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Fate of nitrogen in a tropical peat soil treated with clinoptilolite zeolite

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ABSTRACT

Crop productivity on tropical peat soils is poor partly because of their nutrient deficiency and the acidic nature of tropical peat soils. Ammonium (NH_4^+) and nitrate (NO_3^-) ions are commonly leached from tropical peat soils principally because of high rainfall and lack of clay to retain these nutrients. Leaching of these ions into water bodies causes water pollution through eutrophication which has been implicated in disrupting productive aquatic life. To reverse this act of environmental quality degradation, clinoptilolite zeolite (CZ) could be used to retain nutrients including NH_4^+ and NO_3^- in tropical peat soils for crop use because of the alkalinity and high affinity of CZ for retention of macronutrients. Moreover, this approach could be used as an innovative and sustainable alternative for high use of lime ($CaCO_3$) because of the liming effect of CZ. However, information on using CZ on tropical peat soils is scarcely explored. Thus, CZ was tested in a leaching study to determine its effects on soil total nitrogen (N), exchangeable ammonium, and nitrate retention in a tropical peat soil. Treatments evaluated were: (i) soil only, (ii) nitrogen, phosphorus, potassium (NPK) fertilizer alone, and (iii) combination of different amounts of clinoptilolite zeolite (25%, 50%, 75%, and 100%) and different amounts of NPK fertilizer (50%, 75%, and 100%). The peat soils with CZ improved soil pH, exchangeable NH_4^+, and available NO_3^-. The mixture of CZ and NPK fertilizer reduced NH_4^+ and NO_3^- loss because CZ has high affinity for NH_4^+ whereas NO_3^- was absorbed into the channels of CZ. Co-application of 100% CZ with 100%, 75%, and 50% NPK fertilizer, together with 75%, 50%, and 25% CZ with 100% fertilizer increased total N of the peat soil. Ability of CZ to buffer the soil pH reduces the need for liming. This alternative way of retaining N in tropical peat soils is practical and a sustainable approach for improving peat soil productivity.

Keywords: Organic soils, Soil amendment, Nitrogen retention, Nutrient loss, Water quality

1. Introduction

Peat soils are classified as Histosols or organic soils. They are defined as soils which are formed through partially carbonized vegetation tissues [1]. Biochemical processes by aerobic microorganisms in soil surface layers form peat soils during low subsoil water. According to Hoojier et al. [2], there are approximately 27.1 million ha of peat soils in

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South East Asia. Approximately 2.6 million ha of the peat soils are found in Malaysia [3]. According to Leete [4], over 80% of Malaysia's peatland are found in Sarawak. Peat soils in Malaysia are planted with pineapples and palm oil. Peat soils are classified on the basis of topography and geomorphology, surface vegetation, chemical properties, botanical origin, physical characteristics, and peat swamp genetics [1]. Also, the classification of peat soils is based on decomposition stages. The stages are fibrists, hemists, and saprists. Peat soils in Sarawak are sapric type.

Soil nitrogen is an essential element required for crop growth. Nitrogen exists primarily in organic forms that are not accessible for crop uptake [5,6]. In tropical peat soils, although N content is high, it is mostly found in organic form [1,7]. Organic N present in organic matter is converted to crop-usable inorganic forms through mineralization mediated by soil microorganisms. However, if the N converted exceeds crops' requirement, it causes N loss through NH_3^+ volatilization [8,9], denitrification, leaching, and surface runoff [5]. Availability of inorganic N is low in tropical peat soils due to slow N mineralization and high acidity of the peat soils [1,10].

Tropical peat soils are high in organic matter to provide nutrients for crops through their decomposition to produce for example, ammonium and nitrate. However, the acidic nature of tropical peat soils is not suitable for most plant's growth and development. For example, the low pH (3.5–4.0) of peat soils in Sarawak, Malaysia causes peat soils not able to retain significant amounts of macro and micronutrients. Additionally, excessive rainfall and porous nature of the peat cause leaching of plants nutrients. Furthermore, leaching of most of these plant nutrients in peat soils is due to low or absence of clay to hold cations especially on deep peat soils. Clay has a large specific surface area with predominant negatively charged exchange sites which adsorb and retains nutrients against leaching. These negative charges also react with H^+ and Al^{3+} ions to create a buffering condition against extreme pH changes. Lack of clay to glue peat soil particles in manner that improves water holding capacity and retention of positively charged plant nutrients is one of the reasons for leaching of nutrients in peat soils.

Suitable nutrient management in tropical peat soils is essential for promoting sustainable agriculture [11]. Using clinoptilolite zeolite (CZ) as an adsorbent for nutrient retention is because of the unique features of CZ such as crystalline, nano-porous hydrated aluminosilicates of alkali or alkaline-earth metals, arranged in three-dimensional rigid crystalline system, and framed by the tetrahedral AlO_4 and SiO_4 which meet to form a frame of canals, cavities, and pores [12]. Clinoptilolite zeolite are widely used as slow releasing carriers of fertilizer nutrients [13]. Clinoptilolite zeolite's high cation exchange capacity, pH, void volume, and low bulk density can be used in agriculture as a nutrient carrier, slow nutrient release agent, and liming agent. Besides, the unique ion exchange, dehydration-rehydration, adsorption, and catalytic properties of clinoptilolite zeolite enable them to control the release of nutrients for plant uptake [14,15]. According to Ozbahce et al. [16], CZ improved soil pH and plant productivity because of its high ion-exchange and large adsorptive affinity of this mineral for N. Thus, nutrient use efficiency in farming systems can be enhanced using CZ. This is accomplished by increasing the utilization of $N–NH_4^+$ and $N–NO_3^-$ to enable loss of these nutrients through leaching [17–21].

In spite the fact that the literature is replete with positive the effects of CZ in nutrient management in agriculture and on organic soils such as tropical peat soils, the optimal use of CZ on N retention in tropical peat soils remain largely unexplored. In this present study, it was hypothesized that combining clinoptilolite zeolite with nitrogen, phosphorus, potassium (NPK) fertiliser will enhance N availability through increasing soil pH in addition to reducing N leaching. The research questions of this present study were as follows: (i) how should CZ be used to retain N from fertilized tropical peat soils? and (ii) what is the optimum rate of CZ needed to retain N in fertilized tropical peat soils? Thus, the objective of this present study was to determine the changes in soil total, $N–NH_4^+$, and $N–NO_3^-$ retention upon application of CZ and NPK fertilizer. A study of this kind on N availability using CZ is important for determining the response of tropical peat soils to fertilization apart from improving peat soil and crop productivity. Addition of CZ to tropical peat soils is an attempt to delay nutrients loss from for example, NPK fertilisers, thus minimizing the leaching of nutrients particularly $N–NH_4^+$, and $N–NO_3^-$. This approach reduces the effect of the unbalanced or excessive use of N fertilizers on the environmental quality. The information obtained from this study could contribute to understanding of N fertilizers management on tropical peat soils.

2. Materials and methods

2.1. Peat soil sampling

Peat soil samples were collected from the Malaysian Agricultural Research and Development Institute (MARDI) Research Station, Saratok, Sarawak, Malaysia (1°55′30.9″N, 111°14′15.1″E). The peat soil samples were collected at depth of 0 to 20 cm. The area has an annual rainfall of 3676 mm, a mean temperature of 22.8°C to 32.5°C, and mean relative humidity of the area ranges from 59.7% to 60.1%. Thereafter, the soil samples were air dried and ground to pass a 2 mm sieve for initial physico-chemical characterization.

2.2. Chemical properties of clinoptilolite zeolite

The CZ used in this study was in powder form (sieved to pass 250 mm). The selected physico-chemical properties of the CZ are presented in Table 1. The selected physico-chemical properties of CZ are typical of clinoptilolite zeolite [22].

2.3. Selected physico-chem ical analyses and initial characterization of tropical peat soil

Soil pH and electrical conductivity (EC) were measured based on a ratio of 1:10 soil to water suspension using a digital pH meter [23] (SevenEasy pH, Mettler-Toledo GmbH, Switzerland) and an EC meter (SevenEasy Conductivity, Mettler-Toledo GmbH, Switzerland). The peat soil cation exchange capacity was determined by leaching the soil with 1 M ammonium acetate buffer adjusted to pH 7, followed by leaching with 0.1 M potassium sulfate [24] and steam distillation technique [25]. Total N of the soil was determined using

Table 1
Selected physico-chemical properties of clinoptilolite zeolite

Property	Clinoptilolite zeolite
pH	8.20
Electrical conductivity (mS/cm)	0.13
Cation exchange capacity (cmol(+)/kg)	71
Total N (%)	0.22
Total P (%)	0.01
Total K (%)	0.37
Total Ca (%)	0.67
Total Mg (%)	0.10
Total Na (%)	0.76
Total Fe (%)	0.11
Total Zn (mg/kg)	15
Total Mn (mg/kg)	17
Total Cu (mg/kg)	125

the Kjeldahl method [25]. The method of Peech [26] was used to extract exchangeable NH_4^+ and available NO_3^- after which their contents were determined using steam distillation. Organic matter and total carbon of the peat soil were determined using loss of ignition method [27]. Soil total titratable acidity, exchangeable hydrogen, and aluminium were determined using titration method [28]. The selected soil physico-chemical properties are summarized in Table 2.

2.4. Laboratory leaching experiment

Leaching experiment was conducted for 30 d in the Soil Science Laboratory at Universiti Putra Malaysia Bintulu Sarawak Campus, Malaysia. The treatments evaluated in this leaching study are summarized in Table 3:

The 1 kg of peat soil used for each treatment in this leaching study was calculated based on the bulk density of the peat soil. Rates of the compound fertilizer and CZ used were calculated based on the fertilizer requirement of papaya cultivation on peat soils [32]. The compound fertilizer used in this study was $N:P_2O_5:K_2O$. Peat soil only (S0) was used as a control to determine the losses of N, P, and K from the peat throughout the 30 d of leaching, whereas F1 (Soil + NPK fertilizer) was used to obtain the amount of N,P, and K leached from NPK fertilizer for 30 d without addition of CZ. The inclusion of clinoptilolite zeolite in CZ1, CZ2, CZ3, and CZ4 were compared to obtain the optimum amounts of the amendments used to retain N, P, and K from the NPK fertilizer used (Table 3).

The peat soil and amendments were thoroughly mixed after which the mixture was kept in transparent polypropylene containers before the leaching experiment commenced (Fig. 1). The bottom of the polypropylene container was perforated and covered with filter paper. Distilled water (230 mL) was sprayed to each pot with the peat soil in 5 d interval. The volume of water applied was based on rainy days over 30 d. 5 y (2015–2019) of rainfall data was obtained from Malaysian Meteorological Department, Bintulu, Sarawak, Malaysia such that average amount of rainfall per month was applied. The leachates were analyzed for pH,

Table 2
Selected physico-chemical properties of a tropical peat soil

Chemical analysis	Data obtained (0–25 cm)	Standard data range*
pH in water	3.82 ± 0.0869	3.6
Electrical conductivity (mS/cm)	28.14 ± 0.9541	n.d.
Cation exchange capacity (cmol(+)/kg)	55.33 ± 3.5119	68
Total nitrogen (%)	1.49 ± 0.005	1.41
mg/kg		
Ammonium-nitrogen	135.3 ± 2.55	142.5
Nitrate-nitrogen	70.3 ± 2.17	76.3
(cmol/kg)		
Soil total K	0.53 ± 0.005	n.d.
Exchangeable K	0.12 ± 0.007	0.12
Soil total Ca	3.7 ± 0.05	n.d.
Exchangeable Ca	1.11 ± 0.02	2.55
Soil total Mg	5.72 ± 0.13	n.d.
Exchangeable Mg	1.06 ± 0.03	1.24
(%)		
Organic matter	94.27 ± 3.683	n.d.
Total organic carbon	54.68 ± 2.1361	n.d.

n.d. = not determined;
*Standard data range reported by Keeney et al. [29]; Chefetz et al. [30]; Rowell [31]; Mosier et al. [7].

Table 3
Treatments evaluated in leaching study

Treatment	Soil (kg)	NPK fertilizer (g)	Clinoptilolite zeolite (g)
S0	1	–	–
CZ1	1	–	50
F1	1	50	–
CZ2	1	–	37.5
CZ3	1	–	25
CZ4	1	–	12.5
CZ1F1	1	50	50
CZ1F2	1	37.5	50
CZ1F3	1	25	50
CZ2F1	1	50	37.5
CZ2F2	1	37.5	37.5
CZ2F3	1	25	37.5
CZ3F1	1	50	25
CZ3F2	1	37.5	25
CZ3F3	1	25	25
CZ4F1	1	50	12.5
CZ4F2	1	37.5	12.5
CZ4F3	1	25	12.5

Notes: S1: soil only; F1: 100% fertilizer; F2: 75% fertilizer; F3: 50% fertilizer; CZ1: 100% clinoptilolite zeolite; CZ2: 75% clinoptilolite zeolite; CZ3: 50% clinoptilolite zeolite; CZ4: 25% clinoptilolite zeolite.

ammonium, and nitrate. Soil samples were collected at 30 d of leaching, air-dried, and analyzed for pH, total N, ammonium, and nitrate. Chemical properties of the leachates and soil samples were determined using standard procedures outlined in Section 2.3.

2.5. Experimental design and statistical analysis

The leaching study was arranged in completely randomized design (CRD) with three replications. Data obtained were analyzed statistically using analysis of variance (ANOVA) to detect treatment effects whereas treatment means were compared using Tukey's test ($p < 0.05$). The statistical software used was Statistical Analysis System (SAS) version 9.3.

3. Result and discussion

3.1. Treatments on pH of leachate and pH of peat soil over 30 d of leaching

Effects of the treatments on the pH of the leachates for 30 d of leaching are presented in Fig. 2. Leachate of the soil with conventional practice (F1) recorded the lowest pH throughout the leaching study compared with the treatments with clinoptilolite zeolite. This is possible because most of the nutrients leached from the soil during the leaching study because there is no clinoptilolite zeolite to hold the nutrients. The formation of dissolved NH_3 could be a reason for the lower pH of leachate in F1. This is because H^+ released from NH_4^+ lowered the pH of the leachate. Leachate of the soil alone (S0) demonstrated the second lowest pH throughout the leaching study because of the acidic nature of peat soil (Table 2). Higher pH of the CZ1, CZ2, CZ3, and CZ4 were due to the higher base cations of their clinoptilolite zeolite. Clinoptilolite zeolite-maintained pH of the leachates because of the buffering capacity of this mineral and this finding is consistent with that of Afip and Jusoff [33]. pH of the leachates increased with increasing days of leaching (Fig. 2). The fluctuation of the pH of the leachates is related to the non-uniformity of water movement through the peat soil during leaching.

Fig. 3 demonstrates the soil pH in water at 30 d of leaching for the treatments with CZ alone. The combination of CZ

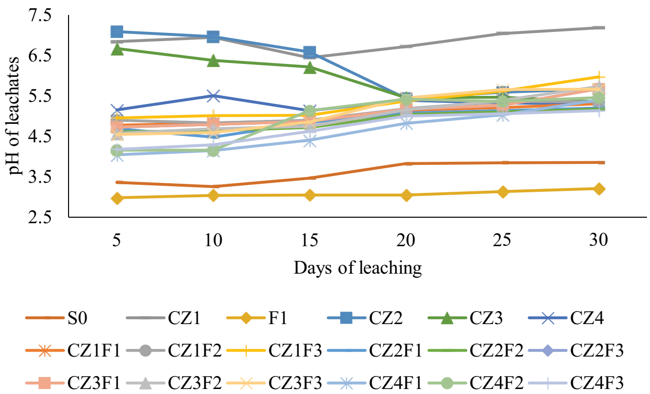

Fig. 2. pH of leachates from peat soils with clinoptilolite zeolite, where S1: soil only; F1: 100% fertilizer; F2: 75% fertilizer; F3: 50% fertilizer; CZ1: 100% clinoptilolite zeolite; CZ2: 75% clinoptilolite zeolite; CZ3: 50% clinoptilolite zeolite; CZ4: 25% clinoptilolite zeolite.

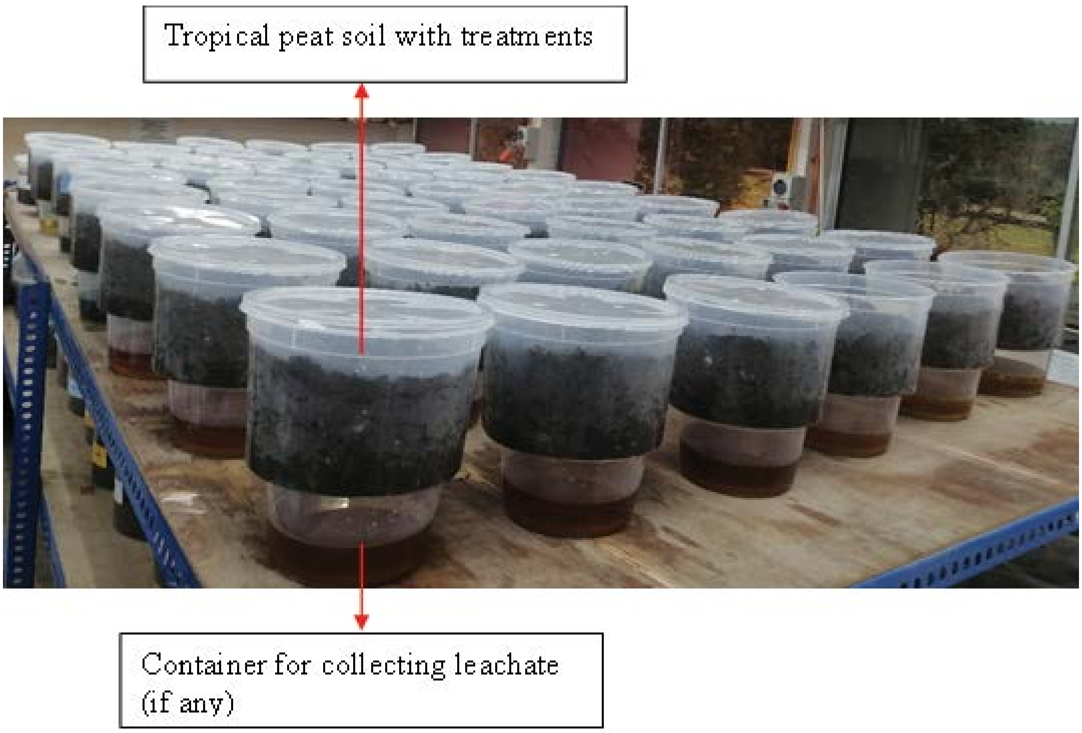

Fig. 1. Set-up of the leaching study (Krishnan et al. [36]).

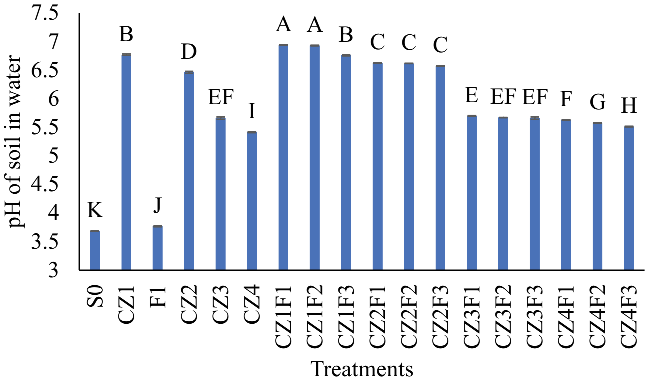

Fig. 3. pH of a tropical peat soil with clinoptilolite zeolite after 30 d of leaching, where S1: soil only; F1: 100% fertilizer; F2: 75% fertilizer; F3: 50% fertilizer; CZ1: 100% clinoptilolite zeolite; CZ2: 75% clinoptilolite zeolite; CZ3: 50% clinoptilolite zeolite; CZ4: 25% clinoptilolite zeolite. Means between columns with different letter(s) indicate significant difference between treatments by Tukey's test at $p < 0.05$. Bars represents the mean values ± SE.

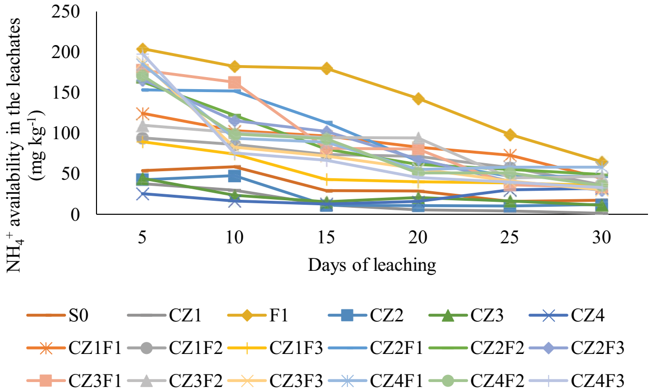

Fig. 4. Ammonium concentration in leachates with clinoptilolite zeolite, where S1: soil only; F1: 100% fertilizer; F2: 75% fertilizer; F3: 50% fertilizer; CZ1: 100% clinoptilolite zeolite; CZ2: 75% clinoptilolite zeolite; CZ3: 50% clinoptilolite zeolite; CZ4: 25% clinoptilolite zeolite.

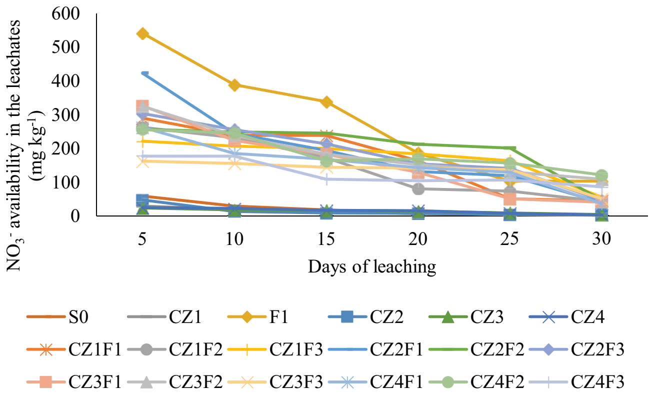

Fig. 5. Nitrate concentration in leachates with clinoptilolite zeolite, where S1: soil only; F1: 100% fertilizer; F2: 75% fertilizer; F3: 50% fertilizer; CZ1: 100% clinoptilolite zeolite; CZ2: 75% clinoptilolite zeolite; CZ3: 50% clinoptilolite zeolite; CZ4: 25% clinoptilolite zeolite.

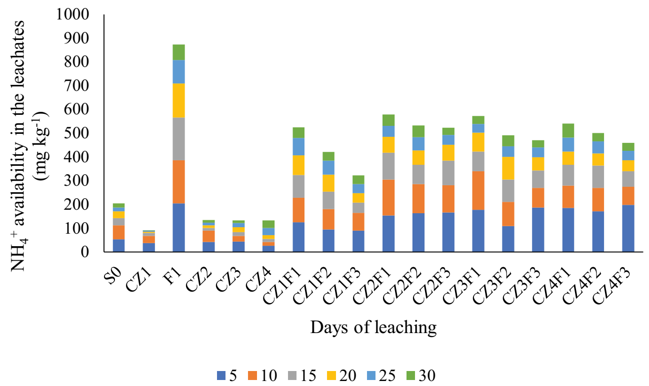

Fig. 6. Cumulative concentrations of ammonium in leachates with clinoptilolite zeolite, where S1: soil only; F1: 100% fertilizer; F2: 75% fertilizer; F3: 50% fertilizer; CZ1: 100% clinoptilolite zeolite; CZ2: 75% clinoptilolite zeolite; CZ3: 50% clinoptilolite zeolite; CZ4: 25% clinoptilolite zeolite.

and NPK fertilizer was significantly higher than soil alone (S0) and soil with NPK fertilizer (F1). The low soil pH of S0 and F1 were due to the higher amounts of basic cations leaching compared with other treatments. Increase in the soil pH following incorporation of CZ is due to the basic nature of the amendment (Table 1). Addition of CZ increased pH of the peat soil because of its Ca, Mg, K, and Na contents [34], thus suggesting the liming effect of this mineral. Chan et al. [35] stated that CZ is marginally alkaline, not acidic and fusing them with fertilizers enhances soil buffering capacity, thus reducing the need for liming. Similar observation had been observed by Krishnan et al. [36] where application of clinoptilolite zeolite increased soil pH. The soil with 100% CZ significantly higher pH compared with other treatments with the soil amendment. The soil pH decreased with decreasing amount of the CZ used.

3.2. Treatments on ammonium and nitrate availability in leachates

The 5-d interval losses of NH_4^+ and NO_3^- from soil in the leaching experiment for 30 d are presented in Figs. 4 and 5. All of the treatments with clinoptilolite zeolite significantly reduced leaching of NH_4^+ from soil compared with soil alone (S0) and the soil with conventional practice (F1) as summarized in Fig. 4. The highest amount of NO_3^- leached from F1 compared with the other treatments with clinoptilolite zeolite (Fig. 5). The lower amount of NO_3^- leached from soil only (S0), CZ1, CZ2, CZ3, and CZ4 could be due to the loss in the form of NH_4^+. Similar observation has been reported by Filcheva and Tsadilas [37] and Polat et al. [38]. The mixture of CZ and NPK fertilizer reduced NH_4^+ and NO_3^- loss because CZ has high affinity for NH_4^+ whereas NO_3^- was absorbed into the channels of CZ.

The cumulative losses of NH_4^+ and NO_3^- for 30 d of leaching were calculated to demonstrate the effectiveness of clinoptilolite zeolite in retaining NH_4^+ from fertilizer (Figs. 6 and 7). Higher amounts of NH_4^+ were leached from F1 compared with the treatments with CZ, regardless of the amount

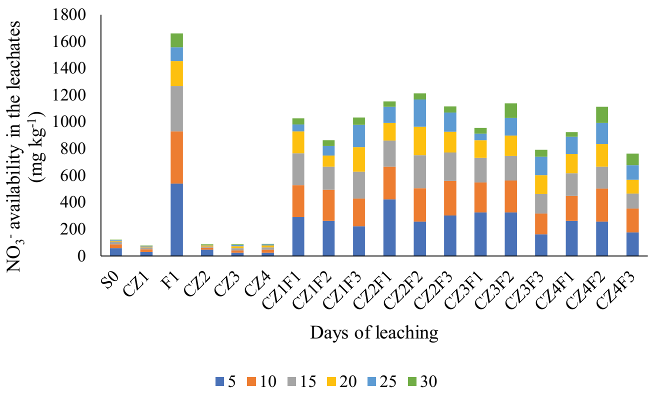

Fig. 7. Cumulative concentrations of nitrate in leachates with clinoptilolite zeolite, where S1: soil only; F1: 100% fertilizer; F2: 75% fertilizer; F3: 50% fertilizer; CZ1: 100% clinoptilolite zeolite; CZ2: 75% clinoptilolite zeolite; CZ3: 50% clinoptilolite zeolite; CZ4: 25% clinoptilolite zeolite.

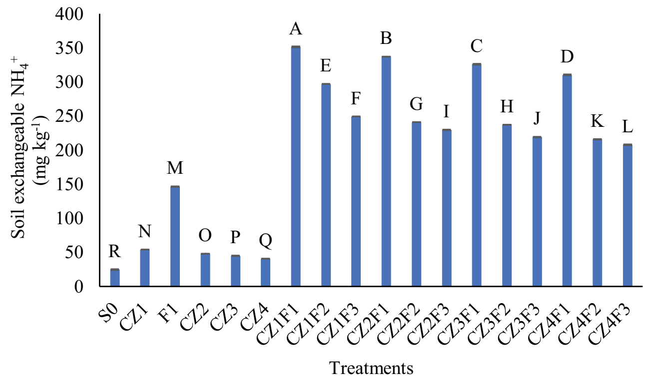

Fig. 8. Exchangeable ammonium of a tropical peat soil with clinoptilolite zeolite after 30 d of leaching, where S1: soil only; F1: 100% fertilizer; F2: 75% fertilizer; F3: 50% fertilizer; CZ1: 100% clinoptilolite zeolite; CZ2: 75% clinoptilolite zeolite; CZ3: 50% clinoptilolite zeolite; CZ4: 25% clinoptilolite zeolite. Means between columns with different letter(s) indicate significant difference between treatments by Tukey's test at $p < 0.05$. Bars represents the mean values ± SE.

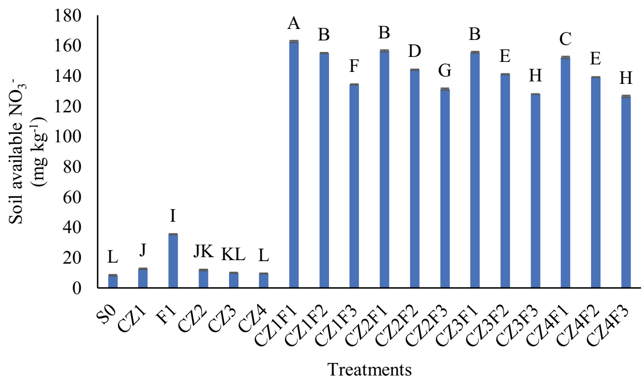

Fig. 9. Available nitrate of a tropical peat soil with clinoptilolite zeolite after 30 d of leaching, where S1: soil only; F1: 100% fertilizer; F2: 75% fertilizer; F3: 50% fertilizer; CZ1: 100% clinoptilolite zeolite; CZ2: 75% clinoptilolite zeolite; CZ3: 50% clinoptilolite zeolite; CZ4: 25% clinoptilolite zeolite. Means between columns with different letter(s) indicate significant difference between treatments by Tukey's test at $p < 0.05$. Bars represents the mean values ± SE.

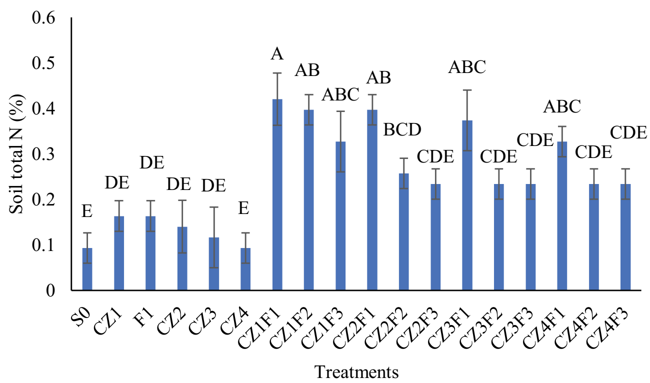

Fig. 10. Total nitrogen of a tropical peat soil with clinoptilolite zeolite after 30 d of leaching, where S1: soil only; F1: 100% fertilizer; F2: 75% fertilizer; F3: 50% fertilizer; CZ1: 100% clinoptilolite zeolite; CZ2: 75% clinoptilolite zeolite; CZ3: 50% clinoptilolite zeolite; CZ4: 25% clinoptilolite zeolite. Means between columns with different letter(s) indicate significant difference between treatments by Tukey's test at $p < 0.05$. Bars represents the mean values ± SE.

of the CZ used (Fig. 6) because of the specific selectivity of CZ for NH_4^+ [39].

3.3. Treatments on peat soil exchangeable NH_4^+, available NO_3^-, and total N of peat soil at 30 d of leaching

At the end of the leaching study, the combination of NPK fertilizer and CZ demonstrated higher content of soil exchangeable NH_4^+ and NO_3^- compared with the peat soil with fertilizer only (F1) (Figs. 8 and 9) because most of the NH_4^+ and NO_3^- were leached from F1 as revealed in Figs. 4–7. These findings suggest that clinoptilolite zeolite can be used as slow-releasing N fertilizer based on NH_4^+ retention. With this reaction, nitrification in peat soils can be controlled or regulated to prevent eutrophication of water bodies through NO_3^- contamination or pollution. Nitrogen losses occur when soils have more incoming water than they can hold. Both NH_4^+ and NO_3^- are prone to leaching [40]. As water moves through soils, the NO_3^- in them moves along with the water. However, because NH_4^+ is positively charged, it is held by the negative charges of clay and humus present in the soil but because of lack of clay in tropical peat soils, leaching of NH_4^+ occurs. This present study has demonstrated that amending tropical peat soil with clinoptilolite zeolite controls N loss from fertilizers because of the small molecular size of the open-ringed structure of CZ which physically protects NH_4^+ ions from microbial nitrification [39]. Mixture of clinoptilolite zeolite and fertilizer reduced NH_4^+ leaching because clinoptilolite zeolite has high affinity for NH_4^+. Exchangeable NH_4^+ are adsorbed in the mineral lattice of clinoptilolite zeolite, whereas available NO_3^- are absorbed into the channels of clinoptilolite zeolite. As reported by Zwingmann et al. [41], CZ has the capacity to hold approximately 20%–30%

of its weight in NO_3^- thus, reducing NO_3^- leaching. Moreover, the use of CZ minimizes rapid conversion of NH_4^+ to NO_3^- [42]. The specific selectivity of clinoptilolite zeolite to NH_4^+ is one of the reasons why clinoptilolite zeolite is used as adsorbent agent to capture N, after which the captured N is stored and released timely released for plant use [43–46].

Soil total N after the leaching experiment without subtracting the contribution of S0 from the other treatments is presented in Fig. 10. The total N concentrations of CZ1F1, CZ1F2, CZ1F3, CZ2F1, CZ3F1, and CZ4F1 were significantly higher than that of F1 (Fig. 10) because the CZ enabled slow release of N from the fertilizer used in this present study. The lower retention of total N in the soil with fertilizer only (F1) confirms the loss of N due to leaching of NH_4^+ and NO_3^- (Figs. 4–7).

4. Conclusion

Treating peat soils with CZ improves soil pH, exchangeable NH_4^+, and available NO_3^-. The mixture of CZ and NPK fertilizer can reduce NH_4^+ and NO_3^- loss because CZ has high affinity for NH_4^+ whereas NO_3^- was absorbed into the channels of CZ. Co-application of 100% CZ with 100%, 75%, and 50% NPK fertilizer, together with 75%, 50%, and 25% CZ with 100% fertilizer increases total N of the peat soil. Ability of CZ to buffer the soil pH reduces the need for liming. This alternative way of retaining N in tropical peat soils is practical and a sustainable approach for improving peat soil productivity. However, further studies are required to consolidate the findings of this present study.

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