Chemical Methodologies 2(2018) 299-307

Chemical Methodologies

Journal homepage: http://chemmethod.com

Original Research article

Experimental Determination of Nutrient Release from Neem Oil (Azadirachta indica) Coated Urea

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ARTICLE INFORMATION

Received: 01 May 2018
Received in revised: 08 June 2018
Accepted: 9 June 2018
Available online: 01 July 2018

DOI:
10.22034/CHEMM.2018.66186

KEYWORDS

Controlled release
Neem coated Urea
UV-Vis spectrometer

ABSTRACT

An accurate estimate of nutrient release pattern from controlled release fertilizer (CRF) is required both for manufacturers as well for farmers because of the fact that it helps them in understanding fertilizer potency and nutrient planning for the crops. Understanding the release pattern of different types of CRF under laboratory conditions as well as in the crop-field is essential in order to determine the fertilizer utilization efficiency on plant growth. The present work explains a simple experimental approach to understand the nutrient release from Neem coated urea (NCU). The coating of Urea with Neem oil (derived from seeds and leaves) inhibits the process of nitrification and reduces the formation of nitrates which in-turn will reduce N_2O emissions. It prevents the loss of urea in the soil. It also controls a large number of pests such as caterpillars, beetles, leafhoppers, borer, mites etc. Also, Neem coating is biodegradable and so it is environmentally-friendly as compared to many non biodegradable polymers used as coating materials in CRF.

UV-Vis spectrometer is employed to determine the amount of released nitrogen. The approach reduced analytical error by preventing sample dilution before measuring. According to laboratory results, the obtained UV-Vis spectrometer proved to be a handy and efficient equipment for studying the nutrient release behaviour from CRF. The release profile presents a sigmoidal shape which is in good agreement with the other works in the area reported in the literature. The obtained experimental results are also in tune with the criteria specified by European Committee for Standardization for CRF.

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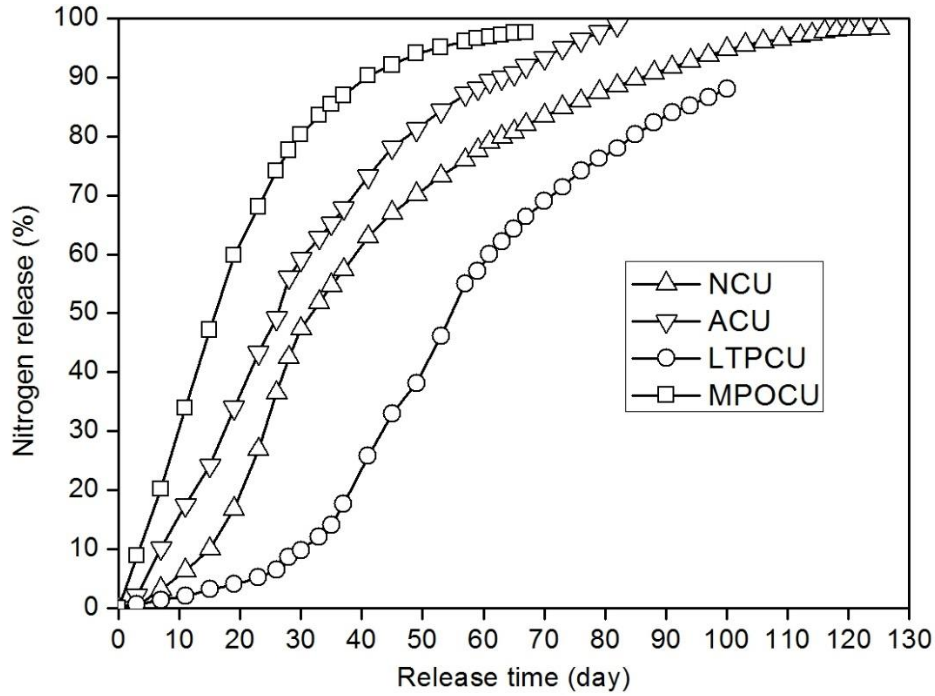
Graphical Abstract



Introduction

Soil fertility is primarily determined by three major elements namely Nitrogen, Phosphorus and Potassium (N, P, K) of which Nitrogen plays a very important role. For this reason, Urea (containing 46% of N) consumption is very high all over the world. Unfortunately, more than half (up to 60%) of the nitrogen leaches out or vaporizes in the form of nitrogen gas, ammonia and nitrous oxide due to the presence of denitrifying bacteria in the soil [1-3]. Therefore, Controlled release fertilizers (CRF), conceptualized and introduced by Oertli [4] in 1962, provide a way to increase nutrient recovery and fertilizer management. In comparison to normal fertilizers, the nutrient release pattern from CRF meets plant requirement and minimizes leaching in an efficient manner whereby improving the overall fertilizer uses [5]. When uncoated urea 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 de-nitrification. When neem coated urea (NCU) is applied to soil, the neem Triterpenes inhibit the activity of nitrifying bacteria resulting in delayed transformation of ammonical nitrogen into nitrite nitrogen. Thus, it ensures slow and continuous availability of nitrogen regarding the crop growth. Coating urea with neem prevents its misuse as well as puts the fertilizer in slow release mode, nourishing the saplings for a longer period, and thus avoiding the repeated use of fertilizer. The process reduces pollution of groundwater. There is an increase in crop yield and efficient pest control management leading to savings. It also increases the shelf life of the product [6].

Current research is focused on CRF applications not only in the laboratory but also in the field crops. Majority of these studies focus on release pattern of CRF because it is has a direct bearing on the determining the effectiveness of these fertilizers on plant growth and in planning plant nutrient management. Goertz et al. studied the release of nitrogen from sulfur-coated urea, and the release was controlled by sulfur thickness [7]. Kochba and Gambash studied the release of nitrate from coated granule where the release occurred by diffusion of urea through a semi-permeable membrane [8]. Dai et al. evaluated the release of nutrient from two resin-coated N, P, K fertilizers [9]. Melissa et al. compared weighing and combustion techniques in studying the release of urea in the field [10].

Although the field tests provide a better demonstration of the nutrient release, 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, determination of nutrient release under laboratory conditions provides a convenient and useful approach to understand nutrient release mechanism. Medina et al. had pointed out that laboratory experiments were successful in predicting nitrogen release rate of slow release fertilizers [11]. Papangkorn et al. and Trinh et al. employed UV-Vis spectrometer in studying the urea release rate from polylactic acid coated and agrium coated urea respectively [12-13].

The present work is based on experimental determination of nitrogen release from NCU to obtain a better understanding of release behaviour. The results of present study are also compared to Nitrogen release from urea coated with other materials.

Experimental

Materials and Methods

NCU calibration curve: NCU (CAS 57-13-6), obtained from Chambal fertilizer and Chemical limited (CFCL) Gadepan, Kota (India), is used to prepare standards for calibration curve. Five NCU solutions were prepared with concentrations: 0, 100, 500, 1000, 2000, 5000, 10000*ppm*. UV-Vis double beam (Make Shimadzu, model UV-1800 available in the research lab of the Chemical

Engineering department) as shown in figure 1 is employed to measure the optical density of the above standards at a wavelength of 210 NM.

Figure 1. UV-1800 Spectrophotometer Double Beam (Malaviya National Institute of Technology, Jaipur)

Nitrogen release test: NCU is used in the nitrogen release test. The particle size distribution was determined for both uncoated urea and NCU using sieve analysis. For this analysis a sample weight of 342 gm was used for both uncoated and NCU. The resultant distribution is shown in figure 2.

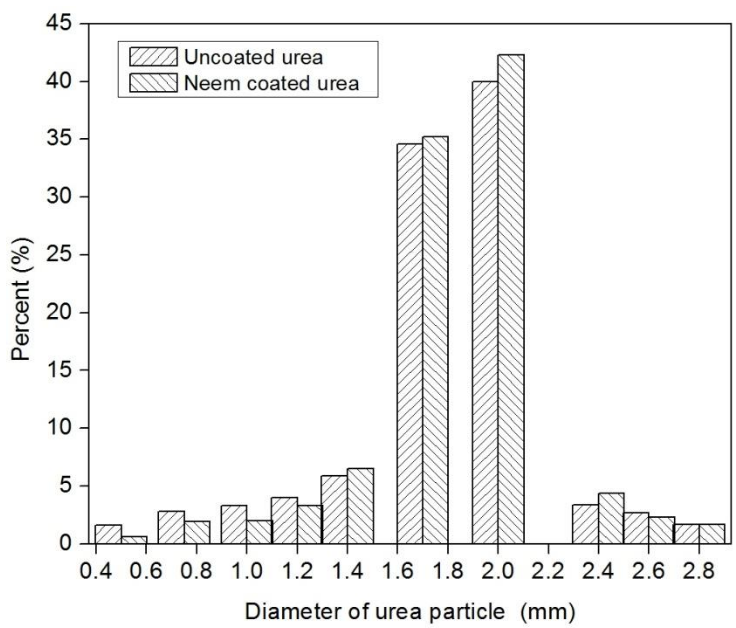


Figure 2. Uncoated and NCU particle size distribution

It can be seen from the figure that maximum particles are available in a diameter range of 1.7mm and 2.0mm. Also, it can be inferred from these results that particle strength also increases due to coating since the less number of particles of NCU are present in lower size distribution ranges. For conducting the release test, 2±0.001gm of NCU particles is immersed into 250ml of distilled water and placed at room temperature as shown in figure 3.

Figure 3. sample for release test

This particular amount of NCU is taken to ensure that urea concentration remains in the range of the calibration curve during the course of conducting the release test. After every 2-4 days, NCU concentration is determined by UV absorbance at 210 nm. Distilled water is added to the fixed point (250ml) before and after the sampling process. The released nitrogen is then calculated from the NCU concentration, and the experiment ends as the released nitrogen reaches 98.27%. Two repetitions were performed in the release test for the sake of attaining accuracy in the results.

Results and discussion

NCU calibration curve: Figure 4(a) shows a NCU calibration curve in distilled water constructedwith standards from 10 to 10000ppm. The obtained calibration curve is empirically given by the equation in which y =0.178x with R^2=0.9949 where x and y present NCU concentration in (ppm) and its absorption in mili absorption unit (mAU) respectively. The curve depicts linearity over a wide range, which helps the measurement of nitrogen release from NCU without dilution of samples.

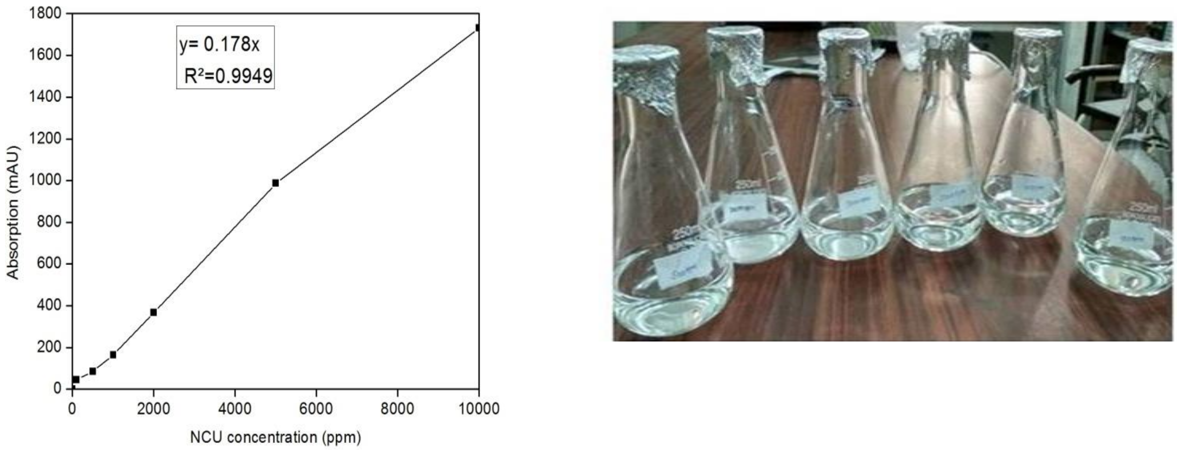


Figure 4. (a): Calibration curve for NCU at 210 nm, (b): Neem coated urea samples

Nitrogen release behaviour: The release profile determined experimentally for Nitrogen release from NCU is shown in figure 5(a). The release profile shows a sigmoidal shape and follows the diffusion stages as described by Shaviv et al. and Lu et al. [14, 15]. In first eleven days, the percent nitrogen release is 6.35%. This increases to 16.82% at the end of 19 days. Thus, up to 19 days the lag period or stage is observed, which is attributed to the fact that, mainly water vapour penetrates into the NCU granule and dissolves a small fraction of solid fertilizer. The driving force responsible for this process is the vapour pressure gradient across the Neem coating. The volume available to the condensed vapour is basically limited to the voids present inside the solid core and those between the core and the coating. A reasonable explanation for the lag period is that some time is needed to fill the internal voids of the granule with a critical water volume.

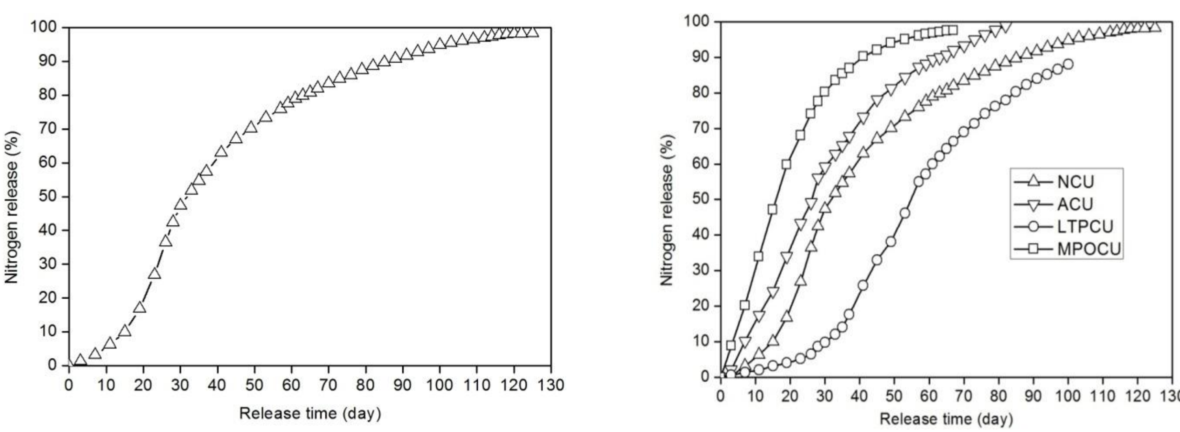


Figure 5(a). Nitrogen release profile in NCU, (b): Comparison of Nitrogen release in NCU with other Coated Urea

In other words, the lag can be due to the time needed for the establishment of a steady state between the flux of water entering the granule and the flux of solute leaving it. Once a steady state is achieved the volume change in the granule is negligible. In terms of thermodynamics, the system can be described as one with zero "net volume flux". Starting from 20th day, the release percentage rises rapidly and reaches to 89.65% at the end of 85th day. This corresponds to the second stage, called the constant release stage, which begins when a critical volume of saturated solution accumulating inside the NCU granule. The rate remains constant as long as the saturated solution in the granule is equilibrated with the non dissolved solid fertilizer. The constant, saturation concentration, yields a constant driving force for fertilizer transport since the concentration of the fertilizer in the external solution is negligible.

The release becomes somewhat slower from 85th day to 125th day, and the amount of nitrogen release reaches 98.27% at the end of the experiment. This is the third stage of the release and named as the decay stage. This occurs due to the fact that once the solid fertilizer in the core is dissolved the concentration of the internal solution decreases due to the continuing concomitant fluxes of nutrient release out and water flow into the granule. Accordingly, the driving force for the release decreases and the release rate decays.

From the experimental data, a regression model is applied to the nitrogen release profile from NCU and the following equation is obtained:

1. $y = 10^{-9} x^6 - 5\times10^{-7} x^5 + 8\times10^{-5} x^4 - 0.0062x^3 + 0.229x^2 - 1.5827x + 1.9284$; $R^2 = 0.9984$

where, x and y represent the release time in days and Nitrogen release (%) respectively.

Experimental data for nitrogen release from NCU was compared with the Nitrogen release data available from the literature for urea coated with different coating materials viz. ACU (Agrium coated urea), LTPCU (Large tablet polymer coated urea) and MPOCU (Modified polyolefin coated urea) and the results are shown in figure 5(b) [16-18].

From the comparison, we can infer that neem coating is effective in increasing the release time of nutrient as compared to other coating materials since for achieving a representative 90% release the time taken is 40 days (MPOCU), 65 days (ACU), 90 days (LTPCU) and 100 days for NCU, thus NCU imparts a better controlled release character to the urea along with the added advantage of coating being biodegradable. Also the nutrient release from NCU (figure 5(a)) is in agreement with three criteria established by the CEN (European Committee for Standardization) [19]. CEN defines the conditions for a fertilizer to be described as slow-release if the nutrient or nutrients declared as slow-release meets defined conditions as shown in table 1 at a temperature of 25°C. The comparison of our data with CEN criteria is tabulated below.

Table 1. Comparison of experimental data with CEN Criteria

S.No.	CEN Criteria	Experimental results from NCU
1.	No more than 15% released in 24 hours	In 24 hours, percent of nitrogen released is 1.47% which less than 15%, so first criteria is satisfied.
2.	No more than 75% released in 28 days,	After 28 days, percent of nitrogen released is 44.44%, which is less than 75%, so second criteria is satisfied.
3.	At least about 75% released at the stated release time	Amount of nitrogen release is 98.27% within 125 days, so third criteria is satisfied.

The Nitrogen release rate is calculated and is shown in figure 6(a). The release rate increases and reaches a maximum at 30 days. After attaining the maxima the release rate decreases and becomes 0.75% /day at about 125 days , The release profile is comparable to the release pattern as shown by other coated fertilizers as shown in figure 6(b).

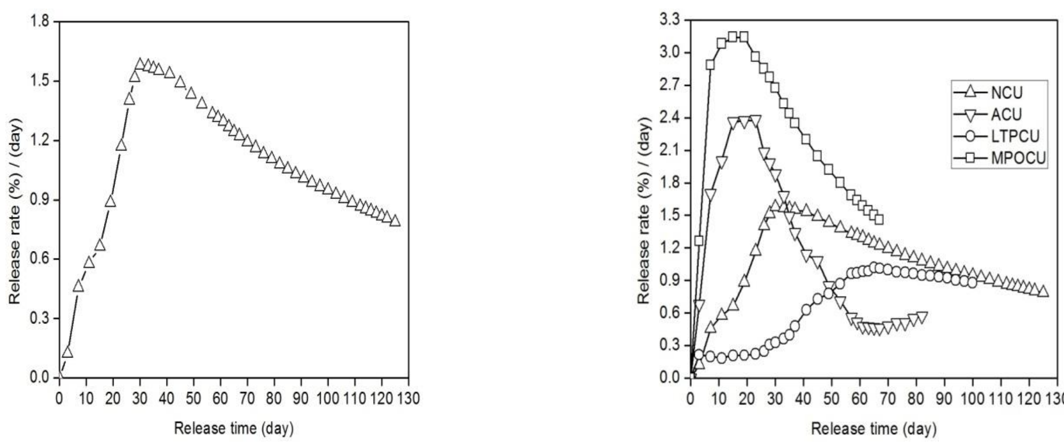


Figure 6(a). Release rate profile NCU, **(b).** Comparison of Release rate profile of NCU with other Coated Urea

Conclusion

In the present study Nitrogen release from NCU is carried out. Experimental results show that nitrogen release percentage follows a sigmoidal behaviour, first, it increase with time and later on becomes constant following a three stage process as discussed previously. The release data also show that NCU match the requirement for controlling release fertilizer. Besides, the use of UV-Vis spectrometer as a promising equipment, in studying the release behaviour is also highlighted.

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How to cite this manuscript: Shiv Om Meena, Manish Vashishtha*, Ankush Meena. Experimental Determination of Nutrient Release from Neem Oil (Azadirachta indica) Coated Urea . Chemical Methodologies 2(4), 2018, 299-307. DOI: 10.22631/chemm.2018.129414.1048.