Preparation and Characterization of a Novel Imidacloprid Microcapsule via Coating of Polydopamine and Polyurea

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Optimization of preparation conditions of microcapsules

Imidacloprid microcapsule samples with polydopamine (PDA) coating or both PDA and polyurea (PU) coatings were prepared under different conditions. The best preparation conditions were obtained when an optimal sustained release of imidacloprid from the microcapsule was achieved. For imidacloprid microcapsule coated with PDA (IMPDA), the effects of dopamine concentrations (2 mg/mL, 3 mg/mL, 4 mg/mL and 10 mg/mL), number of deposition cycles (1, 2 and 3) and temperature (0 °C and 25 °C) on the release of imidacloprid were studied (Table S1). Release profiles of imidacloprid from these microcapsule samples are shown in Fig. S1. It was found that an optimal (slower) release rate of imidacloprid was obtained for

PDA4 sample (dopamine concentration of 10 mg/mL), PDA6 sample (3 deposition cycles) and PDA7 sample (temperature of 0 °C). Although the release profiles of the three samples were similar to each other, the preparation conditions of PDA7 sample were superior over the other two samples (PDA4 and PDA6) as less dopamine and deposition cycle were applied to the preparation process. Therefore, the optimal preparation conditions for IMPDA were as follow: dopamine concentration of 2 mg/mL, deposition cycle of 1 and temperature of 0 °C.

Table S1. Preparation conditions of PDA coated imidacloprid microcapsule samples.

Sample	**C_{DA} (mg/ml)**	**deposition cycles**	**T(℃)**
PDA1	2	1	25
PDA2	3	1	25
PDA3	4	1	25
PDA4	10	1	25
PDA5	2	2	25
PDA6	2	3	25
PDA7	2	1	0

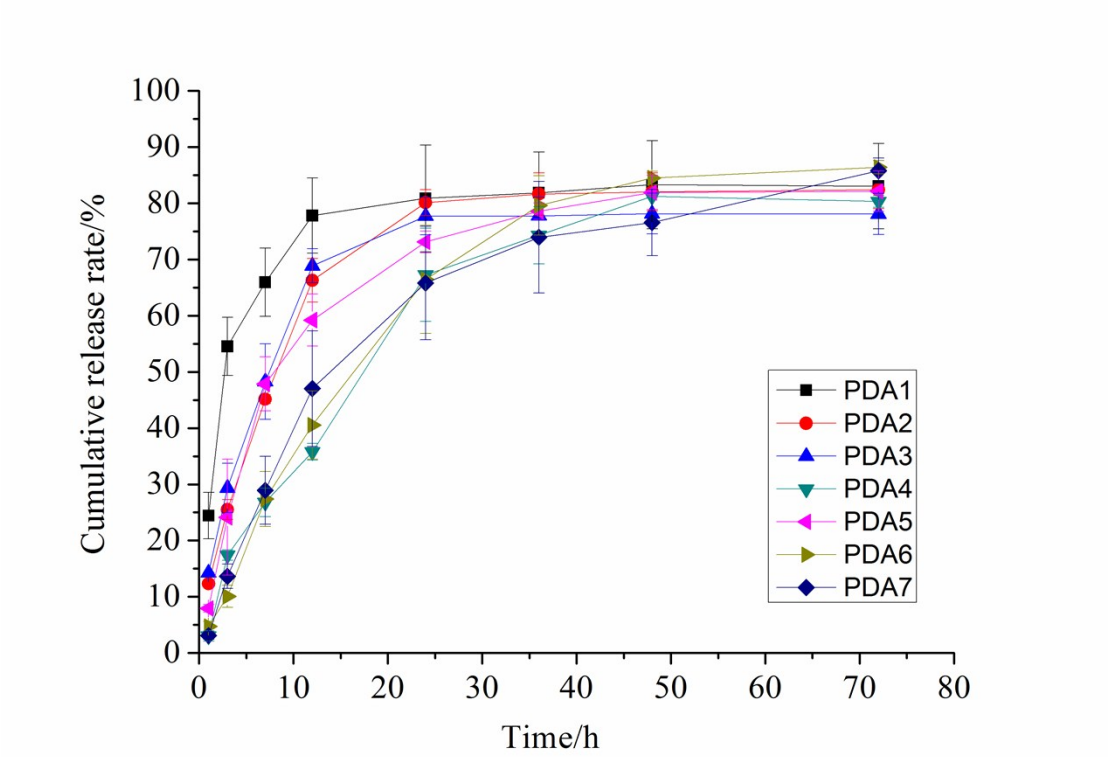

Fig. S1. Release profiles of imidacloprid from IMPDA samples prepared at different conditions.

The PU layer was coated on IMPDA to form IMPU. The effects of isophorone diisocyanate (IPDI) concentrations, cross linker spices [diethylenetriamine (DETA) and triethanolamine (TEA)] and plasticizer addition [dibutyl sebacate (DBS)] on the release of imidacloprid were studied (Table S2), and the release profiles of IMPU samples are shown in Fig. S2. It seemed that PU2 sample had the lowest release rate over the other samples, so the conditions of PU2 sample (DETA of 110 μL + IPDI of 220 μL) were optimal for the preparation of IMPU.

Table S2. Preparation conditions of PU/PDA coated imidacloprid microcapsule samples.

Sample	Condition
PU1	DETA (110 μL)+IPDI(110μL)
PU2	DETA (110 μL)+IPDI(220μL)
PU3	DETA (110 μL)+IPDI(330μL)
PU4	DETA (110 μL)+IPDI(220μL)+DBS(20μL)
PU5	TEA (110 μL)+IPDI(220μL)

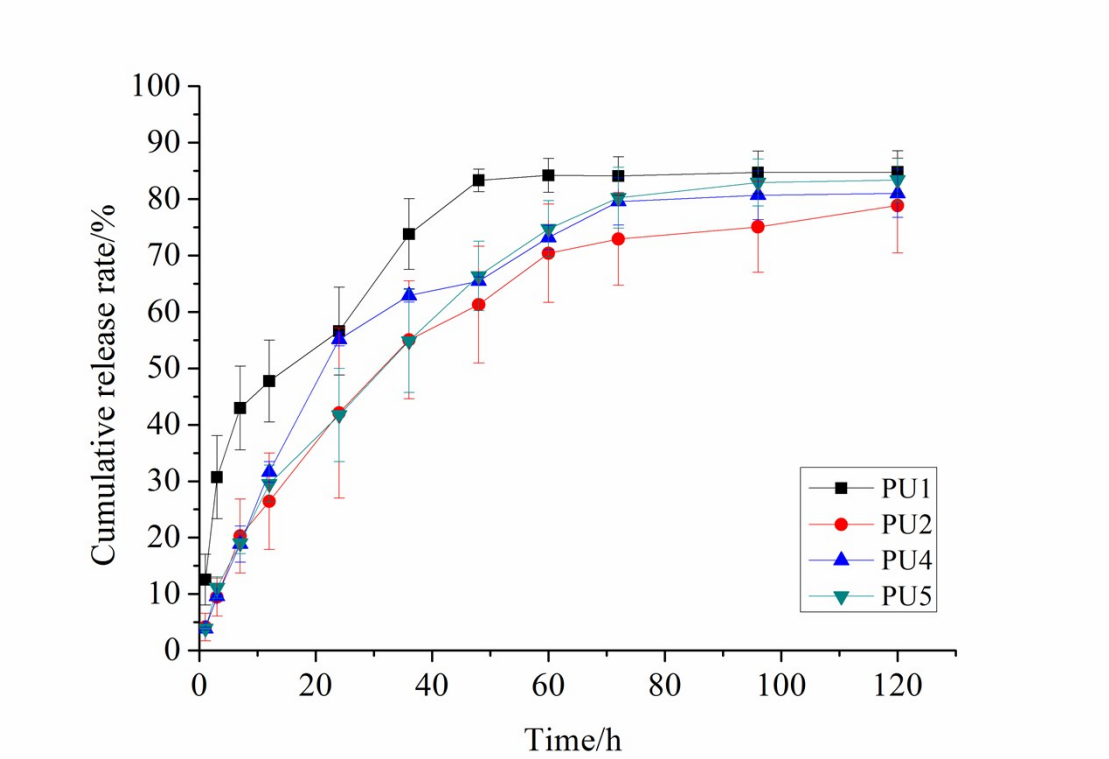

Fig. S2. Release profiles of imidacloprid from IMPU samples prepared at different conditions. The release profile of PU3 sample was not included due to the aggregation of microcapsules during polymerization.

Entrapment Rate and Pesticide Loading

Entrapment Rate. The microcapsule (IMPDA or IMPU) suspension of 1 mL was

added into a conical flask with 100 ml deionized water. The flask was settled in an ultrasonic (100 W) environment at 25 °C for 30 min to dissolve non-encapsulated imidacloprid. Then the extract and the microcapsules were separated with a mutche filter. The extract of 5 mL was introduced to a 10-mL centrifuge tube containing 2 mL dichloromethane. The non-encapsulated imidacloprid was extracted into dichloromethane with stirring at 150 rpm and 25 °C for 30 min. Water was removed using liquid separating and dichloromethane was evaporated at 50 °C for 30min. The residual was redissolved in ethanol and detected by using a WTF UV-2102PC ultraviolet-visible spectrophotometer (UNICO Shanghai Instrument Co., Ltd., China) setting at 270 nm. The residual concentration was converted to the weight of non-encapsulated imidacloprid. Theoretical weight of imidacloprid was measured by weighing a certain amount of IMSC after dried.

Pesticide Loading. After washed three times with distilled water and dried at 50 °C overnight, the microcapsules (~2 mg) were introduced to a 10-mL PP tube containing 2 mL dichloromethane. The encapsulated imidacloprid was extracted into dichloromethane exposed in an ultrasonic (100 W) environment at 25 °C for 24 h. Then the extract was passed through a 0.20-μm syringe nylon filter and dichloromethane was evaporated at 50 °C for 30min. The residual was redissolved in ethanol and measured by a WTF UV-2102PC ultraviolet−visible spectrophotometer (UNICO Shanghai Instrument Co., Ltd., China) setting at 270 nm. The residual concentration was converted to the weight of encapsulated imidacloprid.