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Creation & Characterization of Different Coated Urea Materials & Their Impact as Controlled Release Fertilizers

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ABSTRACT

With the increasing of public concern toward human health and environmental protection, there is a shift toward to develop a cofriendly fertilizer based on natural resources. In this study, a coated urea by different materials such as phosphate slimes (PCU), silica fume (SiCU), bentonite (BCU) with Chitosan as binder were created to enhance the slow release mechanism. Characterizations of the coated urea were done using Scanning Electron Microscopy (SEM) with energy dispersive, X-ray analysis (EDX), Spectroscopy Fourier Transform Infra-Red (FTIR), refractive Index (RI) and Crushing Strength Test. The static release experiment showed that the (PCU), (SiCU) and (BCU) gave a release of 4.0, 4.7 and 4.9 % at the first day and 63.4, 74.0 and 82.7 after 30 days, in the same condition, respectively. Crushing Strength Test was carried out for the same size and showed that (PCU), (SiCU) and (BCU) overcame, the uncoated urea

INTRODUCTION

Nitrogen is the most widely applied plant nutrient element and has commonly been considered the yield limiting one. However, the degree of utilization of nitrogen fertilizers is in the range of 30–50% (Ibrahim and Jibril 2005).

Urea fertilizer is widely used as a principal source of nitrogen in agriculture because of its high N% and low cost of production nitrogen unit comparing with the other nitrogen fertilizers (Ariayathilaka et al., 2008). Unfortunately, urea is highly soluble in water and in regions with high precipitation which leads to loss the nitrogen through volatilization, and leaching in the soil before plants have an opportunity to assimilate it. For this reason, Allison, (1955) and Lundt, (1971) have reported that, as much as 75% of the nitrogen may be lost in areas with high intermittent rainfalls. However, inefficient and indiscriminate use of urea often creates serious environmental pollution as well as economical loss (Al-Zahrani, 1999 and Kent, 2007). Therefore, it were created enormous interest by the researchers to develop a new technology that will help to overcome the limitations of applying urea and high costs of nutrients necessitate economizing their use.

Slow-release fertilizers (SRF) or controlled-release fertilizers (CRF) are designed to slow down the nitrogen release speed, and if possible, to coincide with the nutrient requirement of the plant. They includes (Wang et al., 2014) inorganic materials and/or organic polymers which slightly water soluble covert the granules. Du *et al.* (2007) studied the effect of several SRF) on decreasing ammonia volatilization and N leaching. The results indicated that the development of SRF is a green technology that not only reduces nitrogen loss caused by volatilization and leaching but also alters the kinetics of nitrogen release. Zhang *et al.* (2014) studied CRF encapsulated by graphene films. The results showed that the new coating technology could hold great promise for the development of environmental benign and for crop production. Song *et al.* (2014 found that polygorskite-coated fertilizer could be promising for potato production and be beneficial to agricultural sustainability in semiarid areas. Zou *et al.* (2009) studied effect of different concentrations of natural macromolecular compound on the characteristics of nutrient release in the membrane materials of organic- inorganic compound film-coated urea. The results showed that the concentration of 5% of natural macromolecular compound showed better characteristics of nutrient release and can be utilized as a membrane material combined with inorganic mineral powders to develop film-coated SRF.Accordint to (Xu, 2006; Wu and Liao, 2000; Feng et al., 2005; Pan et al., 2006;and Zou et al., 2006) inorganic minerals include silica, sulfur, gypsum, phosphates, zeolite, bentonite, maifanitum, diatomite, etc. Du et al., (2013) studied the waterborne silicone-acrylate emulsions with excellent performance of nutrient controlled release, which provided novel and improved materials for coated CRF industry. Recent studies have concentrated on choosing membrane materials, particularly for creating a low-price product that is ecofriendly and has beneficial effects. Xiaoyu et al., (2013) used bentonite and organic polymer. Coating urea with bentonite showed decreasing in released nitrogen (EI-Leboudi et al., 1997) Chitosan, the second great amount of natural polysaccharide on earth crust, as a compound of chitin deacetylation is a biodegradable and non-toxic material for environment. Chitosan is widely used to produce controlled release materials in various fields, particularly in controlled release fertilizer (CRF) manufacturing (Muzzarelli et al., 2007and Wu 2008). Chitosan is highly potential to be blended with urea fertilizer for slow release properties due to its unique polymeric cationic character and gel forming properties (Mohamad et al.,2013) The ultimate aim of this study is to develop ecofriendly and low cost coating materials to synthesize coated urea granule, based on Chitosan and different inorganic materials (phosphate slimes, silica fume and bentonite). In this contribution, three different types of coated urea, i.e., phosphate slimes-urea (PCU), silica fume-urea (SiCU), and bentonite-urea (BCU) were prepared and characterized. Different properties of the synthesized materials such as, release time and crushing strength of coated and uncoated urea as well as enhanced slow release of nutrients when a fertilizer is applied were examined.

MATERIALS AND METHODS

Materials:-

Commercial urea granules with a nitrogen content of 46.44% and sizes ranging from 1.0 to 4.0 mm were supplied from El Delta Company for Fertilizers, Talkha, Egypt. The selected superfine powders materials for coating phosphate slimes, silica fume and bentonite were obtained from El Nasr Mining Company, Edfu, Aswan, Egypt, The Egyptian Company for Fertilizers, Kima, Aswan, Egypt and a Kalabasha area (south of Aswan city, Egypt) respectively. Chitosan powder was purchased from Sigma-Aldrich Inc.

Methods:-

Preparation of PCU, SiCU and BCU Granules:-

Urea was used for further experiments. PCU, SiCU and BCU granules has been prepared as follows: urea fertilizer was placed into rotary drum (Egyptian Fertilizer Development Center, Egypt).The schematic diagram is shown in Fig.1, The rotary drum of 50 cm diameter and 30 cm depth was positioned at an angle of 30^o to the horizontal under rotation speed about 35 rpm (Blouin et al., 1971). At this step, Chitosan as binder was sprayed over the urea grains inside the drum. After this, a certain weight of coating powder was spread inside the drum. The process was finished until a compact and homogeneous coating formed on the fertilizer granule**.** The products were collected for drying and analysis. The operational parameters are showed in Table 1

Table 1. Operational Parameters of the Rotary Drum:-

Rotating Speed	**35- rpm**	**Percent of Chitosan**	**4.0 % every batch**
Time of Coating for Batch	20-30 min	Weigh of Coated Materials	700 g every batch
Weigh of urea	5 kg	Drying Temp	٦٠ – ٧0 ˚C

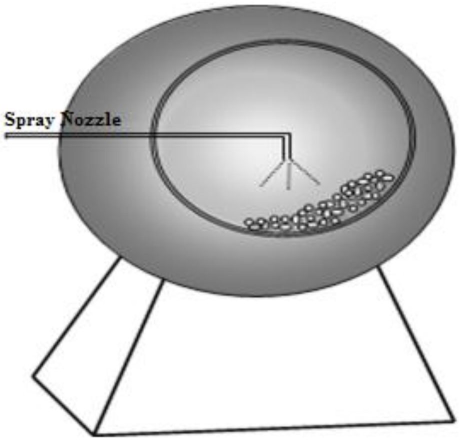

Fig. 1. Schematic Diagram of a Rotary Drum Technique.

a: Total Coating %.

The actual coating % is determined as follows: 20 g of coated urea is crushed and blended with water to accelerate the dissolution of urea. The solution is then filtered, and the remaining insoluble solid material is dried in the oven until constant weight (Salman, 1988) The coating % is calculated from eq1.

$$\% \text{ coating} = \text{wt of residual (g)} / 20 \times 100 \quad \text{-- (1)}$$

b: Nitrogen Content.

The nitrogen content of coated urea is determined by the standard Kjeldahl method (Kjeldahl, 1883). The sample was treated with sulfuric acid to yield ammonium sulfate. The nitrogen content can be calculated by the hydrolysis of ammonium sulfate. The nitrogen content is calculated more accurately from the total coating percentage by eq 2

$$\% \text{ N} = 46.44(100 - \% \text{ coating}) / 100 \text{ –(2)}$$

Table 2. Nitrogen Content in the Coated and Uncoated Urea.

Type of sample	**Coating, %**	**Urea, %**	**Nitrogen, %**
UCU	0.00	100	46.44
PCU	14.39	85.61	39.76
SiCU	14.86	85.14	39.54
BCU	14.62	85.38	39.65

c: Morphology and Microscopic Analysis of the Surface:-

The morphology and relative elemental concentration for samples were analyzed by means of scanning electron microscopy (SEM) (JEOL microscope, model Quanta 250 FEG) equipped with an energy dispersive analysis system of X-ray spectrometer-EDX (Thermo Scientific NSS coupled or linked). Samples were dispersed over a carbon tape pasted on the surface of a metallic disk (stub). Then, the disk was coated with gold in an ionization chamber (BALTEC Med. 020) and then the samples are ready to be analyzed.

d: Fourier Transform Infrared (FTIR) Analysis:-

FTIR (Cary 630 FTIR) analysis was conducted to confirm the presence of urea in the fertilizer samples. Functional groups of chitosan based urea fertilizer were determined using FTIR which is known as a powerful tool for identifying type of chemical bonds and functional groups in a molecule by using infrared absorption spectrum. Fertilizer granules were dispersed in dry KBr powder and ground to obtain fine particles. All spectra were recorded at ambient temperature and the analysis was performed at wavelength range from 349.05 cm^{-1} to 4,000.60 cm^{-1} at scanning speed of 2 mms^{-1} with aperture size of 7.1 mm.

e: Determination of Urea Release by refractive index:-

The test was carried as follow : 50 g of sample was placed with 250 ml distilled water in an erlenmeyer flask and sealed (Lixing. et. al (2010). The Baush& Lomb Abbe- 3L Refractometer was used to measure the refractive index (RI) of urea released in the solution. The refractometer was calibrated daily before measurements against a known refractive index of water and measured at 25°C as a function of time for 1, 2, 3, 6, 9, 12, 15, 18, 21, 24, 27 and the day of 30.Th e value of (RI) is related to the concentration of urea dissolved in water

f: Measurement of Crushing Strength:-

The crushing strength is an important test to ensure that the product can withstand physical handling throughout the supply chain. It is measured by applying a pressure to individual granules, usually of a specified size and noting the required pressure to fracture each granule. The test was performed on the different coated and uncoated urea. To do this, 20 granules was randomly selected from the majority population based on the sieve test. A Chatillon TCM tensile strength tester with capacity of 50-100 k/granule was used for measuring by applying a compressive force on a single granule. So, granules were subjected to a force applied by a metal plunger that was a part of an apparatusand the values were taken triplecats.

RESULTS AND DISCUSSION

Morphology and Microscopic Analysis of the Surface:-

Among visible methods, Scanning Electron Microscopy (SEM) with an energy dispersive analysis system of X-ray spectrometer-EDX is the best to study the morphology and relative elemental composition of the granules. The surface was observed to check for layering and agglomeration. Layering is desired for the particle coating. Surface irregularities and shape of granules was observed. Several granules were selected randomly and observed under optical microscope at magnifications of 500 and 2000X . Images were taken at these magnifications and analysis of these images for changes in surface properties was done. sample images (Figure2 and 3) are shown for uncoated and coated urea granules with different materials. The (SEM) images shown in figure 3 (C, D), (E, F) and (G, H) revels clearly the change in morphology due to coating of urea with different materials. The urea coated by PCU, SiCU and BCU gives more uniform surface as compared to uncoated. The uncoated urea(Figure 3 A-B) formed an irregular surface like a membrane with disordered shape. EXD analysis showed no remarkable presence of any farther elementals ruther than the pure urea (fig 4 A) while the elemental composition of phosphate, silica and bentonite was attained on surface of coated urea (Fig 4 B, C, D.)

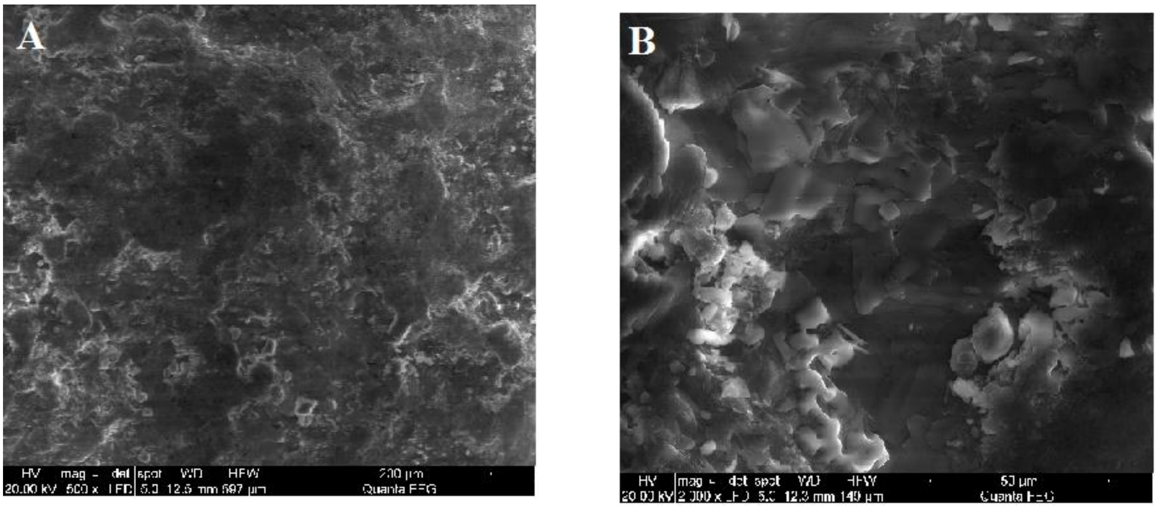

Figure 2 (A, B). Optical microscope image of uncoated urea

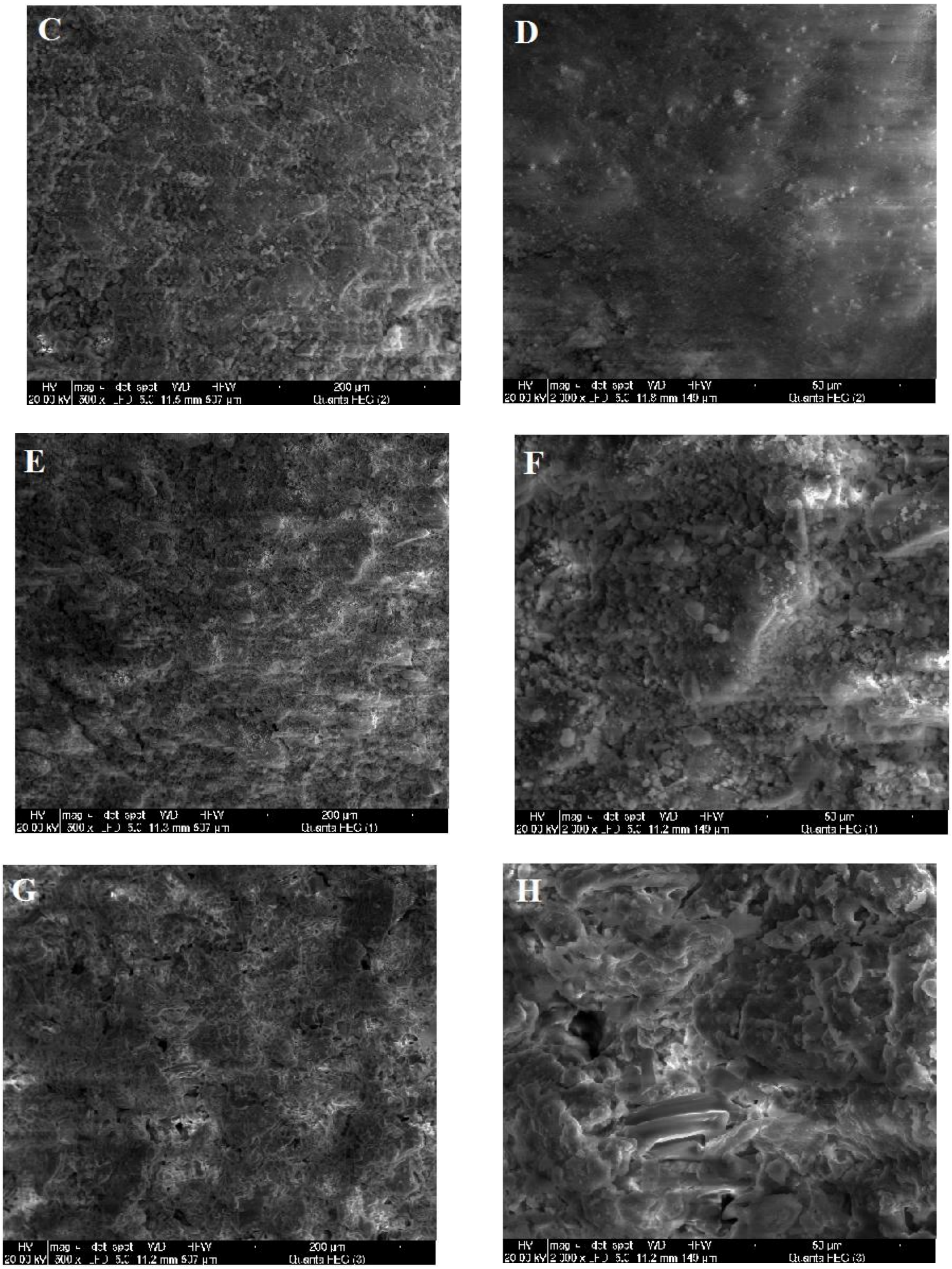

Figure 3. Optical Microscope Images of PCU (C, D), SiCU (E, F) and BCU (G, H) at Different Magnifications (500, 2000 X).

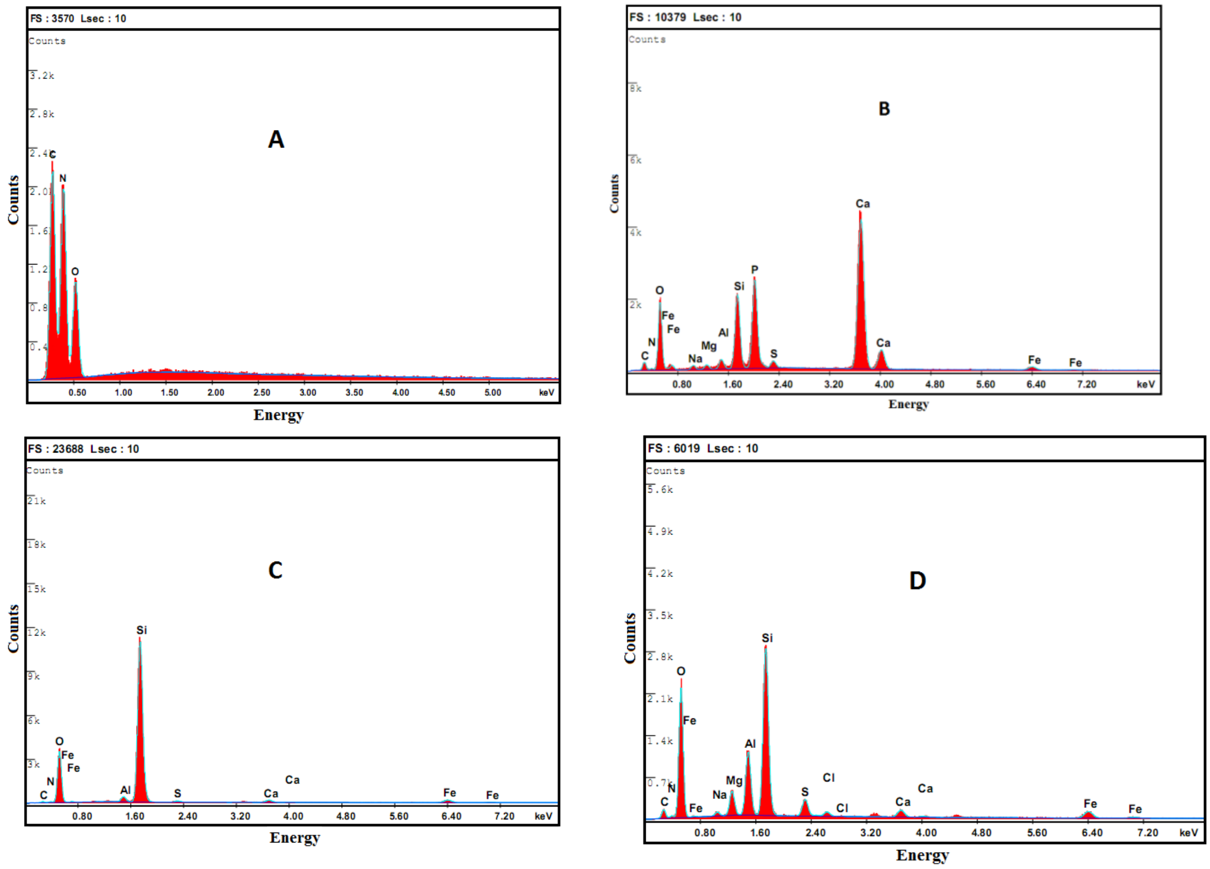

Fig. 4. EDX Diagram of (UCU) (A), (PCU) (B), (SiCU) (C) and (BCU) (D) Materials.

FTIR Spectra Analysis for Uncoated and Coated Urea:-

The FTIR spectra of samples phosphate, silica and bentonite coated urea (Fig 5 A-D) are show in similarities spectra of uncoated samples. Peaks at 3425 and 3322 cm^{-1} of plain urea and the sample formulas are asymmetric and symmetric range vibrations NH_2. Peak at 3252 cm^{-1} of the 3 samples urea (PCU, SiCU and BCU) for OH vibrations of absorbed water molecules. Peak at 1675 cm^{-1} is acarbonyl (CO) and at 1588 cm^{-1} is NH bending vibration and stretching vibration of CH (usually an area of bending vibrations NH) O = C-NH_2. At 400- 1500 cm^{-1} or fingerprint region, all the usual peak of urea samples and the samples look very similar (Costa et al., 2013; Xiaoyu et al., 2013; Muslim et al., 2015). This indicates the proper coating of phosphate, silica and bentonite on the surface of urea.

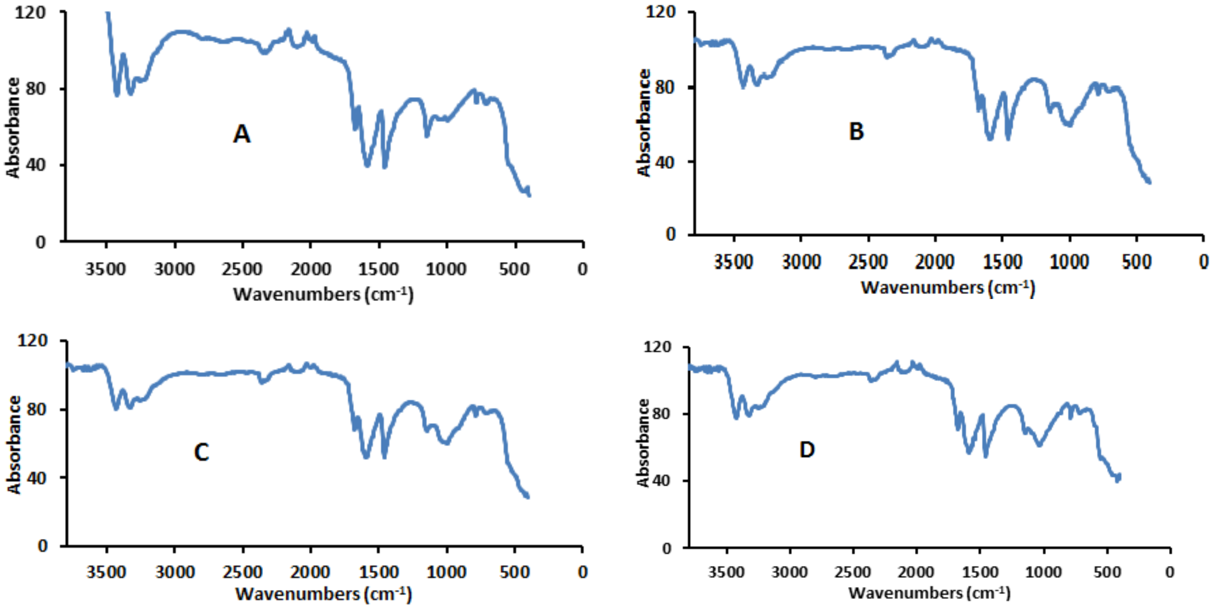

Fig. 5. FTIR Spectra Analysis for UCU (A), PCU (B), SiCU (C) and BCU (D).

Urea Release Using Refractive Index:-

The refractive index is able to be used to measure the concentration of urea in solution directly and the method possesses advantages, such as fast analysis, high efficiency, without any chemical or reagent consumption as well as pollution-free (Xie et al., 2011). Urea release, % (nitrogen content) and refractive index values for different coated urea were measured and compared with uncoated urea. The measuring was continued until the values did not change over a period of time which means that the granules release all the nitrogen content to the water during this period of time. The results are listed in the Table (3) and Fig. (6, 7) that show the variation in urea release of uncoated urea, PCU, SiCU and BCU at the different time. The results showed that the urea release increase time. The results indicated that 4.0, 4.7, 4.9 % of PCU, SCU and BCU, respectively released into the water during the first day and the maximum of release rate appeared on the day 30 which gave 63.4, 74.0 and 82.7%, respectively. On the other hand the uncoated urea released all its nitrogen content within one hour (Muslim et et al., 2015).

These indicated that, slow release properties of PCU, SiCU, and BCU samples obey the Standard of Slow Release Fertilizers of Comité Européen de Normalisation (CEN).According to CEN, a fertilizer can be described as having controlled release properties if the nutrient release is not more than 15% after 1 day or not more than 75% after 28 day (Trenkel 2010).

Shaohua et al., (2012) found that, the released of nitrogen was less than 15% on the third day and reached up to almost 75% on the 30 days in distilled water by preparing cross-linking poly (acrylic acid-acrylic amide)/ bentonite and urea.

Table 3. Urea Released of Coated Urea and Uncoated within 30- Day.

Sample	Urea Release, %	
	1 day	30 day
UCU	100% from The First Day	
PCU	4.0	63.4
SiCU	4.6	73.1
BCU	4.8	81.9

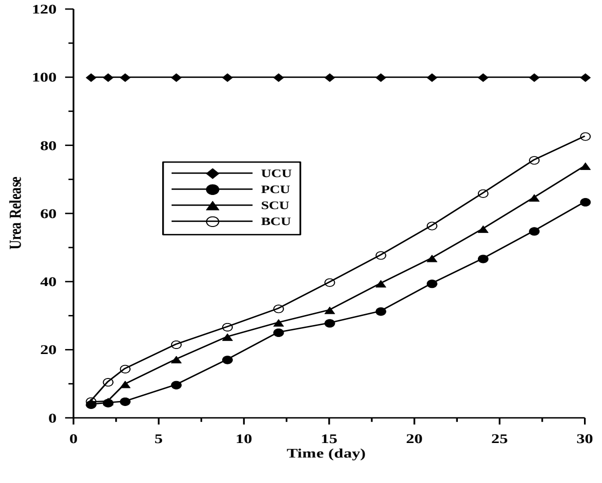

Fig. 6. Urea Release % of Uncoated and Different Coated Urea.

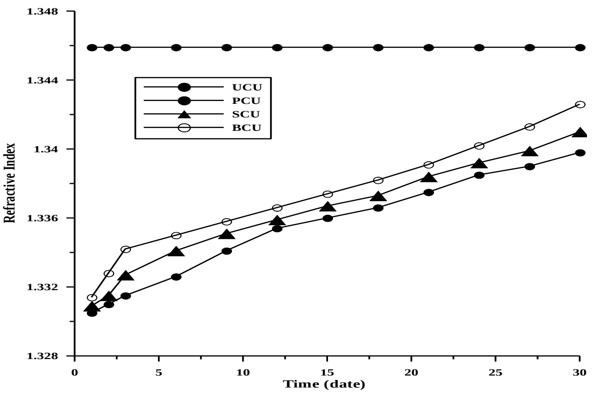

Fig.7. Refractive Index Values of Uncoated and Different Coated Urea.

Crushing Strength:-

It is defined as the force required to crush the particle (Vashishtha et al., 2010)

On comparing crushing strength data, it is important to compare equisize granules, because crushing strength increases significantly with the increase in coated urea fertilizer should have sufficient mechanical strength to withstand normal handling and storage without fracture. The mechanical strength of a granule is influenced by its chemical composition, porosity, shape, surface crystal and moisture content (Salmanet et al., 2003 and Coury et al 2003). Masses average strength expressed in k/granular was determined both before and after coating for particles of size 2.0, 2.8 and 3.1mm from (Table 4 and Fig 8). In all cases, the coated urea with different particle size have a values of crushing strength than that of uncoated urea (Ibrahim et al., 2014).

Table 4. Crushing Strength and Particle Sizes Relationship.

Sample	Crushing Strength Kg/Granular		
	2.0 mm	2.8 mm	3.1 mm
UCU	1.51	1. 85	2.11
PCU	1.80	2.19	2. 38
SiCU	1.81	2.24	2.45
BCU	1.67	1.93	2.26

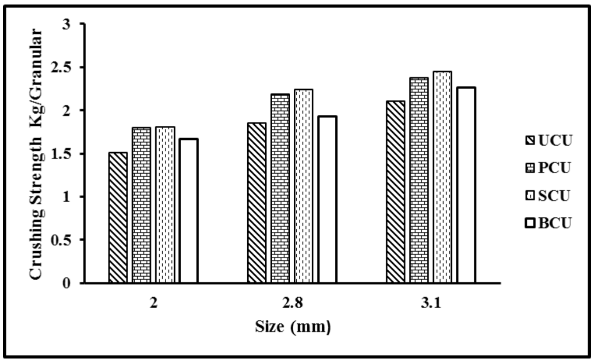

Fig. 8. Crushing Strength and Particle Sizes Relationship.

In addition increasing of the granule size, led to increase force to crush. The same phenomenon was observed by (Walker et al., 1997).

Application:-

Application of the created fertilizer (PCU, SiCU and BCU) against the control and UCU fertilizer and tracing the wheat yield as Ardab/Fed and the residual nitrogen in soil as well reveled the following:-

Data in Table (5) showing the statistical analysis done by ANOVA using least significant difference at $P \leq 0.05$ and showing the effect of slow release urea (PCU, SiCU, BCU) and fast release urea (UCU) on grain of wheat plants during the winter season 2014/2015. It could mentioned that, the application of (PCU, SCU, BCU and UCU) gives high significant increases grain yield than control (6.5 Ardab/ Fad) while uses (PCU, SiCU, BCU) as a source of nitrogen fertilizer gives the high of grains yield than treatment of UCU (13.22 Ardab/ Fad).

It could also notice from the application of (PCU) for wheat plants increases grain yield (20.72 Ardab/ Fad) than all treatments of (SiCU and BCU).

The superior of Slow Release N fertilizer (PCU, SiCU and BCU) than UCU can be attributed to the slow release of N to meet wheat plant requirement, where slow release has allow dissolution rate than urea which reduces nitrogen uptake by plant root. The present results are in agreement with those obtained by (El-Aila et al., 1998; and Yang et al., 2011). Table (5) showing also the residual N in soil after harvest using (PCU) as slow release give residual N in soil 73.03 mg/kg soil which higher than all treatments. The present results are in consistent with those obtained by (Mikkelsen et al., 1994).

Table 5. Effect of PCU, SiCU, BCU and UCU Fertilizers on Grain Yield and Residual N in Soil.

Treatments	Grains Yield, Ardab/ Fad	Residual N mg/kg soil
Control	6.5	16.0
UCU	13.22	43.3
P C U	20.72	73.03
Si CU	19.28	64.6
B CU	18.42	61.6
Significance	**	**
LSD 0.05%	4.357	5.404

CONCLUSION

In summary, the new low cost and ecofriendly Slow Release Fertilizers were developed by coating urea fertilizer granules with different materials such as phosphate slimes (PCU), silica fume (SiCU), bentonite (BCU) and biodegradable polysaccharides chitosan. The static release experiment showed that (PCU), (SiCU) and (BCU) gave a release of 4.0, 4.7 and 4.9 % at the first day and 63.4, 74.0 and 82.7 at 30 day, in the same condition, respectively. Also, the results showed that Crushing Strength Test was carried out for the same size and showed that (PCU), (SiCU) and (BCU) were overcome the uncoated urea (UCU). Therefore, this approach showed a promise in the utilization of abundant, low cost, and natural resource such as phosphate slimes, silica fume, bentonite and polysaccharides as chitosan in the production of the coating material, which could significantly reduce the production cost and make the technique quite ecofriendly.

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تخليق وخصائص لمواد مختلفة لتغليف اليوريا وتاثيرها على الانطلاق البطئ للأسمدة

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مع تزايد الاهتمام الشديد نحو صحة الانسان وحماية البيئة ظهرت توجهات هامة نحو تطوير اسمدة صديقة للبيئة تعتمد اساسا على مصادر طبيعية ز فى هذه الدراسة تم تحضير يوريا مغلفة بمواد مختلفة مثل حجر الفوسفات و السيلكا والبنتونيت بعد تغليف حبيبات اليوريا بمادة لاصقة وهى مادة الشيتوسان وذلك لتحسين ميكانيكية الانطلاق البطئ لعنصر النيتروجين من اليوريا. خصائص ومميزات حبيبات اليوريا بعد عملية التغليف تم دراستها عن طريق جهاز الميكروسكوب الماسح الالكترونى وتحليل العناصر الموجوده بجهاز اشعة اكس بالاضافه الى استخدام جهاز الاشعه تحت الحمراء الاسبكترو سكوبى لتحديد ما اذا كانت روابط جديدة تكونت ام لا . اما تاثير التغليف على انطلاق اليوريا والمدة اللازمة لذلك تم استخدام طريقة قياس معامل الانكسار وتبين من النتائج ان اليوريا المغلفة بحجر الفوسفات والمغلفة بالسيلكا والمغلفة بالبنتونيت انطلق منها نسبة ٤% و ٤.٧% و٤.٩% بالترتيب فى اول يوم بينما بعد ٣٠ يوم وصلت نسبة الانطلاق الى ٦٣.٤% و ٧٤.٠% و ٨٢.٧% بالترتيب . وطريقة اختبار قياس الصلابه لمعرفة تاثير التغليف على قوة وصلابة الحبيبات حيث اعطت نتيجة ان جميع الحبيبات المغلفة تفوقت على الغير مغلفة بينما المغلفة بالسيلكا الاكثر صلابة.