Controlled Release of Biocides in Solid Wood. III. Preparation and Characterization of Surfactant-Free Nanoparticles

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ABSTRACT: Polymeric nanoparticles containing the fungicides tebuconazole and chlorothalonil were prepared by a simple, surfactant-free method and found to have significantly smaller median particle diameters and more stable aqueous suspensions than their surfactant-stabilized counterparts. These more stable suspensions were delivered into southern yellow pine and birch wood with greater efficiency than the equivalent surfactant-stabilized nanoparticle suspensions. We found that the suspensions protected the treated wood against fungal attack by *Gloeophyllum trabeum*, a common brown rot wood decay fungus, and *Trametes versicolor*, a common white rot wood decay fungus, at low tebuconazole and chlorothalonil contents in the wood. Southern pine lost 5% or less of its mass after 55 days of exposure to *G. trabeum* when the tebuconazole or chlorothalonil content in the wood was only 0.4 kg/m^3, while a tebuconazole or chlorothalonil content of 0.8 kg/m^3 in birch wood was sufficient to bring its mass loss to less than 5% after 55 days of exposure to *T. versicolor*. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 86: 615–621, 2002

Key words: surfactant-free nanoparticles; wood preservatives; fungicides; tebuconazole; chlorothalonil

INTRODUCTION

We recently reported on several studies evaluating the preparation of biocide-containing nanoparticles and their efficacy against *Gloeophyllum trabeum* and *Trametes versicolor* wood decay fungi once they are introduced into southern yellow pine (SYP) and birch wood, respectively.[1–3] A general method was developed that was successful with each formulation attempted with minor adjustments based on specific formulations, and nanoparticles were prepared with several different biocides and polymer matrices. We found that biocides introduced into wood using the nanoparticle method had unexpectedly high activity. Wood treated with biocide-containing nanoparticles often showed only "background" mass loss after exposure to *G. trabeum* and *T. versicolor* wood decay fungi using a "wafer" test method,[1] even when the biocide content in the wood was only a fraction of the amount the wood industry usually employs. The reason for the biocide efficacy at low loading levels was unknown. The original advantages postulated for using biocide-containing nanoparticles to preserve wood was that the nanoparticles served as biocide reservoirs and controlled release devices, and so it was thought that the longevity of wood preserved with organic biocides would be increased. It was also postulated and demonstrated that using biocide-containing nanoparticles suspended in water would permit even low solubility biocides such as chlorothalonil to be introduced into wood using aqueous methods, rather than the current and undesirable methods in which such biocides are introduced into wood as solutions in toxic organic solutions, which limits the use of wood treated in this way to applications where it does not come into contact with humans.

However, despite the already demonstrated advantages for the overall approach, the anticipated increases in wood longevity, and the fact that the general method was versatile and able to produce nanoparticles in good yield from all the different formulations attempted, some drawbacks were clear. First, the more hydrophobic nanoparticle formulations consistently afforded larger nanoparticles (typically median diameters of ~ 200 vs. ~ 100 nm for the more hydrophilic formulations); second, even the more hydrophilic formulations were insufficiently stabilized as evidenced from the fact that the nanoparticles at higher suspension loadings were not delivered quantitatively into the wood during the wood pressure treatment, and the undelivered nanoparticles underwent aggregation. The aggregation was especially pronounced with the less stable hydrophobic formu-

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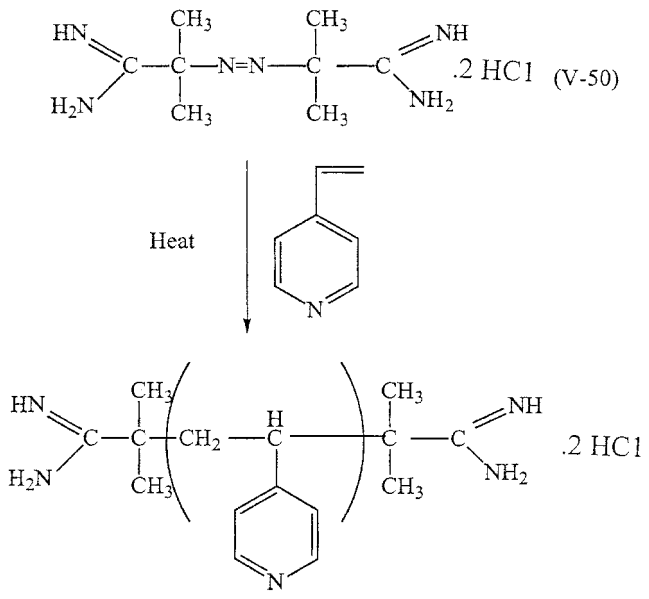

Figure 1 A representation of the surfactant-free V-50/PVP nanoparticle matrix.

lations. Although the median diameter of even the hydrophobic formulations was sufficiently small to pass into the wood pit pores, aggregation was due to the fact that all the formulations possessed sufficient numbers of overly large particles that blocked the pit pores, preventing quantitative particle delivery and inadequate suspension stability. Therefore, although the versatility of the nanoparticle preparative method was advantageous, it was clear that the suspension stability needed to be improved and ideally the particle size should be further reduced.

This article reports the results of an effort to further reduce the size of the nanoparticles, improve the stability of the nanoparticle suspension, and further simplify the nanoparticle preparation method that is the general preparative method. These goals were accomplished by modifying the preparative method to remove the surfactant from the formulation. The surfactant serves to stabilize the nanoparticles by steric stabilization of the surface or ionic repulsion. Ionic stabilization of particles in emulsion polymerization is well known and can be accomplished by added ionic surfactants or by self-stabilization through the incorporation of ionic groups on the particles themselves.[4] In this work it was decided to attempt to eliminate the surfactant component and use self-stabilization methods by preparing the nanoparticle matrix using a free-radical initiator whose initiator fragments might serve to ionically stabilize the nanoparticles (Fig. 1). If this stabilization alone was found to be insufficient, then additional polar or potentially ionic moieties, such as acrylic acid (AA) or methacrylic acid, could be incorporated via copolymerization methods.

EXPERIMENTAL

Instrumentation and materials

Particle sizing was done with a Shimadzu CP-4 particle sizer (centrifugation). Tebuconazole was supplied by Miles, Inc. (Pittsburgh, PA). Chlorothalonil was supplied by ISK Biosciences (Memphis, TN). The water-soluble initiator (V-50) was donated by Wako (Richmond, VA). 4-Vinylpyridine (VP) and all other chemicals were purchased from Aldrich Chemical Company. Wood specimens were SYP (*Pinus* spp.) and birch (*Betula papyrifera*). The fungal tests were conducted with *G. trabeum* (ATCC 11539, a common basidomycete brown rot wood decay fungus) and *T. versicolor* (ATCC 42462, a white rot basidomycete).

Preparation of matrix polymers for self-stabilized nanoparticles

The free-radical initiator V-50 was chosen to provide stabilizing end groups for the matrix polymers. AA was chosen as the polar comonomer when the end groups alone were insufficient to stabilize the nanoparticles.

V-50/PVP

The VP (5.85 g, 40 mmol) and methanol (100 mL) were charged into a 250-mL round bottom flask. The solution was heated to reflux under a nitrogen purge. The V-50 (1.2 mmol) was dissolved in methanol (20 mL) and added to the reaction solution over 10 min. The reaction was allowed to stir at reflux overnight and then allowed to cool to room temperature. Unreacted monomer and solvent were removed under reduced pressure (M_n 43,000 g/mol, 88%).

V-50/PVP-*co*-AA

The procedure was identical to that for V-50/PVP except AA (0.1 g, 0.8 mmol, 2 mol %) was added with the VP to the methanol reaction solution.

Preparation of biocide-containing self-stabilized nanoparticles

Tebuconazole (10 mg) was dissolved in methanol (2 mL) and the solution was mixed with V-50/PVP (50 mg in a 20% solution of methanol). The solution was added to a dropping funnel and dripped slowly into water that had been preheated to 60°C and was being stirred at 450 rpm. Following the addition the suspension was stirred for an additional 0.5 h and then was centrifuged (20,000 rpm for 20 min) and collected by decanting the liquid. The nanoparticles were then freeze-dried to collect the nanoparticles in 92% yield [active ingredient (AI) content ~ 45%, 90% theoreti-

TABLE I
Relative Size and Stability of Surfactant-Stabilized and Surfactant-Free Nanoparticles

	Median particle size (nm)		Size distribution (nm)	
Formulation AI/surfactant	Initial	6 months	As prepared	After 6 months
Tebuconazole/yes	112	ppt	50–350	ppt
Tebuconazole/no	79	188	50–1000	100–2000
Chlorothalonil/yes	169	ppt	50–1000	ppt
Chlorothalonil/no	123	327	50–2000	100–2000

cal]. This procedure is essentially the same as the surfactant procedure except it was carried out using PVP with polar end groups and without the surfactant.

Chlorothalonil-containing nanoparticles were prepared in the same way except using V-50/PVP-*co*-AA. Chlorothalonil is more hydrophobic than tebuconazole and better results were obtained with V-50/PVP-*co*-AA than with V-50/PVP alone. Nanoparticles were collected in 86% yield (AI content ~ 47%, 94% theoretical).

Aqueous AI release rate studies

The method is described elsewhere[1,5] but is based on gravimetric analysis.

Biological studies

We described the method in detail previously.[1,5] All the studies using *G. trabeum* were done using SYP, and all the studies with *T. versicolor* were done using birch wood. The wood block specimens were cubes with the sides cut to 19 mm. The exact mass and dimensions of each block was known. The blocks were then immersed in a nanoparticle suspension with a known mass of nanoparticles to allow the mass of nanoparticles delivered into the wood to be known and therefore and the mass of AI delivered into the wood in those nanoparticles to be calculated. The wood specimens were subjected to a pressure treatment cycle. The undelivered nanoparticles were recovered and characterized for size.

The wood cubes were dried overnight (40°C), cut into four nearly equal wafer sections (two interior and two exterior), and each section was reweighed. The wafers were sterilized in an autoclave for 15 min at 120°C. Forceps were used to place sterile toothpicks on agar plates inoculated with *T. versicolor* or *G. trabeum*, and the labeled wafer sections were then placed directly on the toothpicks. Untreated control wafers were placed in each agar dish so the fungal activity within the dish could be verified. The petri dishes were sealed with parafilm and placed in a walk-in incubator maintained at 80°F and 80% relative humidity. Following 55 days of exposure, the wafers were harvested, cleaned, dried, and reweighed to determine the mass loss. The reported weight losses are the average of at least three cubes.

RESULTS AND DISCUSSION

When nanoparticles were prepared using surfactant stabilization it was evident that more hydrophobic nanoparticles tended to be a little less stable and tended to afford a product with a larger median particle diameter than more hydrophilic nanoparticles. In this early work we selected PVP as the matrix rather than a more hydrophobic polymer and used tebuconazole and chlorothalonil biocides as the AIs. Chlorothalonil is a more hydrophobic AI than tebuconazole and consistently afforded larger and less stable nanoparticles than tebuconazole. Therefore, the relative effects of modifications to the nanoparticle preparation method are assessed by comparing the effects on the chlorothalonil-containing nanoparticles with the effects on tebuconazole-containing nanoparticles and by comparing the results with the surfactant-stabilized nanoparticles.

Particle size and suspension stability

The first study compared the size, size distribution, and suspension stability of the surfactant and surfactant-free nanoparticles over time (Table I). When the surfactant-stabilized nanoparticles were prepared, the nanoparticles had a median diameter of 112 nm when the AI was tebuconazole and 169 nm when the AI was chlorothalonil. The chlorothalonil suspension precipitated within ~ 1 week and the tebuconazole precipitated within ~ 1 month. When the systems were prepared as self-stabilized nanoparticles, the median particle size decreased to 79 and 123 nm, respectively, and both suspensions were stable over the 6-month period over which they were observed. Therefore, the data confirm that this method does reduce the median diameter and gives more stable suspensions, but it was interesting that both self-stabilized formulations yielded a more broad size distribution than did the surfactant-stabilized formulations. This is confirmed when the histograms of the systems, shown in Figure

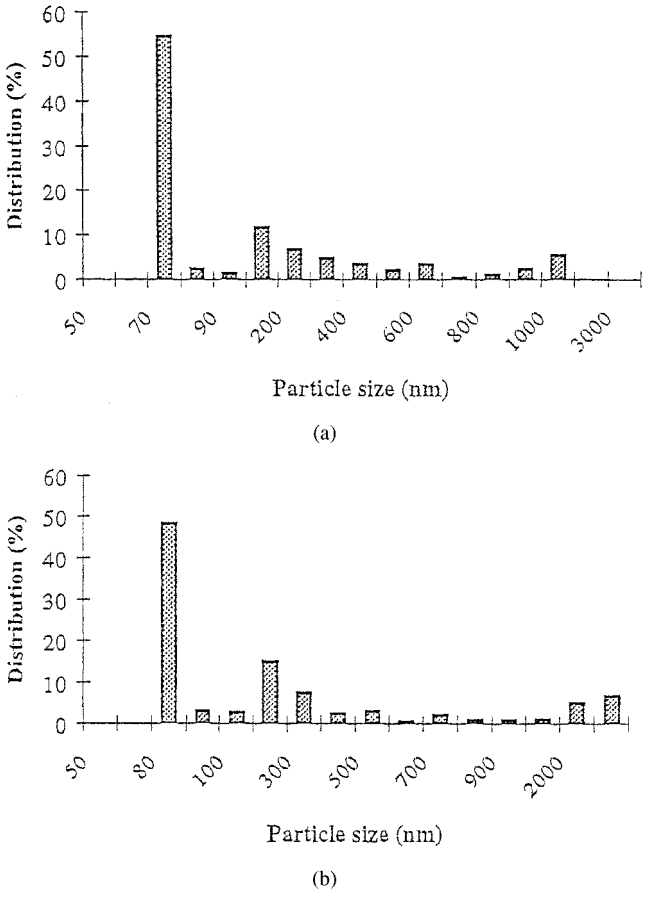

Figure 2 Histograms of surfactant-free nanoparticles containing (a) tebuconazole and (b) chlorothalonil.

2, are observed. The reasons for this are not understood.

Aqueous release rates

The AI release rates of the nanoparticles in water showed that the surfactant-free nanoparticles performed slightly differently from the surfactant-stabilized nanoparticles. The surfactant-free tebuconazole containing PVP nanoparticles [PVP(SF)] released tebuconazole at the same rate as the surfactant-stabilized nanoparticles [PVP(S)] for the first few days, but then the AI was released from the PVP(SF) nanoparticles more slowly than from the PVP(S) nanoparticles [Fig. 3(a)]. The first few days of AI release were probably a diffusion release augmented slightly by a release burst, and then it was only a diffusion controlled release. It was expected that the tebuconazole would release more rapidly from the PVP(SF) than from the PVP(S) because the end groups were expected to increase the hydrophilicity of the nanoparticles. The reason the AI released more slowly after the burst may be the interactions between the ionic end groups and the tebuconazole, which is heterocyclic and possesses a ring nitrogen.

The AI release rates were somewhat different for chlorothalonil [Fig. 3(b)]. Both sets of nanoparticles show an initial burst and then chlorothalonil releases more rapidly from the surfactant-free PVP(S) nanoparticles than the surfactant-stabilized nanoparticles. The burst from chlorothalonil in the surfactant-stabilized particles is exaggerated; in fact, the release rate then falls and a slow diffusion-controlled release is seen. The surfactant-free nanoparticles show a diffusion-controlled release that, following the burst observed from the surfactant-stabilized nanoparticles, shows these nanoparticles release chlorothalonil faster than the surfactant-stabilized nanoparticles. This faster release is more consistent with expectations because the surfactant-free nanoparticle matrix not only has the ionic end groups but also AA moieties in the copolymer and so is more hydrophilic than PVP. The reason for this exaggerated AI burst release from the

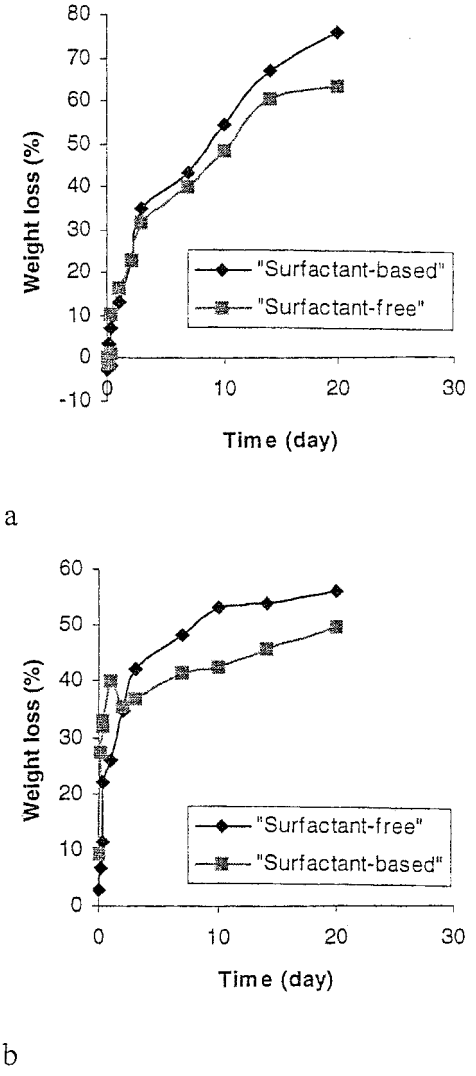

Figure 3 The release of (a) tebuconazole in PVP(S) and PVP(SF) nanoparticles and (b) chlorothalonil in PVP(S) and PVP-AA(SF).

TABLE II
Efficacy of Tebuconazole in Surfactant-Free Nanoparticles Against *Gloeophyllum trabeum*

AI loading (kg/m^3)	AI delivered (kg/m^3)	System	Weight loss (%)		
			Exterior section	Interior section	Average
Control	—	—	—	—	17 ± 3
0.40	0.40	S	2 ± 1	6 ± 1	4 ± 1
0.40	0.39	SF	3 ± 1	5 ± 1	4 ± 1
0.80	0.7	S	2 ± 1	2 ± 1	2 ± 1
0.80	0.79	SF	2 ± 1	3 ± 1	2 ± 1

chlorothalonil is that DSC shows that ~ 22–25% of the chlorothalonil in PVP is present in a crystalline form, and it is thought this may be on the surface of the nanoparticles and quickly desorbs from the surface.[4] None of the chlorothalonil in V-50/PVP-*co*-AA is present in a crystalline state[4] and thus the release is diffusion controlled.

Studies of SYP treated with surfactant-free nanoparticles and exposed to *G. trabeum*

Removal of the surfactant from the formulation altered the AI release rates in water, so it was also possible this would affect the biological efficacy of the nanoparticles. Also, although the median particle size of the surfactant-free nanoparticles was smaller than that of the surfactant-stabilized nanoparticles, the size distribution was broader, so it was not clear if the nanoparticles would be delivered as efficiently into the SYP as the surfactant-stabilized nanoparticles. Therefore, surfactant-free nanoparticles were incorporated into SYP by water-pressure treatments using nanoparticle suspensions with different suspension loadings. Undelivered nanoparticles were recovered and characterized, and the ability of the surfactant-free nanoparticles to protect wood against the *G. trabeum* brown rot wood decay fungus was determined and compared with the original surfactant-containing formulation. The results are shown in Table II for tebuconazole-containing nanoparticles and in Table III for chlorothalonil-containing nanoparticles. To facilitate comparison the tables include data for surfactant-stabilized nanoparticles, identified with an S, and the surfactant-free formulations, identified as SF.

Each agar dish contained a wafer section of SYP that was not treated in any manner to ensure the fungus in that auger dish was active. An averaged value for these wafer sections is reported as a "control." The mass-loss data for the wood wafers treated with tebuconazole-containing nanoparticles shows no difference in the performance of the surfactant-free nanoparticles when compared to the surfactant-containing nanoparticles. However, the data show that the surfactant-free nanoparticles were delivered into the SYP with greater efficiency at the higher loading level of 0.8 kg AI/m^3. This suggests that greater longevity might be expected for wood treated with this system.

The results for the chlorothalonil-treated wood showed greater differences between the surfactant-stabilized and surfactant-free systems. The surfactant-free system afforded some protection to the treated SYP at AI loading levels of only 0.1 kg AI/m^3 while the surfactant stabilized nanoparticles afforded no significant protection to the SYP, even though the surfactant-stabilized and surfactant-free nanoparticle systems were both delivered quantitatively into the treated wood. One logical explanation for the difference might be that the larger surfactant-stabilized nanoparticles might have blocked the pit pores in such a way that nanoparticles were distributed nonuniformly, leaving the interior sections unprotected, while the smaller surfactant-free nanoparticles were distributed more uniformly and protected the wood

TABLE III
Efficacy of Chlorothalonil in Surfactant-Free Nanoparticles Against *Gloeophyllum trabeum*

AI loading (kg/m^3)	AI delivered (kg/m^3)	System	Weight loss (%)		
			Exterior section	Interior section	Average
Control	—	—	—	—	19 ± 3
0.10	0.1	S	15 ± 1	16 ± 1	16 ± 1
0.10	0.1	SF	9 ± 1	10 ± 1	10 ± 1
0.40	0.3	S	13 ± 3	13 ± 4	13 ± 4
0.40	0.39	SF	5 ± 1	6 ± 2	5 ± 2
0.80	0.78	SF	5.1	4.2	4.6

TABLE IV
Efficacy of Tebuconazole in Surfactant-Free Nanoparticles Against *Trametes versicolor*

AI loading (kg/m^3)	AI delivered (kg/m^3)	System	Weight loss (%)		
			Exterior section	Interior section	Average
Control	—	—	—	—	43 ± 11
0.1	0.1	S	—	—	18.6 ± 0.1
0.10	0.11	SF	12	13	13 ± 1
0.4	0.4	S	—	—	13.0 ± 0.4
0.40	0.39	SF	8	7	8 ± 1
0.8	0.6	S	—	—	7.0 ± 0.6
0.80	0.77	SF	4 ± 2	4 ± 2	4 ± 2
1.2	1.1	SF	3 ± 1	4 ± 1	4 ± 1

more uniformly. However, the data show no significant difference for either system in the protection afforded to the interior and exterior wood wafers. An alternative possibility is that the surfactant-free nanoparticle formulation released the AI faster than the surfactant-stabilized nanoparticle formulation. It is possible that the difference in the wood preservative effect was attributable to the chlorothalonil reaching an effective level sooner within the wood with the surfactant-free formulation than with the surfactant-stabilized formulation. Therefore, the wood treated with the surfactant-stabilized nanoparticles underwent some degradation until the chlorothalonil reached effective levels and those levels were reached more rapidly with the faster releasing surfactant-free formulation.

Studies of birch treated with surfactant-free nanoparticles and exposed to *T. versicolor*

Trametes versicolor is a white rot wood decay fungi. In the wafer test method that we used, untreated birch samples lost about 43% of their mass over 55 days. When the birch was treated with tebuconazole-containing PVP nanoparticles using a surfactant-stabilized suspension sufficient to deliver 0.8 kg of AI/m^3 of wood, only ~ 0.6 kg AI/m^3 was actually introduced into the birch. However, when a surfactant-free suspension that was able to deliver 0.8 kg of AI/m^3 of wood was used, it was delivered into the birch almost quantitatively. The surfactant-stabilized and surfactant-free nanoparticle suspensions able to deliver 0.1 and 0.4 kg AI/m^3 were also delivered quantitatively into the birch. Both the surfactant-stabilized and surfactant-free tebuconazole-containing nanoparticles showed some efficacy, even at only 0.1 kg of AI/m^3 of birch, but in both cases the surfactant-free tebuconazole-containing nanoparticle formulations appeared to afford slightly greater protection to the birch. At a suspension loading capable of delivering 0.8 kg of AI/m^3 of wood, the surfactant-free formulation was again delivered almost quantitatively while the surfactant-stabilized system delivered only 0.6 kg/m^3. At this AI loading level it again appeared that the birch treated with the surfactant-free tebuconazole-containing nanoparticles, which lost ~ 4% of its mass, may have been slightly better protected against decay than the birch treated with surfactant-stabilized nanoparticles, which lost 7% of its mass. The surfactant-free nanoparticles were used to prepare a suspension able to deliver 1.2 kg of AI/m^3 of wood, and 1.1 kg of AI/m^3 of wood was delivered into the birch. Birch treated at this level also lost 4% of its mass after 55 days of exposure to *T. versicolor*. Although there was no significant difference in mass loss between the birch treated at this level and birch treated with lesser amounts of the AI, it is thought that longer tests would show a difference in longevity. The results are given in Table IV.

This study was repeated with surfactant-stabilized and surfactant-free chlorothalonil-containing nanoparticles. Again, at suspension loadings capable of delivering 0.1 kg of AI/m^3 of wood, the surfactant-stabilized and surfactant-free nanoparticles were both delivered quantitatively into the birch, and some biological efficacy was seen at this low AI loading level. It appeared that the surfactant-free nanoparticle formulation afforded slightly more protection to the birch than the surfactant-stabilized system. At higher suspension levels the surfactant-free nanoparticle formulation was consistently delivered into the birch more efficiently than the surfactant-stabilized system, but neither formulation was delivered quantitatively at these higher suspension levels. Also, the surfactant-free formulation consistently appeared to afford somewhat greater protection to the birch. For example, when the treating suspension was sufficient to deliver 0.8 kg/m^3, the surfactant-stabilized formulation delivered 0.6 kg of AI/m^3 of birch and after 55 days of exposure the sample lost 12.4% of its mass; however, the surfactant-free formulation delivered 0.7 kg of AI/m^3 of birch and that sample lost only 3.6% of its mass. The reason for the apparently greater efficacy is not clear, but it might again be due to a faster release, allowing the wood to reach a critical AI threshold level more rapidly. Also, it is noteworthy that the surfactant-free nanoparticle suspension could be prepared at

TABLE V
Efficacy of Chlorothalonil in Surfactant-Free Nanoparticles Against *Trametes versicolor*

AI loading (kg/m^3)	AI delivered (kg/m^3)	System	Weight loss (%)		
			Exterior section	Interior section	Average
Control	—	—	—	—	43 ± 11
0.10	0.10	S	—	—	20.1 ± 0.1
0.10	0.10	SF	12 ± 2	15 ± 2	14 ± 2
0.4	0.3	S	—	—	14.6 ± 0.3
0.40	0.35	SF	8 ± 2	9 ± 3	9 ± 3
0.8	0.6	S	—	—	12.4 ± 0.6
0.80	0.7	SF	3.3	3.8	3.6
1.2	0.9	SF	2.6	5.2	3.9

a level sufficient to deliver 1.2 kg of AI/m^3 of birch, but the surfactant-stabilized nanoparticle formulation was too unstable to be able to yield a suspension with this loading level. The mass loss after 55 days of exposure to *T. versicolor* was the same as at 0.8 kg/m^3, but it is expected that this wood with a higher AI content would have greater longevity. The results are given in Table V.

CONCLUSIONS

Biocide-containing nanoparticles were prepared as aqueous suspensions that were stabilized by a conventional surfactant method and a surfactant-free method. The biocides were the fungicides tebuconazole and chlorothalonil. The size and size dispersity of the fungicide-containing nanoparticles prepared with and without surfactant were compared, as was their stability in water over a 6-month period. We found that surfactant-free formulations with both tebuconazole and chlorothalonil afforded nanoparticles with a smaller median particle diameter than the surfactant-stabilized counterparts, and both surfactant-free formulations remained as stable aqueous suspensions for the 6-month period over which they were observed while the surfactant-stabilized suspensions failed over a period of weeks. However, the surfactant-free suspensions had significantly broader particle-size distributions than their surfactant-stabilized counterparts, and yet at higher suspension concentrations the surfactant-free nanoparticles were still delivered more efficiently into the wood than the surfactant-stabilized formulations. All the formulations were used to treat SYP and birch wood, and the treated wood was then exposed to *G. trabeum* and *T. versicolor* wood decay fungi, respectively. All the treated wood was shown to resist fungal decay even when the fungicide loading in the wood was only 0.1 kg AI/m^3 wood, but the surfactant-free nanoparticles appeared to afford more resistance to decay than the surfactant-stabilized formulations. In the chlorothalonil-containing nanoparticles it was thought that this might be due to a faster AI release from the surfactant-free nanoparticles than those stabilized with surfactant, which would allow the AI to reach threshold levels more rapidly. However, this would not explain the higher protection against decay found for wood treated with tebuconazole in surfactant-free nanoparticles because the tebuconazole was released from surfactant-free nanoparticles more slowly than from surfactant-stabilized nanoparticles; thus, the data do not yet support any conclusion for these observations. Once the AI loading level for both tebuconazole and chlorothalonil reached 0.4 kg/m^3 in surfactant-free nanoparticle-treated SYP, it lost less than 5% of its mass after 55 days of exposure to *G. trabeum*; once the AI level for both tebuconazole and chlorothalonil reached 0.8 kg/m^3 in surfactant-free nanoparticle-treated birch, it lost less than 5% of its mass after 55 days of exposure to *T. versicolor* using a wafer test method. Although there is no way to correlate accelerated aging tests with field tests and the AI levels used by industry vary with the specific AI being used and how it is being introduced, it is generally considered that a mass loss of under 5% is a good result and these results were obtained at AI loading levels that are quite low according to industry standards for commercial products. The data also show that the surfactant-free formulations are prepared more easily than the surfactant-stabilized formulations. They also have several advantages, including the fact that the systems used here can be introduced into wood at higher loadings than their surfactant-stabilized counterparts, which should promote wood longevity in field use.

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References

1. Liu, Y.; Yan, L.; Heiden, P.; Laks, P. J Appl Polym Sci 2001, 79, 458.
2. Liu, Y.; Laks, P.; Heiden, P. J Appl Polym Sci, to appear.
3. Liu, Y.; Laks, P.; Heiden, P. J Appl Polym Sci, to appear.
4. Odian, G. Principles of Polymerization, 2nd ed.; Wiley Interscience: New York, 1981; p 335.
5. Liu, Y. Ph.D. Thesis, Michigan Technological University, 1999.