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Environmental Effect of Hydroxyapatite Urea Application on Optimizing Urea Fertilizers for Wheat Plant

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ABSTRACT

This study was intended gradually release of nitrogen into the soil through the covering of urea pellets with hydroxyapatite and increase the wheat yield using zero valent iron spraying. Incubation trial was planed under different conditions (saturated and field capacity) for soil samples to study the performance of the use Hydroxyapatite urea on ammonium ion release for 63 days. A field trial was aimed to examine of hydroxyapatite urea and zero valent iron spraying on improved N-use efficiency, minimize nitrate leaching, and wheat productivity. Urea was used at two rates 80 and 100 % from recommended dose. Data of incubation trial observed that an increase of ammonium ion release with increase in time of incubation by using hydroxyapatite urea. Data of field trial observed that the release of nitrate was slower from hydroxyapatite urea soil than pellets urea, which reached 20 mg L^{-1} for urea pellets and 150 mg L^{-1} for hydroxyapatite urea at two weeks, respectively. Hydroxyapatite urea leads to minimize nitrate leaching into ground water. Also the results showed that the using of hydroxyapatite urea and zero valent iron individually or combined lead to increment in the yield of wheat compared to urea pellets. Yield of treatments was in the following order: urea100+Fe (11%) < hydroxyapatite urea100 (20%) < hydroxyapatite urea100+Fe (27%) compared to urea100 while urea80+Fe (10%) < hydroxyapatite urea 80 (23%) < hydroxyapatite urea 80+Fe (30%) compared to Urea 80. It is concluded from these data that controlled released urea fertilizer improved yield by 27%, and grain protein percentage 50% as compared to urea pellets. The highest increase for nitrogen uptake by wheat plant was observed by using hydroxyapatite urea with zero valent iron spraying. From the results obtained it can summarize that, the efficiency of the use of urea fertilizer can be improved by covering with hydroxyapatite, as well as increasing the productivity of wheat yield by spraying of zero valent iron to reduce the cost price.

Keywords: hydroxyapatite coated urea, slow release fertilizer system, zero valent iron, and nitrate leaching.

INTRODUCTION

Urea is the widely used nitrogenous fertilizer in agriculture because of its high nitrogen content (46%). However, 50-70 percent of the applied nitrogen gets lost due volatilization and leaching (Shaviv and Mikkelsen, 1993). Such losses raise concerns about water contamination and greenhouse gas emissions. Low use efficiency of fertilizer N also reduces economic returns from fertilizer inputs. Nitrogen-use efficiency can be improved by reducing N losses (Engelsjord *et al.,* 1997). Jarosiewicz and Tomaszewska (2003) stated that the slow release fertilizers are a new strategy to minimize environmental pollution due to it's save fertilizer consumption. Tyliszczak *et al.* (2009) they stated that slow release technology, by coating, can be used to reduce the dissolution rate of urea and to increase the efficiency of urea fertilizer. These fertilizers control the release of nutrients with semi-permeable coatings, occlusion, protein materials, or other chemical forms, with slow hydrolysis of water-soluble (Trenkel, 2010). Hydroxyapatite [$(Ca_{10}$ $(PO_4)_6$ $(OH)_2$] nanoparticles are rated as one of the prominent candidates in agricultural applications, which can provide phosphorus nutrient. Much of the cur current literature on HA is however focused on its biomedical applications Zhu *et al*., (2010) due to its excellent biocompatibility and bioactivity, while potential agricultural applications have not been adequately addressed. HA nanoparticles, with its rich surface chemistry owing to the presence of reactive functional groups (Ferraz *et al.* 2007, Han and Misra 2009, Mateus *et al.* 2007, Teng *et al.* 2009, Zhu *et al.* 2010) were explored for surface modification with urea. Application of iron nanoparticles improved agronomic traits of soybean (Sheykhbaglou, *et al.* 2010). Nanoparticles increased water and fertilizers use efficiency (Lu, *et al.* 2002). Nanoparticles improved germination, enhanced growth and physiological activities (Shah and Belozerova, 2009). The objectives of the present research were to use slow release fertilizers by hydroxyapatite urea to optimize urea fertilizer consumption and to reduce nitrate leaching and also to increase wheat yield by zero valent iron spraying.

MATERIALS AND METHODS

Synthesis of hydroxyapatite coated urea

1. Dissolve 19.29 gm of calcium hydroxide in distilled water
2. Prepare 250 ml of 0.6 M H_3PO_4.
3. Add H_3PO_4 to the $Ca(OH)_2$ suspension in dropwise
4. Stirring it vigorously under mechanical agitation(1000 rpm)

The reaction go on according to the following this equation.

$$6H_3PO_4 + 10\ Ca\ (OH)_2 \rightarrow Ca10(PO_4)_6(OH)_2 + 18H_2O.$$

HA nanoparticles synthesized Fig. (H) as described according to Mateus *et al.* (2007) were allowed to settle and the supernatant was decanted. The resulting HA nanoparticles were washed thrice with distilled water. The solid thus obtained was dried at 100°C for 2 h.

Added 12 g wax with 20 ml of petroleum ether. Mixed of wax solution with 2.0 kg urea for 30 min. finally, mixed of urea that coated with wax and hydroxyapatite for their uniform coating.

Without Hydroxyapatite **With Hydroxyapatite**

Fig. H. Urea without and with Hydroxyapatite

Synthesis of zero valent iron

Zero valent iron was formed according to Sun *et al*. (2006).

1. Add 0.8 M Sodium borohydride into 0.2 M Iron (III) chloride hexahydrate by ratio 1:1
2. Mix the producing solution for 5 min at room temperature.
3. Nano zero valent iron was filtrated through 0.45 micron filter paper
4. Nano zero valent iron was washed several times with distilled water to remove the excessive borohydrate.
5. Nano ZVI was dried by N_2 gas
6. Nano ZVI was preserved from the oxidation by maintaining a thin layer of ethanol on the top of zero valent iron.

Effect of Hydroxyapatite Urea Fertilizers on N-Release Incubation of Soil Samples

The aim of this experiment were to investigate the controlling release technology by hydroxyapatite urea for increasing the efficiency of fertilizer by reduce fertilizer's losses and minimize environmental pollution.

1. One kg of soil samples was taken and the different forms of urea fertilizer (1000 mg $^{-1}$ kg).
2. The soil and fertilizer was mixed completely.
3. Soil samples (40 g) were weighed and transferred to separate 800 ml plastic cups and water was added to be field capacity (FC) then saturated (ST).
4. The cups were weighed and the moisture content was maintained constant at FC and ST.
5. Incubated the soil samples at 25 °C.
6. Determinate the ammonium ion in soil samples that taken weekly for 63 days of soil incubation.

Field experiment:

This trail was to use slow release fertilizers by hydroxyapatite urea to optimize urea fertilizer consumption and to reduce nitrate leaching and also to increase wheat (Triticum aestivum L.) cultivars sakha 93 yield by zero valent iron spraying. Experiment was designed in a complete randomized block design. Grains of wheat were growning in season (2016/2017) at the Experimental Farm, Agricultural Genetic Engineering Research Institute, Agricultural Research Center, Giza, Egypt. Field experiment consisted of 1.5 m2 (1x1.5m) in area plots with three replicates for each treatment as well as control. zero valent iron was used at a rate of 100 ppm as spray. zero valent iron spraying application was carried out after 90, 105 and 120 days from growing. Some Physical and chemical properties of the soil under investigate are showed in Table (1). Plants from each treatment were collected to determine growth characters, yield and yield components.

The experiment consisted of eight treatments:

T1: Urea pellets 100% (UP100) as a control
T2: Urea pellets 100% + Fe (UP100+Fe)
T3: Hydroxyapatite Urea 100 (HAU100)
T4: Hydroxyapatite Urea 100 + Fe (HAU100 + Fe)
T5: Urea pellets 80% (UP80)
T6: Urea pellets 80% + Fe (UP80 + Fe)
T7: Hydroxyapatite Urea 80 (HAU80)
T8: Hydroxyapatite Urea 80 + Fe (HAU80 + Fe)

Analytical methods:

Some soil physical and chemical properties were determined as mentioned with Page *et al*., (1982). Micro Kiedahel (Chapman and Pratt, 1961) was used to determination of available ammonium and nitrate. Vanadomolybdate yellow method spectrophotometrically was used to determine Phosphorus content in plant sample according (Jackson, 1973). Fame photometer was used to determine potassium content in plant sample according (Jackson, 1973). Micro-Kjeldahl method was use to determine total nitrogen according to (AOAC., 1970). Total sugars were determined according the method of Smith, *et al*. 1964 and Murphy, 1958. All determinations were performed in triplicate and data represented on dry weight basis as mean values ± standard deviations. At the end of season yield per feddan were calculated. All data were statistically analyzed using Mstatc computer program according to procedures outlined by Freed and Scott, (1986). Nitrogen use efficiency and Economic value was calculated according as FAO (2000).

Table 1. Some soil physical and chemical properties

Parameter	Value
Physical	
Coarse sand%	6.0
Fine sand%	19.9
Silt%	38.5
Clay%	35.5
Soil of texture	loamy clay
Chemical	
pH (1: 2.5, soil suspension)	7.72
Organic matter (%)	1.19
Total nitrogen%	0.05
ECe dS m^{-1}, soil paste	1.75
Soluble cations (meq/L)	
calcium	8.2
magnesium	4.1
sodium	3.5
potassium	1.6
Soluble anions (meq/L)	
carbonate	-
bicarbonate	5.1
chloride	5.3
sulfate	7.0

RESULTS AND DISCUSSION

Effect of Coated Urea Fertilizers on N-Release

Data in Figures 1 and 2 showed that, ammonium ion values of soil samples when added urea pellets and hydroxyapatite urea fertilizers to soil individually under field and saturated capacity. An ammonium ion value was lower under field capacity than saturated. Data observed that the urea pellets has undergone fast hydrolysis than hydroxyapatite urea. Highest releasing of ammonium ion was at the first time by using urea pellets. Ammonium ion reached 387 and 413 ppm for field capacity and saturated, respectively. it worth mention, a decrease of ammonia values with increase in time of incubation. These values reached to 13.8 and 15.9 ppm at end time.

On the contrary, the results of hydroxyapatite urea observed that the value of ammonium at first time was reached 283 for saturated and 208 ppm for field capacity. The release of ammonium ion has decreased hydrolysis in hydroxyapatite urea compared with urea pellets. Hydroxyapatite led to prevent of water from entering into the urea granule. Also, hydroxyapatite perhaps inhibited of urease activity of the soil (Purkayastha *et al*., 1997).

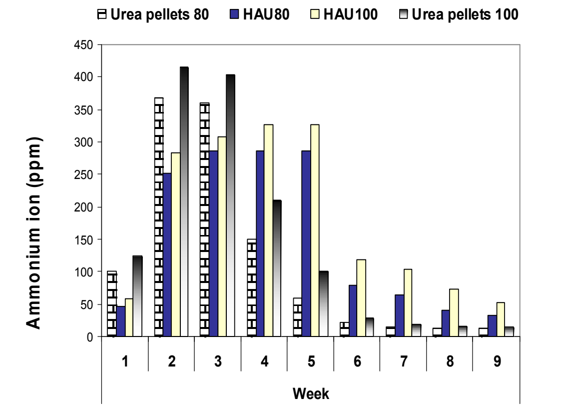

Fig. 1. influnce of hydroxyapatite urea on release of a mmonium ion into soil at saturated acondition

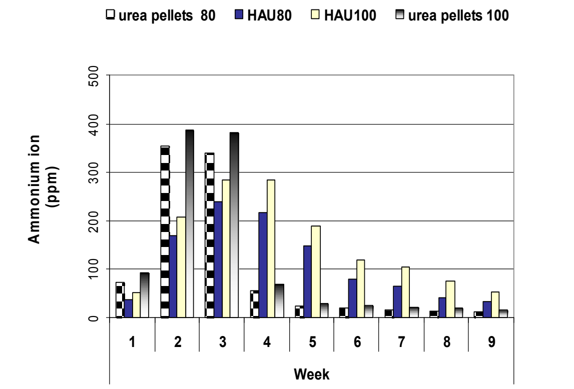

Fig. 2.Influnce of hydroxyapatite urea on ammonium ion release into soil at field capacity

Nitrate concentration in drainage water

Nitrate was estimated in the water samples that collected through the pezomete and the results were reported that nitrate value was higher using urea pellet than hydroxyapatite urea. Nitrate values reached 150 and 20 mg L-1 for urea pellet and hydroxyapatite urea at 15 days, respectively. Nitrate values reached 23 and 25 mg L-1 for urea pellet and hydroxyapatite urea at 105 days, respectively.

Generally, hydroxyapatite urea decreased of nitrate release for a longer period. These results, due to the nitrification inhibition were slow in hydroxyapatite urea as compared to pellet urea.

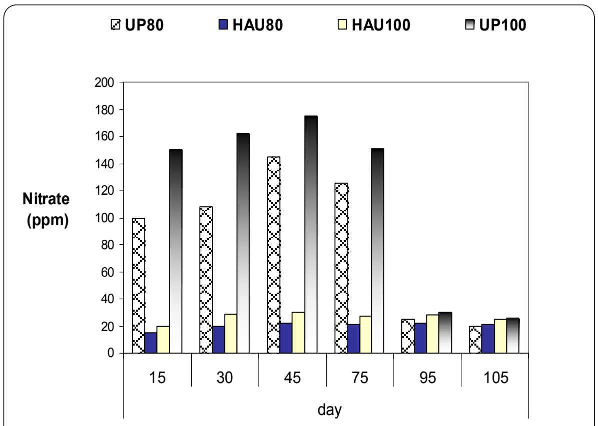

Fig. 3. Nitrate concentration in drainage water sample

Effect of hydroxyapatite urea and zero valent iron spraying on Wheat yields

As shown from Fig. 4, the application of hydroxyapatite urea fertilizers and zero valent iron foliar individually or combined led to increase in grain yield compared to pellets urea. The yield values were in the follow: UP100+Fe (11%) < HAU100 (20%) < HAU100+Fe (27%) compared to UP100 while UP80+Fe (10%) < HAU80 (23%) < HAU80+Fe (30%) compared to UP80. A decrease in yield was observed by using UP80+Fe treatment compared with UP100. An increase in yield was observed by using UP80+Fe compared with UP80. Generally, the increase in grain yield may be nutrients do not leach from the substrate so the plants receive all the nutrients applied (Sastry *et al.* 2010). These results are agreement with the Subbaiya *et al.*, (2012) stated the urea modified hydroxyapatite particles have been employed to agriculture, because of their higher nitrogen utilization efficiency and slow release of the nitrogen to the soil.The highest value of wheat yield was recorded when applied hdroxyapatit urea with zero valent iron spray. Siam *et al.*, (2006) there was a stable complex form with Fe and urea through a ligand-exchange reaction. Rehm and Albert (2006) they reported that, spray of Ferrous sulfate heptahydrate is more effective in wheat plant also yields were bigger for the treatments with micronutrients. Chaudry *et al.* (2007) mentioned that micronutrients increased the wheat yield when applied in single and in combination, along with basal dose of macronutrient.

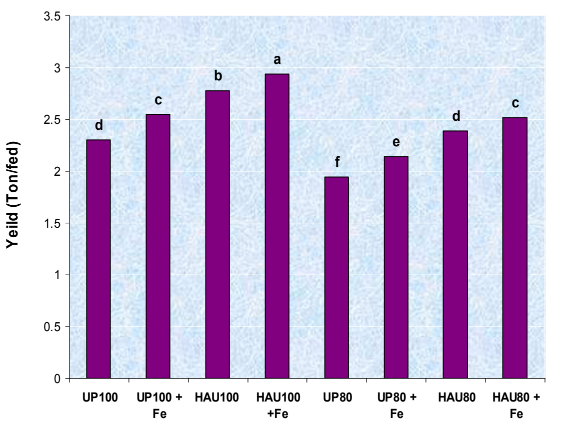

Fig. 4. Influnce of hydroxyapatite urea and nano iron spraying on wheat Yield (ton/fed)

Effect of hydroxyapatite urea and zero valent iron spraying on chemical composition

As compared to the control values (urea pellets), hydroxyapatite urea and foliar application of nano iron led to increase in crude protein and total sugars concentrations in plant grains as illustrated as in Figs (5 and 6). The highest values of sugars and protein were showed in HAU100+Fe followed by HAU100. The increase in grain yield may be nutrients do not leach from the substrate so the plants receive all the nutrients applied. These data agree with the results of Signor and Barbiani (2013) and Liu, *et al.* (2012) mentioned that crude protein was increased by applying slow release fertilizers in maize grains.

Influence of hydroxyapatite urea and zero valent iron spraying on macronutrients of wheat plant

Hydroxyapatite urea and zero valent iron foliar application led to significant increments in macronutrients (NPK) of wheat plant as shown as in Table 2. The highest values of N, P, and K were recorded in HAU100+Fe. Hydroxylapatite ureas lowered the leaching and reduce nitrogen lost by inhibits the nitrification. So nutrients in soil were available. Data are in agreed with Abbas *et al*. (2012), Singh (2013) who stated that iron increased macronutrient uptake. Finally, hydroxyapatite urea with iron spray increased dry matter yield by 27%, N uptake up to 90% as compared to urea pellets.

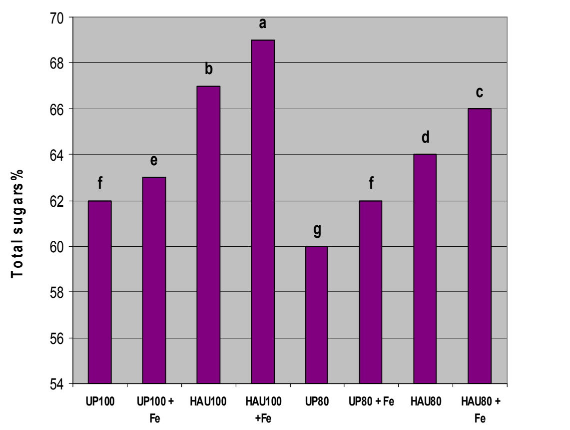

Fig. 5. Influnce of hydroxyapatite urea and zero valent iron on total sugars% of wheat

Nitrogen uptake and recovery efficiency of wheat plant

Nitrogen uptake of different treatments on wheat plant was analyzed (table 3) results observed that urea application with zero valent iron foliar treatments were increased the nitrogen uptake (26.5%) of wheat compared Urea pellets. Hydroxyapatite urea at two rates without or with zero valent iron foliar were increased nitrogen uptake. These increased was 67.6, 75.5, 89.8, 84.7%, respectively. The improvement in wheat yield and grain quality caused by hydroxyapatite coating and zero valent iron foliar may have been due to increased N use efficiency so one possible approach to improve the nitrogen losses from the surface applied urea is to coat it with hydroxyapatite. Recovery efficiency of nitrogen was increased using zero valent iron foliar with or without hydroxyapatite urea at two rates. Increasing reached to 1.27, 1.7, 1.9, 1.33, 1.79 and 1.89-fold than control, respectively.

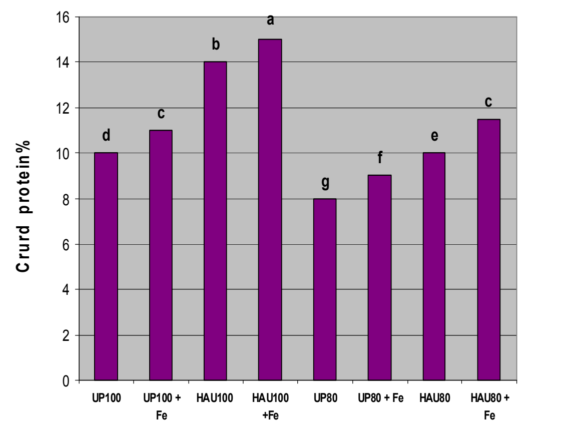

Fig. 6. Influnce of hydroxyapatite urea and zero valent iron on protein% of wheat

Table 2. Influence of hydroxyapatite urea and zero valent iron spraying on macronutrients

Treatment	N%	P%	K%
UP100	$1.740C^{D}$	0.180^{B}	0.229^{BC}
UP100 + Fe	1.910^{C}	0.200^{B}	0.250^{BC}
HAU100	$2.430A^{B}$	0.280^{A}	0.283^{B}
HAU100 +Fe	2.610^{A}	0.320^{A}	0.283^{B}
UP80	1.390^{D}	0.110^{C}	0.199^{C}
UP80 + Fe	1.570^{CD}	0.180^{B}	0.217^{C}
HAU80	1.740^{CD}	0.190^{B}	$0.240B^{C}$
HAU80 + Fe	2.000^{BC}	0.200^{B}	$0.256B^{C}$
LSD at 0.05	0.4600	0.05538	0.01826

Table 3. Uptake and recovery efficiency of nitrogen

Treatment	Grain yield (t/fed)	N %	Uptake (Kg/fed)	Nitrogen use efficiency %	Agrominic nitrogen efficiency (Kg/Kg)
UP100	2.3	1.74	40.0	3.877	29.00
U100 + Fe	2.65	1.91	50.6	4.937	33.67
HAU100	2.76	2.43	67.1	6.582	35.13
HAU100 +Fe	2.91	2.61	75.9	7.470	37.13
UP80	1.94	1.39	26.9	3.215	30.25
UP 80 + Fe	2.26	1.57	35.4	4.279	35.58
HAU80	2.44	1.94	47.3	5.761	38.58
HAU80 + Fe	2.49	2	49.8	6.069	39.42

Nitrogen Use Efficiency%= [(N uptake of N treatment − N uptake of N deficiency treatment) / N unit application]×100.
Agronomic N efficiency (kg/kg)= (yield N treatment − yield N deficiency treatment)/ N unit applied

Economic value:

The results of the analysis showed in table (4), Total return of wheat was depended on grains yield. The cost of growing experiment was calculated. Generally, there was Reasonable profit for some treatments because their investment factor more than 3 (FAO 2000). The maximum profit was obtained from the treatment of HAU100+Fe followed by the treatment of HAU100 then UP100 + Fe. The data also showed that the highest investment factor was recorded to HAU100+Fe treatment followed by the treatments of UP100 + Fe and HAU100 that had net return (6265 and 6484 LE), respectively.

Table 4. Economic value of hydroxyapatite urea and zero valent iron.

Treatment	Grain yield (ton/fed)	ardab/ fed	Total return (TR)	Total cost (TC)	Net return (TR)	Investment factor (IF)
UP100	2.3	15.3	6440	1135	5305	5.67
UP100+Fe	2.65	17.7	7420	1155	6265	6.42
HAU100	2.76	18.4	7728	1244	6484	6.21
HAU100+Fe	2.91	19.4	8148	1248	6900	6.53
UP80	1.94	12.9	5432	1050	4382	5.17
UP80+Fe	2.26	15.1	6328	1070	5258	5.91
HAU80	2.44	16.3	6832	1190	5642	5.74
HAU80+Fe	2.49	16.6	6972	1180	5792	5.91

TR = yield × price (grain + straw) NR = Total return − Total cost of production IF = Net return (NR) / Total cost of production

CONCLUSION

This study evaluated hydroxyapatite and zero valent iron foliar individually or combined as a slow release strategy for sustained release of nitrogen into the soil to save fertilizer consumption and to minimize environmental pollution. The results observed that, hydroxyapatite urea released NH4+ for a longer period than urea pellets. Application of urea pellets fertilizers led to a higher concentration of nitrate in drainage water than hydroxyapatite urea. It is concluded from these results that controlled released urea fertilizer improved yield by 27%, compared with urea pellets so one possible approach to improve the nitrogen losses from the surface applied urea is to coat it with hydroxyapatite.

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التأثير البيئي لتطبيقات اليوريا هيدروكسى أباتيت على تحسين استخدام الأسمدة النيتروجينية الكيميائية لنبات القمح

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وتصف هذه الدراسة استراتيجية للتيسر البطئ للنيتروجين في التربةعن طريق استخدام يوريا هيدروكسى اباتيت وزيادة انتاجية نبات القمح باستخدام الرش بالحديد النانوي. أجريت تجربة التحضين باستخدام عينات التربة لدراسة كفاءة استخدام اليوريا المغلفة بالهيدروكسى اباتيت على تيسر أيون الامونيوم تحت الظروف المشبعة والسعة الحقلية لمدة 63 يوم. اجريت تجربة حقلية لدراسة تأثير اليوريا هيدروكسى اباتيت مع أو بدون رش الحديد النانوي على تحسين كفاءة استخدام الاسمدة النيتروجينية، وإنتاجية القمح، والمكونات الكميائية للحبوب وتقليل تركيز النترات فى الماء الارضى. واستخدمت اليوريا في معدلين 80 و 100٪ من الجرعة الموصى بها. اوضحت نتائج تجربة التحضين أن انطلاق ايون الامونيوم كان أعلى تحت الظروف المشبعة عن السعة الحقلية. كان انطلاق ايون الامونيوم من اليوريا المغلفة بالهيدروكسى اباتيت بطئ ولفترة زمنية طويلة مقارنة باليوريا الغير مغلفة. وأظهرت نتائج التجربة الحقلية أن تطبيق الأسمدة المغلفة من اليوريا أدى إلى قلة تركيز النترات في المياه الجوفية. أدى تطبيق اسمدة اليوريا المغلفة بالهيدروكسى اباتيت وكذلك الحديد النانوي بشكل فردي أو مجتمعة إلى زيادة معنوية في محصول الحبوب من القمح مقارنة باليوريا غير المغلفة. كانت الزيادة فى الترتيب التالى: مقارنة باليويا 100% U100+Fe (11%) < HACU100 (20%) < HACU100+Fe (27%) مقارنة باليوريا 80% while U80+Fe (10%) < HACU80 (23%) < HACU80+Fe (30%) كما أظهرت النتائج انخفاض في محصول القمح باستخدام اليوريا 80٪، وبلغ هذا الانخفاض 16٪ مقارنة مع اليوريا 100٪. وقد لوحظ أن هذا الانخفاض قد قل باستخدام رش الحديد النانو U80+Fe . وبلغت نسبة الانخفاض في الحبوب 6٪. وقد بينت النتائج أن باستخدام اليوريا المغلفة والرش بالحديد النانو زاد المحصول بنسبة 27٪، والبروتين بنسبة 50٪ بالمقارنة باستخدام اليوريا غير المغلفة. ويمكن الاستنتاج من النتائج السابقة امكانية استخدام الهيدروكسى اباتيت وكذلك الرش بالحديد النانو لترشيد استخدام الأسمدة المعدنية النيتروجينة ، وبالتالي الحد من التلوث الناجم عن استخدام هذه الأسمدة، وكذلك خفض تكاليف الإنتاج الزراعي .