# A MICROENCAPSULATED FORMULATION OF LAMBDA-CYHALOTHRIN

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**Abstract** - Demand CS® (lambda-cyhalothrin) has been designed specifically for use in urban pest management. The product is based on microencapsulation technology which encloses the liquid active ingredient in a polymer shell to provide protection against hostile environmental conditions. Electron microscopy and biological assays revealed that capsules remain intact for several months on a range of surfaces and provide control of pests including mosquitoes (*Aedes aegypti*) and cockroaches (*Blattella germanica*). The polymer wall retains the lambda-cyhalothrin for extended periods on inert or chemically active surfaces, reducing losses from adsorption and chemical reaction. However, when an insect picks up a capsule, its contents rapidly diffuse into the lipophilic insect cuticle, leading to rapid knockdown and mortality. Laboratory assays demonstrated that an insect can pick up large numbers of microcapsules from a treated surface, many more than are needed to kill it. Bioassays also provided evidence that contact between social or gregarious insects, such as ants or cockroaches, allows transfer of capsules between individuals with the result of increasing the lethal effect to insects not directly exposed to the treated surface. With the active ingredient not immediately available to the insect, cockroaches and ants were able to pick up a lethal dose of insecticide before any sub-lethal effects could occur. Other effects such as a marginally slower knockdown speed compared to WP or EC formulations were observed, in addition to a reduced flushing effect. The need for formulations specifically designed for use in the urban and household market is highlighted.
**Key words** - Lambda-cyhalothrin, *Blattella germanica, Aedes aegypti, Solenopsis invicta*

## INTRODUCTION

The use of residual surface applications of insecticide remains one of the most cost-effective and versatile means of controlling insect pests in the urban and household environment. The surfaces on to which insecticides are applied vary greatly, but are typically porous which can adversely affect the biological availability of insecticides. Other surfaces may, in addition, be chemically reactive and denature the active ingredient, compromising its persistence. Furthermore, insecticides applied to exterior surfaces are subject to further hostile conditions, most notably photodegradation, which can markedly reduce the persistence of effect.

A potent and persistent insecticidal effect on a range of substrates can be obtained by protecting the active ingredient from the substrate and environmental conditions, and controlling its rate of release. The process of microencapsulation offers this opportunity and this paper discusses the biological behaviour of a microencapsulated formulation of the pyrethroid insecticide, lambda-cyhalothrin (DEMAND CS) developed specifically for use in public health and urban insect pest control situations.

### Capsule Properties

**Construction.** The efficacy and properties of the active ingredient, lambda-cyhalothrin are well documented (Jutsum *et al.*, 1984). DEMAND CS capsules consist of a polyurea wall formed by an insitu interfacial condensation process (Scher, 1983) that has been used extensively by Zeneca including for the production of novel, fast release formulations of lambda-cyhalothrin (Zeon Technology®) (Perrin *et al.*, 1998). The polyurea capsule wall encloses the active ingredient dissolved in a small amount of aromatic oil, the release rate of which can be controlled by varying particle size, wall thickness and cross-link density of the wall. Capsules have a VMD of approximately 12 mm.

**Release rate and mechanism.** The polyurea wall of a DEMAND CS capsule is strongly cross linked, with a high ratio of polymethylene-polyphenyl-isocyanate (PMPPI): toluene di-isocyanate (TDI).



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This confers a relatively low permeability to the capsule contents, (Scher, 1983; Scher *et al.,* 1998) such that the active ingredient remains within the capsules in the container, spray solution, and in dried deposits provided the substrate is inert. Electron microscopic and biological assays have revealed that capsules remain intact for several months on a range of surfaces (Figure 1), enabling control of cockroaches, flies, ants and other pests to be obtained from a single application for several months (Williams, 1997).

A number of mechanisms through which encapsulated formulations exert a toxic insecticidal effect have been proposed. In capsules where the walls are weakly cross-linked, the contents may diffuse through the capsule wall when the spray deposit dries producing a surface deposit which behaves in a similar manner to that obtained from an EC product (Perrin *et al.*, 1998). In contrast, Tsuda *et al.* (1987) and Kawada *et al.* (1995) show how the contents of larger, thin walled capsules (50 mm diameter) can be released by the trampling action of insects disrupting the capsule walls.

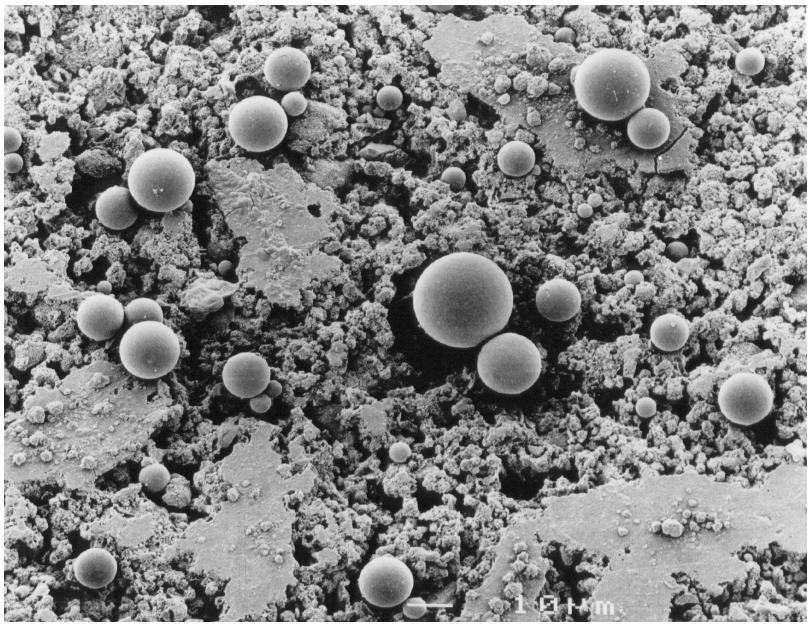

**Figure 1.** DEMAND CS cap-sules on cement 10 weeks after application. Magnification x1400.

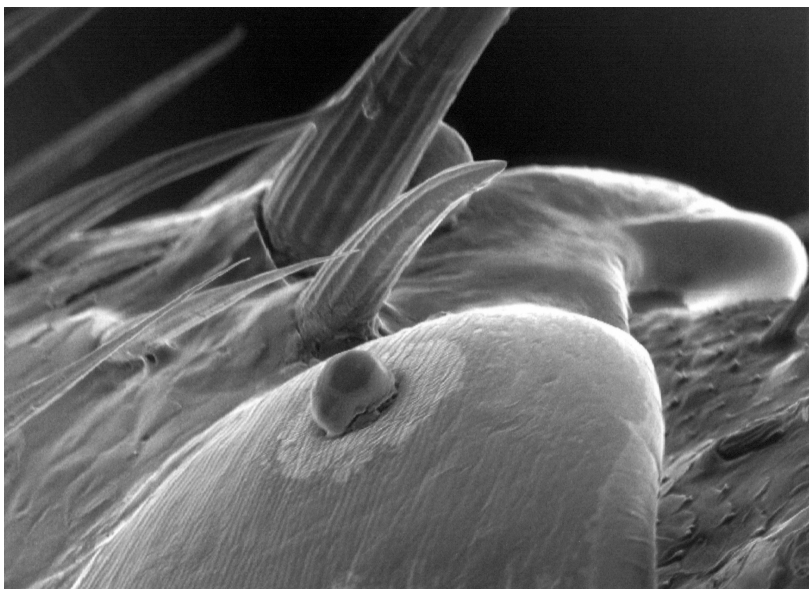

**Figure 2.** DEMAND CS cap-sule on cockroach leg. Magnification x1500.

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In the case of DEMAND CS, insects must pick-up intact microcapsules from most substrates in order to initiate diffusion of the active ingredient through the capsule wall. Indeed, the particulate nature of surface deposits of CS formulations facilitates the collection process. Electron microscopy can be used to assess qualitatively the pick-up of particulate insecticide formulations by insects, however, it is very time consuming to use this technique to provide quantitative data. Using a similar technique to that described by McClarren *et al.* (1986), the number of microcapsules picked-up by cockroaches exposed to a treated substrate was determined. Five replicates each of ten adult male *Blattella germanica* (L.) were exposed for 1 minute to an unglazed tile treated at 30 mg ai/$m^2$. The insects were removed and placed into 20 ml of 0.05% synperonic solution. After sonication for 5 minutes the insects were removed and the suspension centrifuged for 5 minutes at 2,000 rpm. The supernatant was decanted and the pellet re-suspended in 5 ml water. Samples of the suspension were placed in a haemocytometer and examined using a light microscope.

The results indicated that each cockroach picked up a mean of 700 capsules (range 550-1,050) during a 1 minute exposure to the treated surface. This is many times the lethal dose, given that Williams (1997) calculated that there is sufficient lambda-cyhalothrin contained within only 5 capsules (10 mm diameter) to kill an adult *B. germanica*. The opportunity for microencapsulated formulations to transfer apparently excessive doses of insecticide to individual insects may contribute to the effect of improved efficacy against resistant cockroach strains of microencapsulated formulations relative to EC formulations (Koehler and Patterson, 1988).

The lipophilic insect cuticle provides an ideal sink for the active ingredient and its carrier. As the diffusion process proceeds the microcapsules can become closely associated with the waxy insect cuticle. Figure 2 shows a DEMAND CS capsule adhering to the cuticle of a German cockroach (*B. germanica*) surrounded by the capsule contents which have diffused from the core. Once started, the rate of diffusion is rapid, as shown by a speed of knockdown which, although significantly different, is only slightly slower than that obtained from an EC formulation in which the lambda-cyhalothrin is immediately available for uptake by the insect (Table 1).

There are other consequences of formulations which inhibit the initial availability of the active ingredient: the preliminary symptoms of pyrethroid toxicity can result in flushing, that is the expulsion of cockroaches from their harborages. The data presented in Table 2 indicate that the irritant effects associated with pyrethroids are significantly reduced with the CS formulation relative to WP or EC formulations in which the active ingredient is more readily available. Subsequent assays demonstrated that the degree of flushing was not significantly changed by increasing the rate of application up to 40 mg ai/$m^2$ (Weeks, unpublished data).

**Repellency.** Repellent effects, which can lead to cockroaches avoiding exposure to lethal doses of toxicant have been associated with dry deposits of pyrethroid insecticides (Ross and Cochran, 1992). Despite several repeated assays, repellent effects against *B. germanica* could not be consistently demonstrated for WP formulations of lambda-cyhalothrin (Gallo, unpublished data). Repellency was also investigated using fire ants (*Solenopsis invicta* Buren). A ceramic tile beneath each of the established food sources for two laboratory colonies was replaced with a tile treated with DEMAND CS at 15 mg ai/$m^2$. Ants were observed to climb readily on to the treated tiles and start feeding normally. Only after 10-15 seconds did individuals become agitated and start grooming legs and antennae. Subsequently, knockdown occurred within 30 seconds (Gallo, unpublished data). The delayed release of the active ingredient from the capsule may thus allow insects to acquire a lethal dose before any repellent effect can occur.

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**Table 1.** Time for knockdown of 90% of adult male *Blattella germanica* sprayed directly with three formulations of lambda-cyhalothrin at a rate equivalent to 15mg ai/$m^2$ using an automated track-sprayer.

| Lambda-cyhalothrin formulation | $KT_{90}$ (minutes) |
|---|---|
| DEMAND 100 g/l CS | 17.16 b |
| Lambda-cyhalothrin 100 g ai/kg WP | 9.2 a |
| Lambda-cyhalothrin 25 g ai/l EC | 10.33 a |

5 replicates each of 10 insects. $KT_{90}$ for each replicate calculated using Zeneca's KTprog. Treatments with no letter in common are significantly different at the 5% probability level.

**Table 2.** Time for 20% of a cohort of adult male *Blattella germanica* to be flushed from a harbourage sprayed with three formulations of lambda-cyhalothrin at a rate equivalent to 15mg ai/$m^2$ using an automated track-sprayer.

| Lambda-cyhalothrin formulation | $FT_{20}$ (minutes) |
|---|---|
| DEMAND 100 g/l CS | 8.46 c |
| Lambda-cyhalothrin 100 g ai/kg WP | 2.82 b |
| Lambda-cyhalothrin 25 g ai/l EC | 1.5 a |

5 replicates each of 20 insects. $KT_{20}$ for each replicate calculated using Zeneca's KTprog. Treatments with no letter in common are significantly different at the 5% probability level.

**Table 3.** Percentage mortality of untreated *Blattella germanica* following introduction to three adult roaches previously exposed to a residual treatment of lambda-cyhalothrin.

| Formulation | Substrate | 1 HAT | 2 HAT | 3 HAT | 24 HAT |
|---|---|---|---|---|---|
| Lambda-cyhalothrin 10% WP | glass | 88 | 93 | 93 | 98 |
| Lambda-cyhalothrin 10% WP | unpainted plywood | 7 | 10 | 17 | 20 |
| Lambda-cyhalothrin 28% EC | glass | 17 | 20 | 23 | 47 |
| Untreated | glass | 0 | 0 | 0 | 0 |

HAT: hours after treatment (introduction of treated cockroaches)

**Transfer of capsules**

Cockroaches groom legs and antennae in response to exposure to insecticides (Bret and Ross, 1986) and particles (El-Awami and Brent, 1995). This behaviour has been shown to result in the ingestion of capsules which can contribute to the lethal effect of OP microencapsulated products (Tsuji, 1990; Anon., 1992). However, pyrethroids are significantly faster acting than OP compounds and have a less pronounced oral activity. Therefore with microencapsulated lambda-cyhalothrin, it is thought that this route of uptake contributes little to the overall toxic effect of the compound.

Insecticide transfer between cockroaches was demonstrated for WP formulations of pyrethroids in arena tests (Schneider and Bennett, 1985). The phenomenon was investigated for lambda-cyhalothrin by exposing 3 adult *B. germanica* for 30 seconds to a glass sheet treated with a 10% WP or 28% EC formulation of lambda-cyhalothrin at 25 mg ai/$m^2$ . The WP treatment was also applied to an unpainted

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plywood plaque. Immediately after exposure, the insects were placed in an 40 litre arena containing harbourage, food, water and 30 untreated adult *B. germanica*. Results are presented in Table 3. The transfer effect was fast and highly effective, confirming the principle of transfer of a toxic dose between treated and untreated insects. The data indicate that transfer is much more effective with particulate formulations than EC formulations. Secondary mortality also appeared dependent on the efficiency of the pick-up of the primary dose as transfer of WP formulations to cockroaches is more efficient from a glass surface than wood (Chadwick, 1985).

Further tests were conducted to investigate transfer of microcapsules between both cockroaches and ants. An assay against *B. germanica* used an identical protocol to that used for the lambda-cyhalothrin WP and EC formulations (Table 4). To test transfer between ants, two workers of the imported fire ant were confined to a treated ceramic tile surface for 30 seconds. Immediately after exposure, the treated ants were placed in a petri dish containing 10 untreated fire ants. Knockdown assessments were made 1 and 2 hours after the introduction of the treated ants and mortality assessments were made 24 hours after introduction. Results are presented in Table 5. These results demonstrate that cockroaches contaminated with lambda-cyhalothrin microcapsules can transfer them to untreated insects, thus enhancing the insecticidal effect of the initial application. The effect appeared less marked than with lambda-cyhalothrin WP, possibly due to differences in the adhesion of the WP carrier and capsules to cockroach cuticle.

Exposure of fire ants to individuals contaminated with lambda-cyhalothrin microcapsules produced a very fast knockdown and high mortality after 24 hours. There are a number of possible explanations for this impressive effect. It is probable that a single lambda-cyhalothrin microcapsule capsule only need be transferred to pass on a dose lethal to a fire ant, thus fewer contacts are likely to be needed to disseminate a toxic effect. The highly social nature of ants, rather than aggregative nature of the cockroaches, may also further assist this process. Necrophoresis could account for some of the mortality occurring between 2 and 24 hours after treatment. Larger arena or field studies are required to determine the relevance of these findings to the practical situation in the field.

**Residual activity**

The persistence of a residual insecticide application is one of the most important features to be considered when developing formulations for the urban environment. Maintaining a toxic level of biologically available insecticide on a surface is the key to a persistent residual effect. It is the nature of the surface which is perhaps the singularly most important factor in the effectiveness of any residual insecticide. The persistent control effect provided by lambda-cyhalothrin microcapsules against a range of pest species on a range of substrates was documented by Williams (1997). Results from a trial comparing the efficacy of residual applications of DEMAND CS on various surfaces against *B. germanica* are presented in Table 6.

**Adsorption.** Much of the loss of insecticidal activity can be attributed to the migration of the liquid insecticides into the substrate and the greatest loss occurs on porous surfaces such as concrete, plaster and mud. Adsorption can be significantly reduced by adopting particulate formulations such as WP and SCs which are popular in the urban and vector insecticide markets. Microencapsulated formulations are also known to reduce adsorption of the active ingredient into porous substrates (Koehler and Patterson, 1988). Table 7 shows results from a residual test conducted with lambda-cyhalothrin microcapsules and lambda-cyhalothrin 25% CS (Karate Zeon) on a highly porous surface. Karate Zeon is a permeable walled capsule, developed primarily for agricultural use, which releases the core contents soon after the spray deposit dries (Perrin *et al.*, 1998). Consequently, it acts in the same manner as liquid (e.g. EC) insecticides on porous inert surfaces, contrasting strongly with the effect provided by lambda-cyhalothrin microcapsules.

**Degradation.** Substrates such as concrete and plaster also cause the rapid chemical degradation of pyrethroid insecticides resulting in their inactivity (Chadwick, 1985). This effect can be markedly reduced by microencapsulation which protects the capsule contents from the degradative chemical proc-

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**Table 4.** Percentage mortality of untreated *Blattella germanica* following introduction to three adult roaches previously exposed to a residual treatment of DEMAND CS.

| Formulation | Substrate | 1 DAT | 4 DAT | 5 DAT | 6 DAT |
|---|---|---|---|---|---|
| DEMAND 10 CS | glazed ceramic tile | 23 | 30 | 30 | 30 |
| Untreated | glazed ceramic tile | 0 | 0 | 3 | 6 |

DAT: days after treatment (introduction of treated cockroaches)

**Table 5.** Knockdown and mortality of untreated *Solenopsis invicta* following introduction to two ants previously exposed to a residual treatment of DEMAND CS.

| Rate of DEMAND CS (mg ai/m$^2$) | % knockdown 1 HAT | % knockdown 2 HAT | % mortality 24 HAT |
|---|---|---|---|
| 30 | 47 | 53 | 95 |
| 15 | 62 | 65 | 87 |
| 7.5 | 57 | 64 | 99 |
| 0 (control) | 0 | 16 | 16 |

HAT: hours after treatment (introduction of treated ants)

**Table 6.** Mortality of adult male *Blattella germanica* exposed for 60 seconds to various surfaces treated with DEMAND CS at 15 mg ai/m$^2$.

| Surface | 2 WAT | 3 WAT | 4 WAT | 6 WAT | 10 WAT | 12 WAT |
|---|---|---|---|---|---|---|
| Unpainted plywood | 83 | 93 | 99 | 95 | 74 | 67 |
| Cement | 97 | 99 | 99 | 99 | 61 | 1 |
| Unglazed tile | 99 | 95 | 95 | 93 | 91 | 87 |
| Vinyl tile | 99 | 99 | 100 | 100 | - | - |
| Vinyl tile (greasy) | 85 | 75 | 7 | 0 | - | - |
| Untreated (all substrates) | 0 | 0 | 0 | 0 | 0 | 0 |

WAT = Weeks after treatment. 48 hour mortality. Three replicates of 30 insects per treatment.

esses. The polymer wall can provide a greater degree of protection than afforded by the inert components of other particulate formulations, such as WPs. A residual assay conducted on cement highlighted these differences (Table 8).

**Environmental effects**

In addition to the substrate, the environmental conditions also affect the immediate availability and persistence of residual applications.

**Moisture.** An assay was conducted in which treated unglazed ceramic tiles were stored under dry or humid (99% RH) conditions (See Table 9). A slight decrease in the persistence of DEMAND CS on the tiles stored under damp conditions relative to dry conditions was observed. However, the reduction in persistence

was less than that observed with the microencapsulated OP products KNOXOUT 2FM (diazinon) and to a lesser extent, EMPIRE 20 (chlorpyrifos). The poorer performance of these materials under damp conditions was probably caused by the diffusion of the water soluble active ingredient from the capsule followed by adsorption into the substrate. The very low aqueous solubility of lambda-cyhalothrin, allowed the activity of DEMAND CS to remain largely unaffected up to 9 weeks after treatment.

**Grease.** Food-handling establishments frequently require residual insecticide applications for the control of cockroaches. Unfortunately, the grease which is frequently found on surfaces in such places has an adverse effect on both the initial activity and persistence of residual insecticide applications (Newton and Coombes, 1990). Laboratory tests to investigate this effect were conducted by exposing *B. germanica* for 30 seconds to residual applications of insecticide made to stainless steel plates which had been covered with a light layer of unsaturated cooking oil. The amount of oil used did not interfere with the normal movement of cockroaches. The results indicated that the efficacy of DEMAND CS was starting to decline after 21 days, which was sooner than would be expected. Applications made to a clean stainless steel surface would be expected to provide complete control at least 7 weeks (T. Gallo, pers. comm.). With the exception of lambda-cyhalothrin 10% WP, a number of other products were found to be affected to a much greater extent by the greasy conditions (Table 10).

The inactivation is believed to be due at least in part, to the binding of the capsules to the grease layer. Once in contact with grease, the lipophilic core contents can diffuse from the capsule and become dispersed within the grease layer. The difference in efficacy observed between DURSBAN LO (chlorpyrifos EC) and EMPIRE (chlorpyrifos CS) indicated that microencapsulation was a benefit under these conditions, probably by reducing the rate of dispersion of the active ingredient into the grease. There appeared to be no such benefit in the case of diazinon (KNOXOUT).

Further laboratory bioassays using *B. germanica* indicated that DEMAND CS and other products such SUSPEND (deltamethrin) SC were inactivated when applications were made to ceramic tile surfaces contaminated with cockroach faeces (data not presented). Inactivation was immediate suggesting that insects were unable to pick-up of the formulations, possibly due to adhesion of the formulation particles to the substrate. Binding of the active ingredient to the organic matter in the faeces may also contribute to this effect. Inactivation by cockroach faeces does not appear linked to any formulation type or

**Table 7.** Mortality of adult female *Aedes aegypti* exposed for 60 minutes to two lambda-cyhalothrin microcapsule formulations applied to an unglazed ceramic tile at 30 mg ai/m$^2$

| Formulation | % Mortality | | | | |
|---|---|---|---|---|---|
| | 2 WAT | 4 WAT | 6 WAT | 8 WAT | 12 WAT |
| DEMAND 10 CS | 96 | 98 | 98 | 100 | 100 |
| Karate Zeon | 26 | 6 | 12 | 6 | 12 |

WAT: Weeks after treatment.

**Table 8.** Mortality of *Periplaneta americana* exposed for 30 seconds to cement treated with two formulations of lambda-cyhalothrin at 7.5 mg ai/m$^2$ using an automated track-sprayer.

| Formulation | 1 WAT | 2 WAT | 6 WAT |
|---|---|---|---|
| Lambda-cyhalothrin 100 g ai/kg WP | 16 | 0 | - |
| DEMAND 100 g/l CS | 100 | 100 | 56 |

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**Table 9.** Mortality of fourth instar *Blattella germanica* exposed for 5 minutes to unglazed ceramic tiles treated with microencapsulated formulations using an automated track-sprayer.

| Formulation | Rate mg ai/m$^2$ | Condition | 2 WAT | 4 WAT | 9 WAT |
|---|---|---|---|---|---|
| DEMAND 100 g/l CS | 10 | dry | 100 a | 100 a | 100 a |
| DEMAND 100 g/l CS | 10 | damp | 100 a | 100 a | 95 a |
| KNOXOUT 2 FM | 500 | dry | 100 a | 100 a | 100 a |
| KNOXOUT 2 FM | 500 | damp | 97.5 a | 65 b | 7.5 c |
| EMPIRE 20 | 200 | dry | 100 a | 100 a | 100 a |
| EMPIRE 20 | 200 | damp | 100 a | 100 a | 40 b |

Treatments with no letter in common are significantly different at the 5% probability level. Mortality assessed at 24 hours. Four replicates of 10 insects per treatment.

**Table 10.** Percentage mortality of adult male *Blattella germanica* exposed for 60 seconds to a greasy stainless steel surface treated with various insecticides.

| Product | Rate (% product) | 1 DAT | 3 DAT | 7 DAT | 21 DAT |
|---|---|---|---|---|---|
| DEMAND CS | 0.06 | 95 a | 100 a | 89 ab | 78 a |
| Lambda-cyhalothrin 10% WP | 0.06 | 99 a | 99 a | 100 a | 93 a |
| EMPIRE 20 (chlorpyrifos CS) | 0.5 | 92 a | 25 c | 32 bc | 46 b |
| DURSBAN LO (chlorpyrifos EC) | 0.5 | 35 bc | 7 cd | 1 c | 1 c |
| KNOX OUT (diazinon CS) | 1.0 | 0 d | 1 d | 0 c | 0 c |
| Untreated | - | 0 d | 1 d | 0 c | 0 c |

DAT = Days after treatment. 48 hour mortality. Three replicates of 30 insects per treatment. Treatments with no letter in common are significantly different at the 5% probability level.

insecticide chemistry: Braness and Bennett (1989) demonstrated similar results for EC and CS formulations of chlorpyrifos and WP and EC formulations of cyfluthrin. The results from tests on greasy and faeces contaminated surfaces reinforce the need to ensure that surfaces are clean prior to the application of residual treatments.

## CONCLUSIONS

The behaviour and biological properties of lambda-cyhalothrin can be differentiated by employing microencapsulation technology. It has been shown that the thick, non-permeable microcapsule walls used in DEMAND CS, produce a formulation with ideal characteristics for use in the urban and household environment. A persistent residual effect is maintained by protecting the active ingredient from movement into or inactivation by the treated surface. The microcapsules contain the lambda-cyhalothrin for extended periods on inert or hostile surfaces, but when impinged upon an insect the active ingredient rapidly diffuses into the lipophilic insect cuticle. A measurable increase in the knockdown and flushing

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times is seen with these slow release microcapsules when compared to EC or WP formulations. However, these small increases will have no appreciable effect on activity when the product is used in the field. It has been shown that under realistic conditions, insects can pick up large numbers of microcapsules from a treated surface, many more than are needed to cause mortality of the individual. Contact between social or gregarious insects allows transfer of capsules between individuals with the subsequent mortality of insects not directly exposed to the treated surface.

In summary, DEMAND CS has been formulated using microencapsulation technology to provide a product designed specifically for the urban and household environment with the following profile: highly potent active ingredient, persistence of effect on a range of surfaces, including those which are highly porous and chemically reactive, rapid knockdown and kill, ability to be passed between social or gregarious insects. In addition, DEMAND CS has a number of significant improvements in environmental and operator safety which are conferred by microencapsulation (Perrin *et al.*, 1998; Williams, 1997).

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