Fabrication of Slow Release Fertilizer Based on Urea – Saw Dust and Gelatin Binder

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Abstract

The increasing usage of slow release fertilizers has become an essential demand to save fertilizer consumption and reduce the environmental pollution. In this study, two different techniques were used to prepared slow release urea fertilizers (SRUF_X and SRUF_Y) composed of urea, sawdust and gelatin as a binding material. The two prepared fertilizers were characterized by Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray (EDX) and X-Ray diffraction (XRD). Urea static release was studied and indicated a prolongation of the release pattern of urea with approximately three times by incorporating saw dust and gelatin. The kinetics of the static release showed that SRUF_X and SRUF_Y follow non-fickian release. The effect mechanical stress (1, 1.5 and 2 MPa) on the release time of urea was studied and indicated an increase in time of release as the stress increases.

1. Introduction

Environmental pollution and lack of resources are series problems that nowadays face the global. Moreover, the world population is expected to reach 8 billion over the coming years. These problems raised the need for enhancing the quantity and quality of the produced food, this cannot be achieved without fertilization that is used for enhancing plant growth and increasing the yield of crops (FAO-ONU, 2017).

Urea ($\text{CO}(\text{NH}_2)_2$) fertilizer is used as the main source of nitrogen (N), which is one of the major nutrients that is used in large scale in fertilizers manufacture. About 48% of the world population today is alive by means of nitrogen based fertilizers (Leghari et al., 2016; Reetz, 2016). Plants consume about 30-50% of urea, and the remaining ratio of the fertilizer is wasted due to its high solubility into water, Urea leaches into the underground water in the form of nitrates (NO_3^-), runs off with irrigation or volatiles in the form of nitrous oxide (N_2O) causing different forms of pollution. Wasted urea causes reduction of nitrogen utilization efficiency (NUE) besides the economic loss due to the ineffective usage and repeated application of urea fertilizers (Ruby Celsia and Mala, 2014; Mokhtar et al., 2015; Chandini *et al.*, 2019; Zhang et al., 2020).

To elimination or decrease these adverse effects, synthesis of slow release fertilizers (SRFs) became an essential demand. These fertilizers can be fabricated using many techniques with several modifications of the conventional fertilizers, one of these modifications is done to increase the time of nutrient availability for plant by increasing the release time with comparison of conventional fertilizers (Wijesinghe and Weerasinghe, 2015; Lubkowski, 2016). There are some reports that used natural recourses in manufacturing slow release urea fertilizers as bentonite, chitosan, corn starch, biochar from banana peel or corn cup and other biodegradable materials (Fu et al., 2018; Himmah, Djajakirana and Darmawan, 2018; Lateef et al., 2019; Hermida and Agustian, 2019; Hussein et al., 2019).

In this study, sawdust-based slow release urea fertilizers were prepared with gelatin as a binding material; these materials have low cost and biodegradable characteristics that distinguish this work. This

approach will not only help in waste management and pollution prevention, but it will also increase NUE. Compressed composite of urea, saw dust and gelatin was prepared and showed prolonged time of release than uncompressed composite.

2. Experimental

2.1. Material

Saw dust was collected from the residues of white wood from a local source then it was screened, washed couple times with distilled water and then dried at 100 °C for 8h. Gelatin pellets was purchased from a local vender and was then crashed before use. Urea was purchased from Sigma-Aldrich and was dried at 80°C for 8h.

2.2. Preparation of slow release urea fertilizers (SRUFs)

2.2.1. Preparation through recrystallization method (SRUFX)

The proposed method was performed according to (Hermida and Agustian, 2019) with some modifications as follows: The taken amount of urea (90 g) was melted at 130-135 °C on a hot plate then sawdust was added and mixed with stirring for 5 min, or until full combination. On a separate beaker, 6 ml of distilled water was added to 0.2 g of gelatin and heated on hot plate at 60 °C, (a viscous liquid was formed) then, added to the urea-sawdust mixture with stirring till full combination. The obtained SRUF was then dried for 8h at 80 °C.

2.2.2. Preparation through suspension method (SRUFY)

Sawdust (5 g) was added to urea solution (20 g was dissolved in 30 ml distilled water) with stirring for 30 min. A suspension was formed then, dried at 80 °C. On a separate beaker, 18 ml of distilled water was added to 2.4 g of gelatin and heated on hot plate at 60 °C. The formed viscous liquid was added to the urea-sawdust mixture was stirred for 30 min. The final product, SRUF, obtained was then dried for 8 h at 80 °C (Lateef et al., 2019).

2.3. Characterization

The prepared SRUFs were characterized by Fourier Transform Infrared (FTIR) spectra (FT/IR-4100typeA) to identify the organic function groups in the composition. Scanning Electron Microscopy (SEM) was done using (Scanning Electron Microscope model JEOL JSM 6510 Iv) and images were used to identify the morphology of the samples. Energy Dispersive X-Ray (EDX) was recorded by EDS Analysis for the SEM model Oxford X-Max 20) to confirm the elemental composition of SRUFs and X-Ray diffraction (XRD) was recorded using (Philips X'PERT-PRO diffract meter) to indicate the crystal structures of urea.

2.4. Static release experiment

It was carried out according to procedures of (Higuchi, 1963). It was carried out at room temperature, (25 – 30 °C), to determine the release pattern and mechanism of the pure urea and compare it with the prepared SRUFs. The system used for determining the static release of urea in water is shown in Fig. 1.

Generally, 3 g of urea or SRUF was put in a Wassermann tube with one end closed and the other opened. The tube was immersed horizontally in a glass beaker containing 250 ml distilled water to allow release of urea. The system was stirred at 100 rpm. After that, 0.5 ml of water was taken from two different points at the center of the beaker every 30 min. The urea concentration in water samples was determined spectrophotometry using UV-VIS spectrophotometer at 440 nm (Watt and Chrisp, 1954).

2.5. Water absorbance studies (WA)

Water absorbance studies were conducted, by taking 1.0 g of urea, SRUF_X or SRUF_Y (W_1) in Petri-dishes that were previously weighted (W_2). These petri-dishes were placed for 5 days in desiccator filled with water to create moist environment then, weighted again (W_3) to measure water absorption capacity of sample using Eq. 1.

$$\text{Water absorption, WA \%} = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

2.6. Water retention studies (WR)

Water retention experiments (WR) were conducted in pre-weighted cups, add 50 g of sieved soil in cup A (W_A) as control or blank and in cup U, X and Y (W_U , W_X and W_Y , respectively) add 2 g of urea, SRUF_X and SRUF_Y into the cups respectively with 50 g soil and then, 30 ml of distilled water was added in each cup, allowed to contact and reweighed after 24 h (weighed as W_{A1} , W_{U1} , W_{X1} and W_{Y1}). The cups were then retained in a glass box, weighted on daily basis for 30 days (weighed as W_{A2} , W_{U2} , W_{X2} and W_{Y2}) (Hamid et al., 2013; Lateef et al., 2019). Water retention was calculated using Eq. 2.

$$\text{Water Retention, WR \%} = \frac{W_2}{W_1} \times 100 \quad (2)$$

2.7. Urea release kinetics and mechanisms

Urea static release data were analyzed using the Eq. 3 by (Peppas, 1985):

$$Q_t = Kt^n \quad (3)$$

Where, (Q_t) is the fraction of urea released at time t , k is the kinetic constant, and (n) the diffusion exponent which indicates the urea release mechanism.

2.8. Mechanical stress

Samples weighed 0.2 g of each urea fertilizer and the SRUFs were subjected to three different pressures (1, 1.5 and 2 MPa), and they were given codes so that urea samples after subjected to stress were named

U_1 , $U_{1.5}$ and U_2 , the SRUFX samples were X_1 , $X_{1.5}$ and X_2 , also the SRUFY samples were Y_1 , $Y_{1.5}$ and Y_2 respectively. Then they were characterized by XRD to study the effect on the crystal structure. The static release experiment was conducted and compared with the release of 0.2 g of pure urea which was not subjected to any stress to study the effect of mechanical stress on the release rate of these fertilizers.

3. Results And Discussion

Two techniques were used in the preparation of SRUFs: The first is melting and recrystallization of urea (SRUFX). The second is formation of a suspension of urea and sawdust (SRUFY). Before testing for urea release using static release experiment, the prepared SRUFs were characterized by various techniques as follow:

3.1. FT-IR spectra

IR spectra of SRUFX and SRUFY are presented in Fig. 2.

spectra of SRUFY show bands at 3447 and 3362 cm^{-1} , while SRUFX show similar bands at 3440 and 3351 cm^{-1} , which were attributed to the stretching vibrations of asymmetric and symmetric NH_2 group of urea (Xiaoyu et al., 2013, Hosny & Sherif 2015; Hosny *et al.*, 2015). The bands assigned to amide I of gelatin lies in the region 3270-3370 cm^{-1} overlap with that of urea (Sherif & Hosny 2014). The incorporation of Gelatin in the composite was confirmed by the band attributed to proline side chain which appears at 1333 cm^{-1} .

The two strong bands at 1681 and 1626 cm^{-1} were attributed to

$\nu(\text{C}=\text{O})$ and δNH_2 of urea, respectively (Xiaoyu et al., 2013, (Tanong Aewsirie et al. 2008; Hosny et al., 2017).

The spectra exhibit three characteristic bands of cellulose and lignin

$\nu(\text{OH})$ at 3436 cm^{-1} , $\nu(\text{C}=\text{C})$ at 1637 cm^{-1} and $\nu(\text{C}-\text{O})$ at 1122 cm^{-1} . These bands were shifted to lower wavenumber which can be taken as an indication on the formation of hydrogen bond with urea (Zhang et al., 2016; Hosny and Belal, 2020; Hosny et al., 2019). The bands at 1460 cm^{-1} in SRUFY spectrum and the similar one at 1468 cm^{-1} in SRUFY spectrum were attributed to symmetric vibrations of $\nu(\text{NH})$ group.

From the above findings it could be deduced that, the three constituents (urea, saw dust and gelatin) have been incorporated forming a composite.

3.2. Morphology and chemical compositions

Figure 3.A shows SEM image of common urea surface without any modifications. It exhibits the surface of urea as smooth layers confirming that, urea molecules are well ordered (Xiaoyu et al., 2013) and a sediment rock like appears.

SEM images (Fig. 3B and C) show the surface of SRUF_X prepared from melted urea at different magnifications. In Fig. 3B, number of crystals appeared, with length in the range (80-400 nm) and width (40-200 nm), that were likely to be the gelatin crystals. Fig. 3C appears the surface of the prepared urea fertilizer is rough in comparison with common urea alone, as there were some flakes. It appears some deposits of sawdust coated the melted urea surface. The contrast in color suggests the presence of different materials which are the main components (urea, sawdust and gelatin).

SEM images (Fig. 3D and E) SRUF_Y prepared by deposition method at different magnifications. The contrast in color in Fig. 3D indicates that, the surface is not smooth as appeared in the common urea but, it has more rough flakes with some gaps between them. The gap size is the range of 10-300 nm. Also, some particles with spherical shapes appear with diameters 120 and 160 nm.

In Fig. 3E, the surface of SRUF_Y appears as irregular flakes that may refer to the sawdust. These flakes are aggregated and there are some bright spots in the middle that can be attributed to gelatin and urea.

From comparison between the images, it can be seen clearly that the surface of the two prepared SRUFs have different morphologies that affected by the preparations conditions.

The elemental analysis of urea, SRUF_X and SRUF_Y was done by EDX as shown in Fig. 4A, B and C and the elemental compositions of these fertilizers are provided in Table.1. The results confirm the presence of urea in both prepared slow release fertilizers. In SRUF_Y the N weight percent is rather smaller than that in SRUF_X that can be attributed to loss of some urea during the preparation procedures. In both SRUFs the presence of Zn and Cu traces could be attributed to their presence in sawdust.

3.3. XRD and mechanical stress

XRD of common Urea, SRUF_X and SRUF_Y where scanned in range of 15-80 ° (Fig. 5).

All samples show similar peaks at 22.5 and 35.6° correspond to the planes (110) and (210) of urea. However the common urea, without any modifications, had shown a sharp peak at 35.6°, a similar peak with lower intensity at 35.16° appeared in SRUF_X and SRUF_Y, the change in intensity could be attributed to the adsorption of recrystallized urea into the pores of sawdust (Xiaoyu et al., 2013).

The crystallite size was determined from the peak at 35.6° by substituting in Debye-Scherrer equation: $D = 0.94 \lambda / \beta \cos\theta$ (Klug & Alexander 1962). (The wavelength λ is 1.5406 Å for Cu K α radiation. β is the full width at half maximum and θ is the position of the peak) Using this equation, the crystallite size of common Urea, Urea in SRUF_X and SRUF_Y are 57.58, 57.51 and 54.84 nm, respectively.

The effect of mechanical stress on the common urea and the fabricated fertilizers was also studied by XRD as shown in Figs. 6, 7 and 8.

As shown in Fig. 6 urea before subjecting to any stress has a crystalline structure with two sharp peaks at 22.58° and 35.5°, when subjected to stress the sharp peak at 35.5° has become more broader and shifted to 35.22° as the particle size has reduced (Table 2) and the crystalline structure has deformed (Hosny 2015).

Table 1
The elemental composition of urea, SRUF_X and SRUF_Y

Element	Urea		SRUF _X		SRUF _Y	
	Wt%	At%	Wt%	At%	Wt%	At%
C	20.75	24.42	22.26	26.17	21.50	25.36
N	46.89	47.31	45.61	45.98	45.03	45.54
O	31.49	27.82	31.36	27.68	32.65	28.91
Zn	-	-	0.29	0.06	0.28	0.06
Cu	-	-	0.48	0.11	0.54	0.14

Table 2
The crystalline size of urea, U₁, U_{1.5} and U₂ which refer to urea subjected to mechanical stress 1, 1.5 and 2.0 MPa, respectively.

Fertilizer	D (nm)
Urea without stress	57.58
U ₁	43.39
U _{1.5}	35.22
U ₂	27.45

Table 3
The crystallite size of SRUF_X, X₁, X_{1.5} and X₂

Fertilizer	D (nm)
SRUF _X	57.51
X ₁	54.77
X _{1.5}	37.46
X ₂	34.86

The crystallite sizes of compressed samples are smaller than common urea which will affect on their behavior in the static release experiments.

The effect of mechanical stress 1, 1.5 and 2 MPa on SRUFX has been studied. The change in XRD patterns of SRUFX (Fig. 7) indicated that the sharp peak at 35.13° of SRUFX has moved to 35.13° with reduction in intensity and broadening due to decrease in crystallite size after subjecting to mechanical stress as shown in Table.3.

Applying mechanical stress 1, 1.5 and 2 MPa on SRUFY (Fig. 9) showed blue shift in the peak at 35.67° , deformation of the crystal structure and change in crystallite size.

3.4. Water absorbance (WA) and Water retention (WR)

The results of the water absorbance experiment for urea, SRUFX and SRUFY are 32.2, 71.0 and 68.53%, respectively.

The results showed an increase in water absorbance which can be attributed to physical adsorption of water on surface of sawdust which will release into the soil later. Water molecules can occlude also, into the porous structure of sawdust.

The results of WR are shown in Fig. 10 as predicted the water retention decrease by increasing time, in all of the tested samples. The water retention of the soil was the lowest and the retention increases by adding the fertilizers. The highest WR is observed after adding SRUFY then, SRUFX and the last fertilizer is urea. The increasing in WR could be attributed to the porous structure of saw dust.

3.5. Release rate of Urea, SRUFX and SRUFY

The static release experiment was done for the samples of common Urea, SRUFX and SRUFY to study time of release (Fig. 11).

The time dependent results of Urea in distilled water in (mmol/L) for each fertilizer indicated that, common urea is readily soluble in water as expected and reaches a concentration 130 mmol/L after 150 min., but both the synthesized fertilizers continued to release urea with lower dosage for about 510 min. The release rate of common urea is about the third of that for both SRUFX and SRUFY. This behavior can be attributed to the physical connection between urea, sawdust and gelatin as confirmed from FTIR results. The hydrophobic (insolubility) nature of sawdust supports this suggestion. The dilation in the solubility, of urea modified with sawdust and gelatin is suggested as follow: When melted or dissolved urea, comes into contact with sawdust a portion of urea runs into the intermolecular spaces of the sawdust structure which gives it some stability against water. Then, on addition of the gelatin binder, it must coats the sawdust gaps filled with urea on the same time, the rest of urea binds to the sawdust and coats with gelatin increasing the resistance of the prepared fertilizer to water solubility.

Comparison the release % of Urea, SRUFY and SRUFX was carried out in term of urea loss to check the suitable preparation technique (Fig. 12). The results indicated that, the portion of urea released in water is lower for SRUFX in comparison with common urea. The in solubility of SRUFs after modification of urea by sawdust and gelatin, may be attributed to the coordination of urea to metal ions exists in saw dust forming insoluble metal-urea complexes besides coating of urea with gelatin. The highest urea fractions is recorded in case of SRUFY with melted urea, as it can form complex compound in the same way as it reacts in Biuret test (Mikkelsen, 1990). Also a portion may have volatilized during melting as the preparation was done in an open system on a hotplate. SRUFY released a higher fraction of urea *ca.* 20% of the urea lost.

3.6. Urea statics release

The Peppas equation was used for the analysis of static release data of both SRUFX and SRUFY. The kinetic constant (K) and diffusional coefficient (n) were calculated and used to identify the release mechanism of the prepared fertilizers (Xiaoyu et al., 2013).

For SRUFX, the kinetic constant (K) and diffusional coefficient (n) were 1.170003 and 0.6293266 respectively, thus the release rate model of SRUFX was $Y=1.17003 t^{0.6293266}$, this can be used to explain the release mechanism as $R^2=0.9285674$. According to (Hermida and Agustian, 2019) when $0.45 \leq n \leq 1$, urea release mechanism was known as non-fickian (anomalous) and that was the case for SRUFX. While, for SRUFY, the kinetic constant (K) and diffusional coefficient (n) were 2.412692 and 0.5627983, respectively, thus the release rate model of SRUFY was $Y=2.412692 t^{0.5627983}$, with $R^2=0.9512041$. These parameters predict the release mechanism for SRUFY as non-fickian (anomalous).

3.7. Effect of mechanical stress on urea release

The effect of mechanical stress on static release experiment was done for 0.2 g sample of urea without being subjected to any stress in 250 ml distilled water, as a reference sample, then Urea, SRUFX and SRUFY were subjected to three different stress 1, 1.5 and 2 MPa.

From Figs. 14, 15 and 16, it is clear that, the release time increased by increasing the mechanical stress. From Fig. 14, the release pattern of U_2 is much slower than that of common urea. This can be attributed to the reduction of the particles size consequently the pore size of urea leading to decrease in solubility of urea. As indicated from XRD of the stressed samples, there a distortion in the crystal structure of urea, this distortion is another important factor of decreasing solubility where the faces of the crystals are the gaps in which water molecule are accommodated. After deformation of the crystal structure these faces are no longer available to accept water molecules, consequently the solubility has decreased (Hosny 215). From Fig. 15, there is no much difference between the release time of $X_{1.5}$ and X_2 , but, the release time was about 5 times of the that of common urea sample.

The sample of SRUFY, Fig. 16, showed the highest time of release and lowest loss of urea in the static release experiment, when subjected to mechanical stress. There is no large change between Y_1 and $Y_{1.5}$

in the release time of urea. While, Y_2 releases urea with much slower rate exceeds 140 min., about 9 times slower than the release of common urea.

4. Conclusion

Two slow release urea fertilizers (SRUFs) have been prepared with two different techniques through incorporation of urea with natural and environmental safe materials (sawdust and gelatin). They were tested for the urea release using static release experiment, which showed that both prepared fertilizers have a slower urea release than common urea fertilizer. The kinetics of release calculations showed that the release mechanism of both fabricated fertilizers follow the non-fickian release (anomalous). The two SRUFs were subjected to mechanical stress, and showed that the mechanical stress caused increase in the release time and reduction in urea release reached 9 times compared with common urea. Using mechanically stressed urea, sawdust and gelatin composite can present promising slow release fertilizer prepared from natural, safe and cheap materials can reduce the repeated times of use of urea fertilizers and produced on large scale.

Declarations

Authors' contribution N. Hosny, supervision, discussed the data, writing and approving the manuscript. A. Nasser, performed the experimental part and analyses. N.Hasan supervision, S. Moalla supervision and discussed the data.

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Figures

Figure 1

Schematic representation of the used set up for determining the static release of urea in water (1: thermometer, 2: Wassermann tube, 3: glass beaker, 4: magnet, 5: hot plate magnetic stirrer, 6: distilled water7: universal clamp).

Figure 2

FTIR spectra of Urea, Gelatin, Saw dust, SRUFX, and SRUFY.

Figure 3

SEM images of (A) common urea, (B and C) SRUFEX, (D and E) SRUFY at different magnifications.

Figure 4

EDX elemental analysis of Urea (A), SRUFX (B) and SRUFY (C).

Figure 5

XRD analysis of urea, SRUFX and SRUFY

Figure 6

XRD of Urea, U₁, U_{1.5} and U₂.

Figure 7

(A) XRD of SRUFX, (X_1) SRUF compressed at 1MPa, ($X_{1.5}$) SRUF compressed at 1.5 MPa, and (X_2) SRUF compressed at 2MPa, (B) Magnified of the main peak of SRUFX, X_1 , $X_{1.5}$ and X_2 .

Image not available with this version

Figure 8

Caption not included with this version.

Figure 9

XRD of SRUFY, (Y_1) SRUF compressed at 1MPa, ($Y_{1.5}$) SRUF compressed at 1.5 MPa, and (Y_2) SRUF compressed at 2MPa.

Figure 10

Water retention results of Soil, Urea, SRUFX and SRUFY.

Figure 11

The release pattern of Urea, SRUFX and SRUFY

Figure 12

Time dependent release pattern of urea fraction in water.

Figure 13

Urea release kinetics for (A) SRUFEX and (B) SRUFY

Figure 14

Release pattern of U₁, U_{1.5}, U₂ in comparison with urea

Figure 15

Release pattern of X_1 , $X_{1.5}$, X_2 in comparison with urea

Figure 16

Release pattern of Y_1 , $Y_{1.5}$, Y_2 in comparison with urea