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ИЗСЛЕДВАНЕ НА ТРАЙНОСТТА НА АРОМАТ ВЪРХУ ТРИКОТАЖНА ЛЕНТА С ПОКРИТИЕ ОТ EUGRARIT RSPO МИКРОКАПСУЛИ, СЪДЪРЖАЩИ ЕТЕРИЧНО МАСЛО ОТ КАНЕЛА

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INVESTIGATION OF THE FRAGRANCE DURABILITY OF KNITTED FABRIC BAND COATED BY EUGRARIT RSPO MICROCAPSULES CONTAINED THE CINNAMON OIL ESSENTIAL

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ABSTRACT

The aromatherapy textile of cinnamon oil comes from a compound found in the essential oil called cinnamaldehyde. With the antibacterial and anti-inflammatory properties that can help treat certain metabolic, infectious, digestive or respiratory disorders, this compound is also very volatile. Moreover, cinnamon essential oil can help treat dermatitis, but the coumarin compounds found in essential oils can cause skin irritation. Therefore, encapsulation is currently the best solution to improve evaporation and keep the fragrance of essential oils on fabrics longer and reduce large amounts of pure essential oils in direct contact with the skin. In this study, we have investigated the odor fastness of Eugrarit RSPO microcapsules contained the cinnamon essential applied to the surface of interlock fabric knitted by Chief Value Cotton (CVC) yarn (60% cotton, 40% polyester). The fragrance durability of the knitted fabrics coated by pure cinnamon essential oil and the microcapsules contained cinnamon essential oil had been evaluated. The influence of the pressure of knitted fabric band coated by the microcapsule contained cinnamon essential oil induced by extension levels of 21.25%, 57.5%, 68.75%, 83.75% had been studied. The fragrance durability evaluation was based on the combination of the expert method and diluted solution method. The results showed that the fragrance intensity of knitted fabrics treated by pure essential oil was stronger but the diminution of their fragrance was faster than these ones treated by the cinnamon essential oil microcapsules. Besides, the higher the extension applied on the fabrics band, the smaller their fragrance intensity had been maintained.

Keywords: *Microencapsulation, fragrance textile, healthcare textile, interlock knitted fabric, fabric extension.*

1. Introduction

Essential oils are volatile components which produced by different parts of the medicinal plants. These components have antibacterial capacity and have been commonly used throughout the world. They even can be a substitute for chemical antibacterial agents against foodborne bacteria in clothing due to their useful properties [1]. Cinnamon is obtained from several tree species from the genus cinnamomum and is also known as cinnamomum verum. The true cinnamon treeverum is considered as one of the precious medicinal plants that have been widely used in the daily life in Vietnam. Cinnamon essential oil can be also used in aromatherapy, which is the therapeutic use of the plant essential oils that can be absorbed into the body via the skin or the olfactory system. A recent research articles showed the benefits deriving from the use of cinnamon oil in massage for alleviating menstrual pain [2]. Cinnamon essential oil is a dark yellow to brown liquid. It is prepared from the bark of the stem, or leaves and branches by the method of distillation with water or steam distillation [3]. The all parts of the cinnamon tree such as bark, leaves, flowers, roots contain essential oils, especially in the bark with the highest content of essential oils, sometimes reaches 4 - 5 % on mass. The major constituents of cinnamon bark oil includes cinnamaldehyde (76.96 %), cinnamyl acetate (11.07 %), coumarin (5.06 %), the remaining compounds account from 0.11% to 0.92 % on mass. In which cinnamaldehyde (3-phenyl-2-propanal) represents the main component of cinnamon bark oil which has antibacterial and anti-inflammatory properties that can help treat certain metabolic, infectious, digestive or respiratory disorders. Trans-cinnamaldehyde was found to be the major volatile compound which used as a flavor and fragrance ingredient [4, 5]. Though the various potential effects of cinnamon oil and its constituents, including anti-inflammatory, antibacterial, antifungal, and antioxidant, the use of cinnamon oil has been reported to cause problems for some individuals: cinnamon oil has a strong aroma that is unacceptable to some. Furthermore, the direct application of cinnamon oil has been reported to cause skin irritation and allergic reactions [6].

In order to maintain the fragrance longer as well as for the active ingredients to be more effective, a microencapsulation has been widely used to improve the ability to evaporate the fragrance materials for the desired time [7]. Microencapsulation could be understood as a technique to prepare a microcapsules, a small particles that contain an active agent or core material surrounded by a shell of limited permeability. Microencapsulation helps an active material enveloped in microcapsule wall which protects it from external environment. The aroma core is released by diffusion through the microcapsule wall or by rupture of the microcapsules. These microcapsules can be applied to fabric by simple pad-dry sequence or by coating method. During wear, simple mechanical rubbing of fabric gradually ruptures the membrane that releases the active agent for therapeutic, energy boosting, stress busting, moisturising or deodorising effects [8-13].

Many researches have reported the textile applications using the microcapsule contained natural essential oil. G Thilagavathi et al. [9] had prepared the antibacterial fabrics coated by the microcapsule containted the neam oil essential and the oil extracts from mexican daisy. The microcapsules consisted of the oil herbal extracts as active core material and gum acacia as a wall material. The test of antibacterial had conducted using the Staphylococcus aureus and Escherichia coli bacteria. Senem Karagönlü et al. had prepared the thyme oil loaded microcapsules for antimicrobial textile [10]. The microcapsiles using thyme oil as active agent and gelatin and gum arabic as wall materials. The durable aroma finished on cotton fabric using microencapsulation technology had been conducted by L. BHATT et al [8]. Four kinds of natural essential oil (basil oil, lemon grass oil, orange oil, tea tree oil) were used as core material and the gelatin and gum acacia were the membrane of microcapsule in their reaserch. The most efficace essential oil was considered as lemongrass oil. Microencapsulation of fragrance agent and natural volatile oils for application in cosmetic textile had repported by Rumeysa Tekin et al. [11]. The microcapsules with diameters ranging from 10 mm to 80 mm had prepared by interfacial polymerization technique using the fragrance Teddysoft as a core material, PVA as surfactant and the polymer wall of microcapsules was the polyurethane. The hand towels then coated by microcapsules contained

fragrance and by fragrance without microcapsules which mixed with the fabric softener. These hand towels passed the laudering cycles and were let to dry for a week to qualitatively evaluate the fragrance by nine perfumers and by headspace-GCMS analysis. Both the evaluations showed that the hand towels washed with the fabric softener including microcapsules smelt stronger after a week. Moreover, the authors found more the volatile compounds of the fragrance existed on the hand towel treated by the microcapsules than these ones applied directly by the fragrance without encapsulated. The results demonstrated the efficace of microencapsulation technique to maintained the fragrance for textile products.

Odor is a psychophysical phenomenon. For the human senses, the smell considered as the most complex. For odor measurement, there have been different methods which use instruments such as gas chromatograph to determine the odorous gas concentration in ppm and olfactory sensory methods which use the human sense of smell [14]. The odor evaluation of the fragrance fabric was not always easy because of incompliance of the methods for this kind of product.

The fragrance durability by the time or by washed cycles of the garment treated by microcapsules contained the natural essential oil had been reported in many researches but the odor fastness influenced by pressure caused by the extension of knitted fabric band coated by the microcapsule containing the cinnamon essential oil had been seldom noticed in the papers and that was also the aim of the current research.

2.Materials and Methods

2.1 Materials

Knitted fabric: The research has used the interlock fabric knitted by the CVC yarn (60% cotton, 40% polyester) with yarn count of Ne 40/1. The fabric mass was 175.7 (g/m2) and the fabric thickness was 0.8 mm. Its loop length was 2.75 mm. The fabric was knitted on Kingknit machine (Taiwan) with the needle cylinder diameter of 30 inches and machine gauge was E24.

Fragrance agents: The experimental process had used a natural cinnamon essential oil in liquid form and the solution of microcapsules contained cinnamon essential oil (5 % w/w) as aroma agents. The microcapsules (***Figure 1***) that were kindly provided by the project T2021-PC-044 (Vietnam). The cinnamon essential oil - loaded microcapsules were prepared by solvent evaporation method. Their membrane was eudragit RSPO and the quillaja saponin was used as the natural surfactant while ethyl acetate was the non - halogenated solvent applied during microencapsulation process. The microcapsules had nearly spherical shapes with the mean diameter of around 25-30 μm [15].

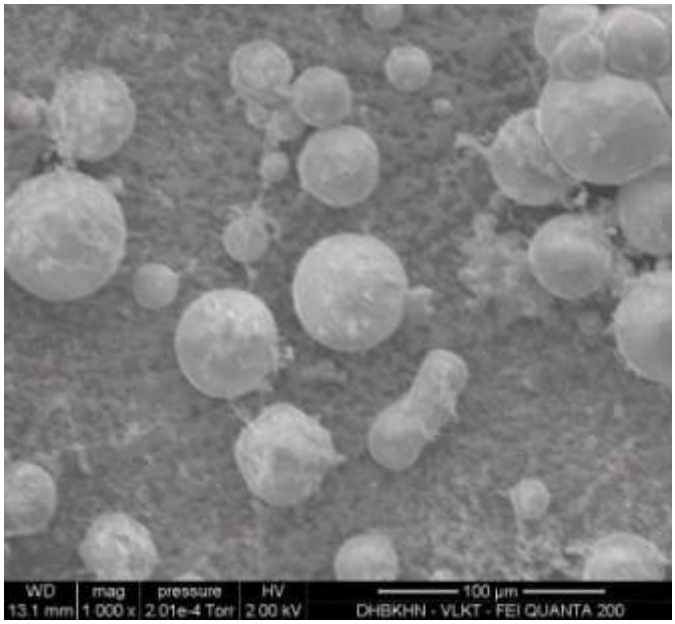

Figure 1 Eugrarit RSPO microcapsules contained cinnamon essential oil.

2.2 Methods

2.2.1 Investigation of microcapsule morphology

The scanning electron microscope (SEM) JEOL model JSM-7600F (USA) was used to investigate the morphology of Eugrarit RSPO microcapsules contained the cinnamon oil essential before and after treating to the knitted fabric. The working conditions SEM was kept at 5.0 kV; LM mode and WD 8.0 mm. For the original microcapsules, a small drop of mother microcapsule suspension was placed on the sample holder of SEM equipment while the fabric coated with microcapsules had been cut on a small piece of fabric, which then was stick to the sample holder with the fabric surface containing microcapsules faced up. The observation was captured at magnifications of x100 and x500 for fabric treated by microcapsules and the observation was taken at magnifications of x10.000 for the details on the microcapsule polymer membrane.

2.2.2 Fragrance evaluation

In this study the fragrance intensity of fabric samples treated by microcapsules contained cinnamon essential oil had been evaluated by expert method in combination with the comparison of diluted sample methodthe. The fragrance intensity was chosen as odor characteristic to evaluate fragrance of the investigated fabrics. The fabric samples treated by microcapsules contained the fragrance agent had been evaluated by expert method in combination with the comparison of diluted sample method based on the standard ASTM D 1292-10 [16] and on the measurement of intensity by magnitude scale procedure.

The aqueous solution of microcapsule had gradually diluted by distil water and has been contained in a small bottle of 10mL. Eleven bottles had been prepared with different ratio of microcapsule volume solution, which was 0%, 10%, 20%, 30%, 40%, 50%, 60%, 70%, 80%, 90% and 100%, corresponding to the fragrance points of 0, 10, 20, 30, 40, 50, 60, 70, 80, 90 and 100. The bottles had then well tight closed during the experiment. The bottle of total distil water is “no odor” and was considered as “blank”. The perfumers have been trained to participate in an evaluation fragrance [16-18]. Every perfumer has smelled the fabric sample and compared to the sample bottles with its fragrance grade then has given out a point in a hundred grades. Before changing to evaluate another fabric sample the perfumer have been demanded to smell the bottle of zero fragrance grade. The perfumers had not any discussion to each other during the evaluation experiment [18].

2.2.3 Influence of fragrance agent kind on the odor durability knitted fabric

Two kinds of fragrance agent have been used: the first was pure cinnamon essential oil and the second was the Eugrarit RSPO microcapsules contained cinnamon essential oil. The investigated fabric was cut into the fabric samples with the size of 50 mm x50 mm. Nine fabric samples for each test of fragrance agent kind and there was total of 18 fabric samples had been prepared for two kinds of fragrance agent.

The experimental process of odor assessment was carried out in an environmental condition with the temperature of 31oC-31.5oC and the relative humidity of 46 %-50 %, which was the typical climate of the south Vietnam. The survey process in the odor assessment was carried out by 6 participants aged from 8 to 45 years old, including 2 males and 4 females in respecting the different olfactory threshold of sex and age of the participants [18].

1mL of solution of Eugrarit RSPO microcapsules contained the cinnamon essential oil was coated evenly to the surface of nine prepared fabric samples. On the other hand, 1 mL of pure cinnamon essential oil was also applied to the surface of the other nine fabric samples. Then all 18 fabric samples have put into the refrigerator at temperature of 2°C and at 20% of relative humidity for 24 hours, that process aimed to harden the microcapsules.

Fragrance durability of the fabric samples were then evaluated for every 60 minutes from the first hour to the ninth hour to compare the fragrance durability of the fabrics treated by pure cinnamon essential oil and by microcapsules contained the cinnamon essential oil.

2.2.4 Influence of the extension on the fragrance durability of knitted fabric band coated by Eugrarit RSPO microcapsules contained the cinnamon oil essential

Four levels of extension knitted fabric band had been prepared by cutting four knitted fabric samples of equal size (8 cm x 3 cm). Then 2ml solution of microcapsules contained the cinnamon essential oil

had coated on the fabric surface. The fabric samples then stored in the refrigerator with the temperature of 2oC and the relative humidity of 20% for 24 hours to solidify the microcapsule on the fabric.

After being stored in the refrigerator environment the fabric samples coated by microcapsules were sewn to form a cylinder band then were looped into four glass jars. Four glass jars with different circumferences as 9.7 cm, 12.6 cm, 13.5 cm, 14.7 cm were used to induce the knitted fabric extension of 21.25%, 57.5%, 68.75%, 83.75%, respectively (***Figure 2***). The fabric surface coated with microcapsule was faced on the wall of the jar.

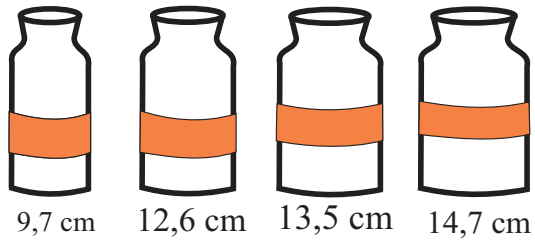

Figure 2 Fabric samples with microcapsules are wrapped in 4 glass vials with different circumferences of 9.7 cm, 12.6 cm, 13.5 cm, 14.7 cm, forming 4 different extension levels.

The fragrance evaluation process was carried out in two sample lots:

*The first sample lot: The knitted fabric band was extended by the jars for 1 hour before fragrance evaluation, which was carried out at the thermostatic cabinet (the temperature was 32°C, and the relative humidity was 58%). After 1 hour in extension, the knitted fabric bands were taken out from the jars and passed to odor evaluation. The fragrance evaluation was obtained 9 times for every 60 minutes from the beginning moment at 0 minute to 540 minutes. The odor panel includes 8 people aged from 21-25 years old who were students of Hanoi University of Science and Technology, they were 4 males and 4 females. The perfumers sniffed each sample in turn and compared it with 11 reference scent bottles on a scale of 0 to 100 rating points then given their personal point of the smell sample.

* The second sample lot: The fragrance evaluation process was similarly caried out to the first sample lot excepted that the during in extension before fragrance evaluation of the knitted fabric bands was 2 hours. The aim of longer duration of knitted fabric band extension was to investigate more detail the behavior of fragrance fabric treated by microcapsule contained oil essential.

3. Results and Discussion

1. Investigation of microcapsule morphology

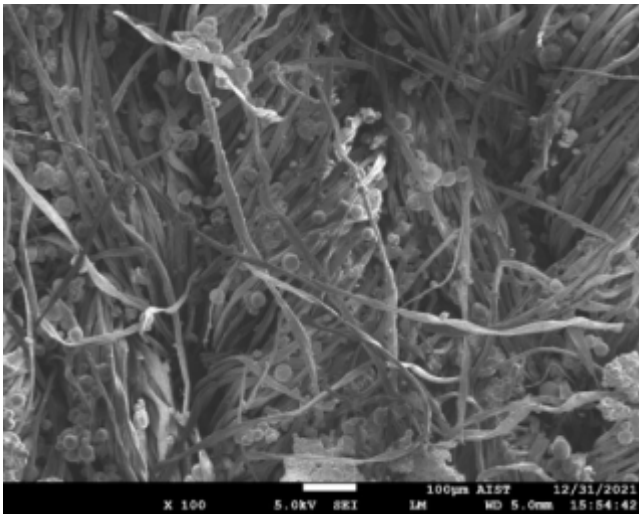

Figure 3 SEM images of knitted fabric surface with the coated microcapsules

Knitted fabrics were always prepared with low twist yarn that gave them specific characteristics such as soft, good on-air permeability and so far. When application of microcapsules on the knitted fabric, the small particles of tens micrometers size had been easily distributed on the surface of the knitted fabric but not deeply in the fabric structure (***Figure 3***). This microcapsule layer on fabric surface may be useful for liberation process of cinnamon essential oil from the knitted fabric band. Moreover, the microcapsules kept their spheric form. They did not be deformed and could guard the cinnamon essential oil for the liberation process.

The more important magnification of x 10 000 helps to observe the structure of the microcapsule membrane (***Figure 4***).

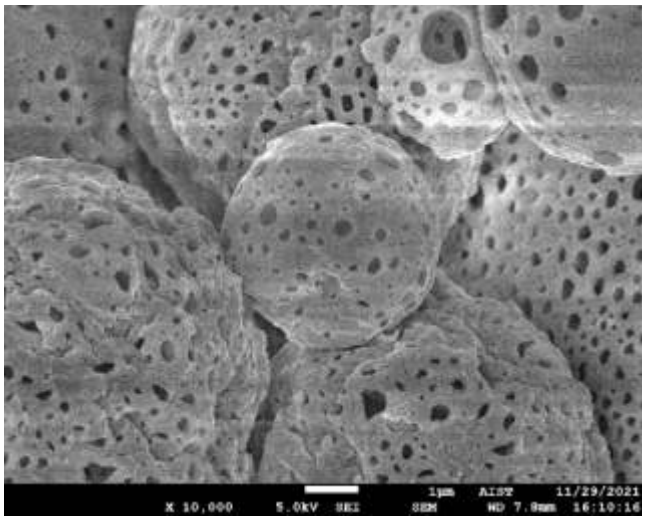

Figure 4 SEM images of microcapsule membrane

Many porous with the different dimensions from some tens nanometer to some hundreds nanometers had been observed in the microcapsule membrane that insists the porous structure polymer membrane of the Eugrarit RSPO microcapsules contained the cinnamon oil essential [15]. The quantity and the size of the porous could be controlled during the microencapsulation and so the liberation rate of oil essential may be change suitable to the application target [15].

2. Influence of fragrance agent kind on the fragrance durability of treated knitted fabric

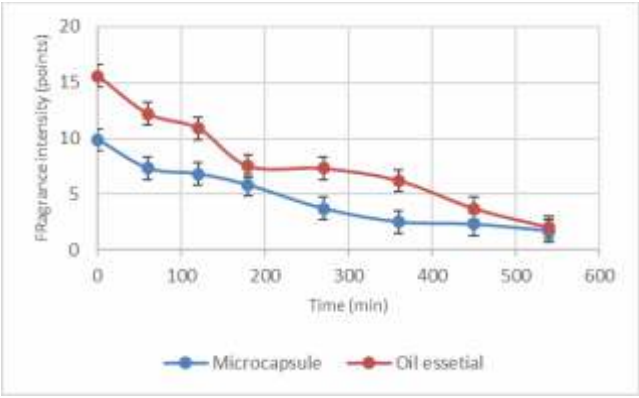

Figure 5 Odor strength of pure cinnamon essential oil evaluated for 540 minutes

In odor to investigate the efficacy of microcapsule on the control of oil essential liberation the pure cinnamon essential oil and the microcapsules containing cinnamon essential oil was used as fragrance agent in this test. The results of the fragrance stability between microcapsules containing cinnamon essential oil and pure cinnamon oil were given by graphs in ***Figure 5*** which showed that after 3 hours of evaluation (at 180 minutes), the odor intensity of the fabric sample treated by pure cinnamon essential oil was decreased from 15.6 points at the beginning to 7.5 points (decreased in 52%) while the odor intensity of that one coated by microcapsules cinnamon essential oil was in diminution from 9.9 points to 5.7 points (decreased in 42 %). It was mentioned that at the beginning, the difference in fragrance intensity between two kinds of knitted band was about 6 points. Moreover, in the time from 360 minutes to 540 minutes, the intensity of pure essential oil is reduced from 6.5 points to 2 points (decreased in 69%) while the odor intensity of cinnamon essential oil microcapsules decreased more slowly from 2.5 points to 1.5 points (decreasing in 40 %). The evaluation after 9 hours (at 540 minutes) showed that the odor concentration of cinnamon essential oil microcapsules was 1.6% and the concentration of pure cinnamon oil decreased to 2% with difference fragrance intensity was only 0.3 points, which showed that pure cinnamon oil evaporates very quickly and the cinnamon essential oil microcapsules were much more durable in keeping their fragrance. The lower diminution rate in odor intensity of the knitted fabric treated by the microcapsules contained the cinnamon essential oil demonstrated that the porous microcapsule membrane plays important role in retarding the evaporation of the cinnamon oil essential from the microcapsules coated in the fabric surface.

3. Investigation the influence of the extension on the fragrance durability of knitted fabric band coated by Eugrarit RSPO microcapsules contained the cinnamon oil essential

The evaluation of the fragrance intensity by two fabric lots after 1 hour and after 2 hours were presented in ***Figure 6***. The results showed that in general, the higher the jar size caused the the greater the pressure exerted on the jar wall, that made more the cinnamon essential oil to be released from the microcapsules via their porous membrane so the fragrance intensity of the knitted fabric band tended to decrease when their extension was increased. But for the first fabric lot (after one hour of extension), the odor intensity of

the knitted fabric band was obtained with 30.37 point, 34.50 points, 26.87 point and 29.75 point corresponded to the extension of at the minimum extension of 21.25%, 57.5%, 68.75%, 83.75%, not very clear tendency on odor intensity, which may be explained that one hour in extension was too short time to make the force on the every microcapsule to push the cinnamon essential oil capturing from the porous polymer membrane.

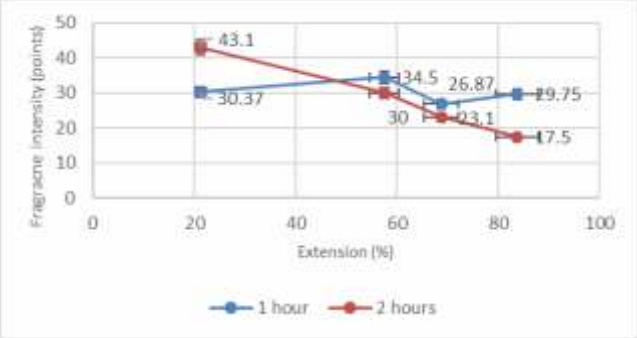

Figure 6 Fragrance stability of cinnamon essential oil microcapsules at the different extension of fabric.

The results of second lot (after two hour of extension) showed more clearly the decreasing tendency in fragrance intensity with higher elongation of the knitted fabric band when they sustained extension in two hours. The knitted band with the lowest extension of 21.25% obtained the maximum average fragrance intensity of 43.1 points, and the sample with the maximum extension of 83.75% had got the minimum average odor intensity of 17.5 points (Fig. 6). These results demonstrated that after two hours sustaining the pressure caused by knitted fabric extension, the force had transmitted though the fabric layer to the microcapsules and had forced the evaporation of the cinnamon essential oil through the porous membrane. The more extension applied, the more force imposed on the microcapsules and the more essential oil had evaporated from the knitted fabric band. In the consequence, the fragrance intensity of the fabric band had decreased when their applied extension was increased. The experimental results showed that liberation of active substance may be controlled from the microcapsule coated on the fabric by the extension applied on the fabric.

4. Conclusion

The problem of long-term retention of essential oil on the fabric surface for the purpose of preserving fragrance or promoting the necessary properties of the essentiel oil for a long time was one goal of the works. Cinnamon essential oil contains many healthful compounds but it is quickly evaporatetes and coumarin compounds easily cause skin irritation when exposed to large amounts of skin. Therefore, using microencapsulation in this study showed the good effectiveness of microcapsules contained the cinnamon essential oil for long-lasting fragrance on the fabric. Moreover, the fragrance intensity of microcapsules contained the cinnamon essential oil was showed decreasing with the higher compressive force corresponding to the larger extension applied on the fabric band. So, the liberation of the active agent such as essential oil from the microcapsules coated on the knitted fabric band could be controlled by fabric extension. The results could be applied on the therapy textile field and medical textile as compression garment.

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