

Rupture Studies on Kokum Wax Coated Urea Briquettes

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Abstract Applying a coating on the commercially available urea briquettes by materials such as kokum wax can assist in slowing down the dissolution of urea from briquettes in wet soils and hence the loss of urea-N occurring by various mechanisms can be minimized. Since Kokum wax coatings succumbed to rupture in wet soils, stability of the coating was assessed by subjecting coated briquettes to urea release in water. Results indicated that both observed lag period and time taken for rupture increased with increasing coating thickness. Time required for rupture increased relatively rapidly with increasing coating thickness than the lag period. Strength of the coating material, if enhanced by methods such as crosslinking, can delay the onset of rupture initiation.

Keywords: *briquette, kokum, wax, urea, release, coating*

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1. Introduction

Most of the fertilizer urea applied in agricultural fields is lost by various mechanisms [1] such as ammonia volatilization, surface run off, leaching and nitrification-denitrification. In this regard, International Fertilizer Corporation developed a technology for large sized urea product [2], viz. urea briquette, which could mitigate urea loss occurring in agricultural fields by various mechanisms. Although the urea briquettes are known to increase agricultural yield per unit mass of applied fertilizer, they suffer from three major disadvantages occurring sequentially [3] as given below;

1. Briquettes dissolve in about 8 to 10 hours in wet soils since they *per se* are not slow release fertilizers.
2. Due to relatively rapid dissolution, high concentration of urea develops around the dissolving briquette. If this high concentration develops near the root zones, it can lead to root burnout, thereby adversely affecting yield from the plant.
3. On account of high concentration of soil urea, even a minor induced convection current through soil can lead to heavy loss of urea. This is a problem with coarse soils having low cation exchange capacity.

Hence, there arises a need to reduce concentration of urea as briquette shrinks. Slow release fertilizers commonly are pelletized formulations coated with a substance or membrane that slows solubility. In light of this fact, applying coating on briquettes has a potential to reduce concentration of urea developed in the surrounding soil. In addition, coated products can supply urea slowly for a longer time, reduce environmental pollution arising

out of leaching losses/surface runoff, facilitates open air storage of the product, and reduce caking during bulk storage [4].

Considering all the above factors, coated briquettes thus developed need to be tested for urea release in wet soils in order to study barrier property of the applied coatings. Presently there are no reported literatures on coated briquettes. Some of the applied coatings are susceptible to ruptures/fissures in agricultural fields depending upon the nature of coating material and consequently prone to accelerated urea release from exposed locations of the coated urea. From this perspective, it becomes necessary to study such instabilities observed, if any, with the applied coatings.

In this paper, a preliminary assessment of effect of kokum wax coating thickness on the coating stability is made from the perspective of rupture developments, by subjecting the coated briquettes to release in water. The results obtained were discussed with regard to the literature reported theoretical models.

2. Materials and Methods

Urea concentration was measured in this study by the method outlined by Pansu and Gautheyrou [5]. Commercial urea briquettes are ellipsoidal in shape, weigh about 2.1g and have average urea content of about 98.5%.

Kokum wax is a biodegradable material obtained from seeds of the fruit kokum (*Garcinia Indica*), a commonly available fruit in the west coast of India. For the present study, commercially available kokum wax with a melting point of 45°C was used. Kokum wax coating (of thickness 230±21µm) was applied by immersing briquettes in

molten wax at 45°C for two minutes and subsequently filtering molten wax through a mesh. Thicker kokum wax coatings on briquettes were applied by repeatedly subjecting coated briquettes to same procedure. To measure the thickness of applied coating, the following technique was used [6]. A coated briquette was placed in water and the coating was allowed to rupture. Peeled off coating was dried and weighed. The dry coating was subjected to coating thickness measurement using a micrometer. The procedure was repeated to get an average coating thickness and weight.

Applied kokum wax coatings on urea briquettes were prone to ruptures and subsequent release from ruptured locations as observed from wet soil trials [7]. Urea release from coated briquettes can occur by diffusion through coating till it ruptures and the observed release rates are very low in this case. Once the coating ruptures, urea dissolves rapidly at the ruptured locations and rate of release accelerates thereafter. Hence, in order to investigate stability of the coating and the various factors that affect the coating rupture, a study was conducted by subjecting kokum wax coated briquettes for urea release in water. It should be noted that the total released urea, delay in urea release (lag period), and the time taken for coating rupture can be noted easily with urea release in water than in wet soils.

In this study, three different thicknesses of kokum wax coating were considered, viz. 350±39 μm, 430±55μm and 1120±130μm for laboratory trials. The study was conducted by subjecting a kokum wax coated briquette to urea release in 100mL distilled water containing 5ppm PMA as urease inhibitor, at 29±2°C. At different time periods during the release experiment, 1mL of water from the beaker was pipetted out and analyzed for cumulative released urea content. Make up distilled water (containing PMA) of 1mL volume was added to the remaining 99mL so as to maintain the quantity of water present at 100mL. Briquettes were observed visually for the presence of cracks, fissures or ruptures during the trial. Initiation of ruptures/cracks/fissures could be visually noted, which simultaneously produced spike in the measured cumulative urea release. Experiments were conducted in triplicates.

3. Results and Discussions

The data from the release experiments are presented graphically in Figure 1 with an error of ±15%.

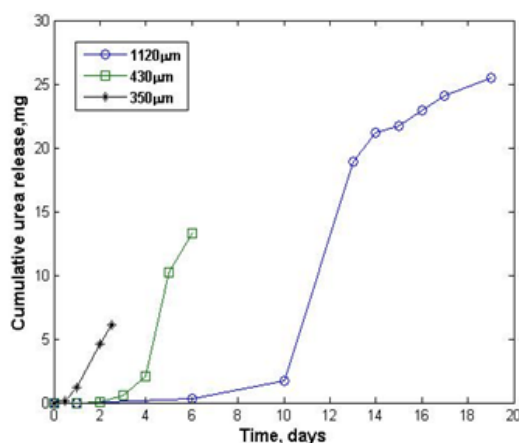


Figure 1. Cumulative urea release versus time from kokum wax coated briquettes in water

Lag period, or the delay in the onset of urea release, was observed for all the three coating thicknesses considered. It can also be observed from Figure 1 that, quantum of release by diffusion through the coating is very limited/negligible; e.g., in the case of 1120μm thick kokum wax coated briquettes, maximum cumulative amount of urea released is 25mg in 19 days, i.e.,

$$\frac{25}{2100} \times 100 = 1.19\% \text{ of urea present in the briquette gets}$$

released through diffusion. Further, single layer kokum wax coated briquettes, with average thickness of 230μm, when subjected to release in water, did not release urea for about 30 minutes and the coating ruptured in about 45 minutes. Lag period and the time taken for rupture versus the three different kokum wax coating thicknesses is graphically shown in Figure 2.

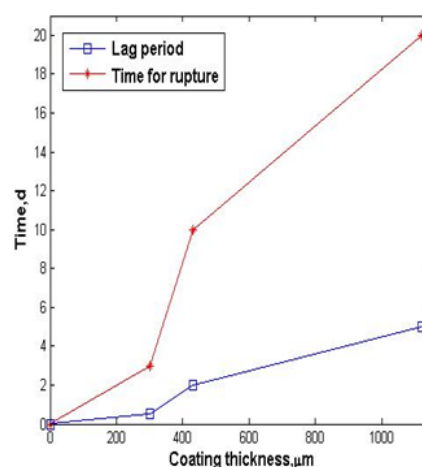


Figure 2. Lag period and time for rupture versus kokum wax coating thickness

It can be observed from Figure 2 that, with increasing coating thickness, the lag periods as well as the time for rupture increases. However, the time for rupture increased more rapidly than the lag period. This indicates that the stability of the coating is better enhanced with coating thickness.

As reported [8], during the lag period, water vapour penetrates into the granule and dissolves a small fraction of the solid fertilizer. The driving force for this is vapour pressure gradient across the coating. The volume for the condensed vapour is limited to the voids present inside the solid core. References [9] and [10] have reported a relationship (equation 1) for the lag period during which water diffuses into the granule through the coating as,

$$t^1 = \frac{\gamma r \delta}{3 p_h \Delta p} \quad (1)$$

γ = granule or briquette internal porosity

$$\gamma = 1 - \frac{\text{briquette density}}{\text{urea density}}$$

Substituting for briquette density (1.31g/mL), urea density (1.32 g/mL) and ignoring pore volume of the coating material,

$$\gamma = 1 - \frac{1.31}{1.32} = 0.0076 \text{ (or } 0.76\%)$$

δ = coating thickness, mm

$$\begin{aligned}
 r &= \text{internal radius of granule, mm} \\
 p_h &= \text{water permeability, mm}^2 \text{ pa}^{-1} \text{ d}^{-1} \\
 \Delta p &= \left(\begin{array}{l} \text{Water vapor pressure saturated} \\ \text{solution vapour pressure} \end{array} \right), \text{Pa} \quad (2) \\
 t^1 &= \text{lag period, d}
 \end{aligned}$$

The internal porosity, γ , is low with briquettes; Lag period is proportional to the product of granule radius and coating thickness, and inversely depends on the driving force (ΔP) and water permeability as per the equation (3). Since the parameters r , P_h and ΔP are constant for the three thicknesses considered, lag period is a linear function of coating thickness. It can be observed from Figure 2, that this relation (equation 3) is approximately linear.

The second stage [8] starts when a critical volume of saturated solution accumulates inside the granule. The rate is almost constant as long as the saturated solution accumulates inside the granule. The rate is governed by the nutrient concentration inside and outside the granule, presence of micropore/macropores of the coating, coating thickness as well as diffusion related properties of the coating material.

A major factor controlling the release from slow release fertilizers is rupture of the coating. Rupture is deemed as 'failure' since urea is expected to diffuse through the coating material rather than release rapidly through the cracks/fissures. In such cases, the rate of pressure buildup inside the granule should be the major factor controlling the release. As reported [10], the time to rupture based on the pressure buildup, t_b , of a single coating can be related to the mechanical properties of the material as (equation 3),

$$t_b \cong \frac{Y_s r \delta}{p_h \Delta \pi M} \quad (3)$$

In the equation(3), Y_s is the yield stress of the coating material (Pa), $\Delta \pi$ is the gradient of osmotic pressure across the membrane (Pa) and M is the Young's Modulus of elasticity of the coating (Pa). Since t_b is directly proportional to coating thickness (δ), with increase of coating thickness rupture initiation gets delayed. It can also be inferred from equation (3) that rupture initiation can be delayed or prevented by opting for stronger coating materials or strengthening the kokum wax coating by crosslinking.

4. Conclusions

Kokum wax coating rupture trials conducted in water indicated that stability and lag period of the coating

increased with coating thickness. Time required for rupture increased relatively rapidly with increasing coating thickness than the lag period. Rupture initiation observed in the case of soft kokum wax coating can be delayed by subjecting the coating material to cross linking in order to enhance the strength of the kokum wax coating.

Abbreviations

M	Young's modulus of elasticity of the coating material, Pa
P_h	water permeability, $\text{mm}^2 \text{ pa}^{-1} \text{ d}^{-1}$
P	water vapour pressure-saturated solution vapour pressure, Pa
PMA	Phenyl Mercury Acetate (inhibitor for enzyme urease)
r	granule (briquette) radius, mm
t^1	lag period, d
t_b	time for coating rupture, d
Y_s	yield stress of coating material, Pa
γ	briquette density, g/mL
δ	coating thickness, mm
$\Delta \pi$	gradient of osmotic pressure across coating, Pa

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