

## Research Article

# Solubility Characteristics and Slow-Release Mechanism of Nitrogen from Organic-Inorganic Compound Coated Urea

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A soil incubation method was used to investigate the solubility characteristics and slow-release mechanism of organic-inorganic compound coated urea at temperature of 10, 20, and 30°C. The membrane microstructure with and without incubation was tested via scanning electron microscopy (SEM). Slow release of nitrogen (N) from different inorganic minerals was analysed by the activation energy from the nutrient solubility system. The rate of nitrogen solubility increased with temperature increasing. The first-order reaction kinetic equation described the solubility process of coated urea. The rate constant  $k$  also increased with temperature increasing. Moreover, the SEM images showed that the microstructure of the coating layer changed into a flocculent structure and the number of tiny pores and holes on the membrane surface increased significantly with temperature increasing, which increased N solubility rate. The Arrhenius equation indicated that activation energy was closely related to  $k$  during the solubility process; the activation energy was reduced with  $k$  rising, which resulted in N solubility rate increasing. Overall, the N solubility rate of coated urea was affected by temperature.

## 1. Introduction

Coated slow-release fertiliser (CRF) is a new kind of fertiliser that provides nutrient release in balance with crop needs [1]. CRF includes inorganic materials and/or organic polymers and is slightly water soluble, with the granular fertiliser at its core [2]. This coating changes the nutrient solubility characteristics and lengthens and controls nutrient release [3]. Studies on CRF have provided new ideas for improving fertiliser use efficiency and minimising pollution to the environment due to fertiliser losses [4]. Du et al. studied effect of several slow-release fertilizers on decreasing ammonia volatilization and N leaching. The results indicated that the development of CRF is a green technology that not only reduces nitrogen loss caused by volatilization and leaching but also alters the kinetics of nitrogen release [5]. Zhang et al. studied slow-release fertilizer encapsulated by graphene oxide films, and the results showed that new coating

technology could hold great promise for the development of environmentally benign controlled-release fertilizer for crop production [6]. Song et al. found that polygorskite-coated fertilizer could be promising for potato production and be beneficial to agricultural sustainability in semiarid areas [7]. Jiao et al. gave some insight to design and characterize new versatile organogelators and soft coated materials with special molecular structures [8, 9]. Wu and Liu studied a double-coated slow-release NPK compound fertilizer with superabsorbent and water-retention. The results showed that this product with excellent slow release and water-retention capacity, being nontoxic in soil and environment-friendly, could be especially useful in agricultural and horticultural applications [10]. Rodrigues et al. found that slow-release N fertilisers are not an alternative to urea for fertilisation of autumn-grown tall cabbage [11]. Paramasivam and Alva and Guertal developed N and irrigation best management practices for citrus in sandy soils to maintain optimal crop

yield and quality and to minimize potential leaching of nitrate below the root zone [12, 13]. Pyoungchung et al. found that, by controlling lignin content and processing temperature of the pellets, one can control the release rate of nutrients present in biochar pellets [14]. Zhong et al. integrated water-retaining and controlled-release fertilizers. The results showed that CRF possessed excellent sustained-release property of plant nutrient and could improve the P release greatly [15]. Rashidzadeh and Olad studied a novel slow-release NPK fertilizer encapsulated by superabsorbent nanocomposite. The results indicated that the presence of the montmorillonite caused the system to liberate the nutrient in a more controlled manner than that with the neat superabsorbent. The good slow-release fertilizer property as well as good water retention capacity showed that this formulation is potentially viable for application in agriculture as a fertilizer carrier vehicle [16]. Zou et al. 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 characteristic of nutrient release and can be utilized as a membrane material combined with inorganic mineral powders to develop film-coated slow-release fertilizer [17]. Muldera et al. developed an economically feasible and biodegradable slow-release coating for urea. The results showed that lignin shows high potential as coating material [18]. Wang et al. found that, with the increase of O-MMT amount, the diameters of the nanofibers decreased and the adsorption rate of MB was evidently improved, and this could be used for CRF. Kondaveeti et al. studied fluorescent agarose-O-naphthylacetyl adduction. The resulting polymer would be of potential utility as a sustained release plant growth regulator and sensory applications [19]. Du et al. studied the waterborne silicone-acrylate emulsions with excellent performance of nutrient controlled release, which provided novel and improved materials for coated CRF industry [20]. Recent studies have concentrated on choosing membrane materials, particularly for creating low-price material that is environment-friendly and has beneficial effects. Fu et al. used g-C<sub>3</sub>N<sub>4</sub> to coat TiO<sub>2</sub> nanocomposites. Xiaoyu et al. used bentonite and organic polymer [21]. Yuan et al. used K-Si-Ca-O compound [22] and Ohshima et al. used a common polymer inclusion membrane for study [23]. However, little research has been conducted on N solubility characteristics and nutrient slow-release mechanisms of organic-inorganic complex coated urea. Slow-release mechanisms are very important to the whole study of CRF and organic-inorganic complex material can largely improve CRF.

In this study, polymers were mixed with inorganic materials and synthesised organic-inorganic complex films using a polymer blending reaction. A rotary coating drum was prepared for CRF. A soil culture method was adopted to investigate N solubility properties, the nutrient release mechanism of the fertiliser, and a foundation for developing and applying new types of CRF. Slow-release mechanisms and immanent causes of organic-inorganic complex coated urea have been investigated in this study. Scientific basis has been

provided for the research and application of environment-friendly, cheap, and new coated slow-release fertiliser.

## 2. Materials and Methods

**2.1. Soil.** The tested soil was brown soil from the Research and Experimental Base of Shenyang Agricultural University. The former stubble crop was corn. Soil samples from 0 to 20 cm deep were collected. The soil physicochemical characteristics were pH, 6.62; soil organic matter, 14.6 g kg<sup>-1</sup>; alkali-hydrolysable N, 84.2 mg kg<sup>-1</sup>; available P, 18.6 mg kg<sup>-1</sup>; and available K, 116 mg kg<sup>-1</sup>.

**2.2. Nitrogen Sources.** The organic-inorganic complex CRFs "BG" and "BF" were the N sources used in the experiment; both had N content of 423 g kg<sup>-1</sup>. The organic materials were based on the modified polyvinyl alcohol (PVA) coded B. The inorganic materials were two kinds of natural secondary minerals. One was functional filler, coded G, which was a metalloid clay mineral mainly composed of amorphous silicon dioxide. It had a small bulk density, large surface area, and strong absorbability. The other inorganic material was porous aluminium silicate, which is a good soil amendment; this material was coded F and mainly consisted of SiO<sub>2</sub>Al<sub>2</sub>O<sub>3</sub> with unique absorbability, ion-exchange, and other features. Organic-inorganic complex films were synthesised using the macromolecule blending reaction between modified PVA and the inorganic materials. Subsequently, the CRFs were prepared in a rotary coating drum.

**2.3. Experimental Design and Methods.** Fertiliser particles of the same size and with a complete coating were used. First, we placed 0.35 g of coated fertiliser in 20 mesh gauze bags (6 cm × 6 cm), which were sealed and tagged. Second, 70 g portions of air-dried soil (mass water content was 26 g kg<sup>-1</sup>) were placed in self-sealing plastic bags with wide covers, and 15.5 mL of distilled water was added to each bag to increase the soil moisture level to 70% of field capacity. The bags were sealed and incubated for 24 hours at three different temperatures (10, 20, and 30°C). Each fertiliser had three treatments. The gauze bags were buried in the soil and cultured. On days 3, 5, 7, 10, 14, 21, 28, 35, and 42 of culturing, gauze bags were removed from the self-sealing plastic bags and carefully washed with distilled water. The fertilisers were dried and then digested in sulphuric acid. Residual N was determined by a Kjeltex 8100 distillation unit, and the N solubility rate of the coated fertiliser at different culture times was calculated. Each treatment was replicated 27 times.

## 3. Results and Discussion

**3.1. Characteristics of N Solubility.**  $y = ae^{b/t}$  ( $a > 0$ ,  $b < 0$ ) is suitable for *curve fitting*. Figures 1 and 2 showed that the N solubility rates increased with increasing temperature and were highly responsive to incubation temperature. These figures also show that the different CRF materials had different N solubility starting times and the BG starting time was comparable to the delayed BF starting time. The lag

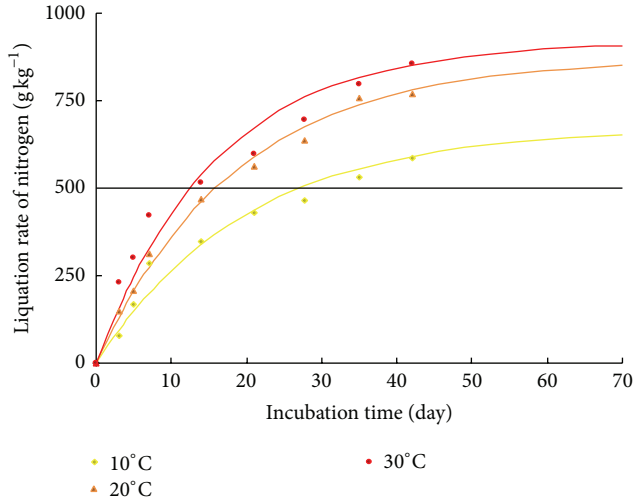


FIGURE 1: The accumulated nutrient leaching rate of BF at different temperatures.

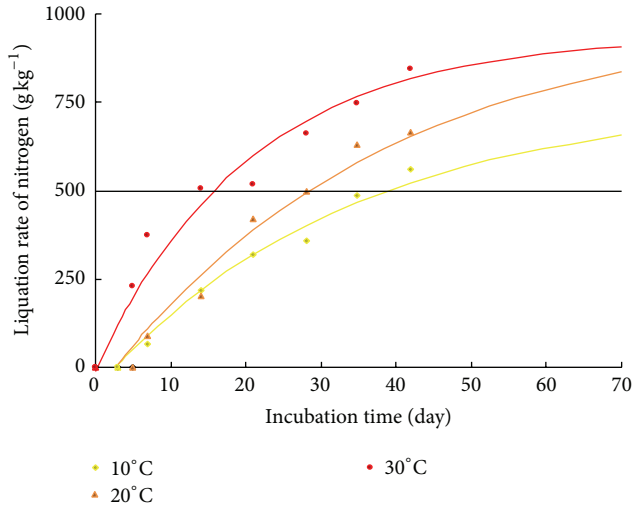


FIGURE 2: The accumulated nutrient leaching rate of BG at different temperatures.

became shorter as the temperature increased, which may have been related to the bond between the BG organic and inorganic materials; such a bond could have enhanced the water-blocking ability of the membrane materials and produced the N leaching lag period. In contrast, BF was released immediately. No relationship was found between the N leaching starting time from BF and culture temperature. The number of days for 500 g kg<sup>-1</sup> N release from the two coated urea was differed by culture temperature. Specifically, BF was released in 27.1 days at 10°C, 16.2 days at 20°C, and 13.5 days at 30°C, whereas BG was released in 42.5 days at 10°C, 28.3 days at 20°C, and 16.8 days at 30°C. Thus, the leaching time for 500 g kg<sup>-1</sup> N was reduced with increasing culture temperature. The results show that higher culture temperature resulted in faster N solubility. Moreover, at the same temperature, 500 g kg<sup>-1</sup> N leached from BG more times

than from BF. Thus, BG achieved a better N release effect than BF. This may have been caused by the BG coating bonding with the urea, thus improving the sustained release effect of the BG material compared to the BF material. The curves in Figures 1 and 2 show the relationship between the N leaching rate (for BG and BF) and time.

**3.2. Dynamic Simulation of N Solubility.** Figures 1 and 2 showed that the N release rates of BG and BF increased with increasing culture temperature and then tended to be constant after a particular time. The rates had a parabolic relationship with time, described by first-order reaction kinetics as follows:

$$-\frac{dc}{dt} = k \cdot c, \quad (1)$$

$$-\int_{c_0}^c \frac{dc}{c} = \int_0^t k \cdot dt.$$

In the case of  $t = 0$ , the reactant concentration is  $c_0$ ,  $t = t$ , and reactant concentration is  $c$ . After integrating the above formula, the expression changed to

$$\ln \frac{c_0}{c} = kt, \quad (2)$$

$$c = c_0 \exp(-k \cdot t).$$

The relationship between nutrient concentration and time is given as

$$N_0 - N_t = N_0 \exp(-k \cdot t), \quad (3)$$

$$N_t = N_0 [1 - \exp(-k \cdot t)],$$

where  $N_t$  is the solubility rate (g kg<sup>-1</sup>),  $N_0$  is the maximum solubility rate (g kg<sup>-1</sup>),  $k$  is the constant of the solubility rate (d<sup>-1</sup>), and  $t$  is solubility time (d).

After CRF was applied, water infiltrated the membrane material, dissolved the fertiliser, and released the nutrients. The release time was also associated with the composition and physicochemical characteristics of coating materials, as reported by several previous studies. Therefore, a lag period was observed during nutrient leaching. On the basis of the first-order kinetic Equation (3), we calculated a more complex relationship:

$$N_t = N_0 \{1 - \exp[-k(t - t_0)]\}, \quad (4)$$

where  $t_0$  is the lag period of release (d).

Figures 1 and 2 show that the two CRFs began to release N under a certain soil moisture content with no lag period. Thus, they can be simulated by the first-order kinetic equation  $N_t = N_0[1 - \exp(-kt)]$ , and the simulated parameters are shown in Table 1. The correlation coefficients ( $r$ ) were all highly significant for the N solubility rate of the two CRFs at different culture temperatures ( $P < 0.01$ ). The constant  $k$  of the two fertilisers was also different between different culture temperatures. Moreover, the value of  $k$  increased with increasing temperature, indicating that temperature



TABLE 1: Simulated first-order kinetic equation parameters for the nitrogen accumulated leaching rate in the BF and BG coated, slow-release urea.

Culture temperature	$k$		$t_0$ (d)		$R$		Se	
	BF	BG	BF	BG	BF	BG	BF	BG
10°C	0.033	0.019	0	2.73	0.9918**	0.9925**	0.125	0.033
20°C	0.053	0.026	0	2.68	0.9816**	0.9906**	0.130	0.073
30°C	0.062	0.046	0	0.21	0.9937**	0.9933**	0.123	0.085

\*\*Data are very significant.

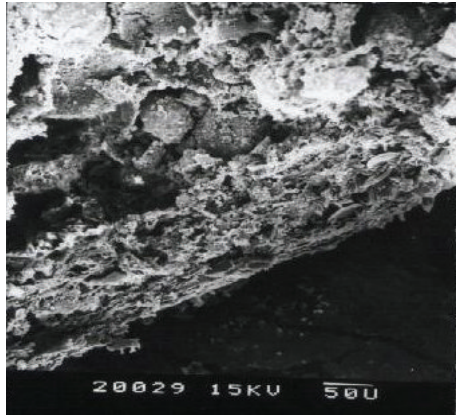


FIGURE 3: Section of 10°C coated fertiliser (×200).



FIGURE 4: Section of 30°C coated fertiliser (×200).

significantly affected the rate of organic-inorganic compound CRFs and that temperature was the main factor for changes in the N solubility rate. Urea solubility within the coating accelerated with the increase in temperature and the rate of water infiltrating the membrane. At the same time, membrane structure changed. As a result, pores on the membrane surface increased and accelerated the N solubility rate.

The BG rate constants were also smaller than those of BF (Table 1). The BG coating material significantly reinforced the capability of slow-release of N from the coated urea. A lag period was observed as N from BG was solubilised. This result indicated that the coatings were linked closely with the granular urea by slowing the material water infiltration rate and delaying the nutrient solubility time. The movement of water increased with the increase in temperature, and the lag became shorter as the temperature increased.

**3.3. Changes in CRF Microstructure.** BG was removed from the soil after incubation at three different temperatures (10, 20, 30°C), and the bags were washed and air-dried. The section with the coated fertiliser and the membrane surface were analysed by SEM. The results are shown in Figures 3 to 8.

Figures 3 and 4 showed that the membrane materials from BG remained in contact with the fertiliser, demonstrating that the coating had permeated the pores on the fertiliser surface. The coating was bound more closely to the granular urea.

The coating microstructure changed into lumpy or fluffy masses with increasing temperature and formed various pores and holes (Figures 5 and 6). In addition, the holes

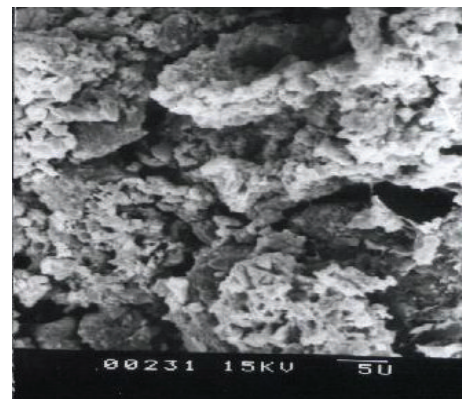


FIGURE 5: Section of 10°C coated fertiliser (×2000).



FIGURE 6: Membrane section of 30°C coated fertiliser (×2000).

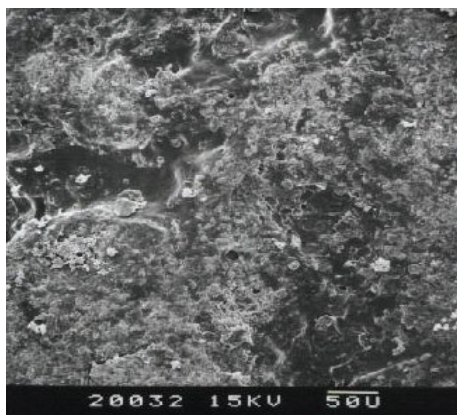


FIGURE 7: Membrane surface of 10°C coated fertiliser (×200).

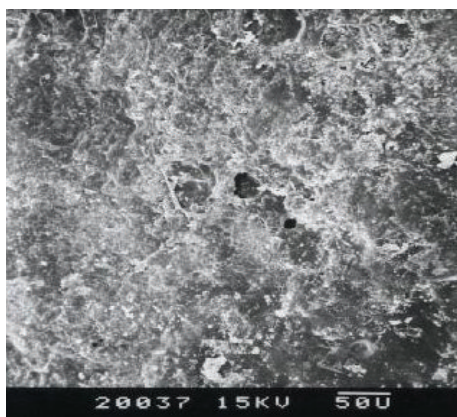


FIGURE 8: Membrane section of 30°C coated fertiliser (×200).

and pores became larger as temperature increased, probably because the coating consisted of PVA/starch cross-linked fluid and hydroxyl groups that can bind with water and adsorb moisture, leading to swelling of the material. The degree of swelling was greater as the temperature increased, resulting in a change in the structure of the membrane. This was also the main reason for the acceleration of the nutrient release rate as the temperature increased. Figures 7 and 8 showed that the membrane surface structure also changed with temperature increasing after the soil incubation. The numbers of pores and apertures were significantly increased at 30°C compared to at 10°C. It is possible that the movement of water accelerated with increasing temperature and that the speed at which water molecules combined with the hydroxyls from the PVA/starch cross-linked fluid increased. Water molecules adsorbed on the membrane surface, dissolved the coating minerals, and reduced the film-forming property of the PVA/starch cross-linked fluid. As a result, the membrane surface developed tiny pores.

**3.4. Relationship between  $k$  and  $E_a$ .** The above analysis indicates that temperature significantly affected the constant

$k$  of CRF. The relationship between the rate constant ( $k$ ) and temperature ( $E_a$ ) followed the Arrhenius equation:

$$k = A \exp\left(-\frac{E_a}{RT}\right), \quad (5)$$

where  $k$  is the release rate constant ( $d^{-1}$ ),  $A$  is a preexponential factor ( $d^{-1}$ ),  $E_a$  is activation energy ( $J mol^{-1}$ ),  $R$  is the gas constant ( $8.31 J mol^{-1}$ ), and  $T$  is absolute temperature (K).

In the Arrhenius equation, the value of  $E_a$  significantly affects the reaction speed as it appears on the index. When the value of  $E_a$  is smaller, reaction speed is greater. In terms of chemical reaction kinetics, a lower  $E_a$  value results in a higher reaction speed and is favourable for the reaction [12]. When the value of  $E_a$  was lower, the N solubility rate of CRF was higher. Therefore, from (5), the value of  $E_a$  could be calculated as the constant of the reaction rate if any two temperatures are known. The expression was deduced as

$$\ln \frac{k_2}{k_1} = \frac{E_a}{R} \left( \frac{T_2 - T_1}{T_1 T_2} \right). \quad (6)$$

Activation energy  $E_a$  can be regarded as a constant in the temperature range of 10–30°C for the N solubility reaction of the CRF. Based on (6), the activation energy  $E_a$  from the N solubility process of BF and BG was calculated as 4016  $J mol^{-1}$  and 5963  $J mol^{-1}$ , respectively. The higher N solubility rate of BF compared to that of BG was due to its lower activation energy. Thus, the abovementioned theory explains that the BF rate was greater than that of BG.

## 4. Conclusion

Nitrogen solubility from organic-inorganic complex coated urea increased with culture temperature increasing. The microstructure in the coating layer changed from a sheet to a flocculent structure with tiny pores and holes on the membrane surface, which increased significantly with temperature increasing, thereby increasing the rate of N solubility. The first-order reaction kinetic equation  $N_t = N_0[1 - \exp(-kt)]$  described the N solubility feature, and the constant  $k$  also increased with temperature increasing. Moreover, the Arrhenius equation,  $k = A \exp(-E_a/RT)$ , indicated that activation energy was closely related to  $k$  during the solubility process, which was reduced with rising  $k$  and resulted in an increase in N solubility rate. The activation energy from the N solubility process changed after the urea coating. Changes in activation energy were an internal factor for the N solubility characteristic of coated urea, and the activation energy was closely related to the material properties and microstructure during the solubility process.

## Conflict of Interests

The authors declare that they have no conflict of interests regarding this work.

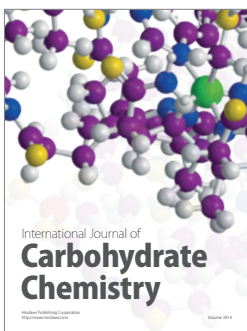
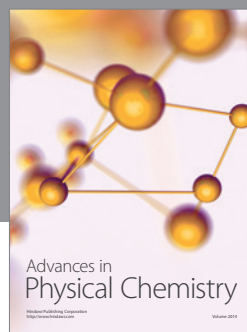
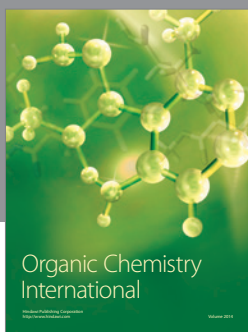
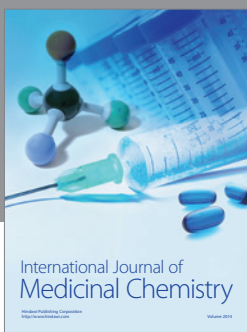


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