties

Clinoptilolite zeolite fertilization in MD2 pineapple cultivation on drained sapric soils significantly affected soil pH, and NH_4^+–N and NO_3^-–N availability. Regardless of plant growth phases, mean soil pH, NH_4^+–N, and NO_3^-–N of the treatments with clinoptilolite (Z1 to Z4) were higher compared with those of the controls (NPK fertilization: NP and unfertilized soils: NC) throughout the MD2 pineapple planting seasons (**Tables 4** to **6**). However, the availability of these nutrients varied with the amount of clinoptilolite zeolite utilized and pineapple growth phases. The higher soil pH in the treatments with clinoptilolite relates to the buffering capacity and gradual build-up of this zeolite mineral, particularly at the aerobic soil layer that leads to an increase in soil pH. The significant retention of exchangeable NH_4^+–N and available NO_3^-–N for sapric soils amended with clinoptilolite zeolite substantiates the ability of this clinoptilolite mineral to adsorb NH_4^+ and NO_3^- ions via ion exchange and mass transport. Furthermore, the significant retention of exchangeable NH_4^+–N and available NO_3^-–

N is consistent with the increase in the peat soil pH because organic nitrogen mineralization increases with increasing pH (Omar *et al.,* 2015). Moreover, the affinity of clinoptilolite zeolite to adsorb and exchange nutrients increases with increasing soil pH (Dogan *et al.,* 2003). The differences in exchangeable NH_4^+–N and available NO_3^-–N retention following clinoptilolite zeolite fertilization (Z1 to Z4) throughout the pineapple growth phases and planting seasons could be associated with the competitive interactions between nutrients and gas molecules for adsorption on the exchange sites of the zeolite framework. In addition, the availability of exchangeable NH_4^+–N and available NO_3^-–N in the sapric soil might have been influenced by the movements of these nutrients via mass flow into the pineapple rooting zone, which is influenced by the preferential flow of the peat soil under heavy and low rainfall conditions throughout the pineapple growth period and planting seasons (Dotaniya and Meena, 2015; Liu and Lennartz, 2019). In this study, the acidic conditions of the sapric soils with the control treatments (NP: NPK fertilization and NC: unfertilized soils) might have impeded organic nitrogen mineralization and this explains the low concentration of exchangeable NH_4^+ and NO_3^- ions in the control treatments.

The significant increase in soil NH_4^+–N and NO_3^-–N buttresses the ability of clinoptilolite to adsorb NH_4^+ ions and NO_3^- onto its framework via ion exchange and mass transport, thus protecting NH_4^+ from microbial nitrification and denitrification. Furthermore, the significant increase in soil pH (**Table 4**) for clinoptilolite zeolite treatments (Z1 to Z4: pH 4.29 to 5.33) compared with the controls (NP and NC: pH 3.44 to 4.01) substantiate that denitrification processes decrease with increasing pH (Saggar *et al.,* 2013; Liimatainen *et al.,* 2014).

Table 4. Mean soil pH of a drained sapric soils throughout the growth phases of the MD2 pineapple plants treated with clinoptilolite zeolite and NPK fertilizers for two seasons

Treatment code	Soil pH (Mean overall at 0 – 20 cm and 20 – 40 cm depths)					
	Season I (Pineapple growth period)			Season II (Pineapple growth period)		
	3 months	6 months	9 months	3 months	6 months	9 months
Z1	$4.32^a \pm 0.08$	$4.66^a \pm 0.19$	$4.57^a \pm 0.06$	$4.76^b \pm 0.18$	$4.46^a \pm 0.14$	$4.72^a \pm 0.04$
Z2	$4.29^a \pm 0.02$	$4.35^a \pm 0.02$	$4.42^a \pm 0.03$	$5.33^a \pm 0.29$	$4.60^a \pm 0.16$	$4.49^a \pm 0.02$
Z3	$4.89^a \pm 0.03$	$4.77^a \pm 0.01$	$4.92^a \pm 0.05$	$4.53^b \pm 0.05$	$4.55^a \pm 0.20$	$4.90^a \pm 0.04$
Z4	$4.50^a \pm 0.01$	$4.51^a \pm 0.09$	$4.51^a \pm 0.01$	$4.89^b \pm 0.22$	$4.54^a \pm 0.09$	$4.62^a \pm 0.05$
NP	$3.49^b \pm 0.03$	$3.46^b \pm 0.02$	$3.47^b \pm 0.02$	$4.01^c \pm 0.07$	$3.97^b \pm 0.01$	$3.92^b \pm 0.12$
NC	$3.47^b \pm 0.03$	$3.39^b \pm 0.01$	$3.44^b \pm 0.02$	$3.82^c \pm 0.05$	$3.81^b \pm 0.02$	$3.88^b \pm 0.04$

Note: Means (value ± standard error) with different alphabets within the same column are significantly different by Tukey's test at $P \leq 0.05$.

Table 5. Mean soil ammonium-N of a drained sapric soils throughout the growth phases of the MD2 pineapple plants treated with clinoptilolite zeolite and NPK fertilizers for two seasons

Treatment code	Soil NH_4^+–N (Mean overall at 0 – 20 cm and 20 – 40 cm depths) (mg kg^{-1})					
	Season I (Pineapple growth period)			Season II (Pineapple growth period)		
	3 months	6 months	9 months	3 months	6 months	9 months
Z1	$930.51^b \pm 8.17$	$476.46^a \pm 5.36$	$312.84^{bc} \pm 8.29$	$357.44^c \pm 5.73$	$134.60^b \pm 5.18$	$420.44^a \pm 9.37$
Z2	$1507.55^a \pm 10.33$	$369.78^b \pm 4.52$	$404.42^a \pm 7.99$	$290.77^c \pm 8.03$	$158.23^b \pm 4.93$	$332.44^b \pm 7.18$
Z3	$681.61^b \pm 7.04$	$394.17^b \pm 6.04$	$281.98^c \pm 6.05$	$428.74^b \pm 4.29$	$226.31^a \pm 7.71$	$297.83^b \pm 5.16$
Z4	$650.34^b \pm 5.99$	$440.47^a \pm 7.15$	$347.36^b \pm 5.74$	$731.65^a \pm 8.81$	$130.38^b \pm 5.39$	$456.12^a \pm 6.25$
NP	$525.36^b \pm 9.18$	$196.00^c \pm 3.03$	$200.72^d \pm 6.38$	$176.67^d \pm 4.63$	$89.78^c \pm 4.04$	$123.42^c \pm 5.01$
NC	$152.11^c \pm 6.93$	$127.07^c \pm 2.81$	$184.50^d \pm 5.19$	$43.71^e \pm 3.58$	$50.09^c \pm 5.23$	$49.77^d \pm 4.81$

Note: Means (value ± standard error) with different alphabets within the same column are significantly different by Tukey's test at $P \leq 0.05$.

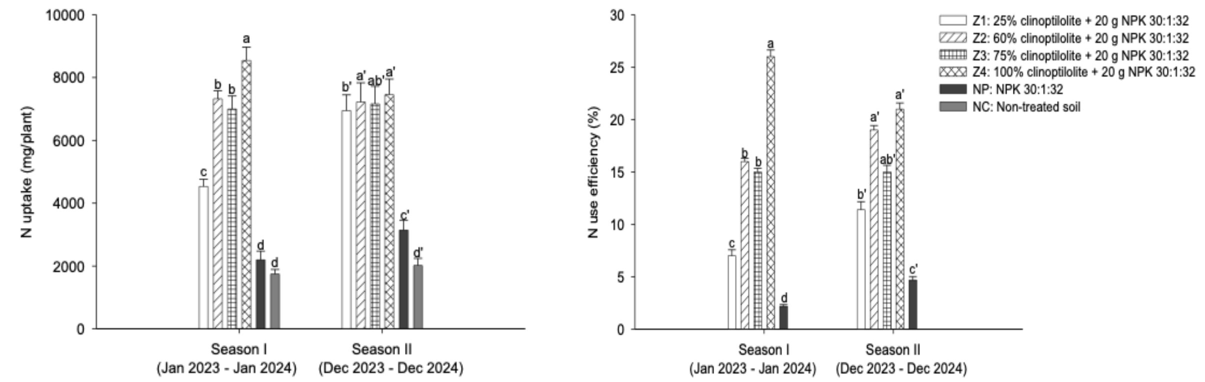
Table 6. Mean soil nitrate-N of a drained sapric soils throughout the growth phases of the MD2 pineapple plants treated with clinoptilolite zeolite and NPK fertilizers for two seasons

Treatment code	Soil NO_3^-–N (Mean overall at 0 – 20 cm and 20 – 40 cm depths) (mg kg^{-1})					
	Season I (Pineapple growth period)			Season II (Pineapple growth period)		
	3 months	6 months	9 months	3 months	6 months	9 months
Z1	$363.01^{b} \pm 3.85$	$149.40^{b} \pm 3.48$	$113.24^{ab} \pm 2.85$	$196.55^{bc} \pm 3.71$	$103.40^{a} \pm 4.49$	$268.90^{a} \pm 5.39$
Z2	$538.29^{a} \pm 9.04$	$228.96^{a} \pm 4.93$	$107.83^{b} \pm 3.13$	$232.07^{a} \pm 5.08$	$105.27^{a} \pm 3.22$	$144.58^{b} \pm 4.43$
Z3	$339.11^{b} \pm 7.62$	$239.67^{a} \pm 4.57$	$150.68^{a} \pm 3.79$	$173.35^{c} \pm 6.14$	$109.70^{a} \pm 4.01$	$197.24^{b} \pm 5.17$
Z4	$326.37^{b} \pm 8.18$	$145.67^{b} \pm 3.51$	$129.35^{ab} \pm 4.01$	$205.31^{b} \pm 4.97$	$96.38^{a} \pm 2.88$	$193.20^{b} \pm 3.89$
NP	$116.65^{c} \pm 4.77$	$92.23^{c} \pm 2.46$	$85.07^{c} \pm 3.41$	$100.51^{d} \pm 3.43$	$59.14^{b} \pm 4.55$	$79.60^{c} \pm 3.45$
NC	$87.29^{c} \pm 3.05$	$83.76^{c} \pm 2.13$	$76.25^{c} \pm 3.77$	$29.11^{e} \pm 3.02$	$33.57^{b} \pm 3.61$	$28.64^{c} \pm 2.95$

Note: Means (value ± standard error) with different alphabets within the same column are significantly different by Tukey's test at $P \leq 0.05$.

MD2 pineapple productivity in sapric soils amended with clinoptilolite zeolite

Irrespective of the MD2 pineapple planting seasons, all the treatments with clinoptilolite zeolite (Z1 to Z4) significantly improved total N and use efficiency of the pineapple plants (**Figure 7**) compared with NPK fertilization (NP) and untreated soils (NC). Likewise, in terms of MD2 yield attributes, clinoptilolite zeolite treatments significantly increased pineapple fresh fruit weight and fruit yield (Table 7). In this study, the fruit weight of the MD2 pineapples treated with clinoptilolite zeolite is between 2.42 to 2.66 kg/fruit, demonstrating an acceptable fruit yield compared with standard NPK fertilization (1.50 to 1.76 kg/fruit). Contrastingly, all treatments including the controls generally showed similar effects on pineapple fruit sweetness (TSS), fruit acidity, and fruit juice pH (**Table 7**). The temporary entrapment of soil exchangeable NH_4^+–N and available NO_3^-–N by clinoptilolite zeolite via ion exchange and mass transport increases the availability of these macronutrients for MD2 pineapple uptake throughout the growth and development of the pineapple plants (Ramadhani and Nuraini, 2018). Moreover, the timely release of NH_4^+ and NO_3^- ions by clinoptilolite zeolite through diffusion following MD2 pineapple requirement explains the increase in N uptake and use efficiency, MD2 pineapple fresh fruit weight and fruit yield. Compared with the controls (NPK and NC), the increase in N uptake and use efficiency, fruit weight and yield of the MD2 pineapple plants concurs with the higher concentration of exchangeable NH_4^+–N and available NO_3^-–N in the zeolite amended sapric soils (**Tables 5** and **6**). Again, these observations give credence to the ability of this zeolite mineral to adsorb and regulate nutrients in sapric peat soils and further demonstrate the importance of N to produce good MD2 pineapple yields. Nevertheless, different amounts of clinoptilolite zeolite (Z1 to Z4) showed no significant difference in terms of MD2 fresh fruit weight and yield. This finding was unforeseen but the reason for this anomaly could be associated with the competitive interactions between gas molecules, ions, and nutrients for adsorption within and onto the clinoptilolite zeolite framework. This may have affected the assimilation, translocation, and utilization of nutrients, particularly N, P, and K for pineapple fruit development (Tewodros *et al.,* 2018). This also explains the comparable effects of clinoptilolite zeolite with that of NPK fertilization on MD2 pineapple fruit sweetness, fruit acidity, and fruit juice pH. In this study, the controls treatments (NPK fertilization and untreated soils) were not effective to improve N uptake and use efficiency, and MD2 fruit yields because of the lower availability of exchangeable NH_4^+–N and available NO_3^-–N and acidic conditions of the sapric soils.



(a) (b)

Figure 7. Total nitrogen uptake **(a)** and nitrogen use efficiency **(b)** of MD2 pineapples planted on sapric soils treated with clinoptilolite zeolite and NPK fertilizers for two planting seasons. Error bars denote the standard error of the mean. Means with different alphabet are significantly different by Tukey's Multiple Range Test at $p \leq 0.05$.

Table 7. MD2 pineapple fruit yield attributes from drained sapric soils treated with clinoptilolite zeolite and standard compound NPK fertilizers

Treatment code	Fruit weight (kg)	Yield (t/ha)	Total soluble solids (°Brix)	Titratable acidity (%)	pH fruit juice
First planting season					
Z1	$2.59^{a} \pm 0.06$	$98.42^{a} \pm 2.38$	$12.01^{a} \pm 0.01$	$0.56^{ab} \pm 0.02$	$4.20^{a} \pm 0.04$
Z2	$2.58^{a} \pm 0.02$	$98.04^{a} \pm 0.63$	$12.40^{a} \pm 0.02$	$0.65^{a} \pm 0.05$	$4.64^{a} \pm 0.08$
Z3	$2.51^{a} \pm 0.02$	$95.38^{a} \pm 0.65$	$12.10^{a} \pm 0.01$	$0.64^{a} \pm 0.04$	$4.32^{a} \pm 0.12$
Z4	$2.66^{a} \pm 0.06$	$101.08^{a} \pm 2.44$	$12.20^{a} \pm 0.02$	$0.57^{ab} \pm 0.05$	$4.28^{a} \pm 0.07$
NP	$1.76^{b} \pm 0.05$	$66.88^{b} \pm 1.76$	$12.00^{a} \pm 0.01$	$0.51^{b} \pm 0.03$	$4.19^{a} \pm 0.09$
NC	$1.60^{b} \pm 0.06$	$60.98^{b} \pm 2.18$	$11.90^{a} \pm 0.03$	$0.48^{b} \pm 0.03$	$3.83^{b} \pm 0.11$
Second planting season					
Z1	$2.53^{a} \pm 0.05$	$96.14^{a} \pm 1.09$	$12.00^{a} \pm 0.02$	$0.54^{b} \pm 0.03$	$4.36^{a} \pm 0.06$
Z2	$2.46^{a} \pm 0.03$	$93.48^{a} \pm 2.45$	$12.00^{a} \pm 0.02$	$0.61^{ab} \pm 0.07$	$4.15^{a} \pm 0.11$
Z3	$2.42^{a} \pm 0.08$	$91.96^{a} \pm 3.24$	$12.10^{a} \pm 0.02$	$0.75^{a} \pm 0.04$	$4.20^{a} \pm 0.04$
Z4	$2.46^{a} \pm 0.08$	$93.48^{a} \pm 3.64$	$12.10^{a} \pm 0.03$	$0.77^{a} \pm 0.05$	$4.24^{a} \pm 0.11$
NP	$1.50^{b} \pm 0.05$	$57.00^{b} \pm 2.85$	$11.96^{a} \pm 0.03$	$0.61^{ab} \pm 0.07$	$4.21^{a} \pm 0.01$
NC	$1.41^{b} \pm 0.01$	$53.58^{b} \pm 4.21$	$11.90^{a} \pm 0.01$	$0.48^{c} \pm 0.02$	$3.97^{b} \pm 0.07$

Note: Average values (± standard error) with different alphabets in the same column are significantly different by Tukey's Multiple Range Test at $p \leq 0.05$.

From the preceding discussion, although different amount of clinoptilolite zeolite applied to the peat soil showed similar effects toward N_2O emission and MD2 pineapple yield, the results of the present study suggest that the minimal application of clinoptilolite zeolite at a rate of 5 g/plant (25%) in combination with compound NPK 30:1:32 (20 g/plant) is sufficient not only to minimize N_2O emissions but also to improve sapric soil and MD2 pineapple productivity. This adjustment to the pineapple fertilization regime could be adopted by pineapple farmers to sustainably manage and cultivate pineapples on drained tropical peatlands without compromising crop productivity considering the current global increase in fertilizer price and operational costs. The gradual accumulation of clinoptilolite zeolite at the drained peat top horizon, which does not degrade easily with time may have been instrumental in minimizing N_2O emissions.

CONCLUSION

Ameliorating drained tropical peat soils with clinoptilolite zeolite decreases soil N_2O emission, reduces soil acidity, improves soil inorganic nitrogen availability and MD2 pineapple yield and N use efficiency. The in-situ field flux monitoring throughout two MD2 pineapple planting seasons demonstrated the ability of natural zeolite mineral of the clinoptilolite species to adsorb

NH_4^+ and NO_3^- ions via ion exchange and mass transport to inhibit microbial nitrification and denitrification processes in drained peat soils. The adsorption and regulation of soil exchangeable ammonium and available nitrate by clinoptilolite for pineapple uptake improves MD2 pineapple productivity. Co-application of clinoptilolite zeolite (25% or 5 g/plant) with compound NPK 30:1:32 at the pineapple vegetative phases (three- and six-month age) and flower induction period (nine-month age) could be adopted by pineapple farmers in their fertilization regime to sustainably cultivate pineapples on drained histosols without compromising environmental productivity. However, long term N_2O flux monitoring for pineapple cultivation utilizing the closed chamber method and eddy covariance is crucial to validate the findings of the present study because environmental conditions namely rainfall and temperature, soil microbial community structure, and organic matter heterogeneity may affect N_2O emissions throughout the pineapple planting seasons.

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