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UREA OR UREA-FORMALDEHYDE LOADING WITH SOME MICRONUTRIENTS NITROGEN AND FOR IRON AVAILABILITY IN DIFFERENT SOIL TYPES

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ABSTRACT: This work was conducted to evaluate the effect of loading some micronutrients on urea or urea formaldehyde for delaying the nitrogen and iron release rate from urea and urea-formaldehyde compared in in different soil types (clay located at 30˚ 01ˋ N, 31˚ 13ˋ E, sandy loam located between 30° 35' 30" N, 32° 14' 50" E and calcareous located at 30°40′N 30°04′E).

Reducing the release rate of urea can increase its availability and minimize negative effects on the environment. A novel fertilizer material that was formed by blending formaldehyde with urea, delayed fertilizer N release in controlled climatic conditions in a greenhouse, through strong retention facilitated by the extensive surface area, porous structure and chemical functional groups in the formaldehyde. Moreover, loading some micronutrients was aimed to increase the nitrogen fertilizers availability.

Data indicated that the nitrogen and iron release rate in urea-formaldehyde sole was slower than urea sole regardless to soil type, while after loading Fe, Mn or Zn on urea and urea-formaldehyde data could be summarizes as the following, the applied nitrogen sources could be represented in the descending order as: Fe-urea formaldehyde> Zn- urea formaldehyde>Mn- urea formaldehyde> urea formaldehyde> Fe-urea> Zn- urea>Mn- urea> urea, respectively. Also, for soil type data indicated that the nitrogen release rate in clay soil was higher than calcareous and sandy soils for all treatment under study.

Key words: Urea, Urea-formaldehyde, Controlled release fertilizers, Micronutrients and Soil types.

INTRODUCTION

Agricultural output needs significant increases to feed the growing population .Fertilizers are essential for plant production systems, with nitrogen being the most limiting nutrient for plant growth. It is commonly supplied to crops as urea. Still, due to volatilization, up to 50 % of the total N application is lost. Slow or controlled release fertilizers are being developed to reduce these losses. The efficiency of classical mineral nitrogen, phosphorus and potassium fertilizers is usually low because a major part of these fertilizers does not reach plant roots and ends up polluting ground waters with nitrates and phosphates (Giroto *et al.*, 2022).

The decomposition of urea-formaldehyde in soil by consecutive series processes as described by Sasson (1979).

$$\text{Urea-formaldehyde} \longrightarrow \text{Urea} \longrightarrow NH_4^+ \longrightarrow NO_2^- \longrightarrow NO_3^-$$

In fact, nitrate ions which are the final produced N-fraction could be taken as a measure for the indicated decomposition process.

Urea–formaldehyde (UF) slow release fertilizer was an organic micro soluble fertilizer made by cross-linking polymerization of urea and formaldehyde under certain conditions, and was also the earliest industrialized slow-release fertilizer (Trenkel, 2021).

Urea and formaldehyde was used to form polymers under certain conditions of temperature, molar ratio, and reaction time. The release period was affected by the degree of polymerization (El-Monem *et al.*, 2009). There has been previous domestic and foreign research and development into urea-formaldehyde slow release fertilizers that more concentrated in single factor and orthogonal design; the deep-level research of forecasting mathematical models is not enough (Jahns *et al.*, 2003; Maslosh *et al.*, 2003). The urea-formaldehyde production lacked a systematic and

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comprehensive approach; hence, it is urgent to advance this technology. The synthesis of urea-formaldehyde mainly focused on a single-factor, while the range of process parameters was larger.

Urea-formaldehyde has become one of the most commonly used slow-release fertilizers worldwide (Ikeda *et al.*, 2014). Urea-formaldehyde fertilizers offer good physical properties and slow release rates; they can promote the formation of an aggregated soil structure, improve soil permeability, and increase penetrating power into crop roots. With fast, long-term functioning, the nitrogen use efficiency may exceed 50% (Yamamoto *et al.*, 2016). In the soil, this may result from microbial hydrolysis into ammonium, carbon dioxide, and water, which involves plant absorption and use. The result would be complete degradation of the fertilizer, which would be environmentally friendly, and thus confer a unique advantage to using these slow-release fertilizers (Trenkel, 2021).

The majority of slow-releasing fertilizers developed to date belong to the first category, that is, either hydrolysis or diffusion controls the release of nutrients to the plants, as in urea formaldehyde, via membrane-coated fertilizers or glass frits, and so forth (Shaviv, 2001). The other category of slow-release fertilizer is that in which nutrients are present in exchangeable or chelate-extractable positions. These mimic the natural forms of available nutrients in soils. Synthetic ion-exchange-resin-based fertilizers4 belong to this category as well as the more recently developed phosphate polymer-based compounds (Ray *et al.,* 1997). Those in the latter group are superior because release rates in hydrolysis and diffusion controlled mechanisms may not match rates of plant uptake, and thus such fertilizers are not always effective. Furthermore, fertilizers with incorporated heavy-metal micronutrients are highly insoluble and have not proved to be successful. Compounds developed for Zn and Cu have overcome some of the major drawbacks associated with earlier slow-release formulations. Uniquely, these are short-chain polyphosphates in which problems of hygroscopicity, stickiness, and water solubility inherent to such partially polymerized linear polyphosphates can be successfully overcome to produce very effective fertilizers (Ishita *et al.,* 2007). The nutrient ions in these compounds have low water solubility but are potentially plant-available by virtue of their high solubility in organic acids such as citrate, and so forth, which are excreted by plant roots *hence*, solubility in organic acids can be used as an index of nutrient availability (Mortvedt *et al.,* 1972).

The objective of this study was evaluation the efficiency of some controlled release fertilizers properties as urea formaldehyde by addition of some plant micronutrients such as iron, zinc and manganese to reduce using traditional mineral fertilizers also to reduce environmental pollution and increase the efficiency of soil applications with less micronutrients loss reaching to maximum response of plant uptake.

MATERIALS AND METHODS

Soil samples were collected randomly from the experimental field area before cultivation to determine the some physical and chemical soil properties. Particle size distribution was carried out by pipette method described by (Estefan *et al.,* 2013).

Samples representing calcareous soil sample was also taken from agriculture research station at Nobaria area (Behaira governorate located between 30° 35' 30" N, 32° 14' 50" E), clay soil was collected from farm of the agriculture research station (Giza governorate located at 30° 01′ N, 31° 13′ E) and sandy loam soil from Ismailia area (Ismailia governorate located at 30°40′N 30°04′E), Egypt. Air dried soils were gently ground to pass a 2 mm sieve, initial physical and chemical analysis of the investigated soils being presented in Tables 1, 2 and 3.

The soil nitrogen content was determined according to Jackson(1983).The Atomic Absorption Spectroscopy method is commonly used to measure of micronutrients used. The extraction by di-ethylene tri-amine penta-acetic acid according to Lindsay and Norvell (1978) is commonly used for evaluating status with respect to micronutrient of Fe, Zn and Mn.

Table 1. Physical properties of the investigated soils under study.

Soil characteristic	Ismailia	Giza	Nobaria
Particle size distribution (%)			
Sand	67.8	27.9	92.9
Silt	12.1	25.3	4.8
Clay	20.1	46.8	2.6
Textural class*	SL	C	S
Soil-water characteristic (%)			
Saturation	38.9	80.5	27.3
Water holding capacity	19.9	26.3	15.6
Wilting point	9.9	13.7	9.4
Available water	10.0	12.6	6.2

*According to the international soil texture triangle**,**

S: Sand (Calcareous soil), SL: Sandy loam and C: Clay

* Using USAD Soil Texture Triangle, after (Bashour and Sayegh, 2007).

Table 2. Chemical properties of the investigated soils under study.

Soil characteristic	Ismailia	Giza	Nobaria
$CaCO_3$ (g kg^{-1})	7.2	2.1	37.2
OM (g kg^{-1})	5.50	4.1	0.6
CEC (cmol$_c$ kg^{-1})	19.45	44.20	6.44
pH (soil paste)	7.6	7.4	7.5
EC (dSm^{-1})	15.6	4.6	3. 9
ESP	15.7	15.6	15.4
Soluble ions (mmol$_c$ l^{-1})			
Ca^{2+}	44.0	10.4	12.0
Mg^{2+}	24.0	14.5	7.9
Na^+	82.3	19.5	18.4
K^+	5.2	1.1	1.3
CO_3^{2-}	0.0	0.0	0.0
HCO_3^-	4.8	5.1	8.5
Cl^-	144	36.0	22.3
SO_4^{2-}	6.7	4.4	8.8

Table 3. Micronutrient contents of investigated soils under study.

Micronutrient	Nobaria	Giza	Ismailia
Fe	12.0	10.4	44.0
Mn	7.9	14.5	24.0
Zn	18.4	19.5	82.3

For preparing urea-formaldehyde was 24 g of urea and 20.3 g of 37% (w/w) formaldehyde were added into a 250 mL round-bottom flask and then stirred constantly. Then, the pH of the solution was adjusted to 9.0 with a 5% KOH solution. The reaction was incubated at 50 °C for

2 h. Fertilizer UF granules were obtained by a plunger-extrusion molding process. For this purpose, the above-reaction product was first solidified at room temperature for 30 min to achieve the desired viscosity for extrusion molding and then extruded into cylindrical strips using a homemade extrusion device. Afterward, the cylindrical strips were placed into the oven and dried at 160 ˚C for 30 min, then cooled down to 80 ˚C, and dried to a constant weight. Finally, the dried, stripped products were cut up, and the cylindrical fertilizer granules were obtained according to (Yang *et al.,* 2018).

Applied micronutrients (Fe, Mn and Zn) as sulphate and mixed with urea or urea-formaldehyde solely used during UF preparation. After maintaining these conditions for 1 h, all contents of the flask were transferred into a large glass dish and dried at 70 ˚C in a drying oven. Finally, the urea or urea-formaldehyde with micronutrients prepared was synthetized and prepared for the analyses.

Each studied soil samples were treated with amounts of different nitrogenous with micronutrients, at a rate of (500 mg N/kg soil) and placed into containers (4.5 cm height and 10 cm in diameter) to be finally left for successive periods of 7, 15 and 21 days, control treats with no fertilization were included. The water content of each soil type was always kept at field capacity.

Analysis of variance was conducted using Minitab version 15. All collected data were statistically analyzed according to (Gomez and Gomez., 1984).

RESULTS AND DISCUSSION

Obtained data was thought to be presented as to include two main headings dealing with released nitrogen accompanied with micro-nutrients from complex urea form as slow release + some micronutrients and normal urea + some micronutrients as well as the studied soil receiving the concerned fertilizers added also included as far as their responses to adopted fertilization designs. The following results were focused on the Iron (Fe) availability in different soil with nitrogen forms and other micronutrients in all treatment under study.

i . First period at 7 days

Data in Table 4, represented the behavior of applied nitrogenous urea-formaldehyde, urea-formaldehyde (UF) complexes with some micronutrients (Fe, Mn and Zn), urea solo and urea complexes with micronutrients (Fe, Mn and Zn) applied in different type of soil as clay, sand and calcareous soil during the first studied time on the nitrogen content in the three soil types under study.

Regard to nitrogen fertilizer forms results indicated that the hydrolysis rate of urea solo was faster than urea-formaldehyde in all soil types under study where the hydrolysis of urea governorated with many factors such urease activity. Obtained results may be explained, according to Sasson (1979) and Kumar *et al.* (1988), on the basis of rapid hydrolysis of urea as compared with the slow release urea form compounds; such hydrolysis is, of course a resultant of mineralization processes.

As showed in Table 4 data revealed that the application of different nitrogen sources under study have different behavior in clay, sandy and calcareous soils where the hydrolysis of all nitrogen sources under study in clay soil was higher compared to sandy and calcareous soils these may be due to the presence of microorganisms in clay soil which activate the processes of nitrification, volatization, mineralization, *etc.* as mentioned by Booth *et al.* (2005) the rates of nitrification based on mineralized organic nitrogen can be equal or even greater than that typically found in neutral pH soils.

Also, microorganisms splitting urea are abundant and widely distributed in soil. In favorable conditions of soil, urea is hydrolyzed quickly to ammonium carbonate, thus it serves after a short period as an energy source for the nitrifying autotrophs. (El-Shinnawi, 1975) found in samples, taken from soils of widely different structure and origin at different depths, that ureolytic bacteria were greatly affected by pH, sampling, and cultural conditions.

Table 4: Available nitrogen from different types of soil treated with or without formaldehyde urea combined with micronutrient after 7 days incubation period.

Treatment (T)	Type of soil (S)		
	Clay	Sand	Calcareous
With formaldehyde			
	Nitrogen concentration, mg kg^{-1}		
Urea-formaldehyde	352.8	366.4	318.0
Urea-formaldehyde + Fe	339.7	398.1	296.6
Urea-formaldehyde + Mn	386.3	365.6	248.9
Urea-formaldehyde + Zn	317.9	380.2	273.5
Mean	349.17	377.58	284.25
LSD at 0.5%	T	D	T×D
	34.2	22.8	12.8
Without formaldehyde			
Urea	842.8	683.4	618.3
Urea + Fe	675.9	560.8	496.6
Urea + Mn	759.7	621.3	548.9
Urea + Zn	691.5	586.7	523.5
Mean	742.48	613.05	546.83
LSD at 0.5%	T	S	T×S
	2.7	4.1	35.3

Urea is expected to have very high mobility in soil. Urea is not expected to volatilize from dry soil surfaces based upon its vapor pressure. Various field and laboratory studies have demonstrated that urea degrades rapidly in most soils. Urea is rapidly hydrolyzed to ammonium ions through soil urease activity, which produces volatile gases, that is, ammonia and carbon dioxide. However, the rate of hydrolysis can be much slower depending upon the soil type, moisture content, and urea formulation (Korrapati *and* Mehendale, 2014).

Regard to micronutrients that complexed with urea or urea formaldehyde data showed that Fe with both urea and urea formaldehyde was the most favorable treatment where the hydrolysis of urea or urea formaldehyde decreased compared to Mn, Zn with urea or urea formaldehyde and control treatment (urea and urea formaldehyde solo).

This could be attributed to Fe ions enhance the hydrolysis inhibitors compound in soil which delay the activity of microorganisms that hydrolyze urea. Fe fertilization considerably increased agronomic efficiency of fertilizer N, when nitrogen applied at proper dose for not to suppress nodulation and fixation capacity of Bradyrhizobium bacteria. Also, the adequate Fe supply increased nitrogenase activity and biological N fixation capacity of soybean (Terry and Jolley, 1994). Moreover, application of Mn combined with different N-Source increased is uptake. Values of Mn-uptake were found to be high in wheat plants grown on the calcareous soil than the sandy soil (Monreal *et al.,* 2016).

The applied nitrogen sources could be represented in the descending order as: Fe-urea formaldehyde> Zn- urea formaldehyde>Mn- urea formaldehyde> urea formaldehyde> Fe- urea> Zn- urea>Mn- urea> urea, respectively.

This results could be attributed that iron play as an urease inhibitors as inhibitors increase N efficiency as they slow conversion from one form of N to another. Urease inhibitors inhibit the urease enzyme, which catalyzes the hydrolysis reaction converting urea to ammonium bicarbonate and then to NH_3 gas and finally to NH_4. The NH_3 gas phase renders the N very vulnerable to volatilization loss if not captured by the soil. This gas loss is greatly minimized if the conversion from urea is slowed by use of an inhibitor, allowing the soil to capture the N more effectively. Although it does nothing to prevent other losses once the transformation takes place (Giroto *et al.*, 2022).

So, the interaction between nitrogen sources, soil types and nitrogen complexes with Fe, Mn or Zn data showed that application of urea formaldehyde complexed with Fe in calcareous soil was the most treatment has a low activity index and consequently more stability time for nitrogen in the soil.

ii. Second period at 15 days

The concentration of nitrogen was followed up over other two periods were at 15 and 21 days. Here, data will be represented the concentration of nitrogen for different sources at the three soil types under study (clay, sandy and calcareous).

Data in Table 5 showed that application of urea only showed decreasing in the nitrogen content due to its activity index was high in the first period. On contrast, the behavior of urea formaldehyde the nitrogen content increased due to the slow-release compositions are characterized by nutrient release mechanism that is based on either (i) diffusion through a membrane/coating or (ii) slow hydrolysis.

Regard to soil type data indicated that the mean value of nitrogen content in the clay soil was the lowest one regardless the above mentioned treatments this may be due to the high activity index of microorganism in clay soil where the combined activity of these ureases determines the rate at which urea is hydrolyzed in soils. The enzyme's activity is influenced by a number of environmental factors (*e.g.*, moisture, temperature, *etc.*) and soil characteristics (*e.g.*, pH, organic matter, depth in soil profile, *etc.*); as a result, complete hydrolysis of urea applied to soils can take anywhere from 1 to 14 days (Wali *et al.*, 2003).

Table 5: Nitrogen release from different type of soil treated with or without formaldehyde urea combined with micronutrient after 15days incubation period.

Treatment (T)	**Type of soil (S)**		
	Clay	**Sand**	**Calcareous**
With formaldehyde			
	Nitrogen concentration, mg kg^{-1}		
Urea-formaldehyde	542.8	573.1	521.6
Urea-formaldehyde + Fe	439.5	487.5	453.4
Urea-formaldehyde + Mn	479.9	543.7	499.2
Urea-formaldehyde + Zn	456.8	410.9	475.9
Mean	479.75	503.80	487.53
LSD at 0.5%	T	S	T×S
	11.8	9.5	10.7
Without formaldehyde			
Urea	442.5	683.4	540.8
Urea + Fe	375.9	596.2	474.2
Urea + Mn	399.7	660.6	497.9
Urea + Zn	381.5	645.1	465.2
Mean	399.9	646.33	494.53
LSD at 0.5%	T	S	T×S
	12.1	11.5	15.9

Results in Table, 5 revealed that the complexing of Fe with urea or urea formaldehyde was followed the same trend in the first period of incubation followed by Zn and Mn these results could be attributed to that Fe acts an essential element for mineral nutrition of rhizobia, nodulation, nodule activity and biological N fixation (O'Hara, 2001).

The interaction effect of urea formaldehyde with Fe in clay soil was the most effective one in delay the nitrogen release in the soil followed by urea formaldehyde with Zn in clay soil, and urea sole in sandy soil was the inferior one.

iii. Third period at 21 days

In the last period (21 days) the nitrogen content in Table, 6 indicated that the rate of nitrogen release in urea sole was decreased clearly compared with other urea complexes, while urea formaldehyde showed different behavior where the nitrogen content increasing overall the 21 days this indicate that urea formaldehyde more stable than urea sole and have the ability to stain in the soil for longer time.

Regard to soil type, the concentration of nitrogen fertilizer in clay soil were, as expected, greatly responded to fertilizer application compared to other soil types under study, particularly with urea as compared with urea formaldehyde sole or complexed with micronutrient whose values followed an order of increasing trend corresponding to that of the activity index which seemed to be reflected on the behavior of urea form compounds in soil as to have N-fractions. Apart from the effects of bicarbonate/high pH on decreasing Fe uptake by the plants, previous studies indicated that bicarbonate/high pH also impair nodule formation and biological N fixation by Brady rhizobium strains in peanut (Tang *et al.,* 1991). The decrease in nitrogen accumulation with an increase in sand fraction of the soil in this study corroborated previous studies that reported lower N accumulation as sand faction of the soil increased (Goos and Guertal, 2019; Lasisi *et al.,* 2020).

Table 6: Nitrogen content from different type of soil treated with or without formaldehyde urea combined with micronutrient after 21days incubation period.

Treatment (T)	Type of soil (S)		
	Clay	Sand	Calcareous
With formaldehyde			
	N concentration, mg kg^{-1}		
Urea-formaldehyde	680.3	751.6	705.2
Urea-formaldehyde + Fe	591.4	645.2	585.7
Urea-formaldehyde + Mn	637.2	680.9	643.9
Urea-formaldehyde + Zn	575.8	665.4	618.1
Mean	621.18	685.78	638.23
LSD at 0.5%	T	S	T×S
	13.7	15.9	18.1
Without formaldehyde			
Urea	261.8	683.4	387.5
Urea + Fe	309.0	596.2	428.4
Urea + Mn	272.9	660.6	399.8
Urea + Zn	301.5	645.1	412.5
Mean	286.30	646.33	407.05
LSD at 0.5%	T	S	T×S
	14.3	17.5	20.4

Complexes of both urea and urea formaldehyde with iron, zinc or manganese results in Table, 6 showed a different behavior for urea or urea formaldehyde where complexes with iron showed a decrease in urea activity index for urea and urea formaldehyde followed by zinc then manganese.

CONCLUSION

As mentioned before the applied nitrogen sources could be represented in the descending order as: Fe-urea formaldehyde> Zn-urea formaldehyde>Mn-urea formaldehyde> urea formaldehyde> Fe-urea> Zn-urea>Mn-urea> urea, respectively. This result could be attributed to iron may act as buffering agent in soil that lead to decreasing the pH soil which is favorable for urea nitrification and lead to decrease the rate of urea volatization.

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تيسر عنصري النيتروجين والحديد بإستخدام اليوريا واليوريا فورمالدهيد المحمله ببعض العناصر الصغرى في أنواع الأراضي المختلفة

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الملخص العربي

أجريت دراسة تحت ظروف الصوبة الزراعية على فترات مختلفة من التحضين (٧ و ١٥ و ٢١ يوم) على أنواع أراضي مختلفة القوام (طينية و رملية طميية و جيرية) وذلك لتقييم تأثير تحميل بعض العناصر الصغرى على كلاً من اليوريا واليوريا فورمالدهيد على تيسر عنصري النيتروجين والحديد والذي تم تسليط الضوء عليهما في هذه الدراسة.

وقد أظهرت النتائج المتحصل عليها أن معدل تسير كلاً من عنصري النيتروجين والحديد عند إضافة اليوريا فورمالدهيد منفردة لأنواع الاراضي تحت الدراسة أقل من المتحصل عليه من اليوريا فقط تحت نفس الظروف.

بينما كان هناك تأثير واضح عند تحميل بعض العناصر الصغرى كالحديد والمنجنيز والزنك على اليوريا واليوريا فورمالدهيد وكان إضافة الحديد المحمل على اليوريا فورمالدهيد أبطأ تيسراً على فترات التحضين تحت الدراسة مقارنة بالمعاملات الأخرى.

وكان ترتيب المعاملات تحت الدراسة كالتالي:

يوريافورمالدهيد- حديد > يوريا فورمالدهيد- زنك > يوريا فورمالدهيد- منجنيز > يوريا فورمالدهيد > يوريا-حديد > يوريا- زنك > يوريا- منجنيز > يوريا ، على التوالي.

وبالنظر إلى نوع التربة المستخدمة في الدراسة وجد أن هناك إختلاف في نسبة تيسر كلاً من النيتروجين والحديد على جميع المعاملات المستخدمة لليوريا واليوريا فورمالدهيد

وأخيراً يمكن تلخيص ما سبق بأن إضافة الحديد محملاً على اليوريا فورمالدهيد للأراضي الطينية كانت أفضل معاملة للحصول على أطول فترة (٢١ يوم) لتيسر كلاً من عنصري النيتروجين والحديد على فترات التحضين تحت الدراسة.