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Research Article

Prediction of Release Characteristics of Film-Coated Urea from Structure Characterization Data of the Film

An efficient method for the prediction of the release characteristics of film-coated urea from its film structure is proposed. A model spray-coating apparatus was built to prepare a plane film to directly measure the film permeability coefficients by simulating the characteristics of randomness, intermittence and multiple layers of the coating, and eliminating particle collision effects. From analysis, the spray-coated film structure can be modeled as comprising a dense structure and a defect structure. Both structures contributed to the penetration flux and the apparent permeability coefficients of the film for water and urea diffusion. The permeability coefficients of the plane films were calculated from the film structure model, and were shown to be close to the measured values.

Keywords: Controlled release, Film coating, Film structure, Permeability coefficient, Release characteristic

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1 Introduction

Film-coated urea is used to increase urea utilization efficiency, and many works have focused on the high performance of controlled-release urea. The production of controlled-release urea by spray coating of polymer latex is an environmentally benign technology, as it does not need a toxic organic solvent [1]. The film material, film structure and release environment affect the release rates [2–4].

Currently, the water dissolution method and soil diffusion method are used for measuring the release characteristics of film-coated urea [5, 6]. The measurements for the whole release process take a long time, especially for long-release-period urea, which can be six months or longer. This is inefficient for the research and development of controlled release urea technology. More importantly, this does not allow the timely checking of product quality in industrial production. Improvements on the water dissolution method for shortening the measurement period, e.g., using an increased temperature, change in pH, etc., are still under exploration [7]. The soil diffusion method also has the same problem of lengthy measurements [8]. There is a need to establish an efficient and accurate method for predicting the release characteristics of controlled-release urea.

For polymer-film-coated fertilizer, the nutrients are released through the film by diffusion [9]. Shaviv et al. [10] analyzed the release process of film-coated urea and suggested that the film permeability coefficients of water vapor and urea are the key parameters determining the release rates. By modeling the release process and measuring the permeability coefficients of water vapor and urea, the release characteristics of the film-coated urea can be predicted. Dirim et al. [11] prepared polyethylene films with dense and simulated holes, measured the permeability coefficients of the film, and showed that the pore structures determined the diffusion flux and permeability coefficients. Xin et al. [12] employed the grid method to find a relationship between the structure and permeability in a porous media, and showed that it is a suitable method for the simulation of the mass transfer process.

A key point for predicting the release characteristics of film-coated fertilizer is to establish a model for the relationship between film structure and permeability coefficients. In this paper, a structure model of spray-coated film was built, from which the permeability coefficient of the film could be calculated using its structure parameters. The relationships between film structure, permeability coefficients of the film, and release characteristics of the coated urea were modeled for efficiently predicting the release characteristics.

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2 Experimental

2.1 Materials

Poly(styrene-butyl acrylate) latex (40 % solid content and average diameter of about 80 nm) was used. The poly(styrene-butyl acrylate) latex was synthesized via semi-continuous emulsion copolymerization of butyl acrylate, styrene, methacrylic acid and crosslinker at 85 °C for 3.5 h, according to reference [13].

Large granular urea of commercial product origins (China Blue Chemical Ltd.) was used. The chemicals used included: urea (analytical grade, Fine Chemicals Co., Ltd., China), 4-Dimethylaminobenzaldehyde (analytical Grade, Sinopharm Chemical Reagent Co., Ltd., China), absolute ethyl alcohol (analytical grade, Modern Eastern Fine Chemicals Co., Ltd., China), hydrochloric acid (analytical grade, Modern Eastern Fine Chemicals Co., Ltd., China), allochroic silicagel (Modern Eastern Fine Chemicals Co., Ltd., China), and deionized water.

2.2 Film Preparation

Cast film, plane film by spray coating, and coated urea films were prepared and used in this study. The cast film was used to measure the film intrinsic permeability of the coating material. The cast film was prepared by coating latex on a glass plate of 100 mm × 80 mm, which was then placed in an incubator (30 °C) to form a film with a thickness of 50–70 µm.

The plane film prepared by spray coating was used to measure the permeability coefficients of the films. The spray coating process, in a fluidized bed, is affected by many factors, and it lacks representative to characterize the film structures by using an individual coated film of a particle. In order to simulate the characteristics of the randomness, intermittence and multiple layers of the coating, and eliminate the effects of particle collision and increase the representativeness of coated film, an experimental simulation apparatus was used to prepare plane films by spraying so as to directly measure the film permeability coefficients [14].

Coated urea was prepared for measuring its film structure and predicting the release characteristics of the film-coated urea. A Wurster fluidized bed was used to prepare the film-coated urea [15, 16]. The coated urea particles were cut and dissolved in deionized water, and subsequently, the films were washed clean and dried for characterization.

2.3 Film Characterization

The permeability coefficients of the plane films were measured using the methods reported by Lan et al. [14]. A scanning electron microscope (SEM, JSM7401, JEOL, Japan) was used for characterizing surface morphology. A mercury porosimeter (Autopore IV 9510, Micromeritics Instrument Corp., USA) was used to measure the pore size distribution of the films.

2.4 Measurement of Coated Urea Release

The measurement was carried out according to the National Standard of China (GB/T 23348-2009). The basic procedures are as follows: 10.0 g coated urea was weighed and put into a mesh bag of nylon, then placed in a sealed flask with 200 mL deionized water at 25 °C; the urea concentration was measured after a time interval by sampling the solution and used to calculate the cumulative release amount, and then the liquid in the flask was replaced by 200 mL of fresh deionized water.

3 Film Structure Model and Permeability Coefficient Calculation

3.1 Structural Characteristics of the Spray Coated Film

The spray coating process has the characteristics of randomness, intermittence and multiple layers. The process is shown in Fig. 1. Latex was atomized by pressurized air and sprayed onto the surface of urea particles. The film was then formed by dewatering via the fluidizing gas. In the fluidized bed, the coating and dewatering processes were repeated many times for a particle. When spray coating was completed, a film of specified thickness was formed [17].

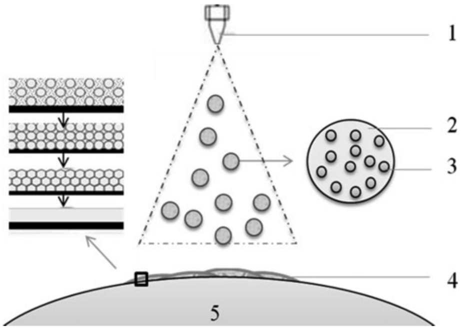

Figure 1. Schematic diagram of spray coating process: 1-nozzle; 2-latex droplet; 3- latex particle; 4-latex droplet spreading; 5-urea particle.

In literature [18, 19], the latex film formation process is divided into three stages, i.e., latex particles get close to each other, deform and touch together, and form a film as the latex is dewatered. In the spray coating process, the latex droplets are random, intermittent and multiple. Researchers [20, 21] have reported that it is easy to form holes, bubbles and phase interfaces in the film since the spray coating process involves droplets (at micron scale), shear and collision.

The pore distribution of the cast film and plane film from spray coating were obtained by mercury porosimeter. The results are shown in Fig. 2. Fig. 2 shows that the pore size distribution of the spray coating film has an obvious bimodal characteristic, with pores of the micron-scale corresponding to a defect structure and pores of the nano-scale corresponding to the intrinsic structure in the films. The cast film mainly showed the nano-scale pores corresponding to the intrinsic structure in the films. The defect structure included bubbles, cracks, etc., with pore sizes of 10–35 µm (Film S1, S2 in Fig. 2), which depended on the spray-coating conditions. The dense structures had pores with sizes mostly less than 100 nm, which depended on the film formation process of latex particles. Fig. 2 also shows that the dense structures had little differences in the cast films. Fig. 3 shows a spray-coating film, which includes the defect structure and the dense structure of the films.

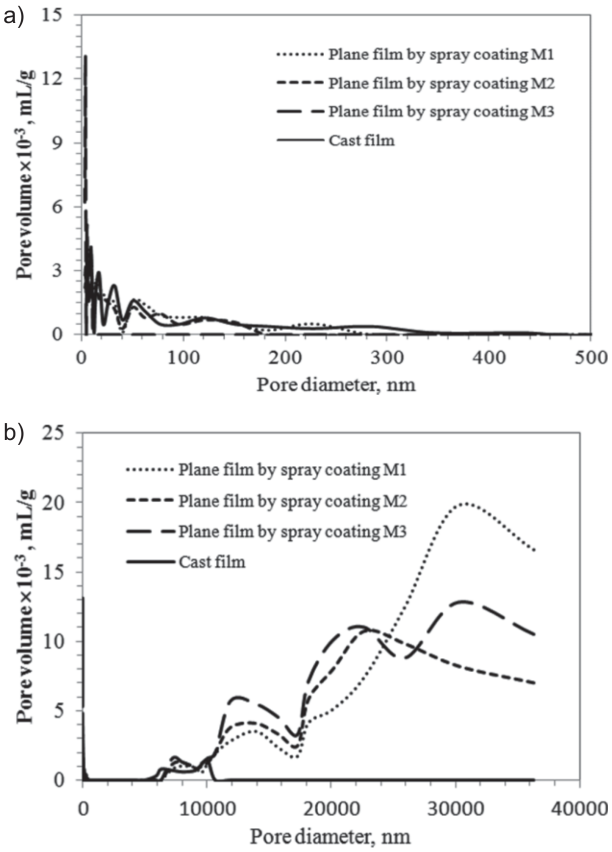

Figure 2. Pore size distribution of spray coating film and cast film: a. 0–500 nm, b. 0–40 µm.

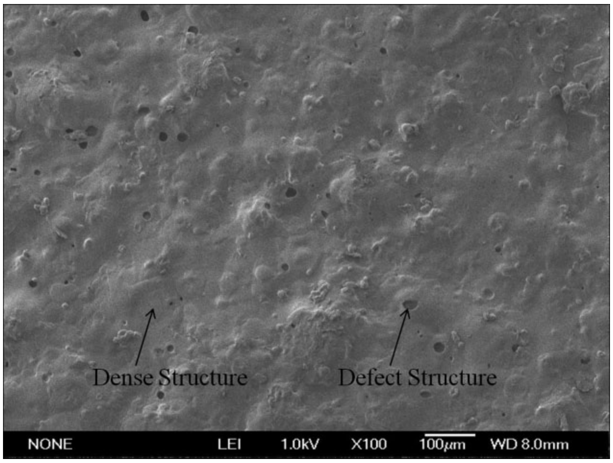

Figure 3. Defect structure and dense structure in a spray coating film.

3.2 Film Structure Model

3.2.1 Assumptions

The above characterization of the film structure indicated that the spray coating film can be considered as comprising a dense structure and a defect structure. The dense structure is similar to the structure of the cast film, and it is essentially the continuous phase of the film. The cast film was prepared at a certain temperature and humidity in an incubator. The temperature and humidity environment of film formation for cast film preparation was similar to that of coated film. Also, the experiments showed that at this environment (the temperature and humidity) did not obviously influence the film permeability. It is reasonable to use cast film to represent the film intrinsic permeability.

It is analyzed that the defect structure is mainly formed by latex droplet accumulation on the urea surface due to the random coating of the droplets. So, the distribution of the defect structure in the film was taken to be random. To simplify this and still retain the essential features, the defect structure was modeled as composed of pores with the average pore size of the spray coating film, with these pores being distributed randomly.

3.2.2 Multilayer Model

Since the film formation can be considered to be the accumulation of a multilayer coating, the film structure was modeled as comprising several layers, which is shown in Fig. 4. Each layer was divided into $N = 500 \times 500$ cubes. The length of the cube side was equal to the average pore size d[1]. The empty cubes represented the defect structure, and the dense cubes represented the dense structure.

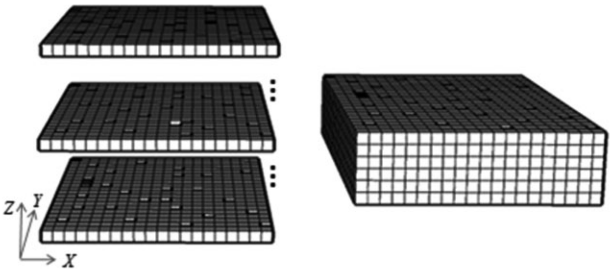

Figure 4. The model of spray film structure.

For one layer, the probability of empty cubes in the grid, P, was calculated as

$$P=m/N \tag{1}$$

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[1] List of symbols at the end of the paper.

where m is the number of pores in one layer, which was calculated from the pore volume and the average pore size, and N was the number of cubes. Then, the probability of dense cubes, Q, was

$$Q = 1 - P \tag{2}$$

The physical film obtained from spray coating can be considered as a superposition of multilayers. At Z direction, the multilayer is comprised of many independent channels. The probability of the pore superposition with different numbers of each channel can be calculated from the assumption of a random distribution of pores. The results are shown as Tab. 1.

3.3 Diffusion in a Through Channel

Nutrients are released from the urea fertilizer by diffusion of molecules or ions. For simplicity, the interaction was not considered in the model. In the measurement of the film permeability, the driving force from water vapor partial pressure and urea solution concentration are constant (the process is governed by Fick's law). In through channel of the film, the diffusion process is governed by Fick's law. For water vapor, the driving force for mass transfer is the partial pressure difference, which is [22]

$$N_h = D_{eh} \Delta P / RTL \tag{3}$$

$$D_{eh} = \frac{\varepsilon}{\tau} \left[\frac{1}{1/D_h + 1/D_{kh}} \right] \tag{4}$$

where N_h is the diffusion flux of water vapor, D_{eh} is the effective diffusion coefficient of water vapor, ΔP is the difference of water vapor partial pressure between both sides of the film, R is the molar gas constant ($8.314\,\mathrm{J\,m\,mol^{-1}K^{-1}}$), T is the temperature of water vapor, L is the thickness of the film, ε is the pore void volume fraction, τ is the tortuosity of the pore, D_h is the molecular diffusion coefficient of water vapor ($2.5 \times 10^{-5}\,\mathrm{m^2 s^{-1}}$), and D_{kh} is the Knudsen diffusion coefficient.

For a urea solution, the driving force for mass transfer is the concentration difference, which is [22]

$$N_s = D_{es} \Delta C / L \tag{5}$$

$$D_{es} = \varepsilon D_s K_{rs} / \tau \tag{6}$$

where N_s is the diffusion flux of the urea solution, D_{es} is the effective diffusion coefficient of urea solution, ΔC is the difference of urea concentrations between both sides of the film, D_s is the molecular diffusion coefficient of urea ($1.38 \times 10^{-9}\,\mathrm{m^2 s^{-1}}$), and K_{rs} is a restriction factor determined by the diameter of the pore and molecular urea.

3.4 Diffusion in Other Channels

The diffusion of water vapor and urea solution through other channels of the film is dominated by the dense parts of the channels, which are governed by Fick's law.

For water vapor [20]:

$$\frac{\mathrm{d}m_h}{\mathrm{d}t} = \rho_g P_h \Delta P A / L \tag{7}$$

where m_h is the mass of water vapor that had diffused from time 0–t, ρ_g is the density of water vapor, P_h is the permeability coefficient of water vapor, A is the sectional area of the channel, and L is the thickness of the dense part in the channel.

For the urea solution [20]:

$$\frac{\mathrm{d}m_h}{\mathrm{d}t} = P_s \Delta C A / L \tag{8}$$

where m_s is the mass of urea that had diffused from time 0 to t, and P_s is the permeability of urea.

3.5 Permeability Coefficient Calculation

The flux in the Z direction was calculated by Fick's law in the 500×500 channels. Mass transfer in the X and Y directions were neglected. For the channel having empty cubes, the equivalent thickness of the channels was used. It was assumed that the mass transfer resistance was concentrated in the dense cubes, and the permeability coefficients in the dense cubes were assumed to be equal to the permeability coefficients of the cast films. The permeability fluxes of water vapor and urea solution were obtained from the accumulation of different channels in the Z-direction. With the total permeability flux, film area, and film thickness, the permeability coefficients can be calculated according to Fick's law.

Since this calculation can only be performed for integer layers and the real film was not usually formed of integer layers, the permeability coefficients of the real film were obtained by interpolation from the curve of permeability coefficients versus the thickness of the multiple layers. In addition, the diffusion of water vapor and urea was affected by the size and amount of pores in the coated film. For a different material of coated film, the hydrophilicity and hydrophobicity of the material affect the permeability of the film. The model can be improved by defining an effective pore size according to the

Table 1. Number of empty cubes in the channels.

Quantity of empty cubes in channels	0	1	2	k*	n
Probability P_k	$C^0_n P^0 Q^n$	$C^1_n P^1 Q^{n-1}$	$C^2_n P^2 Q^{n-2}$	$C^k_n P^k Q^{n-k}$	$C^n_n P^n Q^0$
Number of channels	$P_0 N$	$P_1 N$	$P_2 N$	$P_k N$	$P_n N$

* $k = 0,1,\ldots,n$; n = layer number.

hydrophilicity and hydrophobicity of the material; the details need to be studied further in the future.

Table 3. Transfer flux calculation of a 6-layer model for *S3*.

Number of pores in the channel	Probability	Number of channels	Area [mm^2]	Urea flux [g s^{-1}]	Water vapor flux [g s^{-1}]
0	0.6473	1.62E+5	4.19E–3	9.14E–10	7.82E–8
1	0.2920	7.30E+4	1.89E–3	4.95E–10	4.23E–8
2	0.0549	1.37E+4	3.56E–4	1.16E–10	9.94E–9
3	0.0055	1376	3.6E–5	1.55E–11	1.33E–9
4	3.10E–4	78	2.0E–6	1.32E–12	1.12E–10
5	9.34E–6	2.33	6.05E–8	7.91E–13	6.76E–12
6	1.17E–7	0.03	7.58E–10	7.06E–11	1.76E–9
Total	1.000	250 000	6.48E–3	1.61E–09	1.34E–7

3.6 Comparison of Calculation and Measurement

Plane films were prepared by spray coating in the experimental simulation apparatus [14]. The porosities of the plane films were measured by the porosimeter. In order to explain the calculation process, an S3 film was used as an example, of which parameters were listed in Tab. 2. It is shown that the average pore size of the defect structure was 16.1 μm. The thickness of the S3 film was 102 μm.

The models with 1 layer to 7 layers were built, and the permeability fluxes for each model were calculated, respectively. As an example, the calculation of a 6-layer model for the S3 film is listed in Tab. 3. Then, the permeability coefficients versus the thickness of the multiple layers could be obtained. The main part of the curves is shown in Fig. 5. The permeability coefficients of the film with the same thickness were obtained by interpolation from the curves in Fig. 5.

For different plane films of S1, S2 and S3, the permeability coefficients of water vapor and urea solution were measured and compared with the calculated values. The results are listed in Tab. 4, which showed that the calculated and the measured values agreed well.

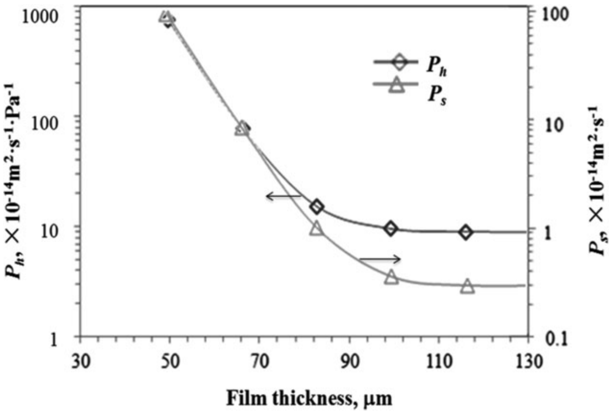

Figure 5. Permeability coefficients vs the film thickness in multilayer model.

Table 2. The defect structure parameters of film *S3* in the model.

Volume of pores [mL g^{-1}]	Average pore size [μm]	Number of pores [g^{-1}]	Number of pore in single layer model m_{S3} [–]
0.0709	16.1	16 748 059	17 484

Table 4. Comparison of the permeability coefficients from measurements and calculations.

	S1	S2	S3
Average pore diameter [μm]	17.7	13.0	16.1
Density of pore [10^7 g^{-1}]	1.62	2.52	1.67
Film thickness [μm]	78	90	102
Permeability coefficient of water vapor, × 10^{-14} m^{2}s^{-1}Pa^{-1}			
Experimental measurement	79.3	19.4	13.4
Model calculation	102	12.8	12.1
Permeability coefficient of urea, × 10^{-14} m^{2}s^{-1}			
Experimental measurement	10.6	0.38	0.41
Model calculation	9.4	0.44	0.37

4 Prediction of the Release Characteristics of Film-coated Urea

4.1 The Model of Film-coated Urea's Release Process

According to the characteristics of the driving force, the release of film-coated urea in water can be divided into three periods: lag period, stable period and decline period, which are shown in Fig. 6 [10, 23]. In the three stages of the film-coated urea's release process, each stage can be regarded as meeting the condition of Fick's law. Fick's law was used in the analysis of the lag period, the stable period and the decline period. The model was established in different stages to predict the release characteristics, which is reasonable, effective and easy.

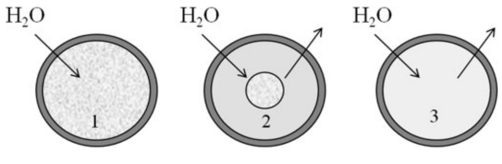

Figure 6. Release periods of film-coated urea at the lag period, the stable period, and the decline period: 1-urea particle; 2-urea saturated solution; 3- urea solution.

In the lag period, water vapor entered the urea particle through the film and dissolved the solid urea. The driving force for this process is the difference of the water vapor partial pressure between the inside and outside of the film. In this period, the release rate is 0. In the measurement, the water transfer rate is

$$\frac{\mathrm{d}m_\mathrm{h}}{\mathrm{d}t} = \rho_\mathrm{g} P_\mathrm{h} \Delta P A / L \tag{9}$$

In the stable period, nutrients began to be released, and the release rate was constant until the urea had dissolved completely. The driving force for this process is the urea concentration difference between the inside and the outside of the film. In the measurement, the urea transfer rate is

$$\frac{\mathrm{d}m}{\mathrm{d}t} = P_\mathrm{s} A \frac{C_\mathrm{sat} - m_\mathrm{s}/V_\mathrm{s}}{L} \tag{10}$$

where C_sat is the saturated concentration of the urea, and V_s is the volume of water.

In the decline period, the solid urea had dissolved completely, and the driving force from the concentration decreased. In the measurement, the urea release rate is

$$\frac{\mathrm{d}m_\mathrm{s}}{\mathrm{d}t} = P_\mathrm{s} \Delta C A / L = \frac{P_\mathrm{s}}{L} A \left[\rho_\mathrm{s} - \left(\frac{\rho_\mathrm{s}}{m_\mathrm{c}} + \frac{1}{V_\mathrm{s}} \right) m_\mathrm{s} \right] \tag{11}$$

where m_c is the mass of core particle, and ρ_s is the urea density.

4.2 Prediction of the Release Characteristics of Film-Coated Urea

In order to assess the model, the release rate of film-coated urea with different film thickness was measured. The pore size distribution of the film coated on the urea particle was measured by a mercury porosimeter. The average film thickness of the coated particles was estimated from the coating quantity. The permeability coefficients of the film were calculated using the multilayer model. The results are listed in Tab. 5.

From the calculated permeability coefficients, the release rate of coated urea can be calculated from Eqs. (9)–(11). Fig. 7 shows the calculated curves of cumulative release versus time, together with the release curves from experimental measurements. It can be seen that the predicted and measured characteristics were close.

The release characteristics of film-coated urea were mainly determined by the permeability coefficients of the film, which depended on the film structure parameters such as the pore size, the pore distribution and the pore density. The calculations showed that the defect structure and the thickness of a film significantly affect the permeability coefficients of the film.

Table 5. Prediction of permeability coefficients from the film structure of the coated urea.

Sample	R1	R2	R3
Average pore size [µm]	14.6	12.5	13.8
Pore density [10^7 g^{-1}]	2.04	1.84	2.82
Film thickness [µm]	40	40	66
Permeability coefficient of water vapor, 10^{-14} m^{2}s^{-1}Pa^{-1}			
Simulation value	365	59.4	16.2
Permeability coefficient of urea, ×10^{-14} m^{2}s^{-1}			
Simulation value	36.2	2.89	1.37

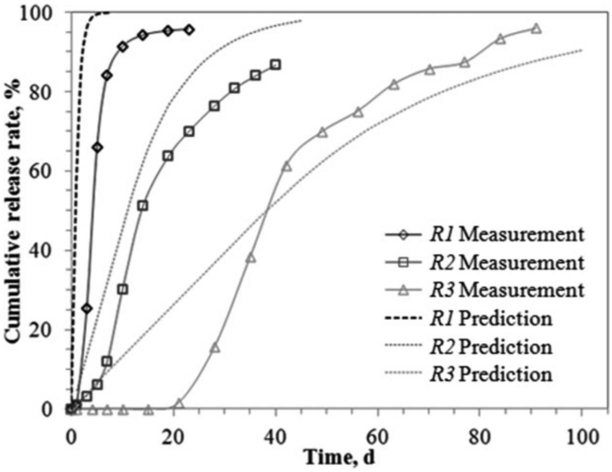

Figure 7. Comparison of the release characteristics between measured and calculated.

The difference between prediction and measurement for curve R3 in the lag period was analyzed, and seen to be caused by the interaction between water and the polymer film. The interaction changes the film structure and the water form in the film, affecting the water transfer character [23]. The film thickness of R1, R2 and R3 were 40 µm, 40 µm and 66 µm, respectively. For a thick film, the interaction affected the release process more obviously. There were few through defect structures in the thick film, which also slowed the rate of water vapor entering the coated urea. Due to the complexity of the process and for simplicity, the interaction of water and the film materials was not considered in this paper. A typical release model of coated urea was used after referring to the literature, i.e., a vapor phase of the water in the film diffusion, driven by the vapor partial pressure, was assumed in this paper [10]. In fact, the transfer of water in the film is a complex process due to the interaction of water and the coated film. Firstly, it needs to be analyzed and confirmed that the water diffusion in the film is in a vapor phase or a liquid phase, or other form, which depends on the properties of the film materials and the film structures. Secondly, the effective pore size of the film structure depends on the characteristics of the film material, including the surface hydrophilicity or hydrophobicity. A great quantity of research work needs to be done to disclose the mechanism and build the model for accurate prediction of the release characteristic of film-coated urea.

5 Conclusion

The characteristics of the randomness, intermittence and multiple layers were included in a multilayer structure model of the spray-coating film. In the model, each layer structure comprised a dense structure and a defect structure. The permeability coefficients of the film were calculated from the structure model and diffusion rates of water vapor and urea solution in the two-phase structure. This was used to predict the release characteristics of film-coated urea. The comparison between calculated and measured results showed that this model of film-structure-to-permeability-coefficients-to-release-characteristics provided an effective way to predict the release characteristics of film-coated urea.

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The authors have declared o conflict of interest.

Symbols used

A	$[\mathrm{m^{2}}]$	the sectional area of the channel
C_{sat}	$[\mathrm{kg\,m^{-3}}]$	the saturated concentration of the urea
ΔC	$[\mathrm{kg\,m^{-3}}]$	difference of urea concentrations between both sides of the film
d	[μm]	average pore size
D_{eh}	$[\mathrm{m^{2}s^{-1}}]$	effective diffusion coefficient of water vapor
D_{es}	$[\mathrm{m^{2}s^{-1}}]$	effective diffusion coefficient of urea solution
D_{h}	$[\mathrm{m^{2}s^{-1}}]$	molecular diffusion coefficient of water vapor
D_{s}	$[\mathrm{m^{2}s^{-1}}]$	molecular diffusion coefficient of urea
K_{rs}	[–]	restriction factor determined by the diameter of the pore and molecular urea
L	[m]	thickness of the film
m	[–]	number of pores in one layer in the film structure model
m_{c}	[kg]	mass of core particles
m_{h}	[kg]	mass of water vapor that had diffused from time 0-t
m_{s}	$[\mathrm{kg\,s^{-1}}]$	mass of urea that had diffused from time 0-t
n	[–]	layer number
N_{h}	$[\mathrm{kg\,s^{-1}}]$	diffusion flux of water vapor
N_{s}	$[\mathrm{kg\,s^{-1}}]$	diffusion flux of the urea solution
N	[–]	number of cubes in the film structure model
P	[–]	probability of empty cubes in the film structure model
P_{h}	$[\mathrm{m^{2}s^{-1}Pa^{-1}}]$	permeability coefficient of water vapor
P_{s}	$[\mathrm{m^{2}s^{-1}}]$	permeability of urea
ΔP	[Pa]	difference of water vapor partial pressure between both sides of the film
Q	[–]	probability of dense cubes in the film structure model
R	$[\mathrm{J\,mol^{-1}K^{-1}}]$	molar gas constant
T	[K]	temperature
V_{s}	$[\mathrm{m^{3}}]$	volume of water

Greek letters

ε	[–]	pore void volume fraction
ρ_{s}	$[\mathrm{kg\,m^{-3}}]$	the urea particle density
ρ_{g}	$[\mathrm{kg\,m^{-3}}]$	density of water vapor
τ	[–]	tortuosity of the pore

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Research Article: A structure model of spray coating film was built, and it can be used to calculate the permeability coefficients of films using its structure parameters. The relationship between film structure, permeability coefficients of the film, and release characteristics of the coated urea were modeled for efficiently predicting the release characteristics.

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