



Research article

A biodegradable biomass-based polymeric composite for slow release and water retention

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ABSTRACT

Slow-release fertilizer has been proven to be more effective than traditional fertilizer for providing a long-term stable nutrient supply. Although such fertilizers have been widely investigated, their water-retention properties and biodegradability have not been fully analysed. Composites of fertilizers and polymers provide opportunities to prepare new types of fertilizer with enhanced properties for real applications. Chicken feather protein-graft-poly(potassium acrylate)-polyvinyl alcohol semi-interpenetrating networks forming a super absorbent resin combined with nitrogen (N) and phosphorus (P) (CFP-g-PKA/PVA/NP semi-IPNs SAR) was prepared. The chemically bonded or physically embedded fertilizer compound could be released from the resin matrix to the surrounding soil under irrigation. The synthesis mechanism, morphology, and chemical and mechanical structure of the synthesized composites were investigated. The reactant doses were optimized through response surface methodology (RSM). A 30-day field trial of the prepared SAR was applied to detect the influence of sample particle size, soil salinity, pH, and moisture content on the slow-release behaviour of N and P. The maximum release values of N and P from the composites were 69.46% N and 65.23% P. A 120-day soil burying experiment and 30-day *Aspergillus niger* (*A. niger*) inoculation were performed, and the biodegradability and change in microstructure were monitored. The addition of SAR to soil could also improve the water-retention ability of the soil.

1. Introduction

With the recent population boom, the conflict between human population and the limited land for crop farming has become a widespread concern (Han et al., 2018). To improve nutrient supply, fertilizer, especially fertilizer containing nitrogen and phosphorus plays irreplaceable roles in crop growth, improving crop yield in a limited area (An et al., 2017). Studies have shown that traditional instantly soluble fertilizer, which has been employed for a long time, can be criticized for the loss of redundant nutrients into the soil, resulting in water contamination, soil deterioration, and potential hazards to organisms. Specifically, most of the unmodified fertilizer granules were dissolved during irrigation, and the nutrients could not be fully absorbed by the crops. For example, some unused fertilizer compounds were trapped in the soil away from the rhizosphere and then converted to nitrites, causing non-point-source pollution and further harming creatures along the food chain (Chen et al., 2018a,b). The entry of unused fertilizer into water bodies through surface or subsurface runoff

could lead to eutrophication, endangering local wildlife and threatening the safety of the drinking water. (Hegedús et al., 2017; Irfan et al., 2018).

Crop yields could be enhanced without compromising the environment through rational water–fertilizer coupling. To avoid the drawbacks of traditional fertilizer and achieve sustainable and efficient application, slow-release fertilizer (SRF) was developed (Yamamoto et al., 2016). SRF could consistently release nutrients to the soil (da Cruz et al., 2017); however, the application of SRF has mainly focused on sufficient nutrient supply, while the other factor, soil water content, has been ignored (Kim et al., 2017) because irrigation could relieve water shortages to some extent. However, arid landscapes require extensive irrigation systems, which has always been a challenging issue. Thus, rational water–fertilizer coupling would be a desirable approach to enhance crop yields without compromising the environment and with decreased irrigation frequency. Super absorbent resin (SAR) has emerged as an excellent fertilizer carrier with various advantages, such as high water absorption, retention ability and fertility improvements

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(Chen et al., 2018b). The high cost of polymer synthesis and potential hazards after application in the soil promoted the introduction of biomass into the preparation process to address the problem (Chen et al., 2018a); the types of biomass have included wheat straw, corncob, and leftover rice (Huang et al., 2018; Wen et al., 2017; Zhou et al., 2018). Chicken feathers, a common byproduct of animal husbandry, have been widely used as feed additives in livestock breeding to provide amino acids (Esparza et al., 2018; Ramakrishnan et al., 2018). The numerous functional groups (-COOH, -NH₂, -SH) in chicken feather protein (CFP) could definitely encourage interaction with monomers and fertilizer components (Boyandin et al., 2017). The protein macromolecule could serve as a backbone in SAR, providing the platform for the polymerization of monomers, enhancing the strength of the whole structure and promoting the fixation of fertilizer in the 3D structure (Poole and Church, 2015). Additionally, the use of biomass as the main skeleton in SRF (Letnik et al., 2017; Xu et al., 2017) would favour the degradation process in soil. There have been few records of the application of chicken feather in fertilizer synthesis, and most existing reports have focused on polymer-coated fertilizer, while the biodegradability of the products was neglected (Xiao et al., 2017).

Although graft polymers showed high performance in terms of water absorbency and retention, their mechanical strength in the soil needed to be enhanced (Kline et al., 2017). Thus, semi-IPN technology was applied to build a more stable structure to resist the outer collapsing force via physically crosslinking a linear polymer with a cross-linked 3D polymer (Mandal and Chakrabarty, 2015; Wen et al., 2017). PVA was chosen to construct this semi-IPN structure.

In this study, we have proposed a facile management system for a common husbandry waste—chicken feathers, as shown in Scheme S1. The extracted CFP was utilized as a backbone macromolecule in the synthesis of CFP-graft-poly potassium acrylate-polyvinyl alcohol semi-interpenetrating networks forming a super absorbent resin combined with nitrogen (N) and phosphorous (P) (CFP-PKA/PVA/NP semi-IPNs SAR) to serve as a slow-release N and P fertilizer. Acrylic acid (AA) was used as a monomer with N,N'-methylene-bis-acrylamide (MBA) as a crosslinker. Through soil tests, the products were proven to exhibit high performance as a fertilizer with good slow-release and water-retaining properties and biodegradability in soil.

2. Materials and methods

2.1. Materials

Urea, K₂HPO₄, K₂S₂O₈, (NH₄)₂Ce(NO₃)₆, Na₂SO₃, acrylic acid (AA), KOH, N,N'-methylene-bis-acrylamide (MBA), polyvinyl alcohol (PVA), polyvinyl chloride (PVC), tetrahydrofuran (C₄H₈O) and the components of Czapek medium were all of analytical grade and purchased from Sinopharm Chemical Reagent Co., Ltd., Beijing, China. Czapek medium was prepared according to the accurate dosages listed in Table S1 after high-temperature sterilization. Fresh chicken feathers were obtained from the local market in Jinan (Shandong, China). The *A. niger* applied in the biodegradation trial, identified as CGMCC-3.5487, was purchased from the China General Microbiological Culture Collection Centre (CGMCC).

2.2. Preparation of CFP-PKA/PVA/NP semi-IPNs SAR

The chicken feathers were washed with distilled water, dried and then ground to a 60-mesh powder. Next, each 1 gm of chicken feather powder was treated with 14 mL of 0.45 wt% KOH and then filtrated to obtain CFP.

Approximately 1 gm of CFP was mixed with initiators (K₂S₂O₈, (NH₄)₂Ce(NO₃)₆, and Na₂SO₃). Next, neutralized AA was added to the mixture, followed by a moderate amount of PVA and crosslinking agent. Then, urea and K₂HPO₄ were added to the reaction system to contribute N and P. The entire reaction was conducted in a water bath (50 °C) and

stirred for another 4 h. Finally, the CFP-PKA/PVA/NP semi-IPNs SAR was dried and ground. CFP-PKA/PVA semi-IPNs SAR for the subsequent mechanistic experiments was synthesized according to the above methods without the addition of N and P.

2.3. Optimization of CFP-PKA/PVA/NP semi-IPNs SAR

Before the RSM study, a series of single-factor preliminary experiments (as described in the Supplementary Materials) were carried out to determine the range of each variable, including the weight ratios of the reactants: X_1 = AA:CFP, X_2 = PVA:CFP, X_4 = MBA:CFP, and X_3 = neutralization degree (ND) of AA. According to these variables, a three-level, four-factor BBD model was applied to confirm the optimized combination of synthesis conditions, as shown in Table S2. The variables were coded by the following equation:

$$X_i = \frac{x_i - x_0}{\Delta x}, \quad i = 1, 2, 3, 4 \quad (1)$$

where X_i represents the coded value of a variable; x_i represents the actual value of a variable; and x_0 represents the difference between adjacent variables.

Based on the experimental data, a quadratic polynomial was applied to describe the relationship between the variables and the swelling capacity of SAR to achieve the optimum preparation conditions. The quadratic model was given as follows:

$$Y = \beta_0 + \sum_{i=1}^4 \beta_i X_i + \sum_{j=1}^4 \beta_{ii} X_i^2 + \sum_{i=1}^4 \sum_{j=i+1}^4 \beta_{ij} X_i X_j \quad (2)$$

where Y is the response function; β_0 is a constant; β_i , β_{ii} and β_{ij} are the linear, quadratic and interactive coefficients, respectively; and $X_{i(j)}$ represents the coded levels of independent variables. The terms $X_i X_j$ and X_i^2 represent the interaction and quadratic terms, respectively.

Design Expert software was used to design the experimental runs, optimize the preparation conditions according to the experimental data and deduce the quadratic Eq. (2) by the BBD model. Analysis of variance (ANOVA) was applied to provide some fundamental parameters such as the coefficient of determination (R^2), the p-value, and the lack of fit to determine the adequacy of the model and the significance of the regression coefficients.

2.4. Characterizations of CFP-PKA/PVA/NP semi-IPNs SAR

FTIR (Thermo Nicolet, NEXUS 2470) was employed in the range of 400–4000 cm⁻¹ by application of the sample to a KBr pellet. The surface morphology was analysed by SEM (FEI, Quanta FEG 250). TGA was conducted by an SDT Q600 V8.3 Build 101 analyser under N₂, and the temperature was increased from 10 to 600 °C at a rate of 10 °C/min. XPS was recorded on an ESCALAB 250 spectrometer (Thermo Fisher SCIENTIFIC, USA) in the range 0–1050 eV.

2.5. Slow-release behaviours of CFP-PKA/PVA/NP semi-IPNs SAR

The release behaviour was detected by soil tests. For each sample, approximately 0.3 gm was enclosed in a non-woven bag (5 cm × 4 cm) and buried in the middle of 250 gm of dry soil from cultivated land at room temperature (25 °C). Three replicates were performed. A certain amount of tap solution was added to maintain the humidity, salinity and pH of the soil. After 1, 3, 5, 7, 10, 15, 20, 25, and 30 days, the samples were removed, washed, and dried, and the contents of N and P in the samples were determined by the Kjeldahl method of distillation and by ultraviolet spectrophotometry, respectively. Based on the single-factor experiment criteria, the influences of each of the following factors on the slow-release behaviour of SAR samples were analysed and discussed: soil pH (5, 7, 9), salinity (0, 50, 100, 500 mM NaCl), moisture degree (MD = 30%, 50%, 70%) and particle size (< 10,

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