

Kinetics of Ammonium Volatilization from Nitrogen Fertilizers as Affected by Sulphur

R. R. Shahin and R. M. M. El-Kilani

Soil and Water Department, Faculty of Agriculture, Cairo University, Cairo, Egypt.

AN INCUBATION experiment for 48 days on a sandy soil was conducted to study the effect of sulphur addition to three kinds of N-fertilizers on the loss of ammonia by volatilization from these fertilizers. The used fertilizers were Urea (U), ammonium sulphate (AS), and di-ammonium phosphate (DAP), either alone or mixed with 0.2 % elemental Sulphur. The volatilized ammonia was received in boric acid and determined by titration.

A kinetic analysis of the reaction rates was done to determine the first order reaction rate for ammonia volatilization. The sulphur addition led to a decrease in the ammonia volatilization first order constants. The first order rate decreased from 0.135 (day^{-1}) to 0.039 (day^{-1}) and from 0.14 (day^{-1}) to 0.067 (day^{-1}) and from 0.132 (day^{-1}) to 0.101 (day^{-1}) for U, AS and DAP respectively. There was a decline in the rate of reaction for the same treatment with time after about 13 days. The first order reaction rates decreased from 0.135 (day^{-1}) to 0.03 (day^{-1}), from 0.039 (day^{-1}) to 0.023 (day^{-1}), 0.140 (day^{-1}) to 0.044 (day^{-1}), from 0.067 (day^{-1}) to 0.012 (day^{-1}), from 0.132 (day^{-1}) to 0.030 (day^{-1}), from 0.101 (day^{-1}) to 0.023 (day^{-1}) for U, U+S, AS, AS+S, DAP, DAP+S respectively.

The experiment was repeated again without adding more sulphur. The residual effect of sulphur relatively decreased the ammonia volatilization first order rate constants. The first order rate decreased from 0.114 (day^{-1}) to 0.074(day^{-1}) and from 0.067(day^{-1}) to 0.052 (day^{-1}) and from 0.042 (day^{-1}) to 0.012 (day^{-1}) for U, AS and DAP respectively. There was a decline in the rate of reaction for the same treatment with time after about 13 days. The first order reaction rates decreased by 50%, 90%, 85% and 80% for U, U+S, AS, AS+S respectively. It increased by about 4% and 130% for DAP, DAP+S respectively.

The nitrogen fertilizers manufacturing industry is one of the most demanding industries for energy. The large increase in oil prices dramatically increases the fertilizers prices. This puts a stress on how to increase the fertilizers use efficiency.

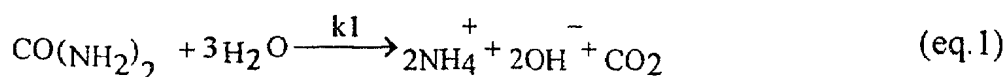
Sustainability of ecosystems also requires reducing the side effects of fertilizer application (e.g., nitrogen fertilization) such as groundwater pollution or air

pollution arising from ammonia volatilization and nitrogen oxides (greenhouse gases) due to denitrification. The losses through volatilization or leaching of nitrates through the soil profile have to be reduced and synchronization of the added fertilizers with the crops need has to be optimized. In the case of inorganic nitrogen addition, the rate of added nitrogen transformation to ammonium is quite rapid and lead to buildup of ammonia in the soil solution which leads to volatilization or rapid transformation to nitrates. A method of reducing this process in the soil solution is technically and economically required. Sulphur is agriculturally invited as one of the compounds used to reduce the buildup of ammonia concentration in the soil solution (Shahin & Soliman, 1998 and Hilal *et al.*, 1990).

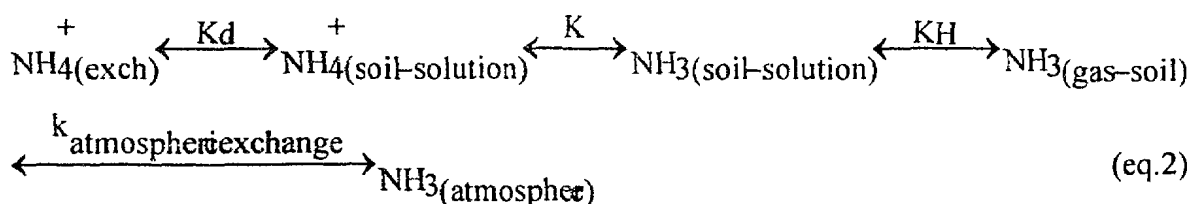
The aim of the present research is to study the effect of sulphur on reducing the ammonia volatilization from different kinds of N-fertilizers. This will be done through studying its effect on the first order rate kinetics of ammonia volatilization.

Theory

When urea and ammonium forms of fertilizers (such as ammonium nitrate, ammonium sulphate, ammoniated phosphate) are added to the soil, they may undergo a series of chemical conversions to ammonia. For urea, the hydrolysis of urea follows the reaction:



The ammonium released from the Urea or added as a fertilizer may then get adsorbed into the solid phase, dissolved in the soil solution or transform into ammonia which may be dissolved or get released into the gaseous phase of the soil which then escapes to the atmosphere rather than becoming a plant nutrient. The whole process is governed by a number of equilibria that may be represented as:



The first step is the equilibrium between adsorbed ammonium ion on the soil particles and the ammonium ion in the soil solution. This can be represented by a distribution coefficient. The second step is the equilibrium between the ammonium ion in soil solution and the dissolved ammonia in the soil solution.

The equilibrium between ammonium (NH_4^+) in soil solution and NH_3 in soil solution can be represented as:



The value of the equilibrium constant (k) depends on soil temperature. Different empirical equations have been presented to calculate its value at a given temperature (Emerson *et al.*, 1975 and Beuteir & Renon, 1978).

$$\log_{10} K = -0.09018 - \frac{2729.92}{T} \quad (\text{eq.4.1})$$

$$\ln(K) = -177.95 - \frac{1843.22}{T} + 31.434 \ln(T) - 0.0545T \quad (\text{eq.4.2})$$

Where T is the absolute temperature. Both equations yield the same values of pK. The values of pK are 10.1 at 0°C, 9.7 at 10 °C, 9.4 at 20 °C and 9.1 at 30 °C (Bates & Pinching, 1950). The relative proportion of NH_4^+ and NH_3 in solution at a given pH can be derived from eq. 5.

$$pK - pH = \log_{10} \frac{[NH_4^+]}{[NH_3]} \quad (\text{eq.5})$$

The third equilibrium is equilibrium between the dissolved ammonia in the soil solution to that in the soil air as dictated by eq.6 (Henry coefficient's law).

Ammonia is in equilibrium between liquid and gas phases according to Henry's law

$$K_h = \frac{C_{\text{ammonia, gas}}}{C_{\text{ammonia, liquid}}} \quad (\text{eq.6})$$

The final equilibrium is an exchange between the gaseous ammonia concentrations in the soil air to that in the layer of air above the soil. This is an aerodynamic exchange process.

Factors that promote these reactions toward the right increase ammonia volatilization. An excessive ventilation near the soil surface will surely deplete all the ammonia in the soil and shift the reaction toward the right (*i.e.*, toward the direction of volatilization).

Modelling Ammonia volatilization

The cumulative NH_3 volatilization as a function of time after urea application follows an exponential relationship reaching an asymptote as time approaches infinity. Different formulations of the exponential such as Logistic, or sigmoidal have been used to describe the shape of curve (Stevens *et al.*, 1989; Demeyer *et al.*, 1995 and Sommer & Ersboll, 1996). Hengirun *et al.* (1999) used a first order kinetic equation to simulate NH_3 volatilization from the soil surface.

$$A_t = A_0 \exp(-k_v t) \quad (\text{eq.7})$$

Where A_t is the total ammoniacal nitrogen remaining in soil at time t ; A_0 is the amount of ammoniacal nitrogen in soil at time $t=0$, k_v is the first order rate constant which account for the transformation to ammonia from urea and its volatilization.

The aim of this research is to determine the value of this first order rate constant and how it is affected by sulphur addition.

Material and Methods

Experiments

Equal amounts of Nitrogen (500 mg) in the form of Urea (U), ammonium sulphate (AS) and di-ammonium phosphate (DAP), either alone or mixed with 0.2% elemental sulphur were added to the surface of 250 gm of a sandy soil, placed in a separate canonical flask, each with a capacity of 500 ml. The characteristics of the soil are given in Table 1.

TABLE 1. The most important characteristics of the used sandy soil.

Properties	Value	Properties	Value
EC(1:1) (dS/m)	0.8	CEC (Cmol/kg)	6.31
pH (1:1)	8.5	Total N (mg/kg)	34.8
(Silt + clay) %	8.6	Available P (mg/kg)	1.85
CaCO ₃ %	3.25	Field Capacity (%wt)	22.4

Distilled water was then added to the soil in each flask to maintain each soil in a state of 100% field capacity. Each flask was then sealed and ammonia free air was then passed through the flasks in the rate of 1.5 L/min. Ammonium free air was prepared by passing air through 0.5 M H₂SO₄. This rate represents a ventilation rate of 3 times the volume of each flask per minute. The whole system was placed in a temperature controlled incubator. The temperature was set at 30±2 °C. Fig.1 is a schematic diagram for the closed system and its ventilation unit. This system was used to continuously remove the volatilized ammonia produced from the hydrolysis of urea and the ammonia volatilized from the tested fertilizers. The volatilized ammonia was received in 50 ml boric acid (2% concentration). The acid was then changed on intervals of 1, 2, 3, 6, 7, 9, 13, 20, 27, 34 and 48 days. The soil moisture was maintained at 100% field capacity by adding distilled water during the execution of the experiment. Ammonia received in the boric acid, was titrated against sulphuric acid (0.05 N) using an automatic pipette for each period. At the end of the experiment, 50 gm of soil in each flask were taken to determine the pH (1:1 extract), EC (1:1), dissolved ammonium by the methods suggested by Keeney & Nelson (1982) and Olsen & Sommers (1982). Sulphate was determined by the use of turbidity technique (Rainwater & Thatcher, 1960).

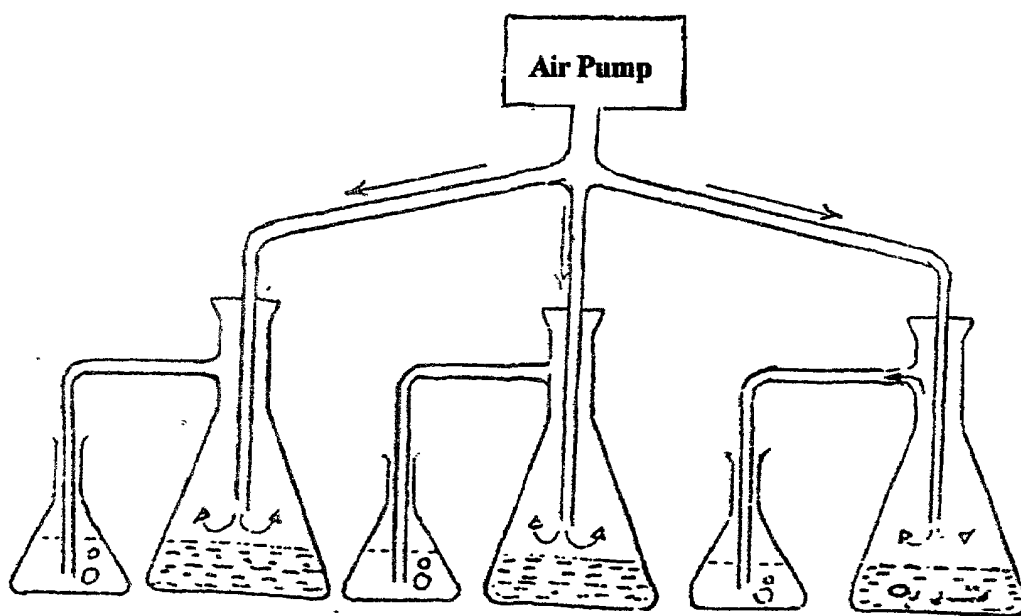


Fig.1. The set up of the experiment.

The experiment was repeated once more on the same soil in each flask by adding the same amount of fertilizers without adding the elemental sulphur. The same procedures were followed to study the residual effect of sulphur on the volatilization of ammonia. The soil moisture and temperature were maintained at 100% field capacity and 30 ± 2 °C during the whole length of the experiment. The volatilized ammonia was then determined for the same periods as the first experiment.

Results and Discussion

Figure 2. a represents the cumulative volatilized ammonia from the tested N-fertilizers up to 48 days of incubation. It is shown from Fig.2a that the cumulative volatilized NH_3 gas versus time has a nonlinear relationship with different rates for the tested fertilizers. Fig.2.b shows the fraction of ammonia remaining in the flask, as a function of time for the tested N-fertilizers. The data also indicated that the addition of sulphur slowed down the rate and reduced the total amount of ammonia volatilization from different N-fertilizers.

The percent of reduction in total NH_3 loss due to sulphur was calculated (Watson, 1990 and Watson *et al.*, 1992) as follows

$$\% \text{ inhibition} = 100(C-T)/C \quad (\text{eq.8})$$

Where

T = Volatilized NH_3 with sulphur.

C = Volatilized NH_3 without sulphur.

The obtained inhibition values were 27% for AS, while the lowest 5.7% was recorded for DAP.

In order to investigate the first order rate kinetics of N-volatilization, the data of Figure 2 a and b was recalculated. It is shown from Fig 2 b the fractional amount of ammonia remaining in the flask is decreasing exponentially as a function of time. This is an indication of the first order behavior of the system. Plotting the value of the natural logarithm of the concentration of the remaining quantity of ammonium, divided by its initial concentration versus time, should yield a straight line with a slope of $-k_1$ (the reaction rate constant).

$$-\ln \frac{a-x}{a} = k_1 t \quad (\text{eq.9})$$

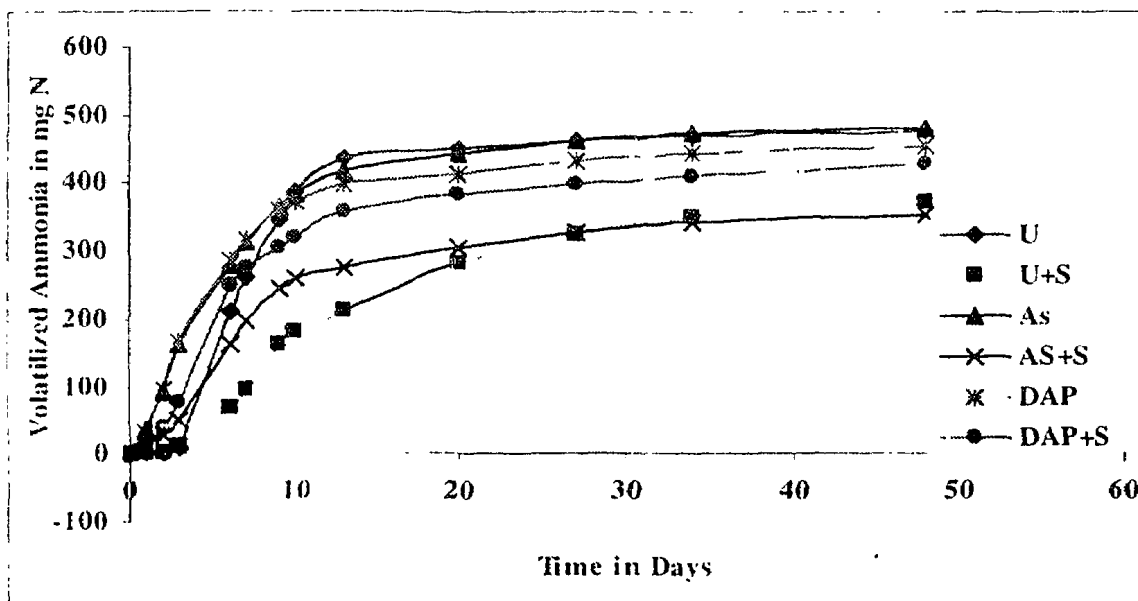


Fig. 2a. The cumulative volatilized ammonia from the tested N- fertilizers as affected by sulphur application.

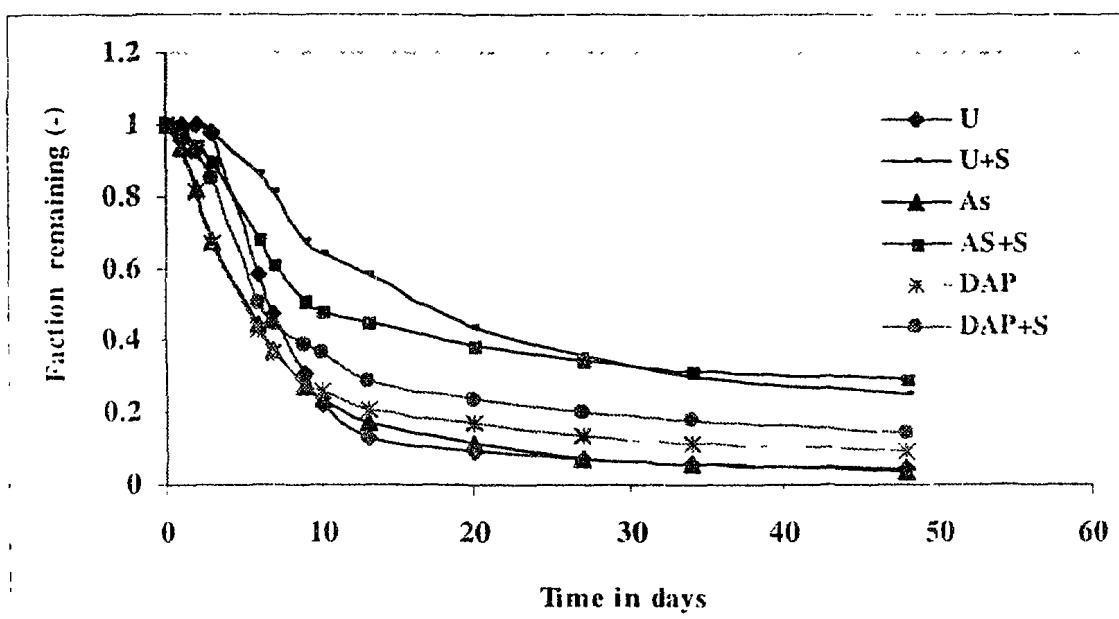


Fig. 2b. The fraction of ammonia remaining in the soil as a function of time for the tested N- fertilizers as affected by sulphur application.

Where a is the amount original ammonia in the fertilizer, x is the amount volatilized and $a-x$ is the amount of ammonia remaining.

Figure 3,4 and 5 represent the attained linear model and the slope of the straight line is the value of the first order reactions rate as shown on each graph. Two stages of NH_3 volatilization were detected. The first stage, up to 13 days has higher rate, followed by a second stage with low rate up to 48 days.

In general, it is shown from Fig. 3,4 and 5 that sulphur addition led to a decrease in the first order rate kinetics. A higher value of the slope indicates a larger k_1 value and indicates higher volatilization for the same concentration. For all fertilizers used, the sulphur has a negative effect on the k_1 value, *i.e.*, it led to a decrease in the rate of volatilization.

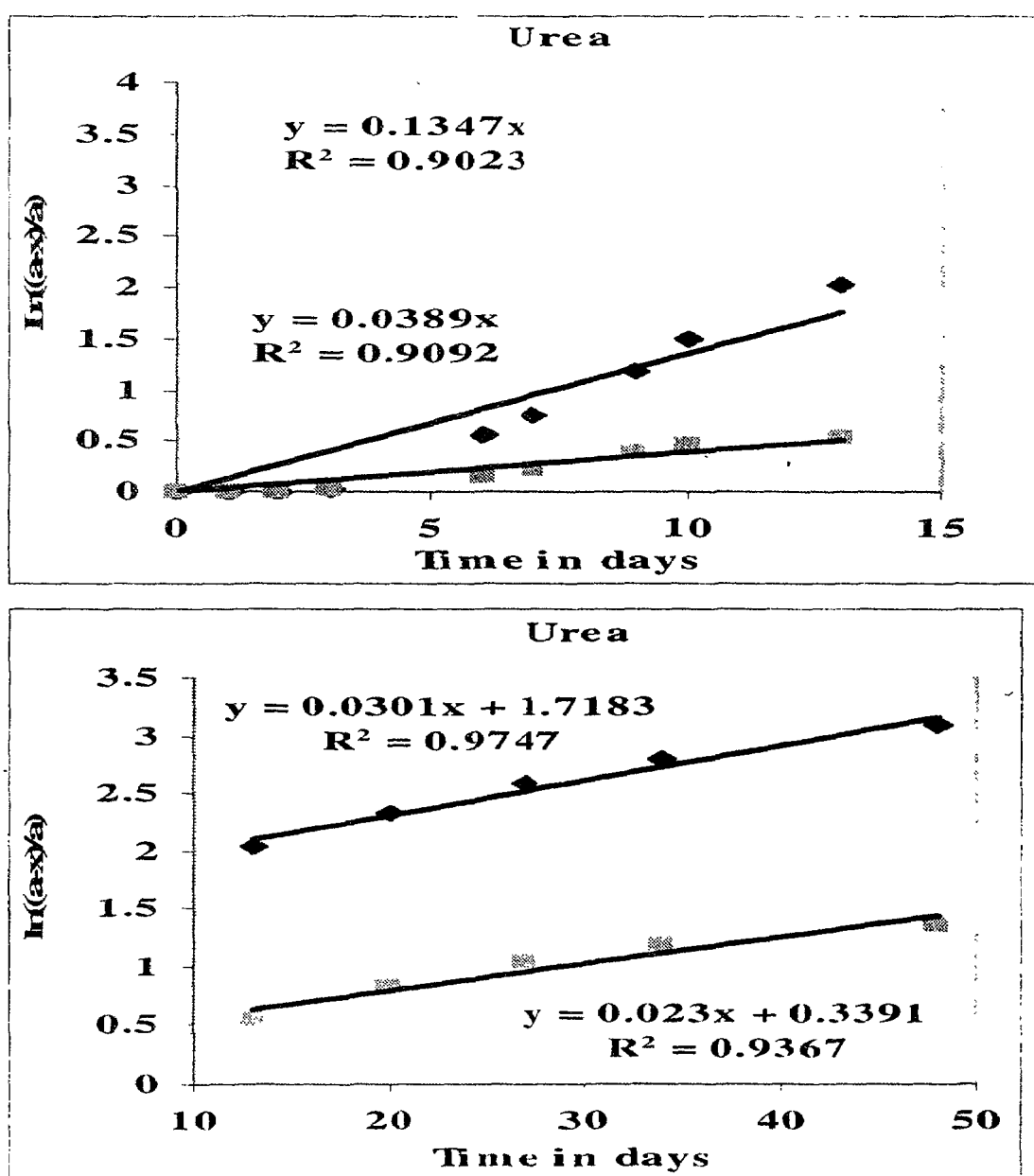


Fig. 3a,b. The effect of sulphur on ammonia volatilization from Urea with sulphur (♦) and No sulphur (◻).

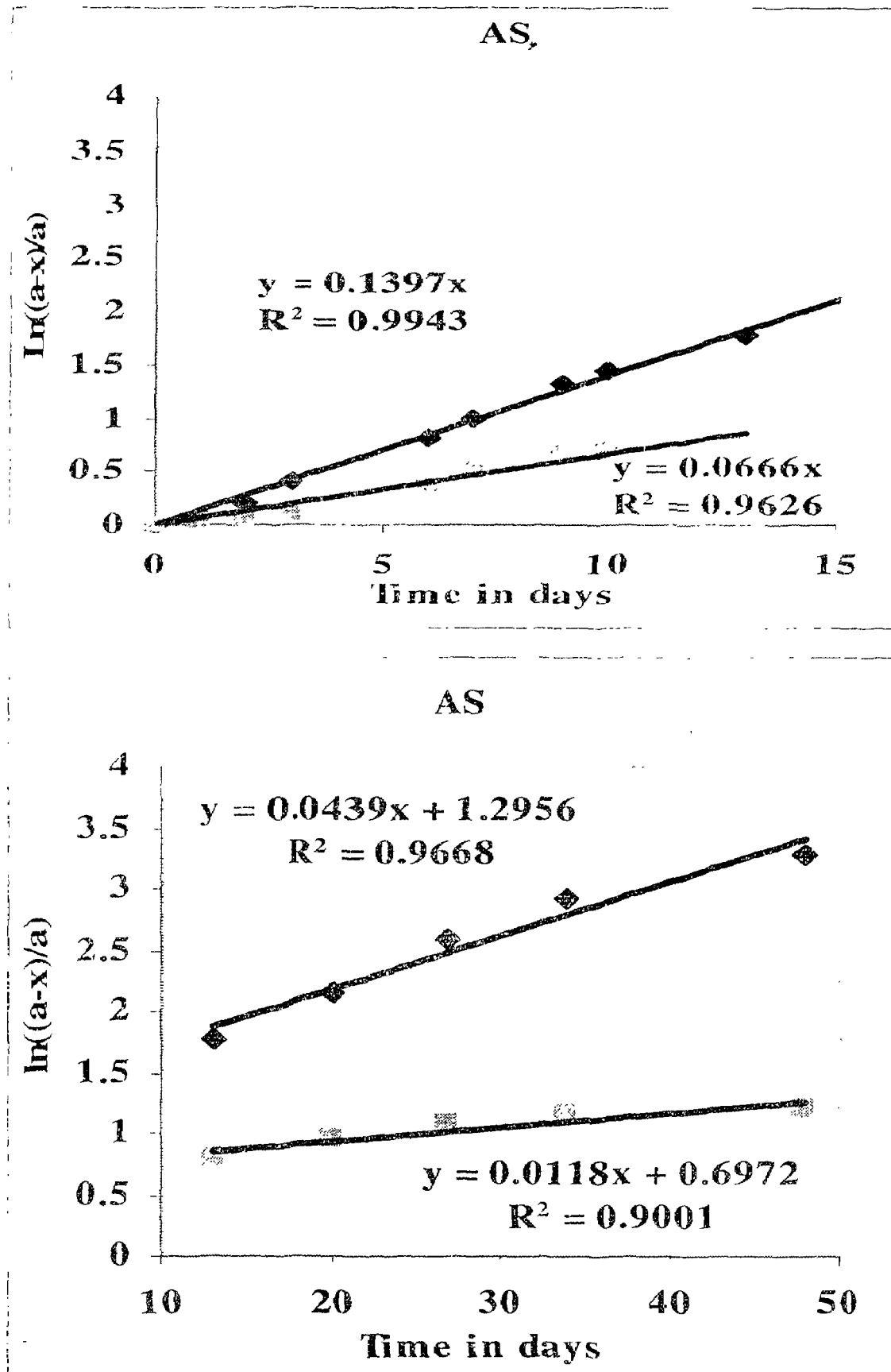


Fig. 4a, b. The effect of sulphur on ammonia volatilization from ammonium sulphate with sulphur(♦)and No sulphur (*).

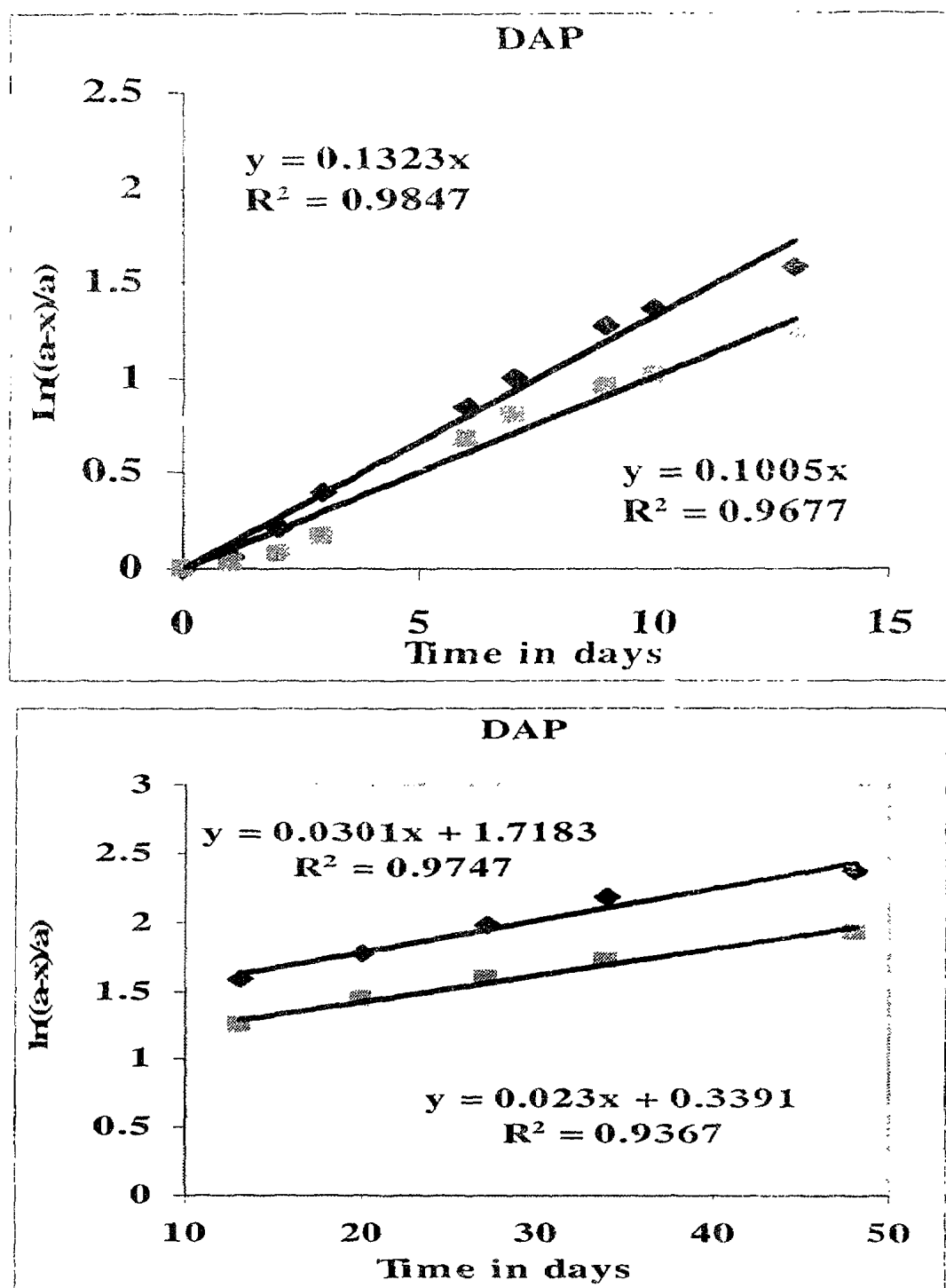


Fig. 5a, b. The effect of sulphur on ammonia volatilization from Diammonium phosphate with sulphur (*) and No sulphur (♦).

Table 2 shows the values of first order rate reactions for all the fertilizers without sulphur (k_0) and with sulphur (k_s). In the columns after every fertilizer (without and with) there is a column which expresses the ratio of the kinetic rate with sulphur over that without sulphur (k_s/k_0). In the rows, the last row expresses the ratio of the kinetic rate at the early stages of reaction (k_a) over that at later stages (k_b).

TABLE 2. The values of the volatilization first order rate reactions.

	Urea(k_0)	Urea + S(k_0)	$(k_0/k_0)_U$	AS(k_0)	AS+S (k_0)	$(k_0/k_0)_{AS}$	DAP(k_0)	DAP+S (k_0)	$(k_0/k_0)_{DAP}$
Early stages (day ⁻¹) (k_a)	0.135	0.039	0.289	0.140	0.067	0.477	0.132	0.101	0.761
Later stages (day ⁻¹) (k_b)	0.030	0.023	0.764	0.044	0.012	0.269	0.030	0.023	0.764
(k_a/k_b)	4.48	1.69		3.18	5.64		4.39	4.37	

When comparing the values of the first order rate reactions, it seems that there was a large decrease in the rate of reaction for all fertilizers with sulphur and without sulphur. It was not possible to obtain a high value of R^2 in the curve fit for the relationship between $\ln((a-x)/x)$ versus time for the whole period (not shown here).

The lowest ratio of (k_a/k_b) was for urea (0.289) and increased in the case of AS (0.477) while it became the highest in the case of DAP fertilizer. This means sulphur has the most effect in case of Urea and became less for AS and even less for DAP.

The decline in the rate of reaction, with time after 13 days, could be due to the accumulation of products of reaction. The decrease was much higher in the case of Urea in comparison to Urea+S (*i.e.*, 4.48 in comparison to 1.69). This could be due to the effect of sulphur on reducing the pH of the media and so it neutralized the effect of ammonia release on increasing the soil pH.

In the case of AS, sulphur addition increases the decline in rate of reaction with time. The obtained data showed that AS fertilization has an acidic reaction in the soil. It lowers the soil pH from 8.5 to 6.96. AS+S treatment lowered the soil pH (1:1) from 8.5 to 6.67. For the case of DAP, there was no effect for sulphur on the rate of decrease of reaction with time (4.39 *vs.* 4.37). The decrease in the reaction rate was the same for both DAP and DAP+S.

The data presented in Table 3 showed the total effect of sulphur blended N-fertilizers, at the end of the experiment, on reducing soil pH, increasing soil EC which may be attributed to the oxidation sulphur to sulphuric acid and the production of sulphate ion and the increase of sulphate salinity in the soil. In addition, the amount of NH_4^+ retained in the soil was increased under the effect of sulphur.

TABLE 3. The total effect of sulphur blended N-fertilizers on soil chemical properties.

	pH	EC(1:1)	NH ₄ ⁺ (mg/kg)	SO ₄ ⁻ (mg/kg)
U	8.5	7.62	65.43	21.76
U+S	7.47	9.15	77.36	38.43
AS	6.96	12.87	72.15	46.98
AS+S	6.67	14.82	94.25	69.11
DAP	7.84	7.25	61.83	22.74
DAP+S	7.41	10.05	89.35	46.59

The effect of sulphur could be understood through an appreciation of its effect on decreasing the pH of the media due to its oxidation to sulphuric acid. This lowers the pH of the soil solution which leads to an increase in the left hand side of eq.5, repeated here for clarity:

$$\pi_K - \pi_H = \lambda \log_{10} \frac{[\text{NH}_4^+]}{\text{NH}_3} \quad (\text{eq.5})$$

Which means a larger value of the logarithm of ammonium ion in the solution with respect to the ammonia gas in the solution. So, the result is a decrease in the concentration in the ammonia in the soil solution which reduces the ammonia gas in the gaseous phase of the soil as dictated by eq.6 (*i.e.*, Henry's law). The pH has also an effect on shifting the reaction given by eq.1 generating ammonium ion to the right. So, the increase in the ammonium concentration in the numerator of eq.5 is not only due to the effect of pH decrease as calculated by eq.5 but also due to the effect of the lowering of the pH on shifting reaction 1 to the right. These two effects are balancing each other but it seems that the first effect dominates and volatilization is reduced. There is also the effect of pH on the activity of urease enzyme. Wyszowska *et al.* (2001) stated that 0.1% elemental sulphur reduced NH₃ volatilization through lowering soil pH from 7.0 to 6.2 which decreased urease enzyme activity by 50% to 75 %.

The residual effect of sulphur on ammonia volatilization

Figure 6 showed the residual effect of sulphur on the NH₃ volatilization from the second dose of the applied N-fertilizers. The initial effect of sulphur reduced volatilized-NH₃ by 52.6 mg NH₃/kg soil (DAP) up to 130.3 mg NH₃/kg soil (AS).

This initial amount was decreased by the residual effect of sulphur to only 25.83 mg NH₃/kg soil (DAP) and 36.2 mg NH₃/kg soil (AS). It is clear from Fig. 6, 7a and b, 8 a and b and 9 and b, showing the results of the second experiment for the tested fertilizers, that sulphur has a residual effect on ammonia volatilization. This could be due to the fact that the used soil is a sandy soil and its buffer capacity is low and all the acidity produced in the flask remained there and was not neutralized by the low buffer capacity of the soil (Ferguson *et al.*, 1984).

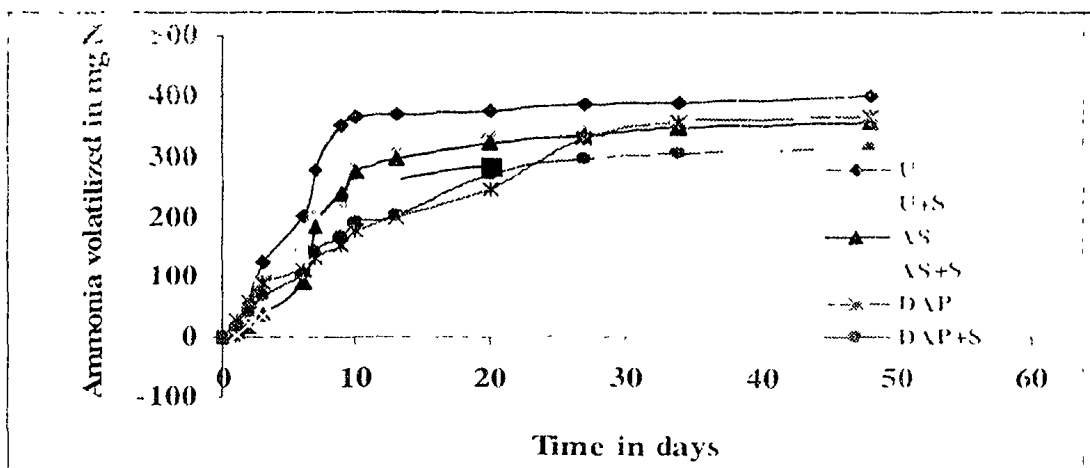


Fig.6. The volatilized ammonia from different kinds of fertilizers as affected by the residual effect of sulphur.

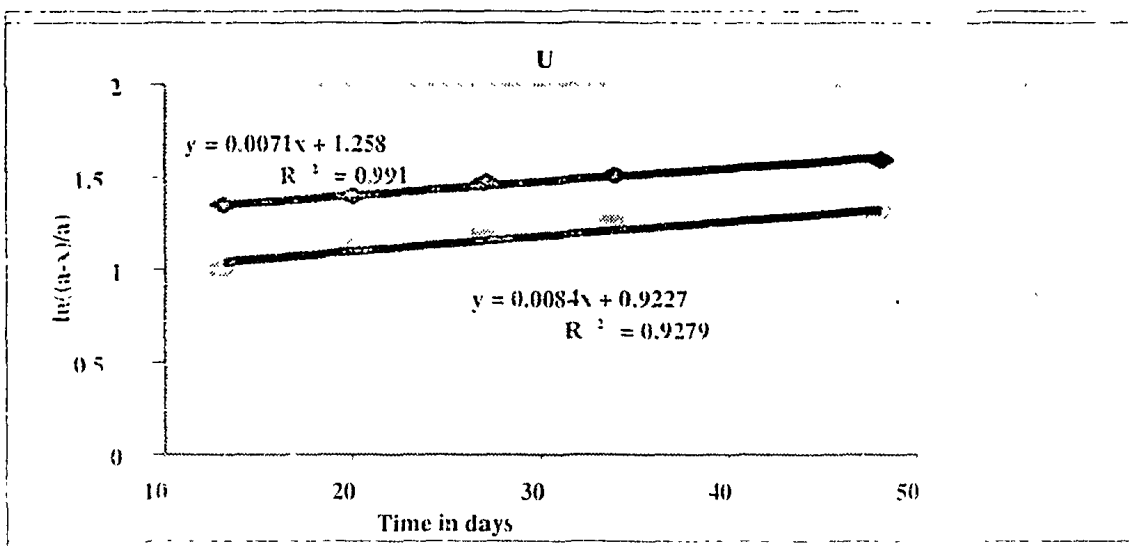
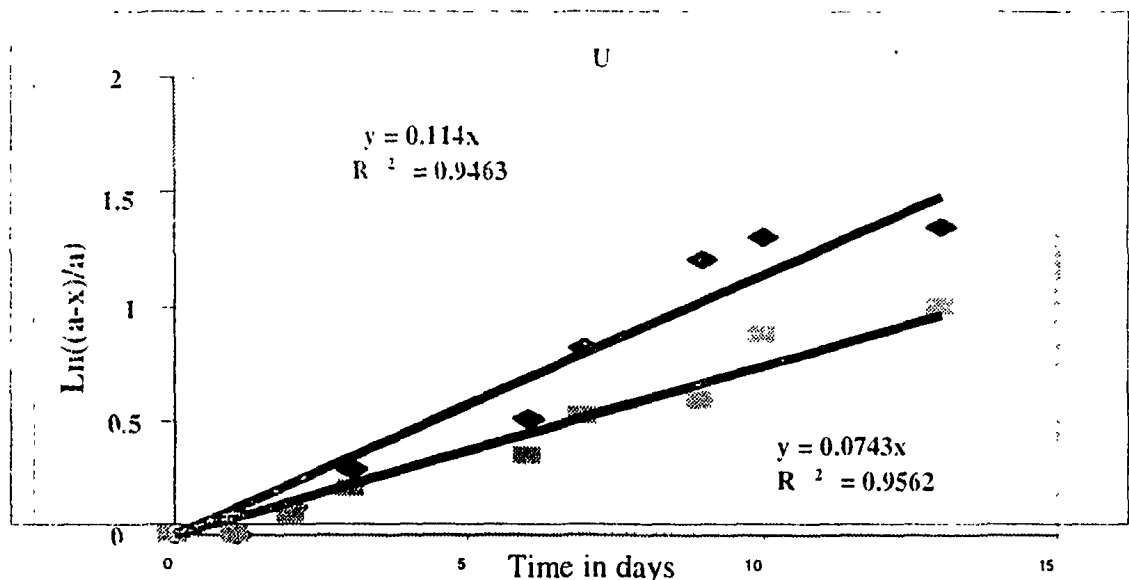


Fig. 7. a, b. The effect of sulphur on ammonia volatilization from urea with sulphur (♦) and No sulphur (◻).

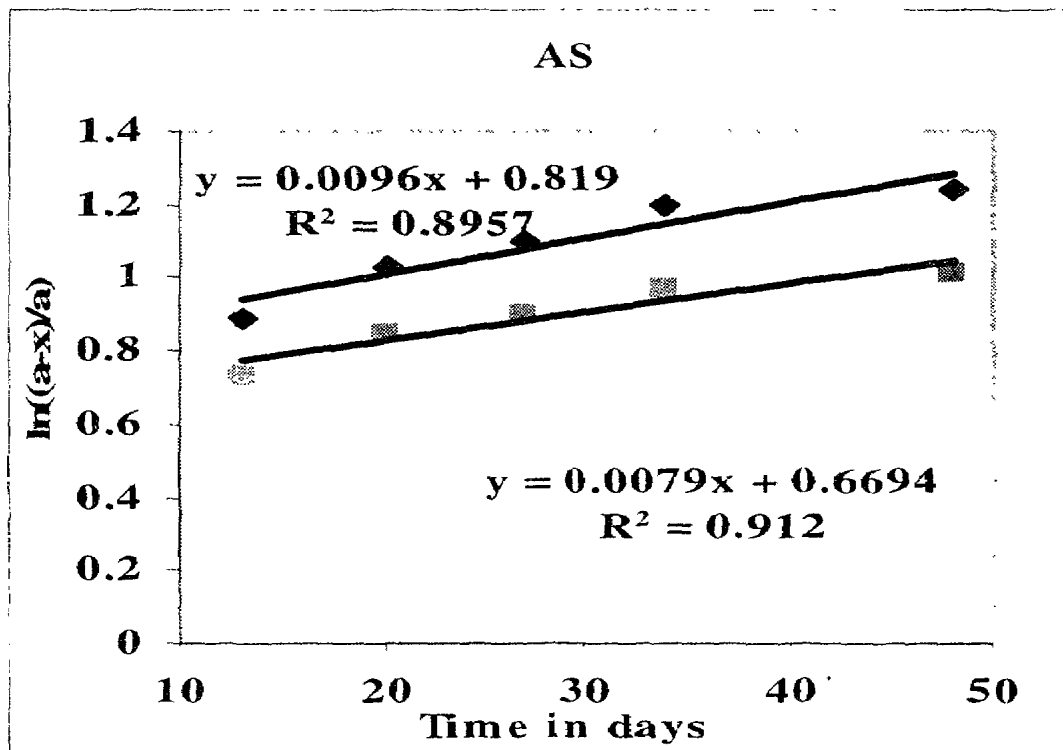
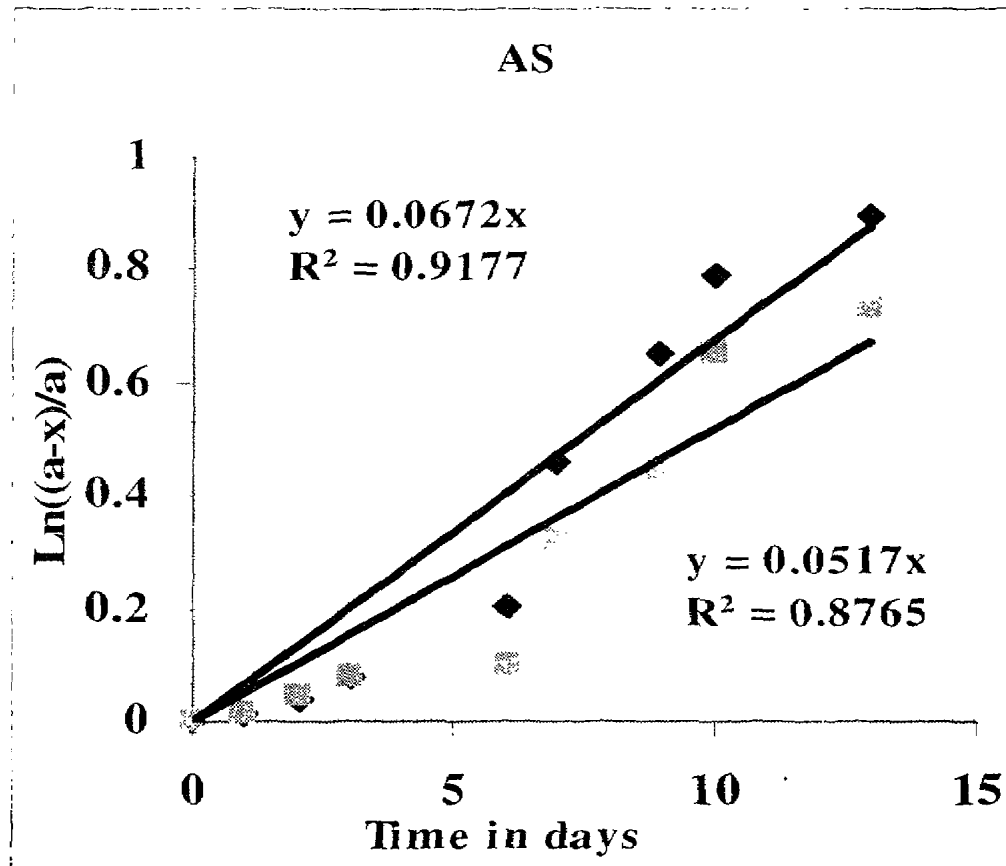


Fig. 8a, b. The effect of sulphur on ammonia volatilization from ammonium sulphate with sulphur (♦) and No sulphur (•).

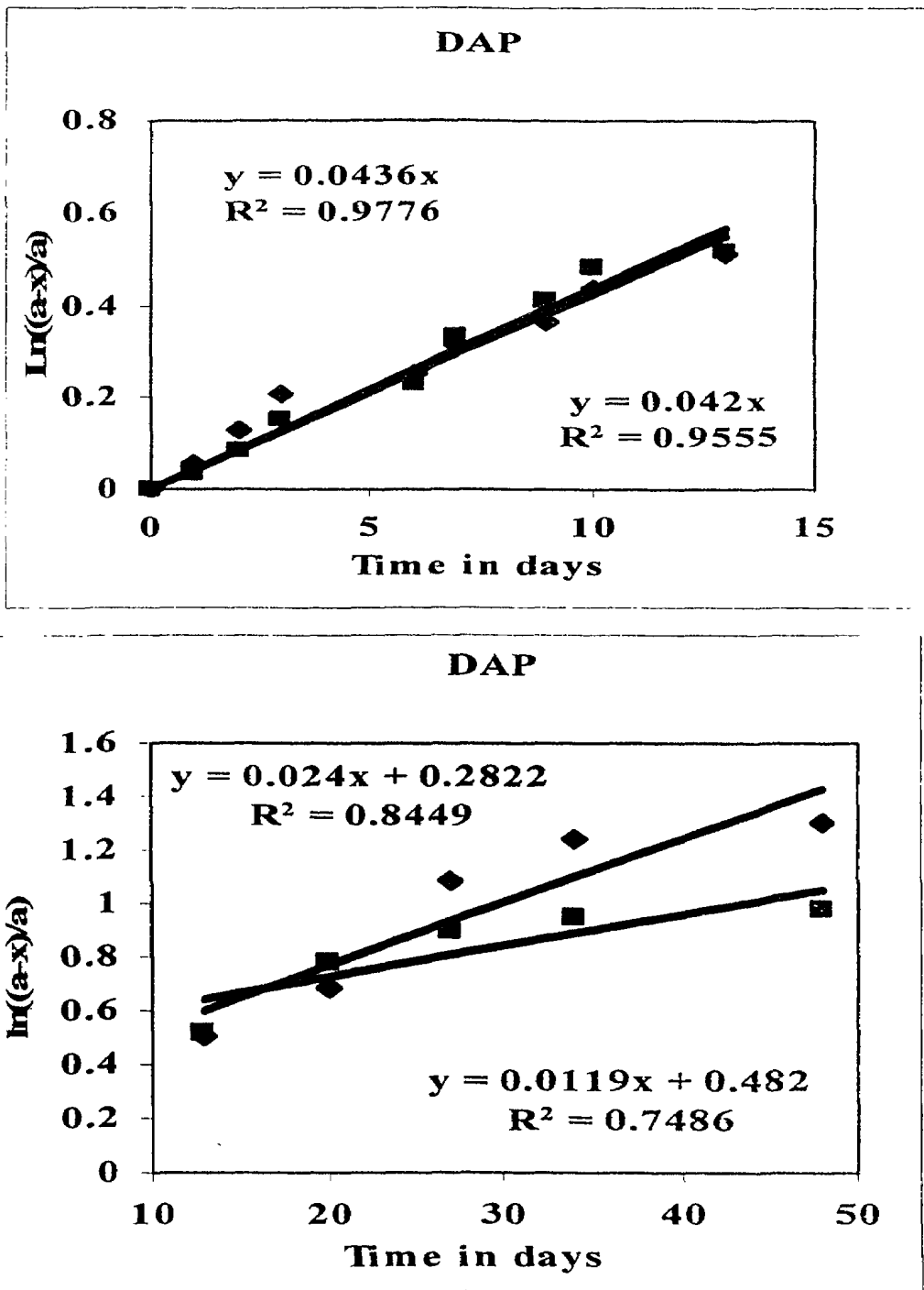


Fig. 9a,b. The effect of sulphur on ammonia volatilization from diammonium phosphate with sulphur (♦)and No sulphur (■).

TABLE 4. The residual effect of sulphur on volatilization.

	Urea (k ₀)	Urea+S (k _s)	(k _s /k ₀) _U	AS (k ₀)	AS+S (k _s)	(k _s /k ₀)	DAP (k ₀)	DAP+S (k _s)	(k _s /k ₀) _{DAP}
Early stages (day ⁻¹) k _a	0.114	0.074	0.652	0.067	0.052	0.769	0.042	0.012	0.283
Later stages (day ⁻¹) k _b	0.007	0.008	1.183	0.010	0.010	1.010	0.044	0.042	0.963
(k _s /k ₀)	16.06	8.85		7.00	5.33		0.96	0.28	

In Table 4, sulphur had an effect on decreasing the volatilization of urea but it was less than in the first experiment. This could be seen by comparing the value of (k_s/k_o) for urea in 0.652 in Table 4 .vs. (k_s/k_o) for urea 0.289 in Table 2. For the case of AS the (k_s/k_o) was 0.477 in Table 2 .vs. 0.769 in Table 4. For DAP, (k_s/k_o) was 0.283 in Table 4 .vs. 0.761 in Table 2. There was more reduction in this ratio. But one should look at the absolute ratios of the reaction. It was very low at the early stages of the second experiment with respect to the early stages of the first experiment for both DAP (0.042 vs. 0.132) and DAP+S (0.012 .vs. 0.101). There was a reduction in the rate of volatilization reaction in early stages of the second experiment for U and AS. Sulphur was less effective than in the first experiment. The ratios (k_s/k_o) was less than one but not so much low as the first experiment. In the later stage of experiment 2, comparing the ratio of (k_s/k_o) (second row of column 3,6 and 9 in Table 4), it became more than one in case of urea(1.183), 1.01 for ammonium sulphate and very close to one (0.963) in case of Dia-ammonium phosphate signalling the end of the sulphur effect.

Conclusions

An incubation experiment was conducted to study the effect of sulphur addition to three kinds of N-fertilizers; Urea (U), Ammonium Sulphate (AS), and Di-ammonium Phosphate (DAP), in an incubated flask, on the loss of ammonia by volatilization from these fertilizers applied to a sandy soil. A kinetic analysis of the reaction rates was done to determine the first order reaction rate for ammonia volatilization. The sulphur addition led to a decrease in the ammonia volatilization first order rate constants. There was also a decline in the rate of reaction for the same treatment with time after about 13 days. By repeating the experiment again with adding a second dose of fertilizers without adding extra sulphur, there was a residual effect for sulphur on ammonia volatilization. There was also a decline in the rate of reaction for the same treatment with time after about 13 days for Urea, Ammonium sulphate respectively. At the later stage of the second experiment, there was no difference between the first order rate reaction constants signaling the end of the sulphur effect.

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حركات تطاير الأمونيا من الأسمدة النتروجينية وتأثرها بالكبريت

رضا رجب شاهين ورشدي محمد محمد الكيلاني
قسم الأراضي والمياه – كلية الزراعة - جامعة القاهرة- مصر.

لقد تم إجراء تجربة تحضين لمدة ٤٨ يوماً علي أرض رملية لدراسة تأثير إضافة الكبريت لثلاث أنواع من الأسمدة علي فقدان الأمونيا بالتطاير من تلك الأسمدة. ولقد كانت الأسمدة المستخدمة هي يوريا وسلفات الأمونيوم وفوسفات ثنائي الأمونيوم ، إما مفردة أو مخلوط مع ٢٠٪ كبريت عنصرى ، ولقد تم استقبال الأمونيا في حامض يوريك وتقديرها بالمعايرة.

ولقد تم إجراء تحليل حركي لمعدلات التفاعل لتقدير معدل التفاعل من الدرجة الأولى لتطاير الأمونيا. ولقد أدت إضافة الكبريت إلي نقص في ثوابت الدرجة الأولى لتطاير الأمونيا. ولقد تناقص ثابت التفاعل الأول من ١٣٥٠ (يوم^{-١}) إلي ٠٣٩ (يوم^{-١}) ومن ١٤ (يوم^{-١}) إلي ٠٦٧ (يوم^{-١}) ومن ١٣٢ (يوم^{-١}) إلي ١٠١ (يوم^{-١}) لسماذ اليوريا وسلفات الأمونيوم وفوسفات ثنائي الأمونيوم علي الترتيب. ولقد كان هناك تناقص في معدل التفاعل لنفس المعاملة بعد حوالي ١٣ يوم. ولقد تناقص معدل الدرجة للتفاعل من ١٣٥٠ (يوم^{-١}) إلي ٣٠٠ (يوم^{-١}) ومن ٠٣٩ (يوم^{-١}) إلي ٠٢٣ (يوم^{-١}) ومن ١٤٠ (يوم^{-١}) إلي ٠٤٤ (يوم^{-١}) ومن ٠٦٧ (يوم^{-١}) إلي ٠١٢ (يوم^{-١}) ومن ١٣٢ (يوم^{-١}) إلي ٠٣٠ (يوم^{-١}) ومن ١٠١ (يوم^{-١}) إلي ٠٢٣ (يوم^{-١}) لليوريا ، اليوريا + كبريت ، فوسفات ثنائي الأمونيوم وفوسفات ثنائي الأمونيوم + كبريت علي وجه الترتيب.

ولقد تم إعادة التجربة مرة أخرى بدون إضافة كبريت. ولقد قلل التأثير المتبقي للكبريت نسبياً مع معدلات الدرجة الأولى للتطاير من ١١٤ (يوم^{-١}) إلي ٠٧٤ (يوم^{-١}) ومن ٠٦٧ (يوم^{-١}) إلي ٠٥٢ (يوم^{-١}) ومن ٠٤٢ (يوم^{-١}) إلي ٠١٢ (يوم^{-١}) لليوريا ، سلفات الأمونيوم وفوسفات ثنائي الأمونيوم علي وجه الترتيب. ولقد كان تناقص في معدل التفاعل لنفس المعاملات بعد مرور ١٣ يوم وقد تناقص معدلات الدرجة الأولى بمقدار ٥٠٪ ، ٩٠٪ و ٨٥٪ لليوريا ، يوريا+ كبريت ، سلفات الأمونيوم ، سلفات الأمونيوم + كبريت علي الترتيب. ولقد تزايدت بنحو ٤٪ ، ١٣٪ لفوسفات ثنائي الأمونيوم وفوسفات ثنائي الأمونيوم + كبريت علي الترتيب.