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In-vivo and in-vitro tick repellent properties of cotton fabric

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Abstract

N,N-diethyl-*m*-toluamide (DEET) and eucalyptol were encapsulated by β-cyclodextrin (β-CD) using the kneading method at four different molar ratios. The products were applied to 100% cotton fabric through the impregnation method. Fourier transform infrared and differential scanning calorimetry analysis results confirmed that the inclusion complex occurred between β-CD and repellent agents at mole ratios of 1:3 and 1:2 for eucalyptol and DEET, respectively. The experiments carried out on *Hyalomma marginatum* showed that DEET and eucalyptol treated fabrics repel, inhibit and kill blood-feeding *Hyalomma marginatum* ticks in-vivo and in-vitro for a period of at least 48 and 72 hours under laboratory conditions. In-vitro essay showed good repellent activities for both DEET and eucalyptol. The results show that eucalyptol has more repellent effect than DEET. In the case of the in-vivo essay, a single washing cycle of eucalyptol: β-CD-treated fabric showed repellent activity.

Keywords

tick, eucalyptol, *N,N*-diethyl-*m*-toluamide, encapsulation, biopesticides, biological control

"Tick" is the common name for the small arachnids in the superfamily *Ixodidae* that, along with mites, constitute the Acarina. They are ectoparasites, living by hematophagy on the blood of mammals, birds and occasionally reptiles and amphibians. Furthermore, they are vectors of a number of diseases, including Lyme disease, Q fever (rare; more commonly transmitted by infected excreta), Colorado tick fever, tularaemia, tick-borne relapsing fever, babesiosis, ehrlichiosis and tick-borne meningoencephalitis and Crimean–Congo hemorrhagic fever (CCHF).[1–3]

CCHF is a widespread tick-borne viral disease, a zoonosis of domestic animals and wild animals, that may affect humans. The pathogenic virus is a member of the *Bunyaviridae* family of ribonucleic acid (RNA) viruses. Typically, after a 1–3 day incubation period following a tick bite (5–6 days after exposure to infected blood or tissues), flu-like symptoms appear, which may resolve after one week. However, signs of hemorrhage may appear within 3–5 days of the onset of illness in the case of bad containment of the first symptoms: first mood instability, agitation, mental confusion and throat petechiae, then soon nosebleeds, bloody urine and vomiting and melena. The liver becomes swollen and painful. Disseminated intravascular coagulation may occur as well as acute kidney failure and shock, and sometimes acute respiratory distress syndrome.[4–10] CCHF has caused deaths in Turkey, especially in the Middle-North region, in the last decade. Several fatal cases have recorded caused by CCHF between 2002 and 2010 in Turkey.[2,5]

The recent trend for functional textiles has paved the way for synthetic and natural agents for preventing insect-borne diseases. Insect repellents featuring many agents have been investigated but only a few have been

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applied to textile material. Due to their poor water solubility and high volatility, researchers have leaned towards several preservation techniques. Besides pyrethroids, such as cycloprothrin,[11] permethrin,[12,13] cypermethrin, prallethrin[14] and *N,N*-diethyl-*m*-toluamide (DEET),[15] these have included organophosphates such as diazinon[16] and essential oils such as limonene,[17,18] citronella[19] and neem seed oil,[20] encapsulated as insecticides with suitable wall materials or molecularly capsulated by host molecules, such as cyclodextrin (CD) derivates. On the other hand, some of these agents are applied to textile surfaces simply without protection by the impregnation method but the washing resistance remained limited.[21,22] The ones used in capsule form are subjected solely to in-vitro studies or experimented commonly against mosquitoes.

Because of its recognized insecticide activity and low mammal toxicity, DEET (*N,N*-diethyl-*meta*-toluamide) is preferred as the active agent. It is the common ingredient of most topically applied commercial insect repellents, which are used to protect against mosquitoes and ticks. It works by blocking insect olfactory receptors and provides an unpleasant smell for insects. From the volatile character, insecticidal effect loss occurs after a couple of weeks.[15,23]

Even though DEET is among the topically acceptable insecticidal substances, it is prejudicial for children under a certain age. However, recently herbal essences have become more enticing to customers not only for their appealing fragrances but also due to their eco-friendly features. Many essential oils have been stated as insect repellents. One of them is eucalyptol, which has proven insecticidal activity by field studies. Eucalyptol (cineole or 1,8-cineole) is a monoterpene that shows low toxicity to mammals. It is the major component of eucalyptus oil, which is used in cosmetic products because of its antibacterial effect and pleasant odor as well. Furthermore, its anti-tick activity has been noted.[24–27]

CDs are natural cyclic oligosaccharides, which consist of six, seven and eight glucopyranose units. β-cyclodextrin (β-CD), which has seven glucopyranose units, has been preferred to use in textile applications. Cyclic structure of CDs gives characteristic three-dimensional (3D) structural features that are regarded as a truncated cone. Cyclic glucopyranose units form an apolar cavity, and hydroxyl groups of glucopyranose units are oriented outside of the cone.[28] Thus, CDs have the capability of encapsulating substance in their apolar cavity. As a matter of fact, substances that contain apolar groups and have suitable dimensions were only molecularly encapsulated by CDs. Molecular encapsulation with CDs is called the inclusion complex, where CDs and active agents are named the host and guest substances, respectively. CDs have been used for scent, cosmetic and dye applications in textiles.[29,30]

Recent studies investigating insect repellent characteristics of textiles including CDs are mainly concerned about grafting of CD derivatives. Abdel-Mohdy et al.[13] surveyed the insecticidal effect of monochlorotriazine β-cyclodextrin (MCT-β-CD) with cypermethrin and prallethrin, separately. Divergently, the complexation process was done on the fabric. In the first step cotton fabrics were grafted with MCT-β-CD via dipping in aqueous solution of the host substance and curing at relatively high temperature. Afterwards fabrics were soaked in a solution containing the insecticide in ethanol and dried. In-vitro insecticidal activity was investigated against mosquitoes. Hebeish et al.[17] also grafted cotton fabric with MCT-β-CD and compared the impregnation method described above and the coating method. Limonene was chosen as the guest substance. Romi et al.[31] applied a similar method for grafting MCT-β-CD to cotton fabric and examined the insecticidal effect of DEET and permethrin.

In the present work one chemical (DEET) and one natural insecticidal product (eucalyptol) were encapsulated by β-CD using the kneading method with four different molar ratios. The effects of mole ratios of preparates were investigated by differential scanning calorimetry (DSC) and Fourier transform infrared (FT-IR) analyses. The products were applied to the 100% cotton fabric through the impregnation method and tick repellent properties of fabrics were observed on rabbits. However, there are several studies regarding insect repellent fabrics, but this study is unique in testing both washing durability and in-vivo tick repellency for preventing such mortal tick-borne diseases.

Experimental method

Materials

Mill scoured, bleached plain weave cotton fabric (specific weight 130 g/m^2) was used as the ear pocket. Figure 1 shows the host and guest molecules. β-CD (1134.98 g/mol) was supplied by Wacker-Chemie AG, Munchen, Germany, as a host molecule. The analytical graded guest molecules DEET (191.27 g/mol) and eucalyptol (154.25 g/mol), employed as insecticides (guest molecules), were developed by Sigma-Aldrich, Taufkirchen, Germany. The formaldehyde-free cross-linking agent Knittex FF-RC was acquired from Huntsman, Istanbul, Turkey. The other auxiliary chemicals were provided by Riedel-de Haën, Seelze, Germany.

Preparation of the inclusion complex

In order to form a weakly water-soluble guest–host couple, the kneading method was performed. In this technique the CD was kneaded intensively with the

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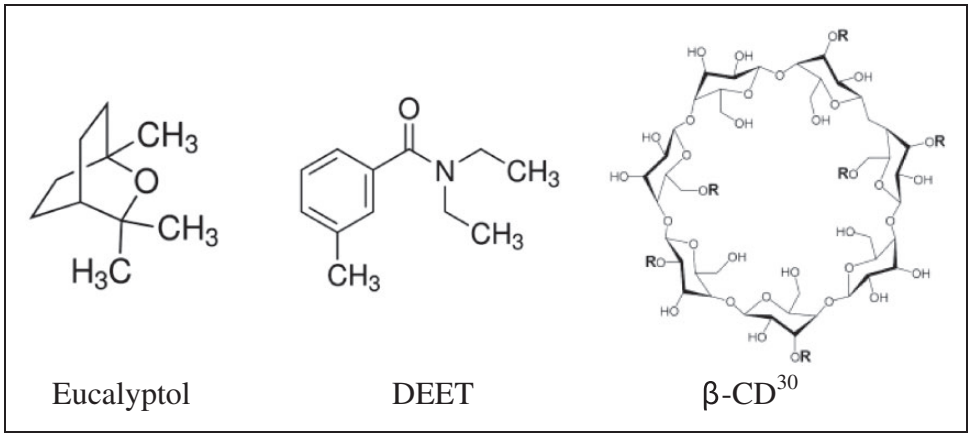


Figure 1. The chemical structure of the active substances and β-cyclodextrin (β-CD). DEET: *N, N*-diethyl-*m*-toluamide.

presence of an organic solvent. The guest compounds, ethanol and β-CD, were added in a mortar and kneaded constantly at ambient temperature with the aid of a pestle until the paste was dried. The inclusion complex was prepared in four molar ratios, separately (host–guest molar ratio = 1:1, 1: 2, 1:3 and 5:9).

Fixation of the inclusion complex onto cotton fabric

To obtain a durable insecticide effect on cotton fabric, fixation has to be applied. After deciding on the optimum inclusion complex formula, the impregnation liquor was prepared. Primarily, products (40 g/l) were dissolved in water and then the cross-linking agent (150 g/l) was added. The pH of the solution was adjusted to pH 5 by CH_3COOH. The fabric sample was impregnated in a solution bath containing inclusion complex and cross-linking agent, and squeezed between rollers to 92% wet pick-up. Fabrics were dried in a circulating air oven at 60°C. Just after drying, the fabric was mounted on pin frames and exposed for 3 minutes to 130°C in a laboratory stenter. The amount of inclusion complexes in fabrics were determined theoretically according to the formula $C_f = \frac{C_s \times Pickup}{100}$, where C_f is the concentration in fabric (g/kg), C_s is the concentration in the solution bath and *Pickup* is the pickup value.

The results were the same for both DEET and eucalyptol. Theoretically, the inclusion complex concentration in the fabrics was 36.8 g/kg.

Washing procedure

β-CD-insecticide complexes treated samples were washed for 1, 10 and 20 cycles with 4 g/l Ece non-phosphate reference detergent (A) at 40°C for 30 minutes, separately, according to ISO 105-C06:2010.[32] The fabrics were rinsed then dried in laboratory conditions.

In-vitro studies

Hyalomma marginatum species of adult ticks were used for both in-vitro and in-vivo essays. The adult ticks were reared in a controlled and sterilized environment and they had no any infections, either viral or bacterial. Adult ticks were kept in an incubator at 12°C. After being taken out from the incubator, the ticks were kept awake for 6 hours at 24°C.

In-vitro studies are carried out using ticks (*Hyalomma marginatum*) that were placed onto fabrics. The life functions of ticks (observation of squirms and activities) were observed after 1, 2, 24, 48 and 72 hours. The observations of life functions were performed under a light microscope.

In-vivo studies

Besides in-vitro studies, in-vivo experiments were also conducted. All the experiments in this study were carried out in compliance with the relevant national laws relating to the conduct of animal experimentation. Furthermore, the study was approved by the local Ethics Committee of the Experimental Studies at Ege University. The rabbits (*Oryctolagus cuniculus*) attended on the trial, which were healthy, 12 months old and weighing 1200–1500 g, were left for 3 days at room conditions for acclimatization. They were given a standard pellet diet and water ad libitum during the study. The animals had free access to nutrients and were maintained under a 12-hour light/dark cycle. The initial average weight and behavior of the animals did not differ significantly at the end of the experiment.

The fabrics used in in-vivo trials, which are in the shape of a truncated triangle, were formed as pockets by sewing the long edges. In order to determine the tick repellency of the fabrics, exactly 10 ticks (*Hyalomma marginatum*) were attached onto each ear of the

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rabbit. Afterwards, DEET and eucalyptol impregnated and washed pockets were placed on each ear. The life functions (observation of squirms, activities and engorgement) of ticks were observed after 2, 24, 48 and 72 hours.

Scanning electron microscopy

Morphology of the inclusion complexes is studied using FEI Quanta250 FEG scanning electron microscope (SEM). The capsulated repellents are placed onto the specimen stubs before coated with gold in order to bring a conductive feature. The measurements are taken at 5 kV accelerating voltage: 1000 × and 2500 × magnification.

Fourier transform infrared spectroscopy

FTIR analysis is done with the aim of proving the complex formation with the changes in the infrared spectrum. Accordingly, the inclusion complexes are measured at a wavelength range of 400–4000 cm^{-1} using a Perkin-Elmer Spectrum BX equipped with attenuated total reflectance (ATR). The resultant spectra were smoothed to remove the noise via official software for the equipment.

Differential scanning calorimetry

DSC was performed using a Perkin-Elmer diamond differential scanning calorimeter for the purpose of distinguishing the complex formation from the simple physical mixture with the help of characteristic endothermic or exothermic peaks. The analyses were conducted in nitrogen medium between 0 and 300°C. The scanning rate was stated as 5°C per minute.

Results

Characterization

Figure 2 depicts the FT-IR spectrum of pure β-CD. The FT-IR spectrum of DEET and its mixtures with β-CD at different molar ratios are given in Figure 3. As seen in spectrum of DEET, there is a characteristic band that appears at 2931 cm^{-1} due to a CH_3 symmetric

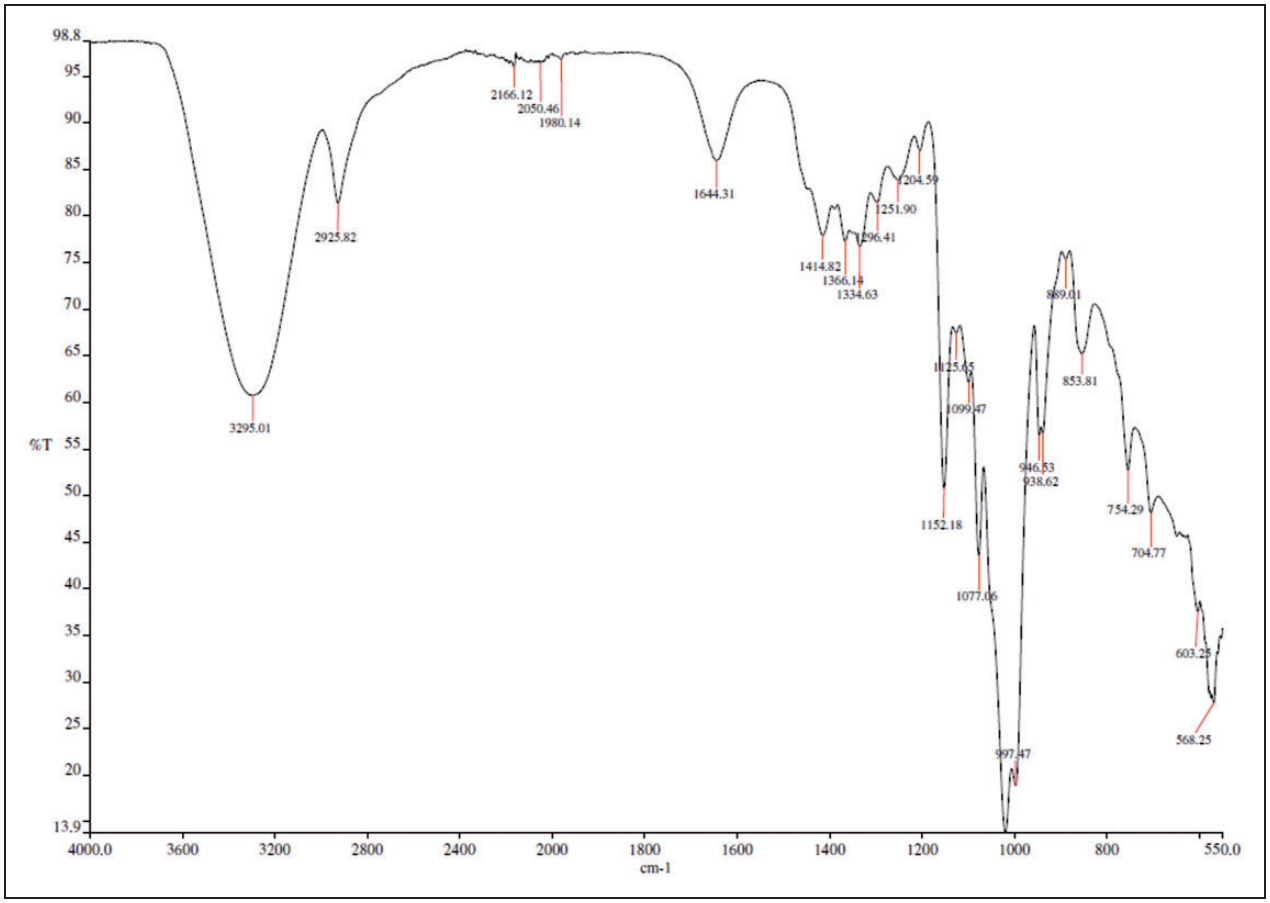


Figure 2. Fourier transform infrared spectrum of β-cyclodextrin.

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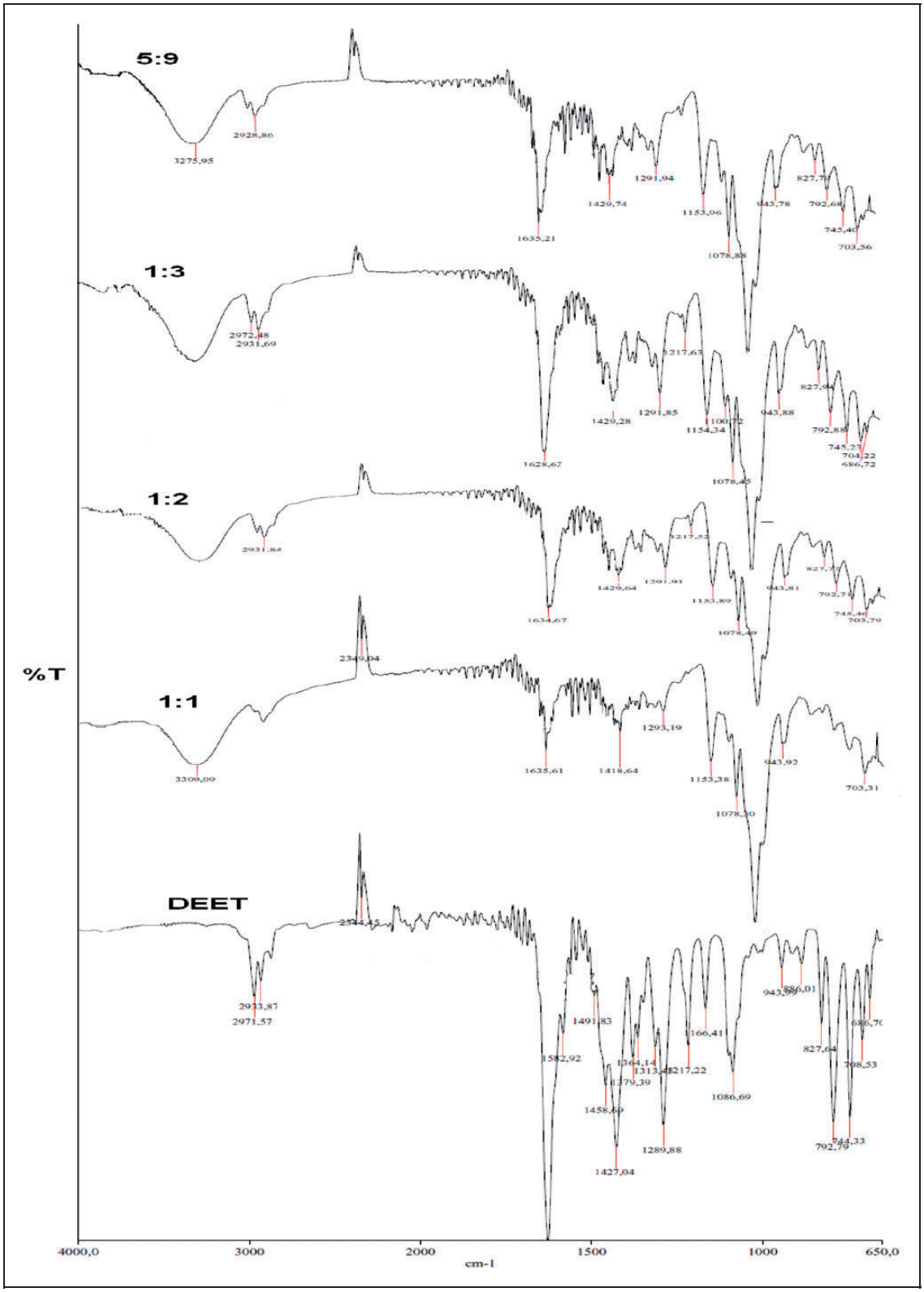


Figure 3. Fourier transform infrared spectrum of *N, N*-diethyl-*m*-toluamide (DEET) and its inclusion complexes with β-cyclodextrin.

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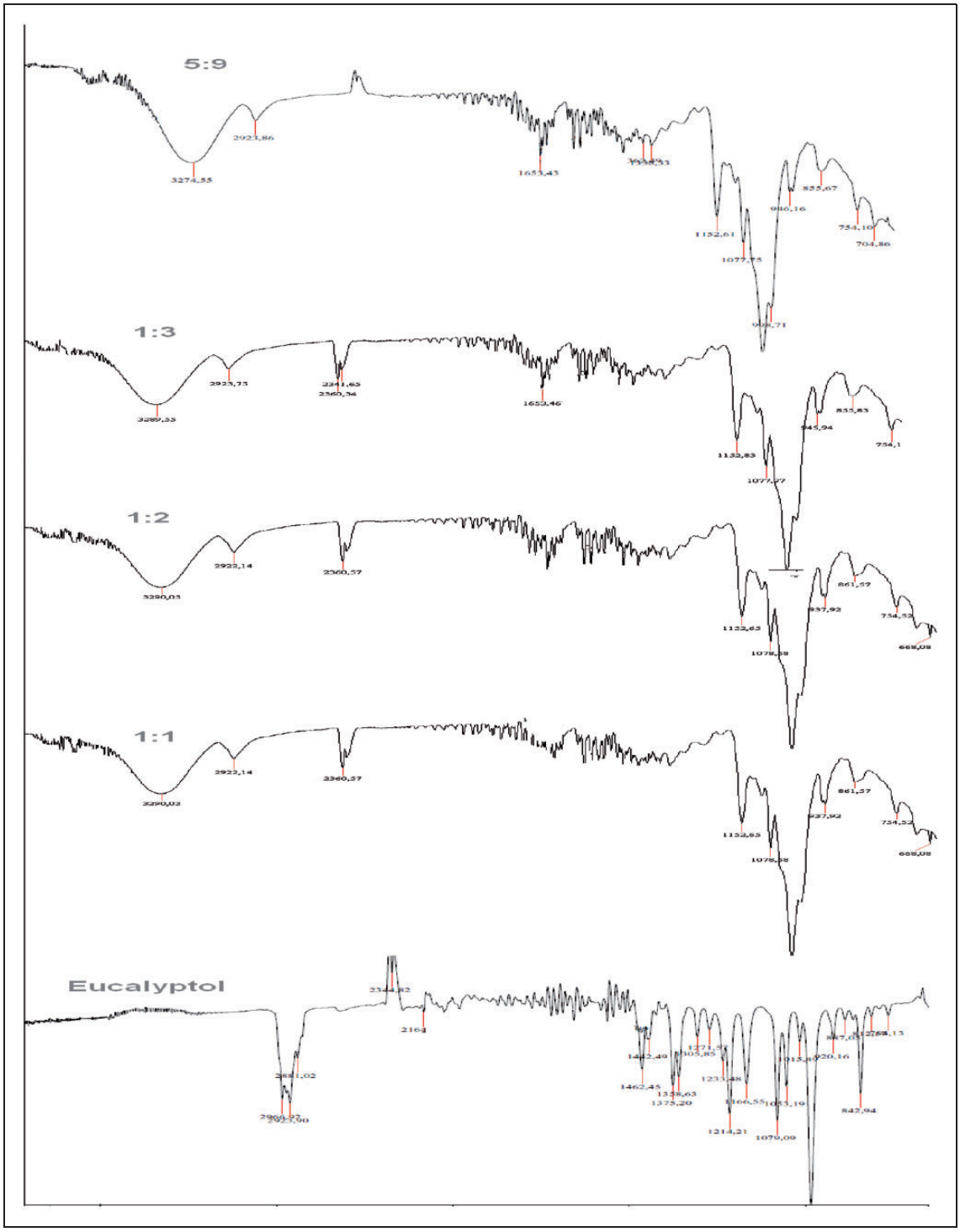


Figure 4. Fourier transform infrared spectrum of eucalyptol and its inclusion complexes with β-cyclodextrin.

stretch. Furthermore, it is found that a band shift at 2973 cm^{-1} is the cause of the CH_3 asymmetric stretch . These two bands can be attributed to the CH_3 group of DEET. Those two bands appear in the spectrums of DEET β-CD mixtures at different mole ratios. A band at around 1635 cm^{-1} can be assigned as C=O stretching of the amide I bound. A broad peak that appeared around 3300 cm^{-1} can be attributed to the OH groups of β-CD.

The FT-IR spectrum of eucalyptol and its mixtures with β-CD at different molar ratios can be seen in Figure 4. The spectrum of eucalyptol is found to be

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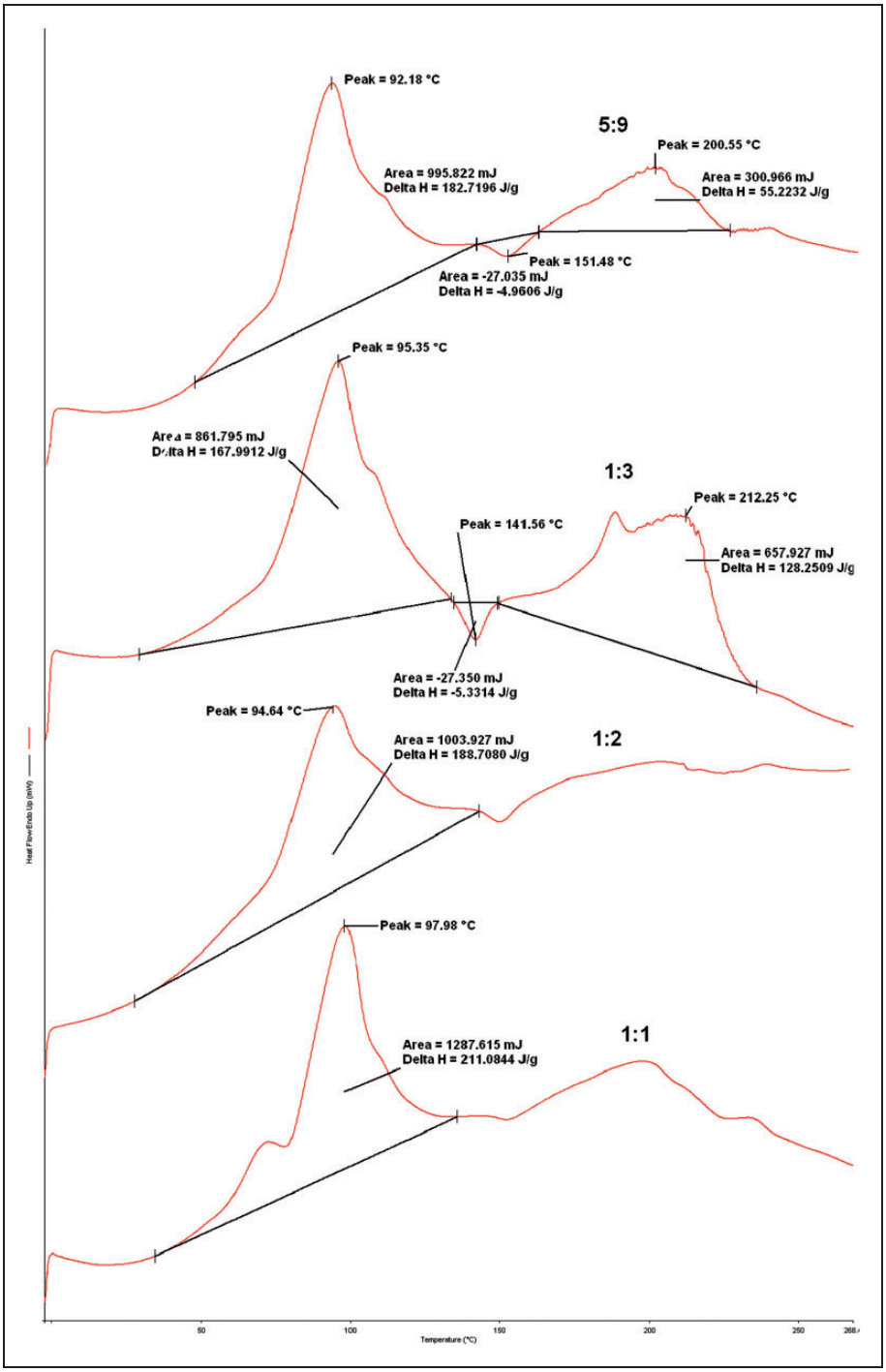


Figure 5. Thermographs of *N, N*-diethyl-*m*-toluamide:β-cyclodextrin complexes.

consistent with the literature. Ciobanu et al.[33] described the carbon–carbon bands between 1359 and 1214 cm^{-1}. While this interaction is distinctive in the spectrum of eucalyptol, it is not seen in the spectra of eucalyptol:β-CD preparations due to the characteristic peak of CD related to saccharide derivates. It is determined that the CH_3 symmetric stretching band shifted at 2923 cm^{-1}. This band is distinctive at spectrums of all molar ratios of eucalyptol:β-CD complexes. Ciobanu et al.[33] described 3D molecular models of inclusion complexes between various essential oils and beta-CD. When examining the molecular model of inclusion complex eucalyptol and beta-CD, the cyclic part of the eucalyptol in the cavity of beta-CD and the

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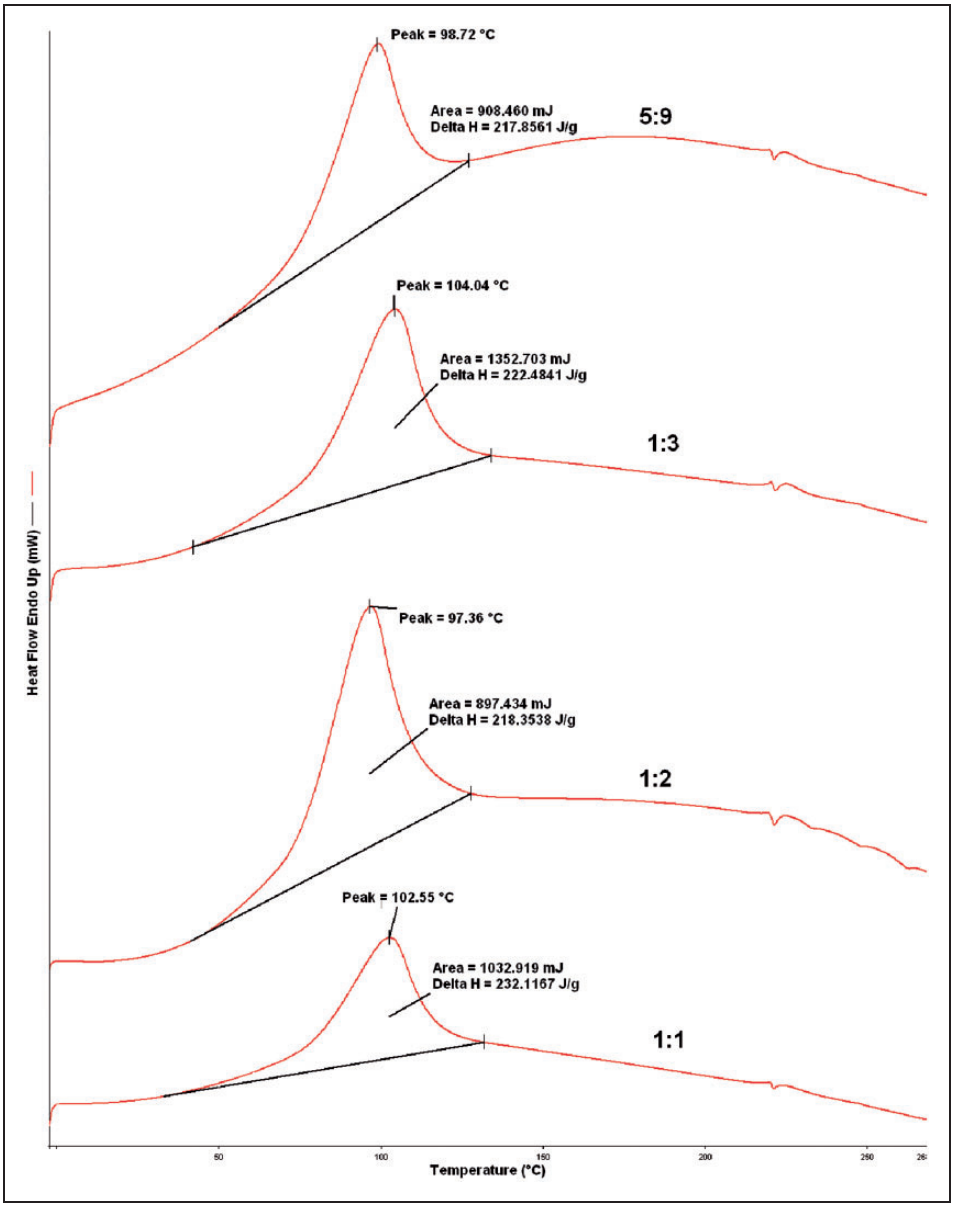


Figure 6. Thermographs of eucalyptol:β-cyclodextrin complexes.

methyl groups were seen as orientated to out of cavity. Hence, this band was distinctive and was observed at spectrums of all molar ratios of eucalyptol: β -CD complexes.

The thermographs in Figures 5 and 6 are given in order to identify the state changes of complexes. An endothermic peak is seen at 100°C due to evaporation of water molecules that have bonded with hydroxyl groups of β-CD. However, the exothermic peak shift at around 150°C is attributed to possible decomposition of DEET. It is realized that the complex has a 1:2 β-CD:DEET ratio, giving lower peaks than the other formulas, which indicates a better thermal resistance (Figure 5). There are distinctive state changes identified aside from expected water evaporation in 100°C. Thus, it is considered that the complexation is accomplished successfully because of the absent peak around the boiling point of eucalyptol, which is at approximately 175°C (Figure 6).

SEM images for microparticles produced by using chosen formulas are given in Figure 7. These repellent-contained particles are compact, do not show observable fractures, cracks or pores and have non-spherical random shapes. It is determined that the eucalyptol particles (Figure 7(a)) are slightly smaller than the DEET particles (Figure 7(b)). The sizes of particles containing eucalyptol are determined to be between 1

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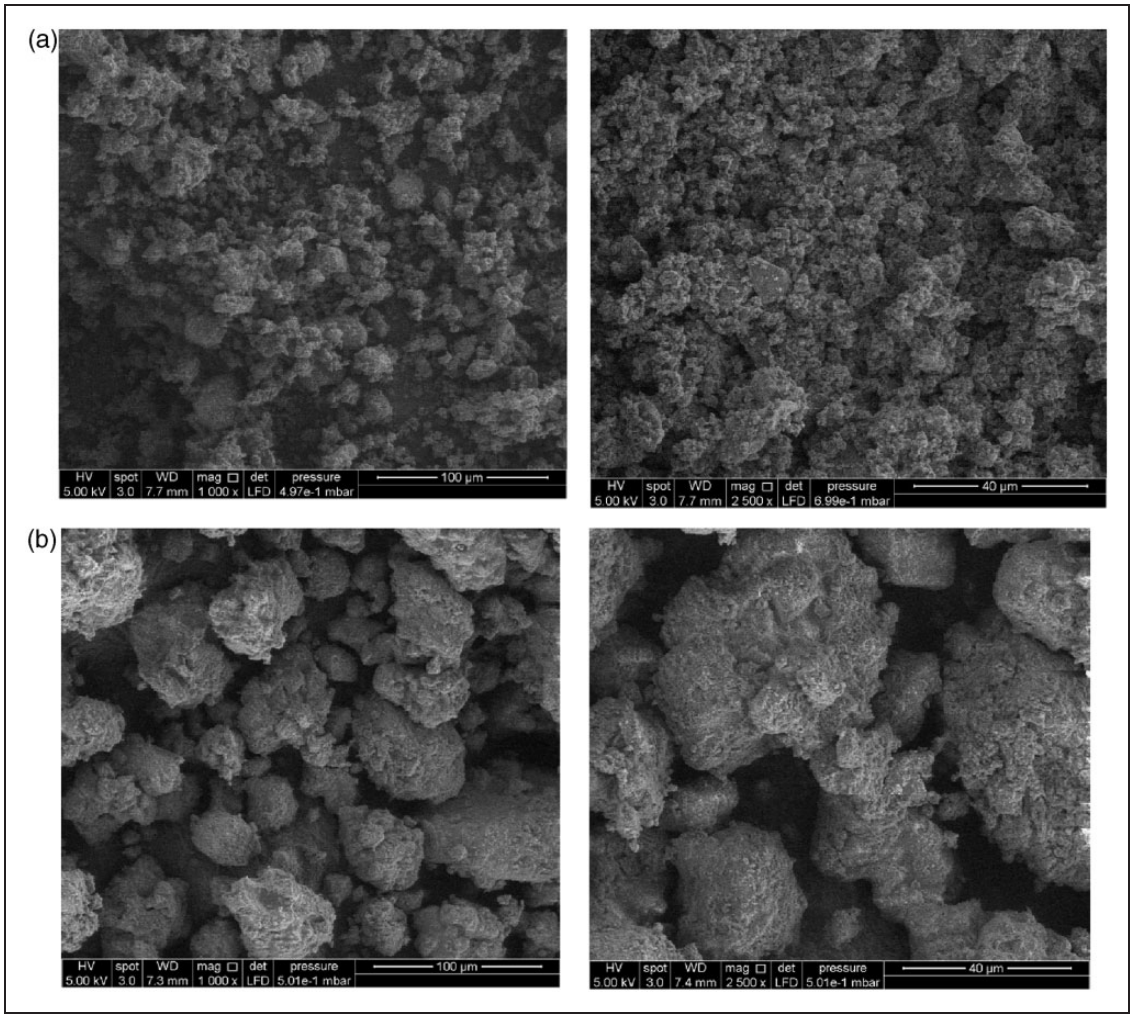


Figure 7. (a) Inclusion complex morphology of 1:3 β-cyclodextrin (β-CD):eucalyptol. (b) Inclusion complex morphology of 1:2 β-CD:*N, N*-diethyl-*m*-toluamide (DEET).

and 20 μm, whereas the particles of DEET are between 30 and 100 μm, approximately.

From all the data acquired, the optimum complex concentrations given the best thermal tolerance are determined, which are 1:3 and 1:2 for β-CD:eucalyptol and DEET complexes, respectively.

In-vitro essays

The life functions of *Hyalomma marginatum* ticks were placed onto treated fabrics at different time intervals (Figure 8). As seen in the chart, after 24 hours, life function is observed only in a few ticks subjected to DEET-impregnated fabrics. At the same time, it is determined that the ticks exposed to fabrics containing eucalyptol could not survive. However, after 48 hours, it was seen that all the ticks had lost their activity, whether subjected to fabrics containing DEET or eucalyptol complexes .

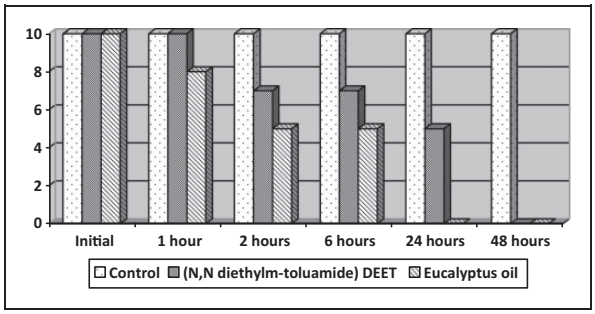


Figure 8. Life functions of ticks at different time intervals (in-vitro).

The effect of washing on ticks' life functions is presented in Figure 9. From the ticks exposed to DEET fabric washed once (Figure 9(a)), only one tick showed activity after one hour. When the number of washing cycles rose, the number of ticks that showed

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vital activities increased. After 24 hours, the activity of ticks resembled that of the control fabric. On the other hand, it was determined that the number of ticks exposed to the washed fabrics containing eucalyptol remained stable after 24 hours (Figure 9(b)). Numerically, fewer ticks that contacted fabrics containing eucalyptol complexes showed vital activity. Increasing washing cycles affected the vital activity of ticks negatively. However, it is seen that activity of fabrics carrying eucalyptol complexes are more stable than fabrics carrying DEET. Hence, it is conceived that eucalyptol complexes showed more repellent and insecticidal activity than DEET.

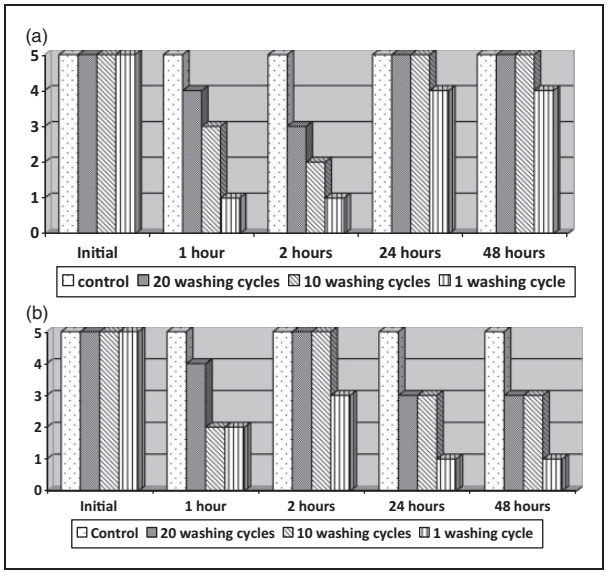


Figure 9. (a) The effect of washing cycles on repellent activity of *N,N*-diethyl-*m*-toluamide (in-vitro). (b) The effect of washing cycles on repellent activity of eucalyptol (in-vitro).

In-vivo essays

In order to evaluate the behavior of fed-on ticks, ears of rabbits were covered with the insecticidal applied pockets, as is seen in Figure 10. When the repellent activity of DEET fabric was examined, it is found out a slight decrease on activity after 24 hours. There was no difference determined between numbers of washing, except fabric washed once. It is claimed that fabrics containing DEET complexes had no repellent effect on host-attached ticks in the in-vivo essay after washing.

The effect of washing durability is depicted in Figure 11. Repellent activity of eucalyptol fabrics was determined by the number of ticks that became detached from the rabbits' ears . Four ticks that meet fabrics washed once escaped after 48 hours . However, other washing cycles had no repellent activity on ticks.

Conclusion

A recent study containing impregnation of nets with DEET repellent conducted in experimental huts indicated that this is a promising approach to overcome the problems associated with pyrethroid-resistant insects.[15] Microcapsule suspensions of pyrethroids are now entering the market as long-lasting indoor residual spray treatments.[34] In order for DEET to become viable as a textile treatment, the repellent will need to be bound within some kind of long-lasting formulation. This route of pick-up is clearly sufficient for DEET to exert a toxic effect, as has been shown in several recent studies on mosquitoes and other insects in laboratory and field experiments.[35,36] Prior reports showed the activity of the essential oil of eucalyptus globules, rich in 1,8-cineole, against the same tick.[37] Nevertheless, it must be noted that this essential oil was lethal in a concentration 10 times higher than the concentration tested in our work. The essential oil of *Melinis minutiflora*



Figure 10. In-vivo essays of repellent activities against ticks.

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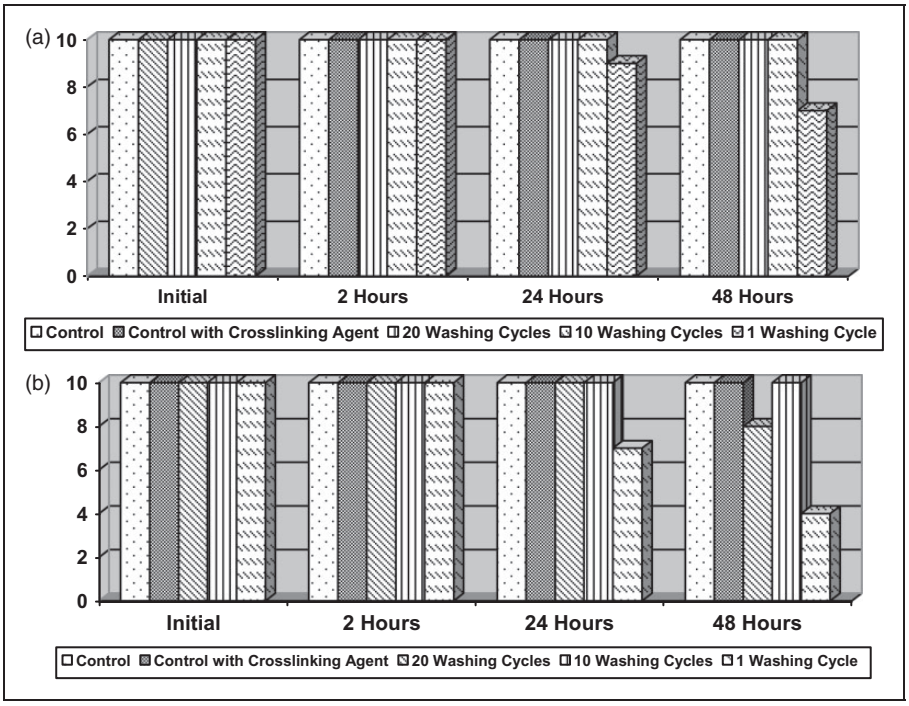


Figure 11. (a) The effect of washing cycles on repellent activity of *N, N*-diethyl-*m*-toluamide (in-vivo). (b) The effect of washing cycles on repellent activity of eucalyptol (in-vivo).

Beauv has shown a virtually 100% lethal effect on the same larvae within 10 minutes of exposure, and this activity was associated with 1,8-cineole presented in 10.6% in the oil.[38] Recent studies reported the use of eucalyptol is repellent against various species of ticks (*Rhipicephalus (B.) microplus, Ixodes ricinus*).[39–41]

The in-vitro essay showed good tick repellent activities for both DEET- and eucalyptol-treated fabrics. Eucalyptol had more repellent effects than DEET. In the case of the in-vivo essay, only a single washing cycle of eucalyptol:β-CD treated fabric showed repellent activity.

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