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Full Length Research Paper

Utilization of activated carbon produced from *Sago hampas* (*Metroxylon sagu*) to reduce ammonia loss from urea

Regis Bernard[1], Osumanu Haruna Ahmed[1]* and Nik Muhamad Ab Majid[2]

[1]Department of Crop Science, Faculty of Agriculture and Food Sciences, University Putra Malaysia, Bintulu Campus, Sarawak, 97008 Bintulu, Sarawak, Malaysia.
[2]Department of Forest Management, Faculty of Forestry, University Putra Malaysia, 34300 UPM Serdang, Selangor, Malaysia.

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Surface-applied urea contributes to NH_3 loss of N through ammonia volatilization, especially in warm and humid regions. Ammonia loss has been a major problem confronting farmers because additional urea is applied to compensate for the loss which increases cost of fertilization. Activated carbon (AC) produced from *Sago hampas* (SH) could be capable of absorbing N in the form of NH_4^+ to minimize ammonia volatilization. The objective of this study was to determine if AC produced from SH could be used to retain ammonium as well as minimizing ammonia loss from urea. Activated carbon was produced from SH by using concentrated H_2SO_4 and $(NH_4)_2S_2O_8$. There were three types of AC produced using different volumes of H_2SO_4 (AC30, AC40 and AC50). The charred product was used as a treatment together with the raw material in order to observe the reduction of ammonia loss using closed-dynamic air flow system for 7 days. The daily loss of $NH_3$was observed and the $NH_4^+$and NO_3^- retention in the soil were determined using standard procedures. Activated carbon produced from SH had a higher CEC and it increased from AC30 to AC50. The treatments did not contribute to the reduction of ammonia loss; however, there was a significant increase in the retention of NH_4^+ in the soil compared to urea alone. The AC was observed to be a better absorbent than SH. AC50 was a better absorbent compared to other treatments due to high acidity and CEC. However, the treatment did not contribute to reduction of ammonia loss, but there was a significant increase of NH_4^+ retention in the soil.

Key words: Activated carbon, ammonia, ammonia volatilization, ammonium, *Metroxylon sagu*, sago *hampas*, urea.

INTRODUCTION

Biological fixation of nutrients especially nitrogen (N) does not compensate for the rapid uptake by the crops. Therefore, efficient use of fertilizers particularly nitrogenous fertilizers such as urea cannot be over emphasized (Ferguson et al., 2002; Abdel-Mawgoud et al., 2007). Urea is the common N-based fertilizer used in agriculture. However, urea is prone to loss in warm and high humid regions via ammonia (NH_3) volatilization, hence reducing N availability when urea is broadcast onto the surface of the soil (Prasertsak et al., 2001; Cai et al., 2002; Latifah et al., 2011d).

There are several factors which influence ammonia volatilization, however this study emphasizes on the pH-buffering capacity and cation exchange capacity (CEC) in order to shift the equilibrium towards ammonium production and retention in the soil so as to minimize ammonia loss (Freney and Black, 1988). In recent years, it has been found that when urea is mixed with materials which are acidic or/and high in CEC, they are capable of decreasing ammonia loss while increasing the

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*Corresponding author. E-mail: osman60@hotmail.com.

Table 1. Selected physico-chemical properties of soil, Sago, and activated carbon (AC30, AC40 and AC50).

Property	Soil	SH	AC30	AC40	AC50
pH (1 *N*KCl)	3.83	3.54	4.36	3.36	1.74
pH (water)	4.89	3.93	5.48	4.44	2.23
[1]CEC (cmol kg^{-1})	21.40	64.38	142.5	160.00	191.67
Soil texture	[1]Clay	nd	nd	nd	nd
Exchangeable Na (ppm)	258	336	39427	40336	41030
Exchangeable K (ppm)	214	359	1001	856	554
Exchangeable Mg (ppm)	101	494	33	33	27
Exchangeable Ca (ppm)	37	1625	142	145	143
Exchangeable NH_4^+ (ppm)	47	23	67	146	173
Available NO_3^- (ppm)	t.e.	1	t.e.	t.e.	1

[1]Clay, (sand = 39.05; clay = 44.92; silt = 16); nd, not determined; AC30, activated carbon (*S. hampas* + 30 ml 50% H_2SO_4); AC40, activated carbon (*S. hampas* + 40 ml 50% H_2SO_4); AC50, activated carbon (*S. hampas* + 50 ml 50% H_2SO_4), t.e., trace.

concentration of ammonium and nitrate ions (Fan and Mackenzie, 1993; Siva et al., 1999; Rosliza et al., 2009a, b; Ahmed et al., 2010; Latifah et al., 2011a, b, c).

Materials which have been used to reduce loss include, Triple superphosphate (TSP), humic acids, fulvic acids,-peat water, zeolite, acid sulphate soil and sago waste water (Ahmed et al., 2006a, b; Taufik et al., 2009; Ameera et al., 2009; Bernard et al., 2009; Rosliza et al., 2009a, b; Ahmed et al., 2010; Latifah et al., 2010, 2011a, b, c). Activated carbon (AC) absorption is widely used to remove pollutants due to their fundamental physico-chemical characteristic which allows them to absorb substances in the form of ions, gas and liquid. Therefore, AC has the tendency to absorb ammonium ions or ammonia (Puziy et al., 2002; Kadirvelu et al., 2004; Alias et al., 2009). Sago starch production from sago palm (*Metroxylon sagu*) contributes to large quantity of fibrous residues (*Sago hampas, SH*) which is approximately 1 ton per working day (Vikineswary et al., 1994). The SH is high in cellulose and lignin. This suggests that the fiber has the potential to absorb ions such as ammonium. This can be achieved if SH is converted to AC to improve its absorption capability (Vikineswary et al., 1994; Quek et al., 1998). Therefore, this study was carried out to determine if AC produced from SH could be used to retain ammonium as well as minimizing ammonia loss from urea.

MATERIALS AND METHODS

The SH used to produce AC was collected from Sagolink SDN. BHD., Mukah, Sarawak, Malaysia whereas, the mineral soil used in the incubation study was Nyalau series (Typic Paleudults). The mineral soil was collected at a depth of 0 to 15 cm at Universiti Putra Malaysia, Bintulu Sarawak Campus. AC was produced from SH using method of Kadirvelu et al. (2004) with modification. The AC was prepared using 3 different volumes (30, 40, and 50 ml) of 50% H_2SO_4. A 10 g of SH was mixed with 1% w/w $(NH_4)_2S_2O_8$, after which H_2SO_4 was added and stirred for 30 min. The charred material was kept in a furnace at 150˚C for 12 h. Afterwards, the material was washed with 50 ml distilled water by centrifugation (11,000 rpm for 10 min) using Sigma Laboratory Centrifuge, 6K15. The washing process was continued with 0.5 M KOH. Distilled water was used to wash the samples twice. The charred material was soaked in 50 ml of 5% $NaHCO_3$ solution for 12 h. Afterwards, the sample was washed with distilled water 3 times to remove excess $NaHCO_3$ and it was dried using a furnace at 120˚C for 12 h. The dried AC samples ground was sieved to pass a 250 µm sieve. The SH and soil samples were sieved to pass a 2 mm sieve. The physico-chemical properties of the samples above were determined as presented in Table 1. The pH of AC was determined in a ratio of 1:10 while that of SH was determined in a ratio of 1:2.5 (Peech, 1965). The CEC of the mineral soil was determined using the ammonium acetate leaching method (Cottenie, 1980) followed by steam distillation (Bremmer, 1965) while soil exchangeable cations (Na, Mg, Ca, and K) were extracted using Bray 1 method (Bray and Kurtz, 1945). Contents of these cations in the mineral soil twere determined using atomic absorption spectrophotometry. The soil texture was determined using the hydrometer method (Tan, 2005). Soil ammonium and nitrate contents were determined using the method of Keeney and Nelson (1982).

The treatments evaluated in 250 g of soil for 7 days were:

T0 = Soil only (control)
T1 = Urea only (control)
T2 = 1.443 g S. *hampas* (control)
T3 = 2.165 g S. *hampas* (control)
T4 = 2.886 g S. *hampas* (control)
T5 = Urea + 1.443 g S. *hampas*
T6 = Urea + 2.165 g S. *hampas*
T7 = Urea + 2.886 g S. *hampas*
T8 = Urea + 0.613 g AC30
T9 = Urea + 0.920 g AC30
T10 = Urea + 1.226 g AC30
T11 = Urea + 0.534 g AC40
T12 = Urea +0.801 g AC40
T13 = Urea + 1.068 g AC40
T14 = Urea + .0485 g AC50
T15 = Urea + 0.728 g AC50
T16 = Urea + 0.970 g AC50

The daily loss of ammonia was measured in a closed-dynamic airflow system which consisted of an exchange chamber (500 ml conical flask) and a trap (250 ml conical flask) both stopped and fitted with an inlet/outlet. Each treatment had an exchange chamber containing the mineral soil which moistened with distilled water to 70% of field capacity. Each treatment had 3 replicates. The inlet of the chamber was connected to an air pump and the outlet was to the trap. The ammonia released from the system was collected

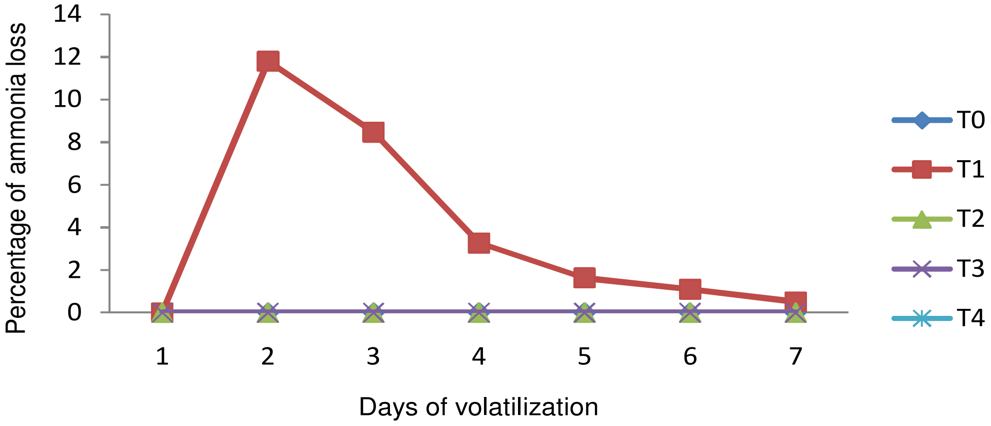

Figure 1. Loss of ammonia over 7 days of incubation for T0, T1, T2, T3 and T4.

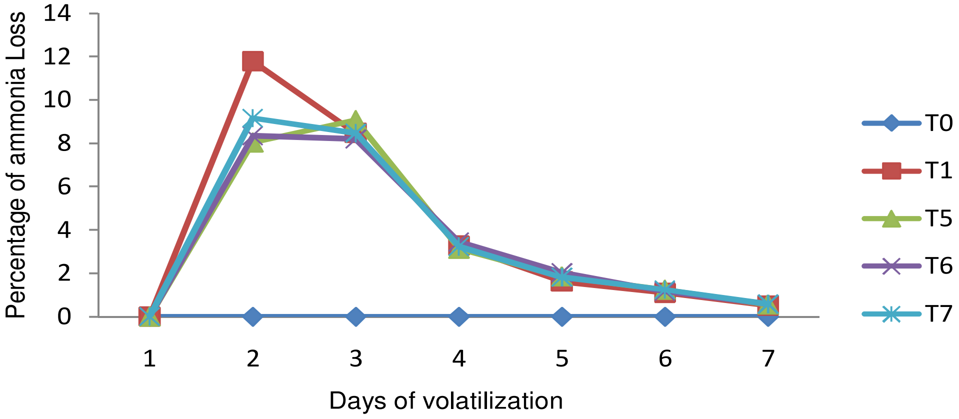

Figure 2. Loss of ammonia over 7 days of incubation for T0, T1, T5, T6 and T7.

separately using a trapping solution (75 ml of 2% boric acid. The solution was replaced daily until the ammonia loss declined to 1%. The solution was titrated with 0.1 M HCl to estimate the amount ammonia loss (Ahmed et al., 2006a, b; Taufik et al., 2009; Ameera et al., 2009; Bernard et al., 2009; Ahmed et al., 2010; Latifah et al., 2010, 2011a, b, c). After the incubation process, the soil samples were analyzed for pH, exchangeable cations (Na, Mg, Ca, and K), NH_4^+ and NO_3^-. The experimental design was a completely randomized design. Treatments effect were detected using analysis of variance while treatment means were compared using Duncan's New Multiple Range Test. The statistical software used was Statistical Analysis System version 9.2.

RESULTS

pH of the mineral soil, SH, and AC in distilled water and 1 M KCl were acidic. Clay content, selected exchangeable cations and CEC of the mineral soil used were found to be higher than the original Typic Palaudults reported by Paramananthan (2000). This could be due to adulteration of the soil caused by human activity in cultivating the soil. SH had a higher CEC but its pH was relatively low even though its exchangeable cations (Na, Mg, Ca, K and NH_4^+) were high (Table 1). All of the three activated samples had higher CEC compared to SH. However, AC50 had the highest (192 cmol kg^{-1}) and this value is comparable to that of a clinoptilolite zeolite (100 to 200 cmol kg^{-1}) (Ahmed et al., 2006b). The pH of the AC was found to be acidic with AC50 being the lowest among the three. Exchangeable Na was higher in AC than in SH due to the addition of $NaHCO_3$. This suggests that the number of washing was not sufficient to remove all of the Na absorbed by the AC. Potassium was higher in AC30 but it decreased with higher volume of H_2SO_4. Calcium was very high in SH (1625 ppm).The daily ammonia loss was measured for 7 days (Figures 1 to 5). The ammonia volatilization peaked on the second day of incubation. The ammonia loss for all the treatments declined on the third day of incubation. This observation is similar to other reports (Ahmed et al., 2006b, 2010; Taufik et al., 2009; Ameera et al., 2009; Bernard et al., 2009; Latifah et al., 2010). However, this incubation study had the shortest period in reducing ammonia loss. All the controls except for T1 did not release ammonia throughout the study. The total loss of ammonia for T5 to T16 was not

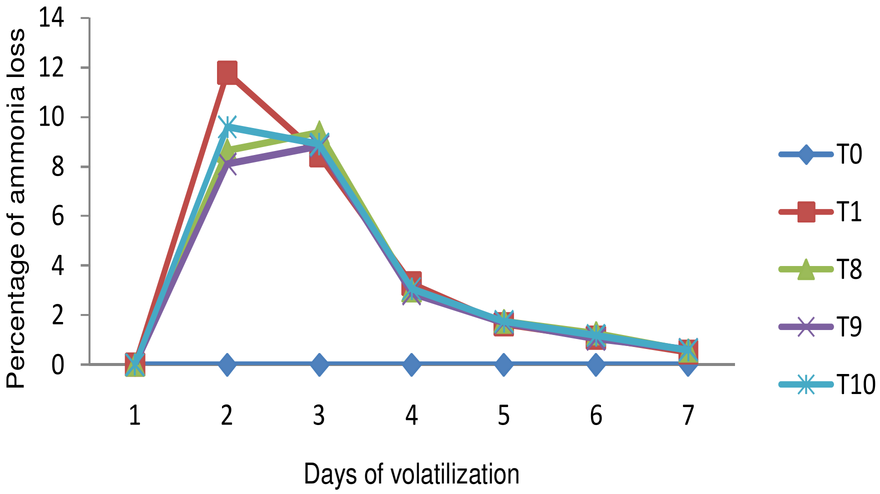

Figure 3. Loss of ammonia over 7 days of incubation for T0, T1, T8, T9 and T10.

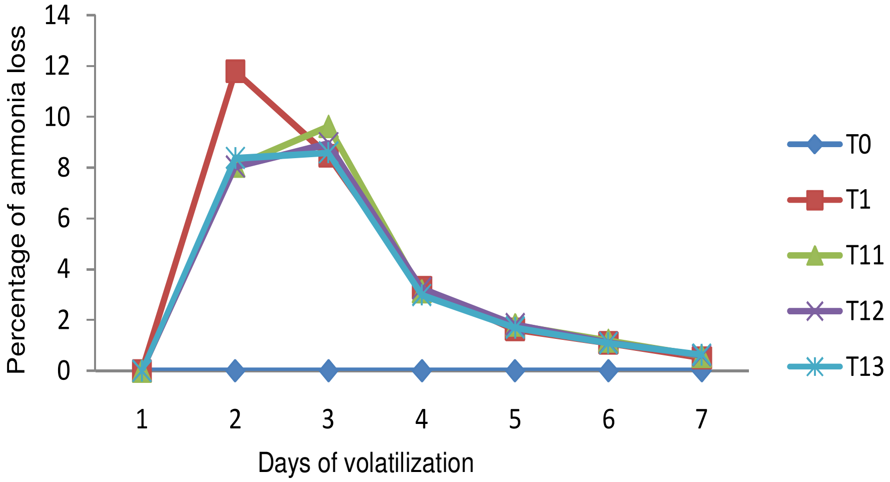

Figure 4. Loss of ammonia over 7 days of incubation for T0, T1, T11, T12 and T13.

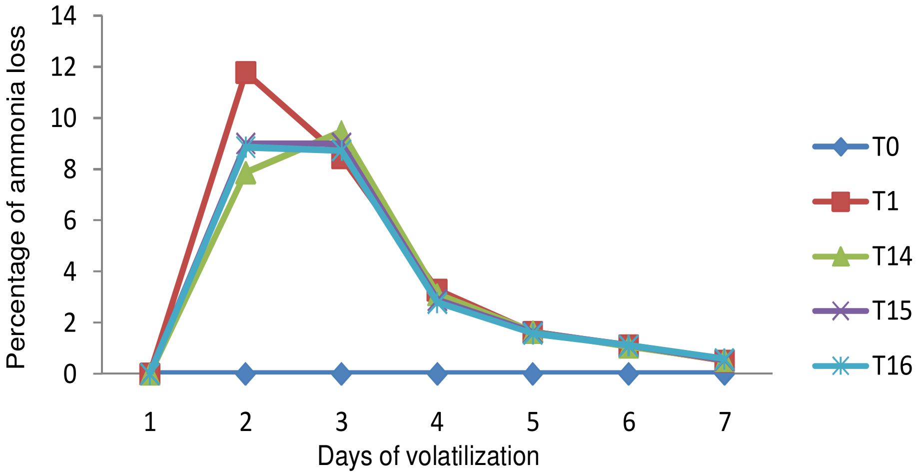

Figure 5. Loss of ammonia over 7 days of incubation for T0, T1, T14, T15 and T16.

Table 2. Total amount of ammonia released over 7 days of incubation.

Treatment	Loss of NH_3 (%)
T0	0^a
T1	26.75^b
T2	0^a
T3	0^a
T4	0^a
T5	23.88^b
T6	23.73^b
T7	24.51^b
T8	24.60^b
T9	23.1^b
T10	25.05^b
T11	24.38^b
T12	24.30^b
T13	23.38^b
T14	23.68^b
T15	24.15^b
T16	23.62^b

Different alphabets within the same column indicate that there are significant differences at $p \leq 0.05$ between mean values using Duncan's new multiple range test, DNMRT.

Table 3. Effects of treatment on exchangeable ammonium and available nitrate accumulation.

Treatment	Exchangeable NH_4^+ (ppm)	Available NO_3^- (ppm)
T0	48.67^a	0^a
T1	1875.20^c	58.33^b
T2	185.53^b	53.25^b
T3	225.33^b	49.04^b
T4	215.66^b	47.77^b
T5	2694.15^d	83.30^c
T6	2853.56^e	85.20^c
T7	2989.70^f	86.89^c
T8	3073.51^g	91.10^c
T9	3118.69^{gh}	91.09^c
T10	3121.45^{gh}	100.48^c
T11	3133.64^{gh}	97.28^c
T12	3128.20^{gh}	105.38^c
T13	3183.04^{hi}	96.36^c
T14	3225.82^{ij}	103.65^c
T15	3252.66^{ij}	96.20^c
T16	3275.61^j	102.03^c

Different alphabets within the same column indicate that there are significant differences at $p \leq 0.05$ between mean values using Duncan's new multiple range test.

significantly different from that of urea alone (Table 2). The treatments with SH, AC30, AC40 and AC50 were devised by calculating the amounts of SH, AC30, AC40 and AC50 required to completely absorb ammonia. Although this increase did not minimize ammonia loss, there was an increase in retention of ammonium in the soil (Table 3). The amount of ammonium retention increased significantly from T5 to T16 compared with

Table 4. Soil pH, exchangeable Na, Mg, Ca and K in the soil after 7 days of incubation.

Treatment	pH		Exchangeable (ppm)			
	1 M KCl	water	Na	Mg	Ca	K
T0	3.79^{a}	4.87^{a}	223.33^{ef}	96.00^{ab}	46.67^{d}	171.33^{a}
T1	6.13^{b}	7.18^{b}	$239.00d^{ef}$	82.33^{bcd}	61.67^{abc}	160.33^{ab}
T2	3.77^{a}	4.84^{a}	221.67^{ef}	97.67^{a}	48.33^{dc}	157.67^{abc}
T3	3.71^{a}	4.83^{a}	233.67^{ef}	95.33^{abc}	56.33^{abcd}	159.33^{ab}
T4	3.77^{a}	4.84^{a}	222.33^{ef}	96.00^{ab}	59.33^{abcd}	148.00^{bcd}
T5	5.74^{c}	6.78^{c}	231.67^{ef}	79.00^{d}	58.33^{abcd}	148.67^{bcd}
T6	5.76^{c}	6.81^{c}	230.00^{ef}	81.67^{cd}	69.33^{a}	156.33^{abcd}
T7	5.74^{c}	6.80^{c}	248.33^{ed}	75.33^{d}	57.33^{abcd}	146.67^{bcd}
T8	5.76^{c}	6.83^{c}	306.00^{ab}	76.67^{d}	56.33^{abcd}	147.00^{bcd}
T9	5.76^{c}	6.81^{c}	313.00^{a}	78.00^{d}	56.67^{abcd}	158.00^{ab}
T10	5.72^{c}	6.84^{c}	268.67^{cd}	$82.18b^{cd}$	55.67^{abcd}	152.67^{bcd}
T11	5.76^{c}	6.80^{c}	280.00^{bc}	$82.03b^{cd}$	63.67^{ab}	156.33^{abcd}
T12	5.73^{c}	6.76^{c}	309.00^{ab}	81.00^{d}	58.67^{abcd}	153.00^{bcd}
T13	5.76^{c}	6.75^{c}	207.00^{f}	78.33^{d}	67.00^{ab}	141.33^{cd}
T14	5.69^{c}	6.74^{c}	208.33^{f}	74.00^{d}	60.67^{abcd}	141.00^{d}
T15	5.78^{c}	6.74^{c}	268.00^{ef}	76.67^{d}	62.67^{ab}	147.33^{bcd}
T16	5.68^{c}	6.76^{c}	269.00^{cd}	76.33^{d}	53.67^{bcd}	144.00^{bcd}

Different alphabets within the same column indicate that there are significant differences at $p \leq 0.05$ between mean values using Duncan's new multiple range test.

urea alone. AC30, AC40 and AC50 improved ammonium retention better than SH. Soil available nitrate contents of T5 to T16 were higher than that of T1. This observation was similar to the findings of Ahmed (2006a, b). With the exception of T0, T2 and T3, the other treatments had effect on soil pH (Table 4). However, their pH values were close neutral, a finding consistent with those of other authors (Ahmed et al., 2006a, b, 2010; Taufik et al., 2009; Ameera et al., 2009; Bernard et al., 2009; Latifah et al., 2010, 2011a, b, c).

DISCUSSION

The SH was found to be high in CEC because of its high content of cellulose and lignin (70 to 90%) (Vikineswary et al., 1994; Quek et al., 1998). This may have improved the porosity of SH. Like most woody fibrous materials, SH is capable of absorbing heavy metals and liquid. This property relates to its CEC (Vikineswary et al., 1994; Quek et al., 1998). The low pH of SH could be attributed to organic acids such as humic and fulvic acids. The increased CEC of AC with 50% (volume basis) increase in H_2SO_4 suggests that with higher amount of H_2SO_4, the SH fibres were rendered more porous. The pH of AC also decreased with increasing volume. This could be partly be attributed to residual effect of H_2SO_4. Although Na was high in AC30, AC40 and AC50, they remained acidic. This suggests that their buffering capacity might have been exceeded.

Although urea amended treatments did not significantly minimize ammonia loss compared with urea alone, the fact that they significantly improved ammonium and nitrate ions retention suggests it was favorable for the formation of these ions over ammonia. Similar findings have been reported by other authors (Ahmed et al., 2006a, 2010b; Taufik et al., 2009; Ameera et al., 2009; Bernard et al., 2009; Latifah et al., 2010, 2011a, b, c). The high retention of ammonium and nitrate ions indicates that these nutrients could be timely and efficiently utilized by plants hence contributing to reduction of these ions being lost through leaching. The low concentration of NO_3^- in the soil compared to NH_4^+ was due to absorption of NH_4^+ by the AC, process which prevented rapid nitrification (Barber and Peterson, 1995; Bernard et al., 2009; Ahmed et al., 2010; Latifah et al., 2010, 2011a, b, c). Accumulation of ammonium under T2, T3 and T4 could be because of crude proteins (1%) in SH (Vikineswary et al., 1994). During incubation, the protein might have been broken down to simple form of N, a process which may have indirectly contributed to increase in ammonium and nitrate. AC was a better absorbent due to its high CEC and large surface area (250 µm) compared with raw SH (2 mm). Besides acidity and CEC, particle size and surface area play an important role in the effectiveness of an absorbent.

Conclusion

Amending urea with different levels of AC did not reduce ammonia loss from urea but it improved ammonium retention compared with SH and urea alone. To consolidate these findings, a similar investigation on organic soils is suggested. Both greenhouse and field experiments using test crop such as *Zea mays* if retention of ammonium ion observed in the laboratory experiment will result in improved urea-N use efficiency and yield.

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