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Study on the preparation, characterization, and release behavior of carbosulfan/polyurethane microcapsules

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ABSTRACT: Polyurethane, a controlled release material, has been widely applied in many fields due to its high thermal and mechanical stability, corrosion resistance, and low cost. In this article, we prepared carbosulfan/polyurethane microcapsules by an interfacial polymerization method using modified isocyanate as the precursor and triethanolamine as a curing agent. The microcapsules were characterized by scanning electron microscopy, Fourier transform infrared spectroscopy, and thermogravimetric analysis, and their release kinetics, chemical stability, and the safety of wheat seeds coating were also detected. The microcapsules had an excellent encapsulation efficiency and loading ability for carbosulfan ~96.23% and 50%, respectively. Furthermore, the microcapsules improved the chemical stability of the carbosulfan and exhibited an excellent sustained release property (above 30 days), which controlled the carbosulfan and carbofuran at an appropriate level for reducing the adverse effects on the environment and agricultural products. The coated wheat seeds germination rate test showed that compared with the emulsifiable concentrate, the microcapsules almost had no effect on the germination rate, plant height, and root length. © 2016 The Authors Journal of Applied Polymer Science Published by Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 43844.

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INTRODUCTION

Although chemical pesticides can be effective for pest control, their indiscriminate use has caused many environmental problems, including the threat to human and animal health and safety, pollution of the atmosphere and groundwater, development of pesticide resistance in the target organisms, reduction in the number of natural enemies, and problems with pesticide residues in agricultural products. Therefore, a reasonable and effective method should be adopted to reduce the pressure on the environment. For example, controlled release techniques can improve the utilization of pesticides, as well as reduce the frequency of agrochemical application.

Microcapsules, one of the controlled release techniques, have been used in many fields, such as food,[1] medicine,[2,3] pesticides,[4–6] environmental engineering,[7] biological Engineering,[8,9] cosmetics,[10] and coatings,[11,12] among others. A controlled release formulation, microcapsules are small particles with sizes between 1 and 100 μm that contain an active agent encapsulated by a natural membrane or a polymeric membrane synthesized by physical methods, chemical methods, or a combination of these methods. The encapsulation is used to protect active agents from oxidation (caused by heat, light, humidity, and exposure to other substances over their lifetime), shield an irritating smell, prevent the evaporation of volatile compounds, and reduce the toxicity of certain active substances.

Microencapsulation is commonly performed by *in situ* polymerization,[13] emulsion polymerization,[14] interfacial polycondensation,[15,16] solvent evaporation,[17,18] colloidal templating,[19] or coacervation/phase separation,[20] with the common shell materials being polyurea,[15,21] polyamide,[22] polyurethane,[12,23] and polystyrene.[24,25] The selection of the preparation method and shell material determines the quality of the final product, including the physical and chemical stability, particle size distribution, release rate and mechanism, and processing cost.

In recent years, polyurethane materials have been widely used in various fields. Polyurethane microcapsules, a polyurethane product, are receiving increasing attention for their high thermal and mechanical stabilities, corrosion resistance, biocompatibility, and low cost. Due to these properties, polyurethane microcapsules have been widely applied in medicine,[26,27] cosmetics,[28] materials,[16,29] and pesticides.[23] Isocyanates, including toluene diisocyanate (TDI), methylene diphenyl isocyanate (MDI), and

their modified derivatives, are common precursors for preparing polyurethane microcapsules. Interfacial polymerization is a convenient technique for the rapid production of polyurethane microcapsules under mild conditions of pressure and temperature. The polyurethane microcapsules are fabricated by forming drops of oil containing an isocyanate in an aqueous phase containing a polybasic alcohol. The isocyanate and the alcohol contact at the oil–water interfaces and quickly react, forming solid polyurethane shells around the oil drops.

Carbosulfan, a carbamate insecticide, is a low toxicity derivative of the highly toxic pesticide carbofuran, which targets a wide range of insects including sucking pests and soil pests. Currently, carbosulfan are generally used for seed coating, the main formulations are emulsifiable concentrate, granules, and dust powder. When the coated seeds were sown into the soil, the active ingredients are readily degraded to the highly toxic pesticide carbofuran by the effect of water and microorganisms (DT_{50}, 21 days, in soil; 1.6 days, in water phase only),[30] which has been held responsible for sporadic kills of fish, wildlife, beneficial insects, and terrestrial and aquatic invertebrates.[31] High concentrations of carbofuran affected the germination rate of the seed and the quality and safety of agricultural products.[32,33]

Currently, the main method of securing the stability of carbosulfan is isolating it from water and light; therefore, the main formulations are emulsifiable concentrate, granules, and dust powder. But when it is used in farmland, it will decompose and generate carbofuran rapidly, which is highly toxic and is potential threat to the environment and safety of agricultural products. After loading carbosulfan into the microcapsules, it is isolated from water and light either in the formulation or application, the stability of carbosulfan is improved and sustained releasing of it was achieved and the carbosulfan and carbofuran were controlled at an appropriate level for reducing the adverse effects on the environment and agricultural products. Currently, only Cao and coworkers reported urea-formaldehyde/carbosulfan microcapsules which were prepared by *in situ* polymerization method.[34] For the urea-formaldehyde *in situ* polymerization method, it is difficult to control the reaction conditions (temperature and pH) and achieve industrialization. Meanwhile, there will be a small amount of formaldehyde residual in the products. For interfacial polymerization, the reaction conditions are mild and easy to control, which is conductive for large-scale production.

In this study, we used modified diphenylmethane diisocyanate as a precursor and triethanolamine as a curing agent to prepare polyurethane shelled carbosulfan microcapsules by using an interfacial polymerization method. The microcapsules showed a high loading content and sustained release behavior. The preparation conditions of the carbosulfan microcapsules, the effect of the pH on sustained release performance and the establishment of a model of the release kinetics, the safety on wheat seeds coating, and environment of the microcapsules were studied herein.

EXPERIMENTAL

Materials

The modified isocyanate (the content of NCO is 29%) was purchased from the Wanhua Chemical Group Co., Ltd. (Beijing, China). Carbosulfan was provided by the Zhejiang Tianyi Agrochemical Co., Ltd. (Shangyu, China) at a purity of 92%. Triethanolamine and stannous octoate were analytical-grade chemicals purchased from the Sinopharm Chemical Regent Co., Ltd. (Beijing, China). The emulsifier (NP-10P) was provided by the Cangzhou Hongyuan Agrochemical Co., Ltd. (Cangzhou, China), and the dispersant (D-425) was provided by Akzo Nobel N.V. (The Kingdom of Netherlands). The acetonitrile and methanol used for HPLC were purchased from Thermo Fisher Scientific Inc. (Massachusetts, USA).

The Preparation of Carbosulfan/Polyurethane Microcapsules

Carbosulfan-loaded polyurethane microcapsules were prepared by interfacial polymerization. Briefly, carbosulfan (16.5 g), NP-10P, modified isocyanate, and stannous octoate (0.1 g) were placed in a beaker and stirred evenly as the oil phase (Table I). The water phase was prepared by dissolving D-425 (2 g) in 100 mL of deionized water. The oil phase was poured into the water phase under high shear (10,000 rpm) uniformly dispersing the oil. Then, the emulsion was transferred to a three-necked flask and stirred at 300 rpm with mechanical agitation. The aqueous solution of triethanolamine (8 g, 25% wt/wt) was slowly added to the emulsion for ~5 min. After the addition, the mixture was stirred at room temperature for 4 h and then heated at 60 °C for 4 h. The shell had completely cured and afforded the carbosulfan-loaded polyurethane microcapsules.

Characterization

Fourier Transform Infrared Spectroscopy. The sample was tested on a Fourier transform infrared (FTIR) spectrometer (Bruker Vector 22, Germany) over potassium bromide pellets, and the wavelength was set from 4000 to 450 cm^{-1}.

Morphology and Structure Analysis. The morphology of the prepared microcapsules was characterized by a scanning electron microscope (SEM, Hitachi S4800, Japan). The sample were prepared by dropping the microcapsule suspension on a quartz wafer, air-dried overnight, then coated with a thin layer of gold by vacuum deposition with a sputter coater (Baltec SCD 050).

The structure of the microcapsules containing 7-hydroxycoumarin was characterized by a confocal laser scanning microscopy (CLSM, OLYMPUS FV1000-IX81; Olympus Corporation, Tokyo, Japan).

Particle Size Analysis. A Mastersizer particle size analyzer (Mastersizer 2000, Malvern Instruments Co., UK) was used to determine the size and distribution of carbosulfan microcapsules.

Thermodynamic Properties of Carbosulfan Microcapsules. The prepared microspheres were evaluated by a differential scanning calorimetry (DSC) (Shimadzu DSC-50, Germany) thermal analyzer under a nitrogen atmosphere.

Measurement of the Encapsulation Efficiency. The carbosulfan microsphere suspension was dispersed in a certain amount of xylene. Then, the mixture was shaken upside down for one minute, followed by standing for 1 min for stratification. The supernatant was analyzed by high-performance liquid chromatography (HPLC, Agilent 1100, USA) with a diode array detector. The HPLC separation of carbosulfan was carried out on a Spuril-C18 column (4.6 mm × 250 mm, 5 μm, Dikma

Table I. The Effects of the Main Parameters on the Encapsulation Efficiency and Particle Size Distribution

						Size distribution (μm)		
Sample	Isocyanate (g)	NP-10P (g)	Shear rate (rpm)	Shear time (s)	EE %	D_{10}	D_{50}	D_{90}
1	4	3	10,000	30	94.76	0.53	2.79	22.15
2	6	3	10,000	30	96.23	0.56	2.08	14.09
3	8	3	10,000	30	96.79	0.56	2.60	18.14
4	6	3.5	10,000	30	95.88	0.54	2.02	17.01
5	6	4.0	10,000	30	91.09	0.40	1.90	22.37
6	6	3	10,000	60	95.87	0.52	2.19	13.40
7	6	3	15,000	30	87.21	0.31	1.52	15.65
8	6	3	5000	30	95.62	2.43	10.72	55.93

Technologies Inc., China) with an isocratic elution of methanol–water (90/10, vol/vol) as the mobile phase. Ten microliters were injected into the HPLC system and separated at 25 °C using a constant flow rate of 1.0 mL min^{-1} at a detection wavelength of 280 nm. The encapsulation efficiency (EE) was calculated according to the following eq. (1).

$$EE(\%) = \frac{\text{mass of carbosulfan in microcapsules}}{\text{initial mass of carbosulfan}} \times 100 \quad (1)$$

Determination of the Release Rate of the Microcapsules. The release rate of loaded carbosulfan from the prepared microcapsules was investigated. A certain quantity of loaded microcapsules was added to a dialysis bag (size: 5 M, MW: 8000–14,000), and then the bag was placed in 20 mL of release medium (the volume ratio of acetonitrile:water is 30:70). The pH values of the release medium were 3, 5, 7, and 9 (adjusted by HCl and NaOH). The release was investigated *in vitro* at 25 °C using a controlled environmental/orbital shaker incubator (Huamei Co., Jiangsu Province, China) at 200 rpm. At different time intervals, 1 mL of solution was sampled, and 1 mL of fresh acetonitrile–water solution was added to the reagent bottle to maintain a constant volume and unsaturated conditions. The sampled solution was analyzed by HPLC under the same conditions mentioned in section "Measurement of the encapsulation efficiency."

Controlled Release Kinetics Studies. The cumulative release of carbosulfan from the microcapsules was determined by an empirical equation according to the Korsmeyer–Peppas model,[2] where M_t is the amount of drug released in time t, M_∞ is the initial amount of drug in the microcapsule sample, k is the first order release constant, and n is the kinetic parameter.

$$M_t/M_\infty = kt^n \quad (2)$$

The Safety of the Carbosulfan Microcapsules. The safety of carbosulfan microcapsules at different pH values was investigated. Certain quantities of carbosulfan microcapsules and carbosulfan emulsion were added to a dialysis bag (size: 5 M, MW: 8000–14,000), respectively. Then, the bag was placed in a 20 mL mixed solution (the same as in section "Controlled release kinetics studies"), with solutions having pH values of 3, 5, 7, and 9 tested. The bags were stored at 25 °C, and at the same time intervals, the concentrations of carbosulfan and carbofuran were tested by HPLC (the analytical method is the same as in section "Measurement of the encapsulation efficiency"). Finally, the curve of the concentration change was drawn according to the measurement results.

Evaluation of the Biological Activity. The effect of the microcapsule suspension when used for seed treatment on the germination rate of wheat was evaluated according to GB/T 3543.4-1995 (China). The wheat seeds were treated by the prepared carbosulfan microcapsule suspension and the commercial seed coating. The treatment concentrations evaluated were 1:25, 1:50, and 1:75 (microcapsules : seed, weight ratio), which corresponded to active ingredient contents of 6.0, 3.0, and 2.0 g a.i./

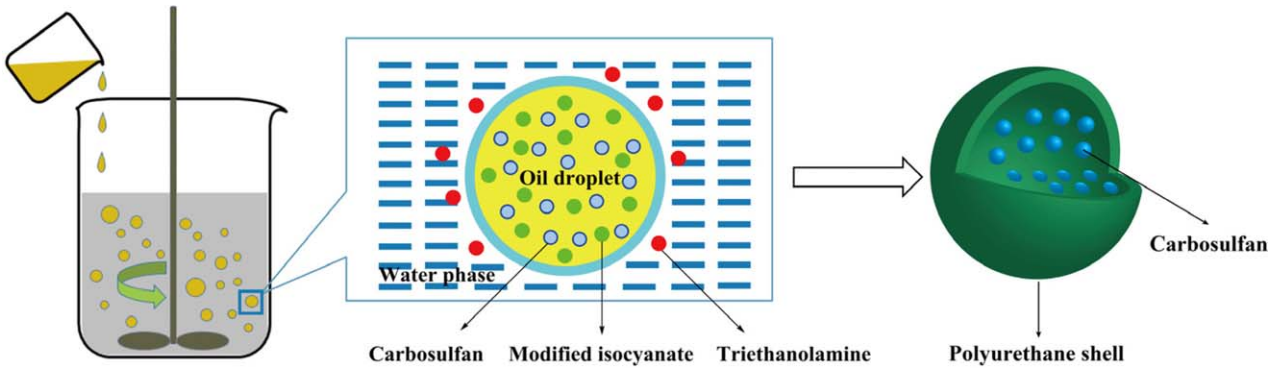

Figure 1. Schematic diagram of the possible formation mechanism of carbosulfan/polyurethane microcapsules. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

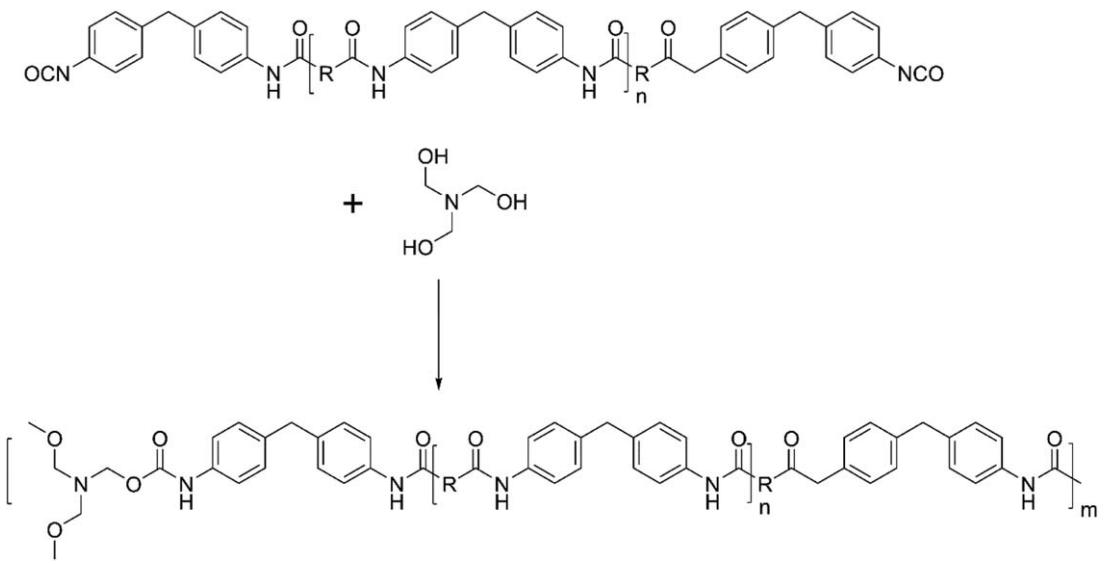

Figure 2. Reaction mechanism for the formation of carbosulfan/polyurethane microcapsules.

kg, respectively. The treated seeds were dried in the air, and then ten seeds were sown in each flower pot (diameter: 12 cm, height: 10 cm), with untreated seeds as a control. Each treatment was repeated twice. The flower pots were placed in the artificial climate incubator (average temperature: 28 °C, light: 8 h/day, 16 h/night). The germination rate, plant height, root length, shoot fresh weight, and underground fresh weight were investigated after 14 days.

RESULTS AND DISCUSSION

Preparation of the Carbosulfan Microcapsules

The preparation procedure and reaction mechanism of carbosulfan microcapsules are illustrated in Figures 1 and 2, respectively. In the first step, the oil phase containing carbosulfan and isocyanate was uniformly dispersed in an aqueous phase under high shear conditions. Then, the triethanolamine was added as a curing agent. The isocyanate and triethanolamine reacted at the oil–water interface and formed polyurethane-shelled carbosulfan microcapsules.

Three of the critical parameters in preparing microcapsules are particle size, distribution, and encapsulation efficiency. It was reported that there are many parameters that affect the size and distribution of the microcapsules, for example: emulsification speed and time, shell/core material ratio, and surfactant content, etc. In this study, the effects of carbosulfan/isocyanate ratio, surfactant content, shear rate, and time on particle size and encapsulation efficiency were investigated to optimize the process for preparing microcapsules. The D_{10}, D_{50}, and D_{90} values indicate the largest particle equivalent diameter when the particle size cumulative distribution reaches 10%, 50%, and 90%, which was used to measure the range of the particle size distribution. As shown in Table I, with an increasing amount of isocyanate, the

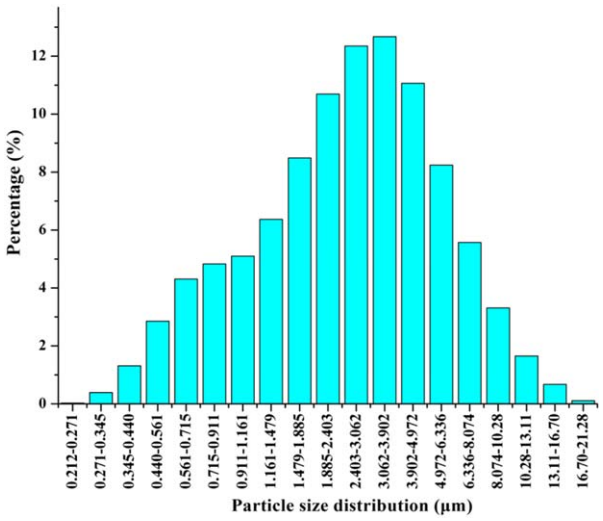

Figure 3. Size distribution of carbosulfan/polyurethane microcapsules (Sample 2). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

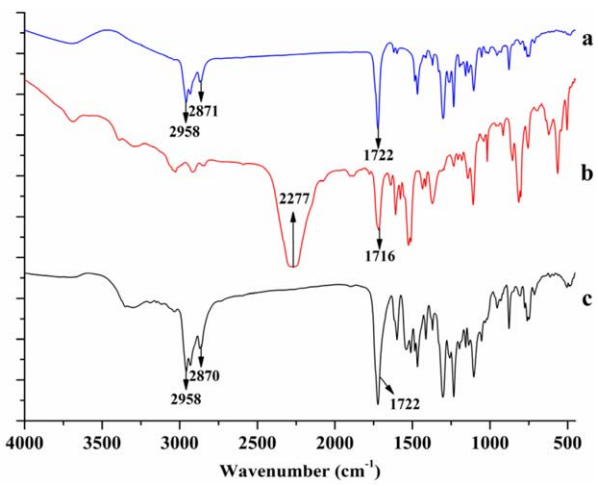

Figure 4. FTIR spectra of carbosulfan (a), the modified isocyanate (b), and carbosulfan/polyurethane microcapsules (c). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

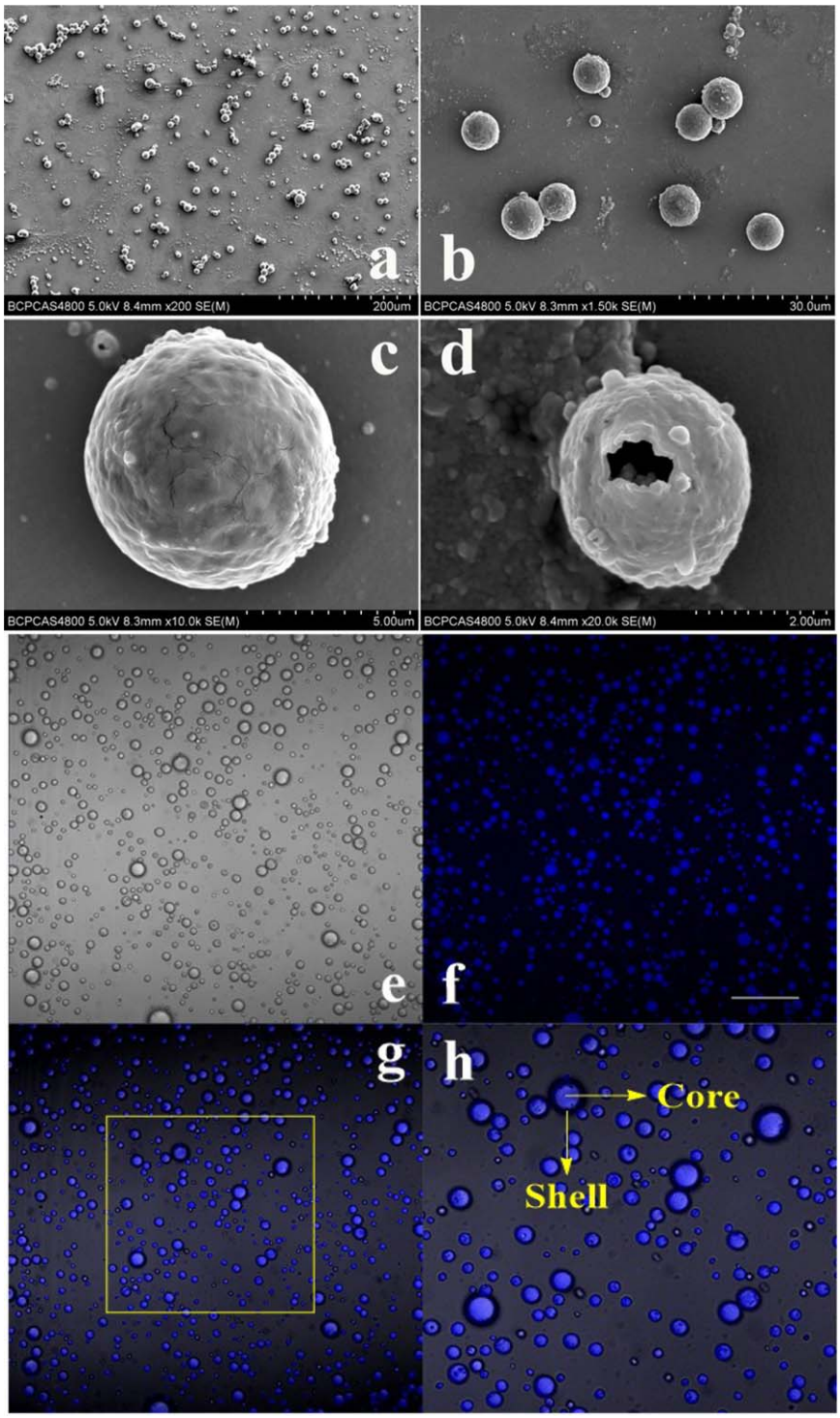

Figure 5. The SEM (a,b,c,d) and CLSM (e,f,g) images of carbosulfan/polyurethane microcapsules. (e) bright field, (f) fluorescence, (g) the overlay image of (e,f,h) partial enlarged of image (g), scale bar = 100 μm. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

encapsulation efficiency increased slightly, and the particle size distribution showed little change. Comparing samples 2, 4, and 5, with an increasing amount of NP-10P, the D_{50} value became small, the D_{90} value became large with agglomeration, and the EE value decreased due to the smaller particle size. It was shown in samples 2, 7, and 8 that when the shear rate was raised to 15,000 rpm, the particle size and the EE decreased; when the shear rate was changed to 5000 rpm, the particle size became large, and the particle size distribution became nonuniform. In the dispersion system, shear stress, surface tension, and viscosity

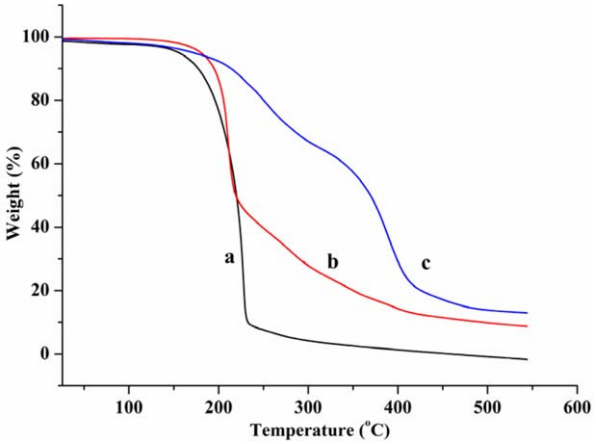

Figure 6. TGA curves of carbosulfan (a), carbosulfan/polyurethane microcapsules (b), and blank microcapsules (c). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

stress influence the dispersion of droplets. Shear stress promote dispersion of the droplets, the surface tension and viscous stress hinder dispersion of the droplets. When the shear stress is stronger than the latter two forces, the droplet was divided to smaller size of droplets continually. The shear stress is proportional to the rate of stirring; therefore, the size of the droplet became smaller with the increasing of the shear rate within a certain range. In this study, with the increasing shear rate, the oil phase emulsified and generates many smaller droplets, therefore the particle size becomes smaller after curing, the mean diameter reduced from 10.72 to 1.52 μm and the span of microspheres showed a decreased tendency. Zhang[35] studied the factors affecting the particle size, the result shows that the particle size becomes smaller with the increase of stirring speed, which is consistent with the result in this study. In addition, there was little effect on the EE and particle size distribution with the use of an extended shearing time (Samples 2 and 6). In summary, we chose Sample 2 as the optimal formula. Sample 2 had an EE of 96.23%, and the particle size distribution (Figure 3) conformed to a normal distribution, with a mean particle size of 2.08 μm.

Table II. The Constants from the Fitting of the *Korsmeyer–Peppas* Model to the Release Date of Carbosulfan from Microcapsules under Different Conditions

Microcapsules	pH values	*n*	*k*	*R*	T_{50} (d)
Sample 2	3	0.5239	12.0830	0.9612	15.04
	5	0.4206	12.2595	0.9913	28.28
	7	0.3427	13.5218	0.9829	45.42
	9	0.4384	10.4636	0.9867	35.44

Characterization of Carbosulfan Microcapsules

The FTIR spectra of carbosulfan, the modified isocyanate, and carbosulfan/polyurethane microcapsules are shown in Figure 4. In Figure 4(a), the peak at 1722 cm^{-1} is attributed to the stretching vibration of a carbonyl group. The peaks at 2958 cm^{-1} and 2872 cm^{-1} are stretching vibrations of C—H attributed to methyl and methylene groups. In Figure 4(b), the peaks at 2277 cm^{-1} and 1716 cm^{-1} are attributed to the stretching vibrations of isocyanate and carbonyl groups, respectively. The characteristic absorption peak of the isocyanate group at 2277 cm^{-1} disappeared in Figure 4(c), indicating that the isocyanate reacted with triethanolamine and formed the polyurethane shell. In addition, Figure 4(c) contained the characteristic peaks of carbosulfan, which proved that the carbosulfan/polyurethane microcapsules had been successfully prepared.

The morphology of carbosulfan/polyurethane is presented in Figure 5. As shown in Figure 5(a,b), the microcapsules of carbosulfan are dispersed without aggregation, and the particle size is uniform. The morphology of the microcapsules is approximately spherical, and the surface is uneven and compact [Figure 5(c)]. In Figure 5(d), it is shown that the microcapsule was ruptured, and through the hole, we speculated that the microcapsules are core–shell structure. The CLSM images of e, f, g, and h containing 7-hydroxycoumarin in the core further testified the core–shell structure, proving that the isocyanate and triethanolamine reacted at a two-phase interface, forming a core–shell

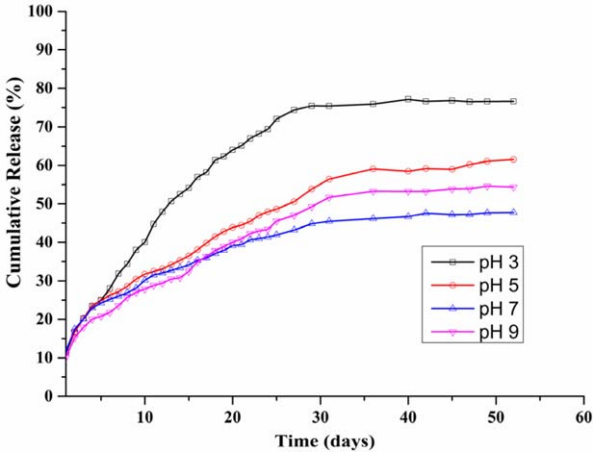

Figure 7. Effect of the pH value on the release behavior of carbosulfan microcapsules (Sample 2). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

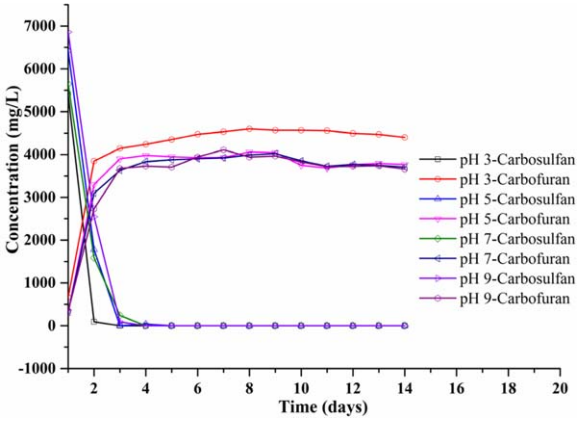

Figure 8. Content changes of carbosulfan and carbofuran in the carbosulfan emulsion under different conditions. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

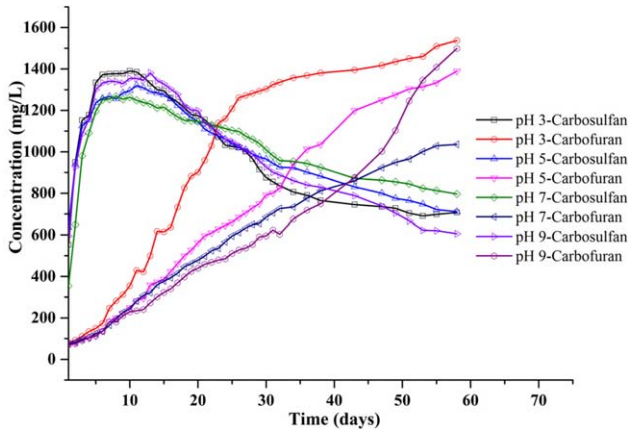

Figure 9. Content changes of carbosulfan and carbofuran in the carbosulfan microcapsule suspension under different conditions. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

structure microcapsule. The core with blue fluorescence is the carbosulfan oil, and the outside shell with circular black shadow is polyurethane.

The amount of loaded carbosulfan and the thermal stability of the prepared microcapsules were detected by thermogravimetric analysis (TGA). The TGA curves of carbosulfan, carbosulfan loaded microcapsules, and blank microcapsules are presented in Figure 6. In Figure 6(a); the weight loss between 150 °C and 225 °C may be due to the volatilization and decomposition of carbosulfan. The weight loss in the range of 210–450 °C can be attributed to the decomposition of the polyurethane shells [Figure 6(c)]. As shown in Figure 6(b), carbosulfan microcapsules lost ~50.0% of their original weights from 175 to 225 °C, corresponding to the ratio of loaded carbosulfan.

Controlled Release Kinetics

The release behavior of carbosulfan microcapsules was studied by a dialysis method under different pH values while keeping the temperature (25 °C) constant at a vibration speed of 200 rpm. Because carbosulfan easily decomposes into carbofuran, the cumulative release amount of carbosulfan is the sum of the following two parts: the detected carbosulfan and the decomposed carbosulfan. Figure 7 shows the effects of different pH values (3.0, 5.0, 7.0, and 9.0) on the carbosulfan release behavior from carbosulfan microcapsules. A sudden release of ~6% occurs at the beginning of the release experiment, which can be attributed to the uncoated carbosulfan. The release rate was highest at a pH of 3, and the cumulative release of carbosulfan reached ~75% on the 29th day, with the next highest release rate at a pH of 5 (cumulative release reached 53% on the 29th day). At pH values of 7 and 9, the cumulative release amounts of carbosulfan reached 44% and 49% on the 29th day, respectively. There are three main release mechanism: diffusion mechanism, erosion mechanisms, and a combination of them. For the polyurethane and carbosulfan, the urethane bond in polyurethane is probably degraded under acid conditions, which has been reported in the literatures.[36] Moreover, carbosulfan is easy to degradation under acid condition,[30] which has promoted the release in a certain extent. So, the release mechanism of carbosulfan/polyurethane microcapsules is affected by the change of pH. The release data were also analyzed by applying the Korsmeyer–Peppas model, $M_t/M_\infty = kt^n$, from which k, n, and T_{50} (time to 50% carbosulfan release from the microcapsules) were calculated in Table II. There was good correlation between the release profiles of carbosulfan from the microcapsules, with the correlation coefficients (r) exceeding 0.9612. The release mechanism depended on the value of n: when $n < 0.45$, diffusion mechanism; $0.45 < n < 0.89$, combined diffusion and erosion mechanism; and $n > 0.89$, erosion mechanism. In Table II, the n value is 0.5239 at a pH of 3, which is between 0.45 and 0.89, so the microcapsules may be released following a combined diffusion and erosion mechanism. The n values are less than 0.45 at pH values of 5, 7, and 9, so the microcapsules may be released following a diffusion mechanism.

The Safety of the Carbosulfan Microcapsules

Carbosulfan is unstable and easily decomposes to highly toxic carbofuran. To determine the safety of carbosulfan microcapsules at different pH values, the carbosulfan emulsion and carbosulfan microcapsule suspension were dispersed in acetonitrile and water solutions (pH values of 3, 5, 7, and 9), respectively. At the same time intervals, the concentrations of carbosulfan and carbofuran in the solutions of each sample were measured by HPLC, and the concentration change curves of each sample are shown in Figures 8 and 9. It is shown in Figure 8 that the carbosulfan was almost completely decomposed to carbofuran on the third day and

Table III. The Evaluation Results on the Biological Activity of Wheat

Treatment concentration	Treatment agent	Germination rate (%)	Height (cm)	Root length (cm)	Shoot fresh weight (g)	Underground fresh weight (g)
1:25	Carbosulfan microcapsules	95	18.7	21.7	4.8	1.0
	Carbosulfan emulsion	40	7.2	13.5	1.6	0.5
	Water	100	19.0	23.6	5.4	1.6
1:50	Carbosulfan microcapsules	97.5	19.3	22.5	5.1	1.5
	Carbosulfan emulsion	50	13.6	16.8	2.9	0.8
	Water	100	20.6	24.3	5.5	1.8
1:75	Carbosulfan microcapsules	100	20.4	23.2	5.2	1.7
	Carbosulfan emulsion	80	16.3	18.5	4.1	0.9
	Water	100	21.2	24.7	5.5	1.7

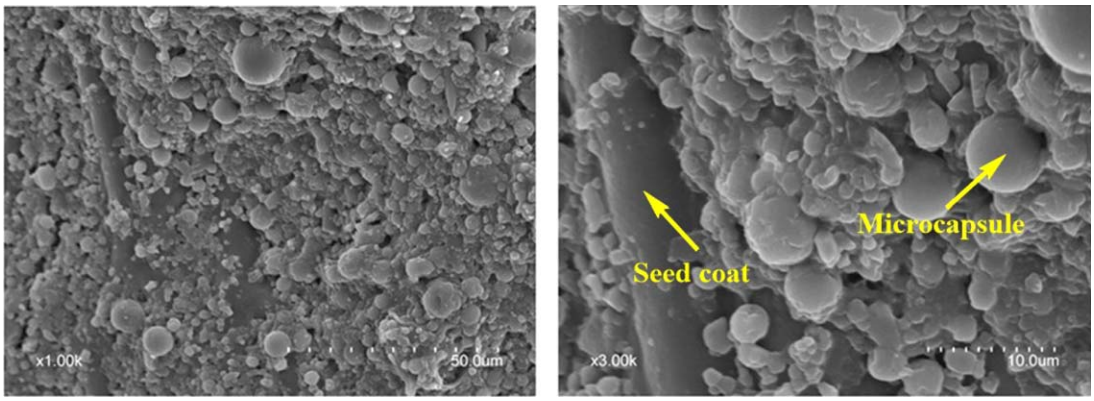

Figure 10. SEM images of the wheat seed coated by carbosulfan microcapsules. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

that the concentration of carbofuran reached up to 4000 mg/L. The system maintained a very high concentration of carbofuran, which will cause environmental pollution and may be leave a residue in agricultural products and impact their quality. It is shown in Figure 9 that the concentration of carbosulfan in solution was above 600 mg/L and that the concentration of carbofuran was below 1500 mg/L on the 58th day. Compared with the carbosulfan emulsion, microencapsulation greatly improves the stability of carbosulfan and allows sustained release for a long period of time, which can also control the carbofuran at an appropriate concentration and reduce the residual risk in agricultural products and environment.

Biological Evaluation

The biological activity of the prepared microcapsules was evaluated by the germination rate, plant height, root length, shoot fresh weight, and underground fresh weight of wheat, and the results are shown in Table III, Figures 10 and 11. The SEM images of the coated wheat seed in Figure 10 show that the microcapsules were well adhered to the surface of the seed without breaking, which proved the good mechanical properties of the microcapsules. For the germination rate, the high concentration and low concentration of the carbosulfan emulsion showed obvious inhibitory effects, this is consistent with Barratt BIP's conclusion.[32] The high concentration of the carbosulfan microcapsules showed a relatively weak effect and the low concentration almost had no effect. In Figure 11, the carbosulfan emulsion showed significant growth inhibition on wheat plants; however, carbosulfan microcapsules had almost no effect on the growth of wheat. The experimental results showed that the carbosulfan microcapsules could sustain release and control

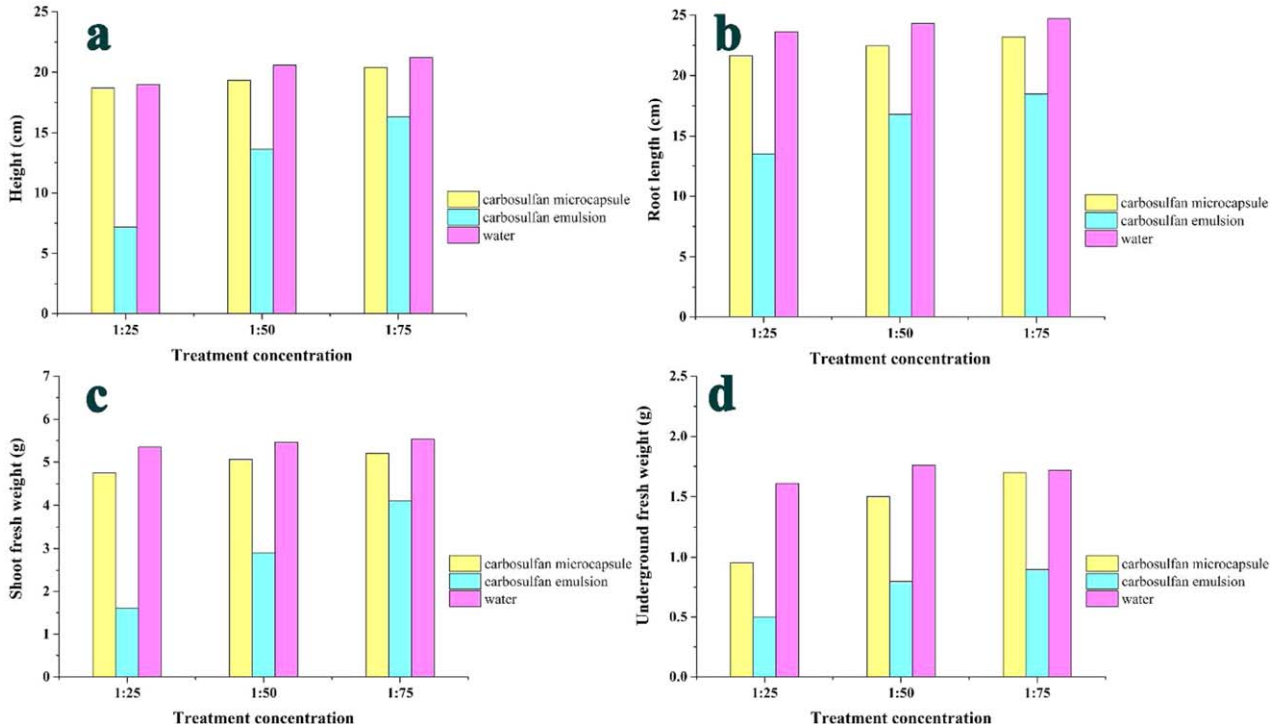

Figure 11. Effects of the treatment agent on the growth of wheat plants, (a) height, (b) root length, (c) shoot fresh weight, (d) underground fresh weight. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

carbofuran and carbosulfan at a relatively low concentration, which exhibited excellent safety for wheat seed coating, non-target organisms, and human health.

CONCLUSIONS

In summary, the carbosulfan/polyurethane microcapsules were successfully prepared by interfacial polymerization using modified isocyanate as the precursor and triethanolamine as a curing agent. The resulting microcapsules, which were characterized by SEM, FTIR, and TGA, had high drug loading, excellent mechanical stability, and sustained release performance. Furthermore, the chemical stability of carbosulfan was improved by microencapsulation, which could control carbosulfan at an appropriate concentration and reduce the risk of wheat seed coating, non-target organisms, and human health.

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