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Research Article

Effect of paper mill sludge (furfural) on soil respiration, urease activity and ammonia volatilization of calcareous soils

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Abstract

Final paper mill sludge (furfural) is a good material for C Source and other nutrients for soil. Incubation tests were done to see how different amounts of furfural affected the release of CO2, urease activity and NH_3 volatilization of two soils (Alzubair and Abul-khasib) collected from Basrah province, south of Iraq. Furfural was applied at 0, 0.5, 1.0, 1.5, 2.0, 4.0, 6.0, 8.0 and 10 % (w/w) of dried soil. The mentioned parameters were measured at 10, 20 and 30 days of incubation time. The furfural rate linearly increased CO_2 evolution till the concentration of 4 %. Increasing furfural concentration beyond 4 % did not affect CO_2 evolution. In contrast, the urease activity was enhanced in the 0-10 % treatments and no clear differences were observed beyond 1.0 %. However, the addition of furfural by causes linearly decreased NH_3 volatilization at all incubation times and in both soils. Control and furfural treatments in Abul - khasib soil gave higher CO_2 evolution and urease activity and lower NH_3 loss than AL - Zubair loamy sand soil.

Keywords: Furfural, paper mill sludge, urease, CO_2 evolution, NH_3 loss.

Introduction

The expansion of agriculture to cover the growing needs of the population to food combined with the negative effect of the biggest problems people have to deal with is climate change. One way to solve this problem is to slow down the decline of the soil, especially in dry and semi-dry areas. That required more attention because of extreme weather conditions. Soils in the south of Iraq have low levels of organic matter because of the weather and the way they are usually farmed and hence decline in their quality. By adding more organic carbon to the soil, organic residues can improve its physical, chemical, and biological qualities improving soil structure and then ensuring an increase in water-holding capacity and nutrient availability (Ilay et al., 2013).

Disposal of industrial wastes in agriculture become a beneficial practice to ensure a cheap source of exogenous organic C and reduce environmental pollution instead of landfilled at the production site or improperly disposed of land application of these residues to plants represents a beneficial alternative to disposal (Camberato et al., 1997). In the province of Basrah, south of Iraq, approximately one ton of paper sludge (furfural) of each 10 tons of corn pods are produced as final produce in a state paper mill. (Gagnon et al., 2000) said that the pulp and paper businesses make a lot of organic waste that has a lot of carbon because it has a lot of cellulose. (Camberato et al., 1997) said that the main sludges from a paper mill don't have many plant nutrients, like N, and have high C/N ratios. On the other hand, the secondary sludges have more N and P and lower C/N ratios. Based on data from individual paper companies, heavy metals in paper production sludges mean that they are rarely considered dangerous materials (Thacker, 1984). Biochemical markers are more aware of changes in soil qualities that happen because of how management affects the health and fertility of the soil (Nayak et al., 2007).

Soil respiration and enzyme activities can be used as early warning signs for the change of organic matter, which in turn can help with nutrient cycles and plant nutrition (Iovieno et al., 2009). Many studies have shown that adding organic matter to soil raises urease activity and soil respiration (Gagnon et al., 2003; Vicena et al., 2022). The loss of ammonia from farm areas is a big problem because it's a plant food source, so it is important economically to farmers (Pote & Meisinger, 2014). Ammonia volatilization losses occur when prilled urea contact with the particles of the soil of PH > 7 (Trenkel, 1997). The addition of organic residues can minimize ammonia loss through adsorbing ammonia by its functional acid groups or through its high CEC, as well as the ability of organic residues to alter soil pH (Taghizadeh-Toosi et al., 2012). (Vanin et al., 2013) found a 35 % reduction in ammonia volatilization upon using poultry litter. (Al-Tameemi et al., 2014) also obtained a precious reduction in ammonia losses in soils treated with HA compared with urea alone.

Locally, no research has been done on how paper sludge affects living things indicators as well as ammonia loss by volatilization. The point of this study was to look at what happens when a lot of different kinds of paper mill sludge (furfural) are mixed concentrations on $C0_2$ evolution, urease activity and ammonia volatilization in two calcareous soils.

Material and methods

Soil and furfural collection

(0–30 cm) of loamy sand dirt from the ground's surface and soil were collected from Al - Zubair and Abul-khasib regions, Basrah province respectively. Samples were left to dry in the air at room temperature, then ground up and put through a 2mm screen to be analyzed and put into an incubator. Table 1 shows some of the chemical, physical, and biological features of the soils that were used. These were found by following the steps outlined by (Black, 1965; Page, 1982). Furfural raw material as an organic side product of the paper production process was collected from a state paper mill, North of Basrah. Furfural was dried at 50° C, crushed, sieved through a 1mm sieve and stored in a plastic bag. Some of the furfural's chemical properties were found using the methods explained by (Page, 1982), which are shown in Table 1.

Incubation Experiments set up

Biological activity of soil (CO_2 evolution)

250g of collected soil samples were placed in 1000 cm^3 glass jars. Furfural was thoroughly mixed with dirt at rates of 0, 0.5, 1.0, 1.5, 2.0, 4.0, 6.0, 8.0 and 10 % based on the dry weight of soil. Urea fertilizer (46%N) was added to the soil at a rate of 1000 mg N kg-1 soil. The moisture content of the mixed sample was adjusted at field capacity. Vail (50 Cm^3) filled with 10 ml of 0.5*N* NaOH solution and laid at the surface of the soil for each jar, and then the jars were sealed tightly to prevent CO_2 leakage. After that, The jars were kept warm in a room that controlled the temperature to 30°C. Three times, the treatments were done again. The amount of water was checked from time to time, and purified water was added as needed.

After 10, 20 and 30 days of incubations a set of jars was withdrawn and CO_2 evolution was determined by titration of the vial Content with standard Cl solution using a phenolphthaline indicator (Nannipieri et al., 1979).

Urease activity in soil

The preparation of soil-furfural mixtures was similar to the experiment described for the CO_2 evolution essay, except for using 5 g of soil. Each treatment was replicated three times. This method from (Tabatabai & Bremner, 1972) was used to test urease activity in a group of samples after 10, 20, and 30 days of incubation. Soils that had been changed were mixed with 9 ml of THAM buffer, 0.2 ml of toluene, and 1 ml of 0.25 M urea solution. This was done at 37℃ for two hours. KCl–Ag2SO4 solution stopped urease from working after it had been incubated. The amount of NH+4-N that was released was tested using steam distillation and given as µg NH+4-N g-1 soil 2h-1.

NH_3 volatilization

As for the CO_2 evolution experiment, 250 g of soil-furfural mixtures were prepared.Each treatment was replicated three times.Soil - air chamber method was used to measure the daily NH_3 volatilization (Fenn & Kissel, 1973). The amended soil samples were placed in conical flasks of 500 cm² volume and tied up with the chamber. A vacuum pump of 2 L air min^{-1} flask $^{-1}$ was used as a power source to replace the volatile NH_3. Then, 20 ml of 2 % boric acid-containing indicators was used as an absorbent solution for NH_3 absorption. Finally, 0.005 M HCL was added to a solution of boric acid to measure how much NH3 was released into the air.

Soil pH

The preparation of soil- furfural mixtures was similar to the CO_2 evolution experiment, except for using 20 gm of soil. Each treatment was replicated three times. After 3,15 and 30 days of incubation, a set of samples was used to measure pH with Lab pH analyzer by adding 20 ml of distilled water (1:1 w / w ratio).

Table 1. Some characteristics of paper mill sludge (Furfural) and Soils used.

Component	Furfural	Al-Zubair soil	Abul-Kasib soil
Water content (%)	7.10	-	-
pH*	8.30	8.00	7.78
EC* (dSm^{-1})	4.55	2.93	6.01
Organic matter (g kg^{-1})	337.67	0.50	6.66
Total N (g kg^{-1})	12.60	0.04	0.38
Phosphorus#(g kg^{-1})	5.30	-	-
C: N	15.54	7.25	10.16
C:P	36.95	-	-
Urease (µgNH^+_4-N g^{-1} soil $2hr.^{-1}$)	-	30	80
Texture	-	Loamy sand	Silty clay

* The EC and pH were found in a solution of 1:5 and 1:1 for Furfural and soil, respectively.

p was estimated as the total content for Furfural and available content for soils.

Data analysis

The two means were used on all measurements. Gen Stats 18.2 was used to analyze variance (ANOVA) to evaluate the effects of furfural concentrations in combination with incubation time on CO_2 evolution, urease activity and NH_3 volatilization. To compare means, the new least significant difference was found at a significance level of 5%.

Results and Discussion

Soil biological activity (CO_2 evolution)

The effect of adding furfural at different concentrations on CO_2 evolution in both soils is illustrated in Fig. 1. It was determined that CO_2 evolution increased till it reached maximum values at a concentration of 4% furfural at both soils and different incubation periods. Increased furfural concentrations beyond 4% seem to be stable. For Al-Zubair soil, CO_2 amounts increased from 1661 mg CO_2 kg^{-1} soil for control treatment to 2900.33 mg CO_2 kg^{-1} soil for 4% furfural treatment and Abul Khasib soil, CO_2 increased from 2013.66 mg CO_2 kg^{-1} soil for control treatment to 2866.33 mg CO_2 kg^{-1} soil for 10 % furfural treatment (average of incubation periods). The differences among furfural concentrations were significant, except values among 4, 6, 8, and 10 % levels. The increase in CO_2 evolution of furfural-treated soils this could be because of changes in the makeup of the microbial community or because the increased energy source makes the microbes grow and do more. Improving the physical qualities of soil also makes it a better place for microorganisms to live (Tejada et al., 2009). The results show that organic carbon and the use of organic amendments, which are related to soil management, limit the actions of microbes. It is well known that low carbon supply controls the flow of energy through the soil's microbes and, in turn, the rate at which soil organic matter is mineralized (Fontaine et al., 2003). A study by (Iovieno et al., 2009) discovered a link ($r = 0.51$; $p < 0.05$) between soil respiration and soil organic carbon. (Vicena et al., 2022) also found a significant increase in microbial respiration in soil treated with organic matter (bushgrass leaf litter) as compared with no addition. However, no difference in CO_2 evolution among all high of furfural (over 4%) This could be because there are a lot of organic materials in it, like more labile organic carbon and complex organic chemicals with COOH and OH groups which change significantly the biochemical conditions and alter microorganisms activity. In addition, the high furfural concentration may change some soil properties such as EC, pH, O_2 availability and nutrient content resulting from changes in microbial community structure, thus soil respiration. (Fettrow, 2018) found no strong correlation between CO_2 evolution and organic matter content (R^2 = 0.066 and 0.2217 for two sites), stating that respiration and organic matter content change with varying levels of soil and environmental conditions.

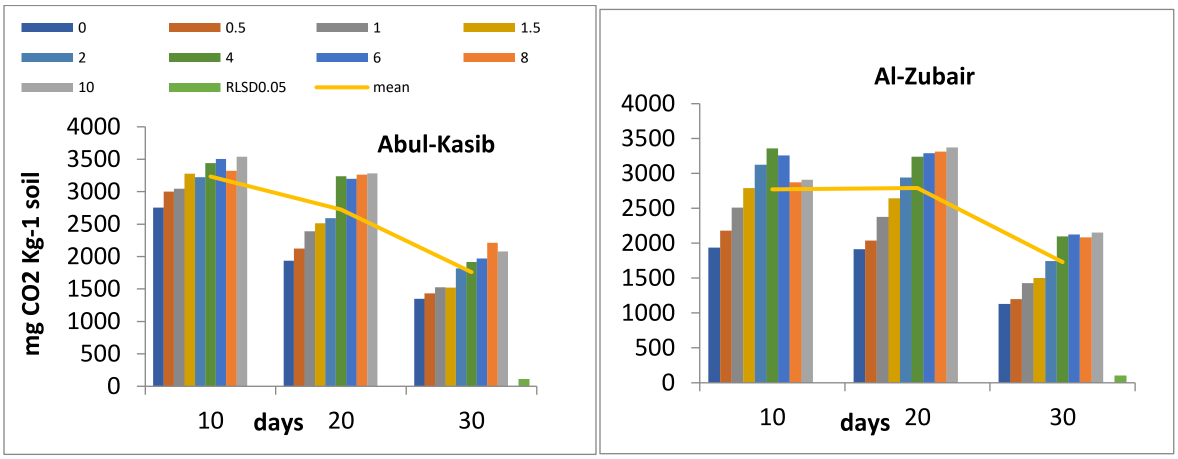

Fig. 1. CO_2 released from Al-Zubair and Abul-Khasib soils under different concentrations of furfural during 10,20 and 30 days of incubation (line represents the mean effect of incubation periods)

Regardless of the treatments, CO_2 evolution decreased with increasing incubation period in both soils (Fig. 1). CO_2 values were 2772, 2792 and 1730 mg CO_2 kg^{-1} soil at 10, 20 and 30 days of incubation, respectively for AL - Zubair soil and were 3234, 2726 and 1760 mg CO_2 Kg^{-1} soil at same periods for Abul - Khasib soil. This clearly shows that using a lot of furfural has a bad effect on the activity of microbes that aren't used again and again. In other words, soils that had furfural added had more respiration in the beginning stages of breakdown. Increasing the amount of fresh soil organic matter leads to a lot more microbial activity, which means that things break down faster (Fontaine et al., 2003). Similar results were seen by (Vicena et al., 2022), who found that trash broke down much faster in the first phase than in the later phase. For each level of furfural, the CO_2 evolution of Abul - Khasib Soil (with higher initial orgonic matter) were more than that of AL- Zubair soil (with lower initial organic matter), but with no statistical differences. The averages for the two soils were 2573.33 and 2431.33 mg CO_2 kg^{-1} soil, respectively.

The control treatments (which did not include furfural) also showed a similar trend. This can be revealed that the CO_2 evolution is relative to available carbon in the soil. The connection between different types of microbes and decomposition seems to rest on how much organic matter is in the soil and how much organic matter is added, which can explained by nutrient availability (Maron et al., 2018). Similar results were obtained by (Fettrow, 2018) who found that the geologic and soil type factors certainly affect organic matter cycle and subsequent transformation of this matter to CO_2. Other writers, on the other hand, discovered that compost had a bigger effect on some microbial activities in soil with less organic matter than on those in soil with more organic matter, which may be explained by harsh microbial conditions due to poor availability of soil organic carbon (Iovieno et al., 2009; Zhang & Zhang, 2016).

Urease activity

The amount of furfural had a big impact on the activity of urease (Fig. 2). The earth in Al-Zubair had the most urease activity treated with furfural concentrations of 1 % with a value of 117.50 ,180.00 and 145.90 µg $NH^+{}_4$-N g^{-1} soil $2hr^{-1}$ for 10, 20 and 30 days of incubation, respectively. Whereas, the highest urease activity in Abul - Khasib soil was observed with furfural concentration of 0.5% with a value of 121.00 ,218.00 and 190.40 µg $NH^+{}_4$-N g^{-1} soil $2hr^{-1}$ for the mentioned incubation days, respectively. Increasing furfural concentration of more than 1% for Al-Zabair soil or 0.5 for Abul- Khasib reduced slightly at the subsequent concentration, and then had no significant differences among other concentrations. The lowest urease activity was observed in soil without furfural application. The higher urease activity in soils treated with furfural may be due to the growth and activity of microbes as well as better soil qualities. The urease enzyme process was more likely to happen after organic amendments were added, and less energy was needed (Su, 2013). (Burns, 1982) said that increasing the activity of microbes leads to the release of external enzymes that may stick to soil particles or get caught by humus particles, protecting the enzyme from proteases. This is in accordance, with the studies of (Abdulkareen & Yassen, 2003; Kuziemska et al., 2020) which showed that treated soil with composter manures caused an increase in urease activity.

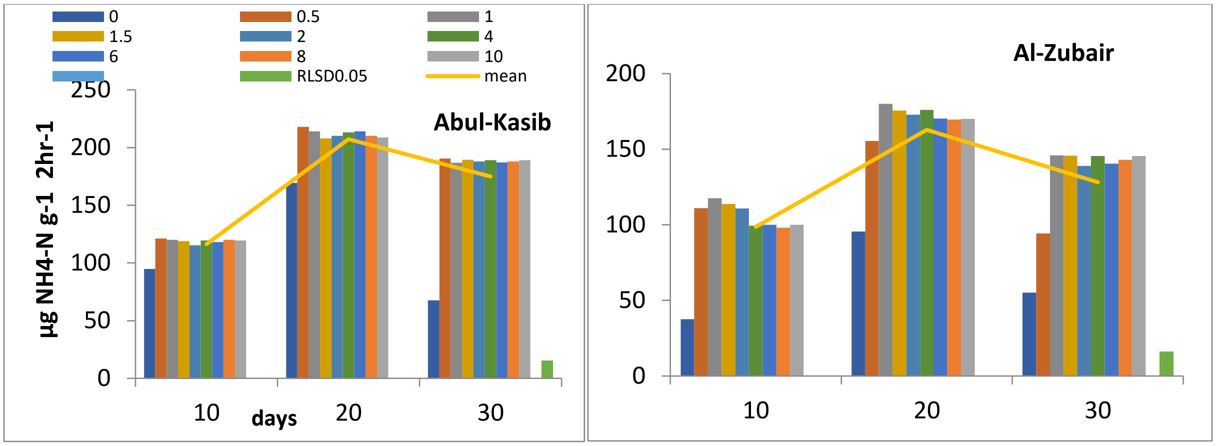

Fig. 2. Urease activity in Al-Zubair and Abul-Khasib soils under different concentrations of furfural during 10,20 and 30 days of incubation (line represents the mean effect of incubation periods)

At large amounts of furfural, urease activity seems to remain unchanged for all treatments. The action was still a lot higher than control, though. The same results were found by Qagnon *et al.*,(1999) who found a reduction in activity of some enzymes at the high levels (32 and 65 *M*g ha $^{-1}$) of paper mill sludge. The excessive increase of furfural may reduce urease activity by inhibiting the survival of microbe due to osmotic stress, altering soil pH and increasing the retention of C (Vandana, 2012). A study by (Gagnon et al., 2000) found that adding 34 Mg ha-1 of paper mill sludge to soil increased the amounts of N and P in the soil, which is a good thing but was excessive and reduced yields as well as enzyme activities. (Leaseburg et al., 2022) also found that adding green manure at the rate of 58 mg g^{-1} soil reduced urease in soil. Increasing the prospect of adsorption of urease on organic colloids at high levels of furfural may also have contributed to decreased urease activity in soil. It was shown by (Paulson & Kurtz, 1970) that the adsorbed urease had a much smaller affinity for the substrate than the native enzyme. (Marzadori et al., 2000) stated that the inhibition of urease the main reason humic acid works is because it holds heavy metals in place. No link between urease action and organic matter content is important was found by many workers (Leaseburg et al., 2022; Pancholy & Rice, 1973). In this work, the activity of urease, with respect to furfural concentrations, was reported to be, at least in part, specific to CO_2 evolution (Fig.1) which indicated the strong relationship between the two parameters and confirmed that any additives to soil altering the life functions of organisms could indirectly affect enzymes activities in soil.

After 20 days of incubation, urease activity was much higher than it was during the first 10 days, then significantly decreased in 30 days of incubation (Fig. 2). This was true for both Soils. The mean values were 98.68, 162.80 and 128.20 µg NH^{+}_{4}-N g^{-1} soil $2hr^{-1}$ at AL- Zubair soil, and 116.30, 207.20 and 175.02 µg NH^{+}_{4}-N g^{-1} soil $2hr^{-1}$ at Abul- khasib soil, for 10, 20 and 30 days of incubation, respectively. Upon furfural decomposition, urease activity will go down at the end of the incubation period because soil bacteria will need less energy. Whereas, the enhancement in urease activity at 20 days of incubation is probably due to the easily decomposable constituents of organic matter being oxidized in the first period (some weeks) of addition, resulting in an increase of microbial population, which will eventually lead only the complex decomposable constituent in soil (Alexander, 1978; Chen et al., 1997). Similar results were obtained by (Abdulkareen & Yassen, 2003) who found that urease activity in soil amended with organic residues was increased at increasing of incubation period from 10 to 30 days, then decreased at 40 days of incubation.

Urease activity in amended and un-amended Abul – Khasib soil was significantly higher than that of counterparts of AL - Zubair soil at all incubation periods. The mean values at Abul – Khasib soil were 116.30, 207.20 and 175.02 µg NH^{+}_{4}-N g^{-1} soil $2hr^{-1}$ at 10, 20 and 30 days of incubation, respectively, while the values at AL - Zubair sail were 98.65 162.80 and 128.20 µg NH^{+}_{4}-N g^{-1} soil $2hr^{-1}$ at the same periods respectively. This result was the same as CO_2 evolution (Fig.1) which was mainly confirmed by high contents of organic matter and clay in Abul - Khatib soil compared with AL - Zubair soil (table 1). Urease activity can be raised by providing enough energy and keeping the protein from breaking down. A significant correlation between soil clay content and urease activity was reported by (Vandana, 2012).

NH3 Volatilization

Data in Fig. 3 showed that volatilized NH_3 significantly decreased at increasing furfural concentrations for both soils and all incubation periods. Controls (no furfural) were the treatment that caused the highest volatilization of NH_3 with approximately 6 times greater than NH_3 loss from the highest concentration (10 %) of furfural. this finding agreed with (Al-Tameemi et al., 2014; Vanin et al., 2013), was found a reduction in NH_3 loss after mixing urea with poultry litter and humic acid. This reduction in NH_3 volatilization can be attributed to the reduction of soil pH in microsites by the production of acidic products. (Eifediyi & Remison, 2010) stated that manure increases soil CEC and prevents NH_3 loss.

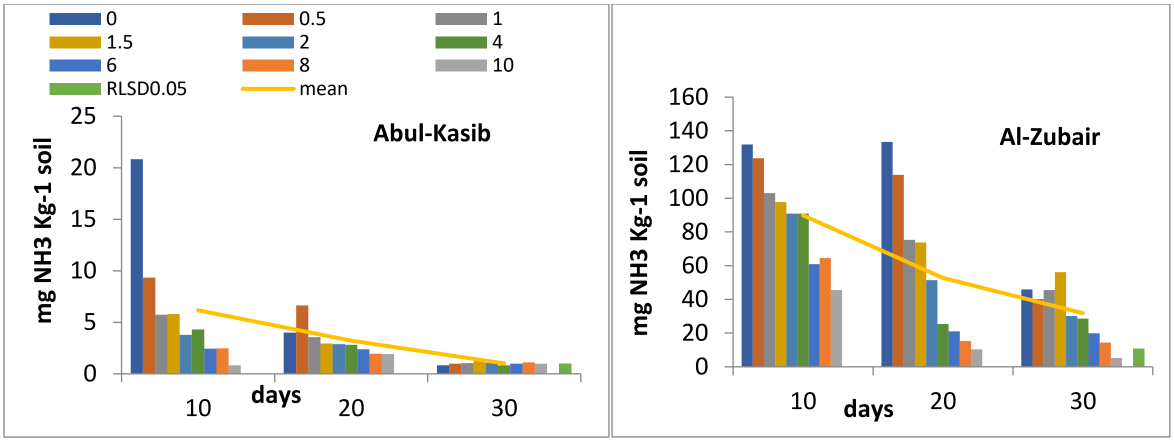

Fig 3. Volatile ammonia from Al-Zubair and Abul-Khasib soils under different concentrations of furfural during 10,20 and 30 days of incubation (line represents the mean effect of incubation periods)

In the present study, decreasing soil pH especially at the end of incubation (table 2) explained the reduction of NH_3 loss as well as, and it was expected that ammonium ions would be protected by function groups resulting during furfural decomposition.

Table 2. Impact of furfural content on soil pH over a range of incubation times

Furfural rate (%)	Al-Zubair soil			Abul-Kasib soil		
	10*	20	30	10	20	30
0	7.90	7.82	7.10	7.80	7.90	7.23
0.5	7.80	7.81	7.00	7.72	8.00	7.11
1.0	7.60	7.61	6.93	7.60	8.05	7.12
1.5	7.73	7.70	6.81	7.60	7.90	6.90
2.0	7.70	7.70	6.80	7.73	7.93	7.11
4.0	7.70	7.80	6.80	7.70	7.90	7.23
6.0	7.77	7.81	6.53	7.70	7.90	7.21
8.0	7.72	7.82	6.73	7.81	8.03	7.22
10.0	7.80	7.80	6.80	7.73	8.00	7.43

*days after incubation.

Ammonia volatilization of 89.85, 52.67, 31.82 mg NH_3-N kg^{-1}, soil for AL- Zubair sail and 6.17, 3.22, 1.01 mg NH_3-N kg^{-1}, soil for Abul -khasib soil were observed at 10, 20, 30 days of incubation, respectively (Fig. 3). That was also true for control treatment. These results clearly showed that approximately 52 % of NH_3 volatilized was lost within 10 days after urea application. Pote and Meisinger (2014) discovered that the rate of NH3-N volatilization dropped from more than 600 g ha-1 hr-1 on the first day to less than 600 g ha-1 hr-1 by the second day. The rate continued to drop each day as most of the NH3-N was washed off the surface by cumulative losses, leaving only small amounts of NH3 to be volatilized. According to (Al-Tameemi et al., 2014), ammonia loss began three days after HA was mixed with urea and then decreased continuously until the end of the experiment (60 days).

Mean results showed that soil type significantly affected NH_3 volatilization including control treatments (Fig. 3). Actually, NH3 volatilization from AL-Zubair soil was much higher than that from Abul-khasib soil, which was only 0.98 to 20.83 mg NH3-N Kg-1 soil. It ranged from 5.10 to 131.83 mg NH3-N Kg-1 soil. This is because the loose soil doesn't hold much water and has low CEC values, which causes NH3 to be lost (Grant, 2004; Zhao et al., 2002). (Liu et al., 2017; Yaseen, 2010) both came to the same conclusions.

Conclusion

The application of furfural (paper mill sludge) to Abul -khasib and AL- Zubair soils markedly affected CO_2 evolution, urease activity and NH_3 loss. Furfural promoted CO_2 evolution as a soil biological activity indicator and consequently promoted urease activity. Despite the increasing urease activity after the addition of furfural, NH_3 volatilization was significantly decreased. These findings indicated that furfural can be considered as a soil conditioner that enhances microbial activity and minimize loss of N in soils, improving urea use efficiency since furfural is presented in high amount with low collection and preparation cost. The addition of furfural and more than 4 % concentration is economically not feasible due to there being no significant effects on CO_2 evolution and urease activity, although NH_3 loss has been reduced.

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