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## Neem (Azadirachtaindica) oil coated urea a novel controlled release fertilizer: physical and chemical analysis of structure and its nutrient release behaviour

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Abstract: The development of controlled release fertilizers (CRF) is a green technology that not only reduces nitrogen loss caused by volatilization and leaching but also alters the kinetics of nitrogen release, which, in turn, provides nutrients to plants at a pace that is more compatible with their metabolic needs. With an increasing awareness towards human health and environmental protection, there is a rapid shift towards the development of eco-friendly fertilizer based on natural resources. Majority of the literature on CRF pertains to organic and inorganic material based coatings on fertilizers and among them in case of neem oil coated urea (NCU) most of the literature is confined to elucidate coating effect on increasing the crop yield and plant growth [57-61]. However, literature lacks any comprehensive study on NCU as a CRF, covering major aspects such as its characterization, determination of nutrient release rate, and comparison with other CRF. The present work is an attempt to fill this gap in scientific knowledge about NCU. In the present study, neem oil coated urea (NCU) was prepared to achieve the controlled release mechanism necessary to meet crop requirements. Characterizations of the uncoated urea (UCU) and NCU were done using scanning electron microscopy (SEM) with energy dispersive Xray analysis (EDX), fourier transform infra-red (FTIR), refractive index (RI), and crushing strength test. To establish the superior behaviour of NCU as a CRF, the results were also compared with literature data of other CRF, namely phosphate slimes (PCU) and bentonite coated (BCU) with chitosan as a binder. The nutrient release experiments showed that NCU gave a release of 1.03 and 45.03 % at the first day and day 30, respectively. Crushing strength test was carried out for the same size particles, and results showed that NCU has better mechanical strength as compared to UCU, PCU, and BCU.

Keywords: urea; nutrient release; refractive index; fertilizer.

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## INTRODUCTION

Earth's population has witnessed an exponential growth in the past few decades and has now reached approximately 7.0 billion, and this is further expected to reach 9.5 billion by 2050.1-3 This population growth, on one hand, has fueled the global food requirements and the per capita food requirement is expected to double by 2050 from the present levels.<sup>4</sup> On the other hand, cultivable land area is diminishing due to rapid industrialization, increasing urbanization, desertification, and land degradation.<sup>5,6</sup> These intimidating factors pose a serious threat to the global food security and need an immediate response and solution. Different interventions to meet the challenges of food security have already been undertaken worldwide; prominent among these is to improve the efficiency of agricultural systems to produce more food from a given area of land. One such intervention to meet the increasing food demands is to employ enormous quantities of fertilizers in the agricultural sector, although this has led to adverse environmental impacts. The use of fertilizers bears a direct or indirect impact on the soil biota and its functioning.<sup>7</sup> Hence, it is the need of the hour to develop such systems, which, along with ramping up production, also alleviate environmental problems.<sup>8</sup>

Fertilizers are chemical compounds used to improve agricultural productivity as they are able to supply the necessary nutrients required for the plant growth<sup>9</sup>. Urea is among the most widely used N fertilizers, which contains 46 % nitrogen.<sup>3,10</sup> However, a large proportion of urea gets hydrolyzed before its uptake by the plant.<sup>11</sup> When uncoated urea (UCU) is applied to the soil, the urea (Amide) nitrogen is rapidly converted to ammonical nitrogen and subsequently to nitrite and nitrate forms. Nitrogen in these forms, besides being absorbed by plants, is also rapidly lost from the soil due to leaching, run off, volatilization, and denitrification, leading to environmental pollution and disturbing the soil ecology<sup>12</sup>. Controlled release compounds are widely used in biotechnology, medical purposes, and predominantly as fertilizers in agricultural production.<sup>13,14</sup> Controlled release fertilizers (CRF), in contrast to conventional fertilizers, can overcome the loss of nutrients caused due to off stream losses, leaching, and volatilization since they have more sustainable release characteristics and better uptake and stability.<sup>15</sup> A large varieties of commercial CRF are produced in which fertilizer products are coated with alkyd resin, polyurethane, and polyolefin; however, these materials do not degrade appropriately in soil solution phases and thereby, their accumulation over a period of time results in loss of soil fertility and defeats the very purpose of carrying out the sustainable agriculture of arable lands. However, it is crucial to take into account the materials that were utilized for the urea coating, especially in terms of how quickly they degrade.<sup>16</sup>

Recent studies have explored the possibilities of choosing coating materials that have twin advantages of being not only eco-friendly but also cost effective one





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such study was reported by using bentonite and organic polymer.<sup>17</sup> Also, Neem oil coated urea (NCU) is fast emerging as an important CRF because of its multiple benefits. Neem, *Azadirachtaindica* is a native to the arid regions of the Indian subcontinent, where it grows to 12-24 m high. It can be propagated easily by seed, or by 9 to 12 month old saplings can also be transplanted. Fresh fruit yield per neem tree ranges between 37 and 50 kg per year. Forty kg fruit yields nearly 24 kg of dry fruit (60 %), which in turn gives 11.52 kg of pulp (48 %), 1.1 kg of seed coat (4.5 %), 1 kg of husk (25 %), and 5.5 kg of kernel (23 %). The kernel gives about 2.5 kg of neem oil (45 %) and 3.0 kg of neem cake (55 %). Neem oil extracted from the seeds of the neem tree has insecticidal and medicinal properties due to which it has been widely used in pest control. The cake (containing 5 percent N) left after oil extraction is generally used as manure and also for making neem cake-coated urea.

Neem oil contains melicians (generally known as neem bitters), of which epinimbin, deacetyl, salanin, and azadirachtin are the active fractions, which showed dose dependent nitrification inhibition action. When NCU is applied to soil, the neem triterpenes inhibit the activity of nitrifying bacteria, which results in delayed transformation of ammonical nitrogen into nitrite nitrogen. This leads to a substantial reduction in the loss of fertilizer and pollution of groundwater. NCU ensures slow and continuous availability of nitrogen throughout the crop growth, nourishing the saplings for a longer period and thus avoiding the repeated use of fertilizer.<sup>18</sup> Other potential benefits of using NCU includes an increase in crop yield, efficient pest control management leading to savings, increases in the shelf life of the product, and preventing its misuse as feedstock for use in other chemical industries.

#### MATERIAL AND METHODS

#### Materials for Preparation of NCU

Commercial UCU granules with a nitrogen content of 46.44% (label specification) and distribution of particle diameter from 0.5mm to 2.8mm (determined using sieve analysis) were supplied from Chambal Fertilizers & Chemicals Limited (CFCL) Gadepan, Kota (India). The urea particles were coated with Neem oil supplied by Shubhra Industries, Jaipur (India).

Various methods are available for making coated urea, which acts as CRF. Among the available techniques, the fluidized bed coating process, being one of the oldest and experimentally simple to design and operate, for forming small coated particles, also holds promise for making NCU of reasonable quality. The line diagram of the experimental set up used is shown in Fig. 1(a). The NCU obtained after coating is shown in Fig. 1(b).





Fig. 1(a) Experimental set up of fluidized bed coater, (b) NCU obtained from UCU after coating in Fluidized bed coater

The fluidized bed was made from a flexi glass. A spraying nozzle of an air-atomized nozzle was centrally set above the fluidized bed. A batch of UCU 210 g in weight was put in the fluidized bed, which was fluidized at higher than their minimum fluidization. For coating, 20 ml of neem oil at room temperature and flow rate of 0.35 ml min<sup>-1</sup>, under a pressure was atomized with compressed air and sprayed onto the urea particles in the fluidized bed for coating. Steady state was indicated by a constant bed temperature and constant head. Final product samples were collected after 20-30 min of fluidization. By this time, urea particles were uniformly coated with the neem oil.



#### Physical and chemical analysis

| Table I. | Properti | es of N | eem oil | and U | Jrea |
|----------|----------|---------|---------|-------|------|
|          |          |         |         |       |      |

| _   |                           |  |                              |
|---|---------------------------|--|------------------------------|
| Neem oil  |                           | Ure                                      | a                            |
| Iodine index                                      |                           |  |                              |
| Kinematic viscosity [30°C]<br>(m <sup>2</sup> /s) | 43.75                     | Molecular weight                         | 60.06                        |
| Saponification value                              | 199.86                    | Odor                                     | Odorless                     |
| Physical state at room temperature                | Liquid<br>(golden yellow) | Absolute viscosity<br>[20°C] (kg/m-s)    | 0.185                        |
| Acid value  | 32.538                    | Density (kg/m <sup>3</sup> )             | 1320                         |
| Cloud point (°C)                                  | 13                        | Melting point (°C)                       | 133-135                      |
| Pour point (°C)                                   | 7.0                       | Flowing time (20°C) (Second)             | 25-40                        |
| Density at room temperature (kg/m <sup>3</sup> )  | 918.2                     | Solubility in water (kg/m <sup>3</sup> ) | 1079 at 20°C<br>1670 at 40°C |
|   |                           |  |                              |

#### Particle size analysis

The particle size of fertilizer products and/or fertilizer raw materials is defined as the particle diameter ranges of the test material. Particle size affects agronomic response, granulation techniques, storage, handling, and blending properties. The particle size distribution was determined for both UCU and NCU using the sieve analysis (dry sieving as per IFDC S-107).<sup>19</sup> For this analysis, a sample weight of 210 g was used for both UCU & NCU, and the resultant distribution is shown in Fig. 2. It can be seen from the figure that maximum particles are available in the diameter range of 1.7 mm and 2.0 mm. Also, it can be inferred from these results that the particle strength increases due to coating (reduction in particle breakage after neem oil coating) since less number of particles of NCU are present in lower size distribution ranges.

## Percent coating

The actual coating % is determined by taking a 10 g NCU sample, immersed in 100 ml of distilled water. After thorough shaking, the urea gets dissolved in the water, and as a result, the coating was liberated from NCU. Subsequent filtration and water evaporation gives the coating % which is calculated using Eqs. 1.

Coating 
$$\% = \frac{[M_i - M_o]}{M} \frac{X}{M} 100$$
 (1)

Where,  $M_i$  = weight of filter paper with urea sample, after evaporation (g)  $M_o$  = weight of filter paper (g) M = weight of urea sample (g) % coating= [0.57] / [10]\*100 = 5.7 %



Nitrogen content

The nitrogen content of NCU is estimated using the standard Kjeldahl method. The sample was treated with sulphuric acid to yield ammonium sulfate. The hydrolysis of ammonium sulfate yields the nitrogen content. Also, the nitrogen content can be calculated mathematically using the total coating percentage as given in the Eqs.2. Nitrogen content values of UCU and NCU obtained experimentally and using Eqs. 2 are almost the same, which are shown in Table II. The value obtained by Eqs. 2 is used in the present work.

$$N\% = 46.44 \frac{(100 - \% Coating)}{100}$$
(2)

TABLE II. Nitrogen content in UCU and NCU

| Sample | Coating, % | Urea, % | Nitrogen, % |
|--------|------------|---------|-------------|
| UCU    | 0.00       | 100     | 46.44       |
| NCU    | 5.7        | 94.3    | 43.79       |

#### Dustiness factor

Dustiness is a physical property of fertilizer. Due to the large quantities of fertilizer produced and raw materials handled in bulk, their dustiness is of particular concern and can cause problems like significant material losses during processing, handling, and application thereby resulting in loss of revenues. Also, environmental pollution and exposure of employees to hazardous levels of dust is not indicative of a responsible organization. So, dustiness of fertilizer is an undesirable property. The dustiness was measured by taking 10 g of NCU in a bunker funnel, in which compressed air (10 Psi of pressure) is passed from the bottom. After 5 min, the sample is removed from the bunker funnel and weighed. The weight loss is then calculated. The amount of weight loss is called dust. In the present case, loss in mass of the

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sample = 0.037 g, so % Dustiness = 0.037/10\*100 = 0.37 %, which is in agreement with the values reported in the literature for other CRF.

#### Dissolution rate

The UCU obtained from the market and NCU samples prepared as discussed above were analyzed for the dissolution rate for which a 5 g of sample particles (of same diameter of 1.7 mm) were put in a beaker containing 50 ml of double distilled water. For mixing, a magnetic stirrer was used at constant speed. The time required for the complete dissolution of urea was noted. The temperature was also varied to see its effect on dissolution, and as expected, the dissolution rate was slower for NCU as compared to UCU. The dissolution rate (indicated by lower dissolution time) increases with temperature, as can be seen from the results in Table III.

#### TABLE III. Dissolution rate of UCU and NCU

| Sample              |       | Diss  | solution time | / s   |       |
|---------------------|-------|-------|---------------|-------|-------|
| (diameter = 1.7 mm) | 30 °C | 35 °C | 40 °C         | 45 °C | 50 °C |
| UCU                 | 324   | 300   | 287           | 239   | 210   |
| NCU                 | 482   | 445   | 426           | 372   | 357   |
|                     |       |       |               |       |       |

Crushing strength

The crushing strength is an important parameter that indicates the ability of particles to withstand physical handling throughout the supply chain. Experimentally, it is measured by applying pressure to individual granules, usually of a specified size, and noting the required pressure to fracture each granule. In the present work, the test was performed on different sizes (1.7 mm and 2.0 mm) of NCU and UCU. A tensile strength tester (Make Shimadzu available at material research center of Malaviya National Institute of Technology, Jaipur) was used for measuring crushing strength in which granules were subjected to a force applied by a metal plunger that was a part of the apparatus, and the values were noted.

#### Morphology and microscopic analysis (SEM) of the surface

Scanning electron microscopy (SEM) with an energy dispersive analysis system of X-ray spectrometer (EDX) is one of the powerful analytical tools to study the morphology and relative elemental composition of the granules. Thus, morphology and relative elemental concentration of UCU and NCU were determined using scanning electron microscopy (SEM) (Make: Nova Nanosem-450 FEI, available at material research center of Malaviya National Institute of Technology, Jaipur), which was equipped with an energy dispersive analysis system of X-ray spectrometer, EDX. For analysis, UCU and NCU 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, and the samples were ready to be analyzed.

#### FTIR Analysis

Fourier transform infrared (FTIR) spectroscopy is a powerful tool to study the chemical species and the functional groups present in a sample. FTIR spectrophotometer (Make: PerkinElmer company available at material research center of Malaviya National Institute of Technology, Jaipur) was used to analyze the fertilizer samples. The UCU and NCU granules were dispersed in dry KBr powder and ground to obtain fine particles. These particles were analyzed at wavelength range from 4500 cm<sup>-1</sup> to 400 cm<sup>-1</sup>. All spectra were recorded at ambient temperature.





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#### X-ray diffraction (XRD) Analysis

It is a rapid analytical technique primarily used for the phase identification of crystalline materials and can provide information on unit cell dimensions. The analyzed materials are finely ground homogenized, and the average bulk composition is determined by XRD (Make: 'X' Pert powder, available in Material Research Center in Malaviya National Institute of Technology Jaipur). It works on Bragg's law (2d sin  $\theta = n\lambda$ ). The XRD patterns were recorded in the range of 2 $\theta$  equal to 20°-80°. It was used to explain the interference pattern of X-rays scattered by the crystals.

#### Experimental determination of nutrient release from NCU in water domain by refractive index

An accurate estimate of the nutrient release pattern from CRF is required both for manufacturers as well for farmers, since it helps them in understanding fertilizer potency and nutrient planning for the crops. Although the field tests provide a better estimation of the nutrient release behavior, however, it suffers from inherent constraints of being influenced by the variation in environmental conditions like temperature, soil moisture, soil pH, soil microbes population and porosity, etc. Thus, the determination of nutrient release under laboratory conditions provides a convenient, useful, and nearly accurate approach to understand the nutrient release mechanism. Other researchers had also pointed that laboratory experiments were successful in predicting the nutrient release rate of CRF.<sup>20</sup> In actual practice, the estimation of nutrient release in the soil is more pertinent since it depicts the actual behavior under field conditions; however, as explained, the mechanism of nutrient behavior in CRF is mainly governed by water penetration into the core through the coating. Thus, the study of nutrient behavior in the water domain gives a fairly accurate picture of nutrient release, and this is experimentally done first, followed by studies in the soil domain to improve the predictions. In the present work, the nutrient release in water from NCU is experimentally determined using a refractometer.

To determine the urea release, 50 g sample (UCU and NCU) were placed in 250 ml distilled water in a separate Erlenmeyer flask and sealed. The refractive index (RI) of both the samples were measured using a refractometer (Make: ATAGO available at Thermodynamics Research Lab in Malaviya National Institute of Technology Jaipur) to get the urea release in the solution. The instrument was calibrated daily before measurements against a known refractive index of water, and RI of urea samples were measured at 25 °C and 40 °C as a function of time for 3, 7, 11, 15, 19, 23, 26, and 30 days. The value of (RI) is related to the concentration of urea dissolved in water.

#### RESULTS AND DISCUSSION

#### Morphology and microscopic analysis of the surface

SEM pictures of the UCU and NCU's surfaces and sections were utilized to investigate the morphological alterations brought on by the neem oil coating. Some portions of UCU (Fig. 3a) were rough, but the majority of it was smooth. While Fig. 3b confirms agglomeration. Fig. 3c, d shows the layer that results from the application of neem oil; it was smooth compared to UCU. Layering is a desirable trait for the particle coating. An irregularity in coating thickness and shape of granules was observed. UCU and NCU particles of 1.7 mm diameter were selected randomly and observed under SEM at magnifications of 500x and 1000x. SEM images are shown in Fig. 3a and 3b for UCU and Fig. 3c and 3d for NCU.





Fig. 3.SEM image of UCU [a(500x) and b(1000x)] and NCU [c(500x) and d(1000x)] granules

In the case of UCU, long crystals were tightly cemented with the finer ones on the urea surface (Fig. 3(a-b)). This morphology can be attributed to the fact that uncoated granular urea production involves agglomeration. Some pores and gaps were also visible. However, in the case of NCU (Fig. 3(c-d)), the coating imparts more homogeneity to the surface, and since neem oil coating is dense, no visible signs of gaps or cavities in the coating layer were observed. The enclosing of urea granule gaps or cavities due to neem oil layering altered its release behavior, making them CRF as demonstrated by NCU SEM results. To get an estimate of coating thickness, NCU particles were cut with a sharp knife, and these broken particles were scanned using SEM, as shown in Fig. 4(a-b). A variable coating thickness lying between 51.9  $\mu$ m to 65.56  $\mu$ m was observed.

As can be seen in the images, the coating layer is very dense, and the contact between the coating layer and the urea granule was thorough. This dense coating is responsible for imparting controlled release behavior to urea. It is reported that the coating thickness affects the release pattern of nitrogen from fertilizers.<sup>21</sup>





Fig. 4.SEM [a (500x) and b(1000x)] image of a section of NCU granules

## EDX of UCU and NCU

The EDX of UCU (Fig. 5a) and NCU (Fig. 5b) showed no remarkable difference in the presence of any extra elements in NCU compared to UCU. However, a lower peak of nitrogen in the case of NCU suggests that about 2 % reduction in total N was observed for NCU compared to UCU. It is due to the inherent nature of the coating process, in which the urea granule undergoes spraying, wetting, and drying processes, which may cause some loss of nitrogen. However, variations in the contents of O, C, Fe, and Al were also noted.

## XRD Analysis for UCU and NCU

The XRD spectra of the UCU and NCU of particle diameter 1.7 mm were compared in Fig. 6. The figure makes it clear that both particles had crystalline structure and the peaks of urea, urea ammonium nitrate, and ammonium nitrate were visible only, and no characteristic peaks of other impurities were found to be seen. The main cause of this variation in peak intensity was that neem oil blocks the sites when it penetrates the micro cracks of UCU. This was attributed to the strong physical adherence of neem oil with the outer coating of the urea base. All the recorded peaks correspond to the components of the fertilizer. Since no new peaks in the spectra of NCU are observed, it may be inferred that no new phases are formed during the coating process. This shows that the interactions between the neem oil and urea are basically physical and not chemical in nature.









Fig. 6. XRD Diagram of UCU (a) and NCU (b)

FTIR spectra analysis for UCU and NCU



Fig. 7. FTIR diagram of NCU and UCU (both at 1.7 mm dia)

The FTIR spectra of UCU and NCU (Fig. 7) demonstrate that all the peaks were in the same positions. Peak shifting was not observed. Both the samples show similar strong peaks of the amide group, N–H, at 3500 cm<sup>-1</sup>, indicating the presence of urea molecules. This is followed by the double peaks of C=O bonds at around 1682 cm<sup>-1</sup> and the C-N bond at around 1465 cm<sup>-1</sup>. No additional functional groups were observed in NCU; only changes in the intensity of the functional groups are seen, implying that the interaction between the UCU and the neem oil is physical.

## Determination of nutrient release using refractive index

The refractive index can be used to measure the concentration of urea in solution directly and thus provides a fair estimate of nutrient release from the



sample. This method offers distinct advantages like fast analysis, high efficiency, and free from chemical or reagent consumption. In the present work, the percentage of urea release in terms of nitrogen content and refractive index values for NCU was measured and compared with UCU and other materials such as PCU and BCU using chitosan as a binder. The PCU and BCU data taken from literature<sup>22</sup> is compared to show the superior slow release behaviour of NCU.

Fig. 8 shows the NCU calibration curve in distilled water constructed with standards from 0 to 250000 ppm at 25 °C and 40 °C. The two temperatures are selected to see the effect of temperature on the release mechanism. The curve depicts linearity over a wide range, which helps in the measurement of nitrogen release from NCU without dilution of samples.

The refractive index values for NCU at 25 °C and 40 °C and corresponding urea release percentages (using the calibration curves) are plotted in Fig. 9a and 9b, respectively. The nutrient release rate increases with increases in temperature. This temperature dependency helps in ascertaining the urea availability to the crop as per ambient and corresponding soil temperature.

In order to compare the controlled release efficiency of NCU with UCU and other coated fertilizers, namely PCU and BCU, the refractive index values and urea release percentages at 25°C were measured. The results are shown in Fig. 10 (a) and (b), respectively.

The results show that 1.03, 4 and 4.8 % of Nitrogen of NCU, PCU, and BCU, respectively, were released into the water during the first day, and the release rate values at day 30 were 45.03, 63.4 and 81.9 %, for NCU, PCU and BCU respectively. On the other hand, the uncoated urea released all its nitrogen content within one hour.



Fig. 8. Calibration curves for NCU at different temperatures

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Fig.10. UCU, BCU, PCU, and NCU (a) Refractive index values (b) Urea release percentage

These release data shows the slow release properties of NCU, PCU, and BCU samples, which also follows the standards of slow release fertilizers of Comité Européende Normalization (CEN). According to CEN, a fertilizer can be described as having controlled release properties if nutrient release is not more than 15% after 1 day or not more than 75 % after 28 days. Also, release from NCU is slower for any particular time as compared to PCU and BCU; this indicates better control over nutrient release when urea is coated with neem oil. So NCU is a novel CRF, which exhibits excellent slow release behavior apart from being biodegradable and neem oil acting as biopesticide and insecticide.

## Crushing strength

The crushing strength of NCU and UCU for two diameters, namely 1.7 mm and 2.0 mm, were measured using a tensile tester, and results are shown in Table IV.

TABLE IV. Crushing strength of NCU and UCU

| Sampla |      |              |
|--------|------|--------------|
| Sample |      | (dia=2.0 mm) |
| UCU    | 1.49 | 1.62         |
| NCU    | 2.50 | 2.96         |
|        |      |              |

From the table values, it can be inferred that NCU particles have higher crushing strength as compared to UCU of the same size, indicating its higher mechanical strength. Higher crushing strength also leads to a reduction in attrition during storage and transportation and also reduces the dustiness factor. The crushing strength increases with the increase in the size of particles for the same sample.

In order to compare the crushing strength of NCU with PCU and BCU, literature values were taken, and the results are shown in Table V. These particular CRF particles were selected because the literature values of the crushing strength of these CRFs were available for particles with the same urea core radius and coating thickness as the NCU prepared. From the table, it is clear that NCU has higher crushing strength as compared to PCU and BCU, indicating its better performance as CRF due to improved strength.

| I   | Tullin (and 2.0 mill) |
|-----|-----------------------|
| UCU | 1.62                  |
| NCU | 2.96                  |
| PCU | 1.80                  |
| BCU | 1.67                  |

TABLE V. Comparison of crushing strength of NCU, UCU, PCU and BCU

## CONCLUSION



In order to raise the crop productivity, contemporary technologies must be used, to improve food output. Using CRF is one such strategy which is used worldwide, in the present work various aspects of an environmentally friendly CRF namely NCU is scientifically explored using various analytical and experimental techniques to determine its suitability as an effective CRF. NCU was prepared using neem oil as a coating material in a fluidized bed. Crushing strength test results showed that NCU (2.96 Nmm<sup>-2</sup>) has better mechanical strength as compared to the UCU (1.62 Nmm<sup>-2</sup>), PCU (1.80 Nmm<sup>-2</sup>), and BCU (1.67 Nmm<sup>-2</sup>). Higher crushing strength aids in safe storage and transportation of a CRF. Analysis carried using FTIR and XRD showed that the neem oil coating did not react, and no new chemical interactions take place and the adherence of oil is mainly due to



surface forces. SEM images shows a variable coating thickness lying between 51.9  $\mu$ m to 65.56  $\mu$ m. Its release characteristics show controlled release behavior because strong adherence of oil fills the void in the urea particle, which manifest itself in an increased nutrient release time. NCU release characteristics were also in accordance with prescribed standards of CEN. To establish the superior behaviour of NCU as a CRF its release rate results were also compared with literature data of other CRF, namely phosphate slimes (PCU) and bentonite coated (BCU) with chitosan as a binder. The results show that 1.03, 4 and 4.8 % of Nitrogen of NCU, PCU, and BCU, respectively, we're released into the water during the first day, and the release rate values at the day 30 were 45.03, 63.4 and 81.9 %, for NCU, PCU and BCU respectively. On the other hand, the uncoated urea released all its nitrogen content within one hour. Therefore, NCU shows a promise of becoming a preferred CRF using abundant, low cost natural resources that is neem oil. In summary, NCU proves to be a novel CRF.

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## ИЗВОД

#### УРЕА ОБЛОЖЕНА NEEM (AZADIRACHTAINDICA) УЉЕМ, НОВО ЂУБРИВО СА КОНТРОЛИСАНИМ ОСЛОБАЂАЊЕМ: ФИЗИЧКА И ХЕМИЈСКА АНАЛИЗА СТРУКТУРЕ И ПОНАШАЊЕ ПРИ ОСЛОБАЂАЊУ ХРАНЉИВИХ МАТЕРИЈА

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Развој ђубрива са контролисаним ослобађањем (CRF) је зелена технологија која не само да смањује губитак азота узрокован испаравањем и испирањем, већ и мења кинетику ослобађања азота, што заузврат обезбеђује хранљиве материје биљкама, темпом који је компатибилан са њиховим метаболичким потребама. Уз све већу свест о људском здрављу и заштити животне средине, долази до брзог померања ка развоју еколошки прихватљивог ђубрива заснованог на природним ресурсима. Већина литературе о CRF се односи на превлаке на бази органских и неорганских материјала на ђубривима. У случају урее обложене уљем Neem (NCU) литература је ограничена на разјашњавање ефеката премаза на повећање приноса усева и раст биљака. Међутим, у литератури недостаје било каква свеобухватна студија о NCU као CRF, која покрива главне аспекте као што су његова карактеризација, одређивање брзине ослобађања хранљивих материја и поређење са другим CRF. Овај рад је покушај да се попуни овај јаз у научним сазнањима о NCU. У овој студији, уреа обложена уљем Neem (NCU) је припремљена да би се постигао механизам контролисаног ослобађања неопходан за испуњавање захтева усева. Карактеризације необложене урее (UCU) и NCU су урађене коришћењем скенирајуће електронске микроскопије (SEM) са енергетско дисперзивном рендгенском анализом (EDX), инфрацрвеном спектроскопијом (FTIR), мерењем индекса преламања (RI) и тестом чврстоће на дробљење. Да би се утврдило супериорно понашање NCU као CRF, резултати су упоређени са литературним подацима других CRF, односно фосфатне слузи (PCU) и



бентонита (BCU) обложених хитозаном као везивом. Експерименти ослобађања хранљивих материја су показали вредности од 1,03 (први дан) и 45,03 % (тридесети дан). Испитивање чврстоће на дробљење је спроведено за честице исте величине, а резултати су показали да NCU има бољу механичку чврстоћу у поређењу са UCU, PCU и BCU.

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