KAUNAS UNIVERSITY OF TECHNOLOGY

DEIMANTĖ PLAŠČINSKIENĖ

DEVELOPMENT AND EVALUATION OF PROPERTIES OF TERRY FABRICS WITH AROMATIC MICROCAPSULES

Summary of Doctoral Dissertation
Technological Sciences, Materials Engineering (T 008)

Kaunas, 2021

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KAUNO TECHNOLOGIJOS UNIVERSITETAS

DEIMANTĖ PLAŠČINSKIENĖ

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INTRODUCTION

Microcapsules (MC) are used in many fields, such as medicine, food industry, textile industry. In all these areas, the capsules are attractive because of the ability to release the material embedded in them at a specific location and at the right time. In medicine, capsules are beginning to be used as drug delivery systems for treatment of various diseases, even cancer. They are used in food industry to embed fats, vitamins, minerals, flavors, dyes, and other substances. In textile industry, microcapsules are particularly widely used, because when applied to a textile product, they provide new properties, such as fire resistance, antibacterial, insect repellent, and fragrance. Cosmetic textiles is another use of capsules in the textile industry, where textiles, due to the active substance incorporated in the capsules, which is gradually released, can soften the skin, supply vitamins, tone the body, or emit odor. The capsules with embedded essential oils allow enjoying aromatherapy constantly. In addition, some essential oils as well have antibacterial effect, which is very important for products used in humid environments, where a favorable medium for the growth of bacteria and fungi appears.

Due to special structure of terry fabrics, they are most often used in humid environments (bathrooms, saunas, or kitchens), as their terry surface is able to absorb a larger amount of water, compared to the flat fabrics. Various textile finishing processes are used to create value-added products, one of which is the coating of terry cloths with scented microcapsules. For the products to be high quality, it is important to analyze how the finish of the microcapsules changes the structure and geometrical parameters, physical and mechanical properties of the terry fabric. One of the most important physical properties of terry fabrics is absorption and air permeability, as it depends on how comfortably the user will wear the product, how it will suit the environment in which it is intended to be used. Long lastingness, durability and appearance of the product depend on the mechanical properties.

The aim of the doctoral dissertation is to create ramie/cotton terry fabrics with flavor and investigate the influence of microcapsule finishing on the structure and properties of the terry fabrics.

The objectives of the research were the following:

– to evaluate the efficiency of attaching microcapsules to different type of fibers: cotton, ramie, artificial bamboo, linen, and bleached linen yarns;

– to determine the influence of microcapsule finishing on the structure and geometrical parameters of terry fabrics and bending rigidity;

– to determine the abrasion resistance and weight loss during the abrasion of terry fabrics that are treated with microcapsules with essential oil as well as resistance by applying the washing process.

–to evaluate the air permeability of terry fabrics treated by microcapsules and the air permeability of fabrics with microcapsules after the abrasion and washing process; to determine the dependence between the air permeability and weft density of fabrics that are treated with microcapsules at different binder concentrations, as well as the number of abrasion cycles;

– to investigate the resistance to the pile loop extraction of terry fabrics after the finishing and washing process of microcapsules; to determine the dependence between the loop extraction force and binder concentration;

– to determine the influence on the static and dynamic absorption of fabrics, water vapor absorption, and water evaporation process of terry fabrics after the finishing and washing process of microcapsules; to determine the dependences between the spot area and inspected period and between the remained water ratio and the drying time.

Scientific novelty and practical importance. With the growing interest of consumers in the added value of textile products that are made of natural fibers, the dissertation analyzes natural fiber yarns and ramie/cotton terry fabrics treated with microcapsules with eucalyptus essential oil. There is almost no literature on the microcapsule coating of different natural fiber yarns; the microcapsule-coated cotton fabrics are the most commonly analyzed. Therefore, it is important to investigate the possibilities of attaching microcapsules to yarns of different fibrous compositions. Ramie is a very soft and gentle fiber; thus, the products made from ramie yarn are light and comfortable. It is similar to flax fiber in its morphological structure and sorption properties and is more valued for the low creepiness and surface gloss. However, ramie fiber break cyclically flexed; therefore, it is often used with other natural, artificial, or synthetic fibers. In textile products, the combination of ramie fiber with cotton achieves better physical and mechanical properties. Cotton fiber has good sorption and strength properties. The terry fabrics with the most suitable fiber composition for microcapsule insertion were selected for the study. In order to create added value fabrics, ramie/cotton terry fabrics were treated with microcapsules with a core material of eucalyptus essential oil. There is no literature on terry fabrics treated with fragrant microcapsules; therefore, it is especially important to analyze the physical and mechanical properties of ramie fiber, which is rarely used in the production process. The qualitative indicators of terry fabrics with microcapsules were investigated in the dissertation. It is possible to predict the properties of fabrics with a complex structure by using the results of the research and mathematical dependencies. The results of the work can be applied to the prediction of quality indicators of other, similarly structured textile products with microcapsules and the development of new textile materials.

Approbation of the research results. The results of this research were presented in 7 scientific publications, including 4 articles published in *Clarivate Analytics Web of Science* listed journals with a citation index (IF/AIF>0.2), 3 of them in the international scientific conference proceedings. The results were presented at 5 international conferences.

Structure of the doctoral dissertation. This doctoral dissertation consists of an introduction, including objectives, literature overview, experimental part, 3 chapters of results and discussion, conclusions, a list of references (136 entries), and a list of scientific publications on the topic of the dissertation. The material of the dissertation is presented in 102 pages, including 7 tables and 55 figures.

CONTENT OF THE DISSERTATION

Introduction presents the relevance of the study, defines the aim and the research objectives, outlines the defended statements, and discusses the scientific novelty as well as practical significance of the doctoral dissertation.

The first chapter, Literature Review, introduces general information about the properties of microcapsules, manufacturing, coating possibilities of textile materials, analyzes methods of microcapsules loaded on textile materials and properties of terry fabrics.

The second chapter, Research Methodology, describes the object of the research and the methodologies of the experimental investigations.

Experimental materials

The spun yarns of five different fibers were selected for the impregnation with microcapsules: cotton (50 tex), bleached linen (50 tex), grey linen (68 tex), artificial bamboo (74 tex), and ramie (67 tex). Terry fabrics (Table 1) that were analyzed in the experimental work were made from ramie/cotton yarns. The impregnation of yarns and terry fabrics was performed under conditions shown in Table 2 of commercial microcapsules, containing eucalyptus essential oil (LJ Specialities, UK). The terry fabrics with MC were affected by domestic washing and drying procedures, investigating 5–25 cycles (for $S_{5\text{-}25}R10{,}5_{160}I_{80}$ samples). The domestic washing procedure was performed according to ISO 6330:2012 standard.

Table 1. Characteristics of ramie/cotton terry fabrics

Fabric variant	Pile high, mm	Nominal linear density, tex			Fabric density, dm^{-1}	
		Ground weft	Ground warp	Pile warp	Pile and ground warp	Weft
$R6_{80}$	6	cotton, 50	plied cotton, 2 x 25	ramie, 67	250	80
$R6_{100}$						100
$R6_{120}$						120
$R6_{140}$						140
$R6_{160}$						160
$R10{,}5_{160}$	10.5					160

Table 2. Conditions of microcapsule impregnation

<table>
<tr><th rowspan="2">Prepared samples</th><th rowspan="2">Concentration of the microcapsule, g/l</th><th rowspan="2">Binder concentration, g/l</th><th colspan="2">Drying and fixation temperatures, ºC</th></tr>
<tr><td>drying</td><td>fixation</td></tr>
<tr><td rowspan="6">cotton, bleached linen, grey linen, bamboo, ramie yarns</td><td rowspan="12">30</td><td rowspan="6">80</td><td rowspan="12">100–110</td><td>120</td></tr>
<tr><td>130</td></tr>
<tr><td>140</td></tr>
<tr><td>150</td></tr>
<tr><td>160</td></tr>
<tr><td>170</td></tr>
<tr><td rowspan="6">ramie/cotton terry fabrics</td><td>20</td><td rowspan="6">150</td></tr>
<tr><td>35</td></tr>
<tr><td>50</td></tr>
<tr><td>65</td></tr>
<tr><td>80</td></tr>
<tr><td>95</td></tr>
</table>

Experimental methodology

All samples were conditioned under standard atmospheric conditions according to standard LST EN ISO 139:2005, e.g., 20±2 °C temperature and 65±4 % relative humidity for 24 h before the experiments.

The surface and morphology of microcapsules as well as the yarns and terry fabrics that were treated were examined by scanning electron microscopy (SEM) Hitachi S-3400N (USA) and Quanta 200 FEG (USA) at the acceleration voltage of 5–20 kV.

The mass and linear density of the yarns were determined by using electronic scales KERN EW 150-3M (KERN & Sohn GmbH, Germany) at the accuracy of 0.001 g.

In order to identify the properties of terry woven fabrics, the following structural, physical, and mechanical parameters of these terry woven fabrics have been investigated:

Structural and geometric parameters of terry woven fabrics

Area density of woven fabrics. The woven fabric area density has been identified referring to LST EN 12127:1999 standard (“Textiles - Fabrics - Determination of Mass per Unit Area Using Small Samples”).

Thickness of fabric was set according to the standard LST EN ISO 5084:2000 by Textil-Dickenmesser DPT 60 digital instrument (Hans Schmidt & CO GMBH, Germany).

Physical properties of terry fabrics

Static water absorption was measured according to method BV S1008 "Bureau Veritas Consumer Products Service BV S1008". The static water absorption was calculated according to the following equation:

$$W_{st} = \frac{m_w - m_d}{m_d} \times 100; \quad (1)$$

where W_{st} is the static water absorption, %; m_w is the weight of wet sample, g; m_d is the weight of dry sample, g.

Dynamic water absorption experiments were performed by Petrulytė and Baltakytė (2009) methodology, using SMZ 800 Nikon Stereoscopic Microscope and Coolpix 4500 Digital Camera. The absorption process was filmed from the start moment until the last moment, i.e., from the moment when the drop of distilled water (of 0.110 g) fell onto the surface of the fabric, until it was absolutely absorbed by the fabric. The experiments were performed by filming from the upper side of the fabric. The areas of the liquid spots were measured by investigating the pictures of video records, and the changes in the spots areas over the time were calculated according to the following equation:

$$W_{din} = \frac{M - PM}{PM} \times 100; \quad (2)$$

where W_{din} is the change in spot area, %; M is the change in the spot area at intermediate moment, mm^2; PM is the change in the spot area at the start moment, mm^2.

Water vapor absorption in Woven Fabrics. The water vapor absorption investigation was conducted referring to LST EN 13515:2004 standard.

Water evaporation from Woven Fabrics. The water vapor evaporation investigation was carried out referring to FTTS-FA-004 standard, in which the information on the investigation of the fluid evaporation process by using the drop method is provided.

Air permeability of woven fabrics. The air permeability testing was conducted by referring to LST EN ISO 9237:1997 standard, defining the method of measuring air permeability in flat textile fabrics. The air permeability testing was performed by using the LI4DR instrument (Karl Schroder KG, Germany). The air permeability was calculated according to the following equation:

$$R = \frac{q_v}{A} \times 167; \quad (3)$$

where LO is the air permeability, $dm^3/(m^2 \cdot s)$; q_v is the airflow rate, dm^3/min; A is the tested area, cm^2; 167 is the calculation from $dm^3/(cm^2 \cdot min)$ to $dm^3/(m^2 \cdot s)$ coefficient.

Mechanical properties of terry fabrics

Bending rigidity was investigated by using a standard non-contact device, referring to standard methodology that is corresponding to ISO 9073-7 standard.
Resistance to pile loop extraction of Terry Fabrics. The experiments were performed according to LST EN 15598:2008 standard. The resistance to pile loop extraction was assessed by using the Zwick/Z005 (Zwick GmbH & Co. KG, Germany) stretch testing machine and testXpert® software.
Abrasion resistance of Woven Fabrics: Specimen Mass Loss. This method is based on the following standard: "Textiles - Determination of the Abrasion Resistance of Fabrics by the Martindale Method - Part 3: Determination of Mass Loss" - LST EN ISO 12947-3:2001.
The third chapter, Results of Investigations, presents the results of the experimental investigations and their analysis.

Investigations of possibilities to graft microcapsules with eucalyptus essential oil on natural fibers

Traditionally, the fixation of microcapsules (MC) onto textiles is achieved by applying a binder and the thermal curing procedure, during which the binder is converted into a tough polymer that forms a network on the textile to hold the microcapsules. In this work, the yarn samples that were grafted with eucalyptus microcapsules were analyzed by SEM: mass and linear density of the yarns were determined. SEM micrographs with MC that were adhered on cotton, bleached linen, grey linen, ramie, and bamboo yarns are shown in Figure 1. It could be seen that the impregnation on all yarns and microcapsules that was investigated was effective on the surface of the fibers as well as in the deeper layers between the fibers.

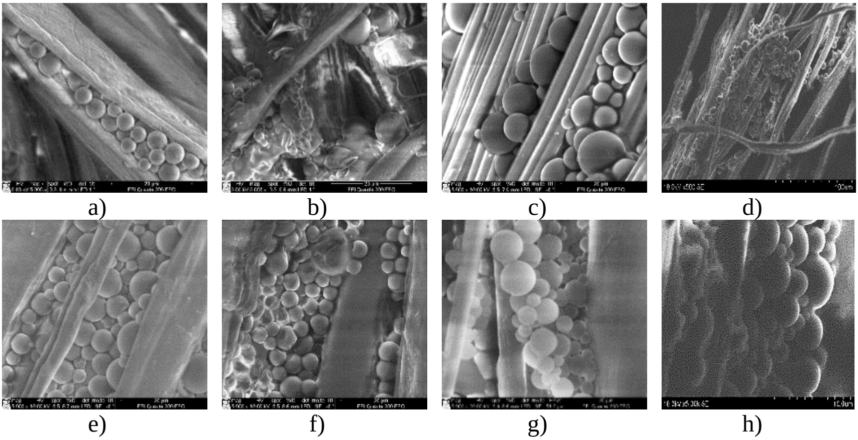

Figure 1. SEM micrographs of different yarns at different thermos-fixation temperatures (T_F) (magnification x5000): a) cotton yarn, when T_F is 120 ºC; b) cotton yarn, when T_F is 170 ºC; c) bamboo yarn, when T_F is 150 ºC; d) grey linen yarn, when T_F is 150 ºC; e) ramie yarn, when T_F is 140 ºC; f) ramie yarn, when T_F is 170 ºC; g) bleached linen yarn, when T_F is 140 ºC; h) bleached linen yarn, when T_F is 160 ºC

The presence of obviously unchanged eucalyptus MC could be seen on the yarns that were investigated when they were treated with a thermo-fixing temperature (T_F) of 120–140 °C. As it can be seen from Figures 1 a, c–e, g, the MCs have regular spherical shape and have no deformations in their shell. A thermo-fixing temperature of 120–150 °C caused no changes in the MC on yarns of various fibers, since the soft and smooth surface of eucalyptus MC could be observed. Moreover, it has been noticed that generally, the MC are distributed individually without significant agglomeration. As surfaces of grey and bleached linen fiber and ramie fiber are not smooth enough, the MCs are deposited on specific contact surface. Because of their specific structure, cotton fibers adhered to MCs on the surface and between fibers as well as in the inner fiber cavities and sags (see Figure 1 a). Bamboo fibers were attached to the MCs well, but the smooth surface of the fibers conditioned the enveloping character of the fixation. The Figures 1 c, d show that few misshapen microcapsules could be seen when the T_F was increased up to 150 °C. Meanwhile, Figures 1 b, f show yarns that are heated to 170 °C, in which surface damage to MCs is clearly noticed, being a typical appearance for many items; some MC were deflated and cannot be observed as spheres. Besides this, some MCs were broken entirely and became empty after such treatment, confirming that MCs remained on the fibers and even inner fiber cavities; but the fragrance had partly gone, and in single cases, even absolutely gone. From the SEM analysis, it has been determined that with an increase in T_F, generally, the larger MCs were damaged faster and then the smaller ones hardly at all. It is very

relevant to investigate what changes happen in the fiber mass after the treatment with MCs and the binder. The add-on as a significant index for textile impregnated with MCs was analyzed by other authors as well (Marti et al., 2012; Salaun et al., 2010), investigating the amount of capsules and active agents in fibers and quantifying them by the weight changes between the untreated and treated with MCs fabrics.
This study shows that there is an increase in the mass (Δm) of different fiber composition yarns: cotton, grey linen, bleached linen, ramie, and bamboo yarns after treating with eucalyptus MCs at different T_F. It has been found that the mass of cotton yarns after the treatment with MCs changed by 2.7–3.9 %. The Δm of bleached linen and ramie yarns after the treatment changed by 7.1–17.3 % and 10.1–13.0 %, respectively. The change in the mass of bleached linen and ramie yarns is important with the highest increase at 160 and 140 ºC T_F, respectively. Whereas the change in the mass for cotton yarns has the lowest value (2.7 %) at a thermo-fixing temperature of 170 ºC. Additionally, a substantial difference in the increase in the mass was found by analyzing both linen yarns: the Δm of bleached linen yarns was 11.1 % higher compared with the grey ones, when the T_F was 160 °C. The microcapsule shell material cannot interact with the surface of the fiber; hence, a polymeric binder is used to attach MCs to the fiber surface, making a thin layer, which holds MCs for a longer time. Since the Δm of ramie yarns were found to be higher compared to the grey linen, bleached linen, and bamboo yarns, and bearing in mind the well-known advantages of ramie for loop making (highly absorbent, comfortable to wear, especially in a warm and damp environment, durable, resistant to rotting and mildew, withstanding high water temperatures during laundering, etc.) and the popularity of cotton for ground sets, the ramie/cotton fiber combination was used for weaving terry fabrics.

Investigation of MC treatment on terry fabrics area density and thickness

In this experiment, terry fabrics were treated with MC by using different binder concentrations. The experimental area density (PT) results were mathematically determined, and it has been found that the area density of terry fabrics increased to 282.9–486.3 g/m^2, when studying terry fabrics of different weft densities and impregnated with binder concentration of 20 g/l. By treating the binder concentration with 95 g/l, the PT of terry fabrics increased to 292.6–510.3 g/m^2. The regression analysis was performed, and the informativeness of the design of the PT of terry fabrics that were treated, taking into account the weft density and the binder concentration, was investigated. Using the informativeness criteria (R.A. Fisher's criterion), it has been found that the obtained mathematical models that are expressing the dependence of the area density of the treated terry fabrics and the weft density are informative at a binder concentration of 20 g/l and 95 g/l. The finish of the MCs has an effect on the thickness of the fabric, as the binder stiffens the loops of the fabric, which remain on the stand during the compression and are able to withstand the load. The thickness of the grey terry fabrics varied from 2.09 to 3.43 mm, whereas for the treated with MCs terry fabrics, this value ranged from 2.87 to 4.75 mm, depending on the binder concentration and weft density. When analyzing the influence of the washing process on the fabric thickness, it has been found that the thickness of terry fabric decreases after 25 washing cycles from 11.57 % to 8.01 % at different compressions.

Investigation of MC treatment on terry fabrics bending rigidity

The bending rigidity (BR) of terry fabrics that were treated with MCs, regarding the weft density and binder concentration, showed an increase compared to the grey terry fabrics. The bending rigidity of grey terry fabrics varied from 0.65 to 1.33 Nm^2 when analyzing the warp direction and from 0.46 to 1.16 Nm^2 for the weft direction, regarding all intervals of weft density that were investigated. Meanwhile, for the treated samples, it has been found that BR increased from 5.57 ($R6_{100}I_{20}$ variant) to 12.94 Nm^2 ($R6_{140}I_{95}$ variant) when analyzing the warp direction and from 2.35 ($R6_{80}I_{50}$ variant) to 10.78 Nm^2 ($R6_{140}I_{95}$ variant) when analyzing the weft direction. As the binder filled the voids, interstitial cavities, inter-fiber and inter-yarn spaces, sealing the pores of the fabric, the textile became rigid. Besides this, the BR increased by 5.3–14.0 times in the warp direction and by 5.1–12.8 times in the weft direction when varying the binder concentration at all intervals that were investigated compared with the grey fabrics. It means that the terry fabrics tended to be considerably more resistant to bending due to the treatment with MCs and binder (Petrulyte et al., 2017a).

Abrasion resistance of terry fabrics

The influence on the abrasion resistance of ramie/cotton terry fabrics depends on the binder concentration. By changing the binder concentration from 20 g/l to 95 g/l, the weight loss of the $R6_{80}I_{95}$ variant was found to be 3.78 % lower than $R6_{80}I_{20}$ variant, after 25,000 abrasion cycles.

The research deals with the abrasion properties of grey fabric as well as MC treated fabrics and washed ones (5–25 washing cycles). The appearance of the terry fabric changed after the abrasion, i.e., after 25,000 abrasion cycle loops on the surface of the terry fabric are removed, the fabric became finer, but no holes appeared in the fabric. When the number of abrasion cycles increases, the mass loss of the fabric increases because of the removing of the loops. During all investigated abrasion cycles, the mass loss of the fabric with MCs without washing decreased up to 88.9 % compared with grey one. After 100–25,000 abrasion cycles, the mass loss changed up to 5.8 % and up to 16.3 %, investigating fabric with MCs without washing and the washed fabric (25 washing cycles), respectively. After 25,000 abrasion cycles, the mass loss increases up to 1.33 times and up to 1.97 times when investigating fabric with grey one and after 5 and 25 washing cycles, respectively.

Investigation of MC treatment on terry fabrics air permeability

Air permeability is one of the prime factors affecting the thermal comfort of the wearer in clothing and textiles. The air permeability of each textile sample that has been performed by using different binder concentration was measured and compared in this research. The decrease of air permeability after impregnation is presented in Figure 3. The maximum value of the air permeability treated with MCs terry fabric was 1146.7±38.7 mm/s ($R6_{80}I_{95}$), and the minimum value was 228.2±24.1 $dm^3/(m^2 \cdot s)$ ($R6_{160}I_{50}$). The results proved that the treatment of fabric with MCs and binder affects the structure of the fabric. After the treatment with MCs, the gaps in terry fabric loops and yarns change. The changed structure of the textile causes air permeability.

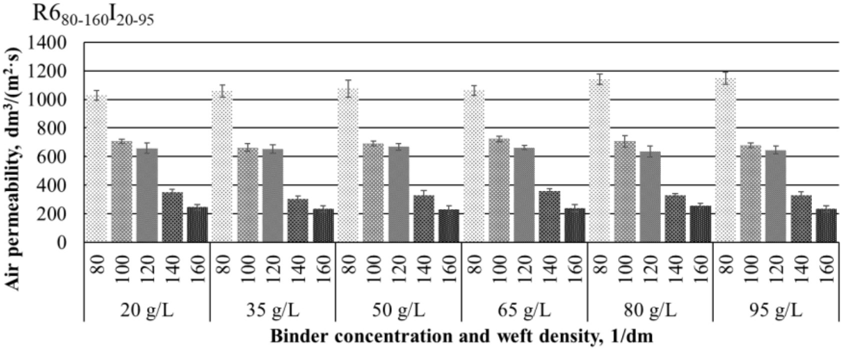

Figure 2. The air permeability of ramie/cotton terry fabrics after MCs treatment

In the investigation of air permeability of terry fabrics after the abrasion, it has been found that when increasing the number of abrading cycles, initially, the air permeability decreases. The air permeability of the $R6_{80}I_{20}$ and $R6_{80}I_{95}$ variants reduced by about 40 % and up to 87 % after 1,000 and 25,000 abrasion cycles, respectively, compared to the non-mechanically exposed fabrics. The air permeability of $R6_{160}I_{20\text{-}95}$ variants decreased by 1–30 % and 54–82 %, respectively, after 1,000 and 25,000 abrasion cycles, compared to the specimens that were not abraded. When investigating ramie/cotton terry fabrics with different washing cycles ($S_{5\text{-}25}R10{,}5_{160}I_{80}$), the air permeability decreased by 1.8 times, compared with grey terry fabrics. Investigating the air permeability of terry fabrics treated with MC after the washing process revealed that the air permeability decreases with increasing number of washes. When washing terry fabrics treated with MC, the loops retain their stable shapes, the fabric does not change its appearance, but it is less air permeable compared to the grey fabrics.

Resistance to pile loop extraction of terry fabrics

The pile area of terry textile is very significant; thus, the poor resistance of pile yarns against pullout is one of the most important problems of quality of terry fabric structures. This study deals with the displacements and forces associated with pulling a loop from the terry woven fabric, regarding the fabric structure and treating with aroma-microcapsules. The influence of treatment with MC of terry textile is visible in the increased stiffness. If to assess the effect of the modification of the structure of the fabric, the measurements of the resistance to pile loop extraction were carried out before and after the deposition of aroma-micro-spheres on the textile. The obtained results proved that the difference between the resistance to pile loop extraction for the grey and modified terry fabrics depends on the changed structure. The lower values in the change (by 0.4–1.1 times) of resistance to pile loop extraction were obtained for the terry fabrics with maximum weft density, i.e., of 160 dm^{-1}, and the differences generally increased with the increase of binder's concentration from 20 to 95 g/l. Whereas the higher values in the changes (even up to 2.8–3.6 times) were determined for the lower weft densities of the fabric, i.e., 80–100 dm^{-1}. Finally, it was concluded that the weft density of terry woven fabric treated with microcapsules and different binder concentration affected the loop yarn pullout (Petrulytė et al., 2017b).

The resistance of terry fabrics to pile loop extraction is discussed, taking into account the fabric structure and the effect of washing. The washing with detergent changed the structure of the fabric because the fabric was affected by a variety of factors during the washing cycle: chemical, mechanical, and heat impacts. As a result, after washing, the gaps between the loops and the yarn changed, and the loops became hairy, bulky, and fluffy. In addition, the fabric structure has become more compact. The high number of washes resulted in the loss of the loose fibers from the yarns.

The resistance to pile loop extraction of fabrics treated with MC and washed for 5–25 wash ranged from 694.93 mN (pull distance 5 mm) to 5516.79 mN (pull distance 25 mm). In addition, a consistent increase in the effect of washing cycles was found: with the increase of washing to 10, 15, 20, and 25 cycles, the resistance to pile loop extraction increased by 4.5 and 8.8 %, 40.3 and 47.1 %, 53.4 and 57.6 %, 71.0 and 79.4 %, respectively, compared to the samples washed by 5 washing cycles.

Investigation of MC treatment on terry fabrics sorption properties

One of the main functions of terry textile products is to absorb moisture, which depends on the fabrics warp and weft density, the height of the loop as well as the raw material and the impact/finishing. The dynamic and static water absorption, water vapor absorption, and water evaporation were investigated for ramie/cotton terry fabrics. The aim of the research was to determine how the dynamic

absorption of terry fabrics changes after the MC treatment and what effect on the dynamic absorption has the washing process of fabrics treated with the MC. When analyzing the dynamic absorption process of ramie/cotton terry fabrics, it has been observed that the drop applied to the grey fabric loses its round shape in the first few seconds. A significantly slower process takes place on the fabric treated with fragrant microcapsules where the drop retains its shape for up to 40 s. The process of absorption of grey terry fabric proceeds quite rapidly: the drop of water is absorbed during the first 10 s, but the absorption process up to 30 s of the observation period is intense as well. The studies showed that by this observation moment (30 s), the spot area had increased by 38.3 %. The $ZR10.5_{160}$ variant, after 70 s observation time, increased the spot area to 45.3 % from the start moment. Such dynamic absorption results may have been determined by the loop height (10.5 mm) and the ability of the ramie fiber to absorb moisture intensively, because the high loops form larger surface volume absorbing water. The spot area of grey terry fabric varied from 72.6 to 133.5 mm^2 throughout the observation period.
The dynamic absorption process of terry fabric with MC treatment is slow, and the change in the spot area is recorded for up to 600 s. The studies showed that the change in the spot area up to 10 s is 1.2 % with an increase in the observation time up to 70 s; the spot area increases up to 7.6 % compared to the start moment. The spot area of the $R10,5_{160}I_{80}$ variant varied from 38.4 to 78.2 mm^2 throughout the observation period.

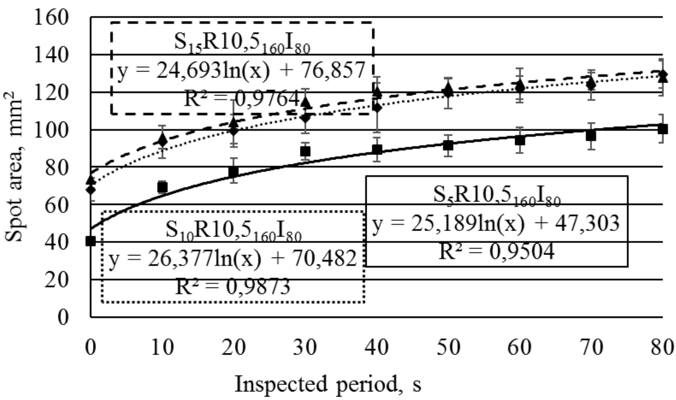

Figure 3. The spot area of ramie/cotton terry fabrics after 5, 10, and 15 washing cycles

When determining the changes in the dynamic absorption of terry fabric with MC treatment after washing (Fig. 3), it has been observed that the dynamic absorption process is increasing up to 10 washing cycles, and after 15 washes, the absorption begins to decrease. After 5 wash cycles, the absorption increases by 43.9 % and

57.3 %, compared to the R10.5$_{160}$I$_{80}$ variant after 10 s and 80 s of observation time, respectively. After 10 wash cycles, the spot area increased by 41.4 % after 10 s of observation time, compared to the R10.5$_{160}$I$_{80}$ variant. The spot area of the samples after 20 and 25 wash cycles increased from 80.9 mm^2 to 105.9 mm^2 and from 62.3 mm^2 to 99.8 mm^2 after 80 s. During the washing process, terry fabrics are subjected to water, mechanical, thermal, and chemical effects that affect the changes in fabric and loop structure.

The static water absorption studies of ramie/cotton terry fabrics showed that the absorption decreases by 37.4 % after the MC treatment. An increase of absorption has been observed after the washing process. After 5 wash cycles, the absorption was increased by 2.9 %, compared to the fabrics with MC treatment. Increasing the number of wash cycles to 25, the absorption of the terry fabric reduces. The static water absorption is affected by the washing process, which changes the porosity and structure of the fabric. During the washing, the fabric under the influence of mechanical, thermal, and chemical effects changes the shape of the loops and removes some of the fibers and chemicals that are used in the MC treatment.

The water vapor absorption studies of ramie/cotton terry fabrics have shown that the water vapor absorption increases by 10 % after the terry fabric treatment with MC. The washing process gives fabrics better water vapor sorption properties. After 25 washing cycles, the water vapor absorption of terry fabrics increased by 25.2 %.

In a study of the water evaporation process of terry fabrics, it has been found that the evaporation process took up to 120 min from the start moment until the absolute water evaporation for grey fabric and the treated fabric with MC without washing. The water evaporation for treated fabrics lasted 10 min shorter (i.e., till 110 min) and then washed after 5 washing cycles, whereas for the fabrics after 10–20 washing cycles, the water evaporated till 130 and 110 min, respectively. The moisture transport passed faster when analyzing the terry fabrics, which were washed 25 washing cycles, i.e., 100 min was the last period. It has been found that when analyzing 40th minute of the evaporating period of grey terry fabric, the remained water ratio was 54.83 %, and it is the highest value of all variants. Furthermore, the remained water ration for terry fabric after the treatment with MCs without washing was 52.74 %. Whereas when treated terry fabrics were affected by 25 washing cycles, the remained water ration was the lowest (40.93 %), and the difference was 13.9 %, compared with ZR10.5$_{160}$ variant. Analyzing different fabric structures, the fabrics show similar character of water evaporating over the time. Meanwhile, the washed terry textile demonstrated different particularity of water evaporation: the drying rate of R10.5$_{160}$I$_{80}$ fabric variant is the slowest, compared with all other variants, analyzing 10–60 min of the evaporation period.

CONCLUSIONS

1. The attachment of microcapsules to cotton, ramie, bamboo, linen, and bleached linen fibers was effective.

• The weight of bleached linen yarn after the microcapsule coating increased by 17.3 % at a fixation temperature of 160 ºC, but the deformations and damage to the capsule walls have been observed as well. Due to the high fixation temperature, the core component of the MC, i.e., the eucalyptus essential oil, can be released.

• The effective attachment of the MC has been established with ramie yarn. The microcapsules are attached to the surface of the fiber and are inserted into the gaps in the fiber.

2. The MC treatment of ramie/cotton terry fabrics affects the fabric structure and bending rigidity.

• The MC treatment of terry fabrics increases the area density. Depending on the weft density and the binder concentration, the area density of ramie/cotton terry fabrics increased from 8.2 % to 27.1 %, compared to the grey fabrics. The relationship between the weft density and surface density were determined by linear equations (R^2 up to 0.8937), when terry fabrics were treated with microcapsules using binder concentrations of 20 and 95 g/l.

• The MC finish increases the thickness of the loop fabrics from 20 % to 45 %, because the binder stiffens the loops of the fabric, which are able to withstand the applied load during the compression.

• The MC finish for terry fabrics increases the bending rigidity of terry fabrics. Comparing terry fabrics with MC treatment with grey fabrics, it has been found that the bending rigidity increases up to 14 times in the warp direction and up to 12.8 times in the weft direction. The relationship between bending rigidity and the weft density was determined by a linear equation (R^2=0.8100) when terry fabric was treated with microcapsules at a binder concentration of 80 g/l.

3. The resistance to abrasion of terry fabrics depends on the weft density and the finish given to the fabric. After MC treatment, the fabrics became more resistant to abrasion, but the washing process reduces the resistance to mechanical impact. The weight loss after 5 washing cycles was found to be 1.33 times higher than for the untreated fabrics and 1.97 times higher than for fabrics treated with MC at 25,000 rpm. A strong relationship has been found between the mass loss of terry fabric during the abrasion and the number of abrasion cycles, described by the polynomial equations with a determination coefficient of R^2 up to 0.9989.

4. When analyzing air permeability of terry fabrics, it has been found that the MC and binder treatment for terry fabrics has a negative effect on the air permeability of fabrics. The binder reduces the gaps in the yarns and between the yarn systems, increasing the area density of the fabrics. The air permeability of the fabrics after the abrasion process is reduced to 80 %, and after 25 washing cycles, reduced to 70 %, compared to the fabrics with MC treatment. The strong relationship between the air permeability and weft density was found in terry fabrics that were treated

with microcapsules at different binder concentrations, and it is described by linear equations (R2=0.9201–0.9604).

5. The resistance to pile loop extraction depends on the fabric structure properties and the binder concentration. The MC treatment increases the resistance to pile loop extraction by 2.1 and 1.5 times at pullout distances of 5 and 25 mm, respectively. The washing process increases the resistance to pile loop extraction. It has been found that the extraction force after 25 washing cycles is 3.9‒3.4 and 1.9‒2.3 times higher, compared to the grey fabrics and the fabrics treated with MC with an extraction distance of 5‒25 mm. The dependence between the pulling force of pile loop and the binder concentration is described by the linear equations (R^2=0.7620–0.7969).

6. The sorption properties of ramie/cotton terry fabrics are influenced by the microcapsule treatment and the washing procedure.

• The dynamic absorption process in terry fabric with MC treatment is slower compared to the grey fabrics. When analyzing the changes in dynamic absorption after the washing process, it has been observed that the absorption increases up to 10 washing cycles, and after 15 washes, the absorption begins to decrease. The determined dependence of the change in the spot area at the observation time is described by logarithmic equations with the determination coefficient R^2 up to 0.9847.

• The static water absorption of terry fabrics reduces after the microcapsules treatment by 37.4 %.

• After the MC treatment, the water vapor absorption increased by 10.0 %, and after 25 wash cycles, it increased by 25.2 %, compared to the grey fabrics.

• The water evaporation studies have shown that the evaporation process is more affected by the number of wash cycles than the MC treatment. The relationship between the remained water ratio and the drying time was determined and described by logarithmic equations (R^2=0.9400–0.9983).

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PRESENTATION OF SCIENTIFIC RESULTS IN SCIENTIFIC CONFERENCES

1. Worldwide Conference "14th Autex World Textile Conference", 2014, Bursa, Turkey, poster presentation.
2. International School-Conference "16-th International Conference-School Advanced Materials and Technologies", 2015, Palanga, Lietuva, poster presentation.
3. Worldwide Conference "16th Autex World Textile Conference", 2016, Liubliana, Slovenija, two poster presentations.
4. International School-Conference "17-th International Conference-School Advanced Materials and Technologies", 2016, Palanga, Lietuva, poster presentation.
5. International Conference "Baltic Polymer Symposium 2016", 2016, Klaipėda, Lietuva, poster presentation.

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Education:

1994–2007 Kaunas, Dainavos Secondary School.
2007–2011 Bachelor studies of Industrial Engineering, Faculty of Design and Technologies, Kaunas University of Technology.
2011–2013 Master studies of Polymers and Textile Engineering, Faculty of Design ant Technologies, Kaunas University of Technology.
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REZIUMĖ

Mikrokapsulės naudojamos daugelyje sričių, tokių kaip medicina, maisto pramonė, tekstilės pramonė. Visoms šioms sritims kapsulės patrauklios dėl galimybės jose įterptą medžiagą atpalaiduoti tam tikroje vietoje ir tinkamu laiku. Medicinoje kapsulės pradedamos naudoti kaip vaistus pernešančios sistemos įvairioms ligoms gydyti, net ir vėžinėms. Maisto pramonėje naudojamos riebalams, vitaminams, mineralams, kvapams, dažams ir kitoms medžiagoms įterpti. Tekstilės pramonėje mikrokapsulės naudojamos ypač plačiai, nes padengus jomis tekstilės gaminį, suteikia naujų savybių, tokių kaip: atsparumas

užsiliepsnojimui, antibakteriškumas, vabzdžių atbaidymas, spalvos keitimas, kvapas. Kosmetinė tekstilė gali būti atskira kapsulių panaudojimo sritis tekstilės pramonėje, kai tekstilės medžiagos dėl kapsulėse įterptos veikliosios medžiagos, kuri palaipsniui atpalaiduojama, gali minkštinti odą, tiekti vitaminus, tonizuoti organizmą ar skleisti kvapą. Kapsulės su įterptais eteriniais aliejais leidžia nuolat mėgautis aromaterapija. Be to, kai kurie eteriniai aliejai turi ir antibakterinį poveikį, o tai labai svarbu gaminiams, naudojamiems drėgnoje aplinkoje, kur palanki terpė daugintis bakterijoms ir grybeliams.
Kilpiniai audiniai dėl savo ypatingos sandaros dažniausiai naudojami drėgnoje aplinkoje (vonios, saunos patalpose ar virtuvėje), nes savo kilpiniu paviršiumi geba absorbuoti didesnį kiekį vandens, palyginti su lygiais audiniais. Siekiant sukurti gaminius su pridėtine verte, naudojami įvairūs tekstilės medžiagų apdailos procesai, vienas jų – kilpinių audinių padengimas kvapiosiomis mikrokapsulėmis. Kad gaminiai būtų kokybiški, svarbu išanalizuoti, kaip mikrokapsulių apdaila keičia kilpinio audinio sandaros ir geometrinius rodiklius, fizikines ir mechanines savybes. Vienos svarbiausių kilpinių audinių fizikinių savybių yra absorbcija ir laidumas orui, nes nuo jų priklauso kaip komfortabiliai vartotojas dėvės gaminį, kaip jis tiks aplinkoje, kurioje yra skirtas naudoti. O nuo mechaninių savybių priklauso gaminio ilgaamžiškumas, patvarumas ir išvaizda.

Darbo tikslas ir uždaviniai

Šio darbo **tikslas** – sukurti aromatinius ramės/medvilninius kilpinius audinius ir ištirti mikrokapsulių apdailos įtaką kilpinių audinių struktūrai ir savybėms.
Šiam tikslui pasiekti buvo iškelti *uždaviniai*:

- įvertinti mikrokapsulių prisitvirtinimo efektyvumą prie natūralių pluoštų: medvilnės, ramės, bambuko, lininių ir balintų lininių verpalų;
- nustatyti mikrokapsulių apdailos įtaką kilpinių audinių sandaros ir geometriniams rodikliams bei audinių lenkiamajam standumui;
- ištirti kilpinių audinių, padengtų mikrokapsulėmis su eteriniu aliejumi, atsparumą dilinimui, taip pat audinių, padengtų mikrokapsulėmis ir paveiktų skalbimo proceso, atsparumą dilinimui, nustatant masės nuostolius;
- įvertinti kilpinių audinių, padengtų mikrokapsulėmis, laidumą orui, bei audinių, padengtų mikrokapsulėmis, laidumą orui po dilinimo ir skalbimo proceso. Nustatyti audinių padengtų mikrokapsulėmis, esant skirtingoms rišiklio koncentracijoms, priklausomybes tarp laidumo orui ir ataudų tankumo, taip pat dilinimo ciklų skaičiaus;
- išanalizuoti kilpinių audinių mikrokapsulių apdailos ir skalbimo proceso įtaką pūko kilpos atsparumui ištraukimui. Nustatyti priklausomybę tarp kilpos ištraukimo jėgos ir rišiklio koncentracijos;
- nustatyti kilpinių audinių mikrokapsulių apdailos ir skalbimo proceso įtaką audinių statinei ir dinaminei absorbcijai, vandens garų absorbcijai ir audinių

džiūvimo procesui. Nustatyti priklausomybes tarp dėmės ploto ir stebėjimo laiko bei tarp vandens likučio koeficiento ir audinio džiūvimo trukmės.

Darbo mokslinis naujumas ir praktinė vertė

Didėjant vartotojų susidomėjimui tekstiliniais gaminiais iš natūralių pluoštų ir turinčiais pridėtinę vertę, disertacijoje pasirinkta analizuoti natūralių pluoštų verpalus ir ramės / medvilninius kilpinius audinius, padengtus mikrokapsulėmis su eukaliptų eteriniu aliejumi. Literatūros apie skirtingų natūralių pluoštų verpalų padengimą mikrokapsulėmis beveik nėra, dažniausiai analizuojami mikrokapsulėmis padengti medvilniniai audiniai. Todėl svarbu ištirti mikrokapsulių prisitvirtinimo prie skirtingos pluoštinės sudėties verpalų galimybes. Ramė yra labai minkštas ir švelnus pluoštas, todėl gaminiai iš ramės verpalų būna lengvi ir patogūs. Savo morfologine sandara ir sorbcinėmis savybėmis panašus į linų pluoštą ir labiau vertinamas dėl mažo glamžumo ir paviršiaus blizgesio. Tačiau ramės pluoštas cikliškai lankstomas lūžinėja, todėl dažnai naudojamas su kitais natūraliais, dirbtiniais ar sintetiniais pluoštais. Tekstiliniuose gaminiuose ramės pluoštą derinant kartu su medvilnės pasiekiama geresnių fizikinių ir mechaninių savybių. Medvilnės pluoštas pasižymi geromis sorbcinėmis ir stiprumo savybėmis. Tyrimams pasirinkti kilpiniai audiniai, kurių pluoštinė sudėtis labiausiai tinkama mikrokapsulėms įterpti. Siekiant sukurti pridėtinės vertės turinčius audinius, ramės / medvilniniai kilpiniai audiniai buvo padengti mikrokapsulėmis, kurių šerdinis komponentas – eukaliptų eterinis aliejus. Literatūros apie kilpinius audinius, padengtus kvapiomis mikrokapsulėmis, nerasta, todėl ypač aktualu išanalizuoti, gamybos procese retai naudojamo ramės pluošto, kilpinių audinių sandaros, fizikines ir mechanines savybes. Disertaciniame darbe ištirti kilpinių audinių su mikrokapsulėmis kokybiniai rodikliai. Naudojantis atliktų tyrimų rezultatais ir matematinėmis priklausomybėmis, galima prognozuoti sudėtingos sandaros audinių savybes. Darbo rezultatai gali būti pritaikomi kitų, panašios struktūros, tekstilinių gaminių su mikrokapsulėmis kokybinių rodiklių prognozavimui ir naujų tekstilės medžiagų kūrimui.

Darbo sandara ir apimtis

Bendra darbo apimtis – 102 puslapiai. Darbą sudaro įvadas, literatūros apžvalga, darbo metodika ir tyrimo rezultatų skyriai, išvados bei 136 šaltinių literatūros sąrašas. Darbe pateikti 51 paveikslas ir 7 lentelės.

Išvados

1. Mikrokapsulių prisitvirtinimas prie medvilnės, ramės, bambuko, lininių ir balintų lininių verpalų yra efektyvus.

- Balintų lininių verpalų masė po padengimo mikrokapsulėmis padidėjo 17,3 %, esant 160 ºC fiksavimo temperatūrai, tačiau pastebima mikrokapsulių sienelių deformacijų ir pažeidimų. Dėl aukštos fiksavimo temperatūros gali būti atpalaiduotas mikrokapsulių šerdinis komponentas – eukaliptų eterinis aliejus.

- Efektyvus mikrokapsulių prisitvirtinimas nustatytas padengus jomis ramės verpalus. SEM analizė patvirtino, kad mikrokapsulės prisitvirtinusios prie pluošto paviršiaus ir įsiterpusios į pluošto tarpus.

2. Kilpinių ramės / medvilninių audinių padengimas mikrokapsulėmis su eukaliptų eteriniu aliejumi turi įtakos audinių sandarai ir lenkiamajam standumui.

- Kilpinių audinių apdaila mikrokapsulėmis su eukaliptų eteriniu aliejumi padidina audinio paviršinį tankį. Priklausomai nuo ataudų tankumo ir apdailos metu naudotos rišiklio koncentracijos, ramės / medvilninių kilpinių audinių paviršinis tankis padidėjo nuo 8,2 % iki 27,1 %, palyginti su nepadengtais mikrokapsulėmis audiniais. Audinių, padengtų mikrokapsulėmis naudojant 20 ir 95 g/l rišiklio koncentraciją, priklausomybė tarp ataudų tankumo ir paviršinio tankio, aprašoma tiesinėmis lygtimis (R^2 iki 0,8937).
- Mikrokapsulių apdaila kilpinių audinių storį padidina nuo 20 % iki 45 %, nes rišamoji medžiaga sustandina audinio kilpas, kurios suspaudimo metu geba priešintis suteikiamai apkrovai.
- Kilpiniams audiniams suteikiama mikrokapsulių apdaila padidina audinių lenkiamąjį standumą. Lyginant padengtus mikrokapsulėmis kilpinius audinius su žaliais audiniais, gauta, kad lenkiamasis standumas padidėja iki 14 kartų metmenų kryptimi ir iki 12,8 karto ataudų kryptimi. Audinių, padengtų mikrokapsulėmis, esant 80 g/l rišiklio koncentracijai, priklausomybė tarp lenkiamojo standumo ir ataudų tankumo, aprašoma tiesine lygtimi ($R^2 = 0,8100$).
- Kilpinių audinių atsparumas dilinimui priklauso nuo ataudų tankumo ir audiniui suteikiamos apdailos. Po padengimo mikrokapsulėmis audiniai tapo atsparesni dilinimui, tačiau skalbimo procesas sumažina atsparumą mechaniniam poveikiui. Nustatyta, kad dilinant masės nuostolis po 5 skalbimo ciklų yra 1,33 kartų didesnis nei neapdorotų audinių, ir 1,97 karto nei padengtų mikrokapsulėmis, po 25000 sūkių skaičiaus. Nustatytas stiprus ryšys tarp audinių masės nuostolio dilinant ir dilinimo ciklų skaičiaus, aprašomas polinominėmis lygtimis, kurių apibrėžties koeficientas R^2 iki 0,9989.

3. Išanalizavus kilpinių audinių laidumą orui, nustatyta, kad kilpinių audinių padengimas mikrokapsulėmis sumažina audinių laidumą orui. Audinių laidumas orui po dilinimo proceso sumažėja iki 80 %, o po 25 skalbimo ciklų apie 70 %, palyginti su padengtais mikrokapsulėmis, neskalbtais audiniais. Nustatytas, stiprus ryšys tarp audinių, padengtų mikrokapsulėmis, esant skirtingoms rišiklio koncentracijoms, laidumo orui ir ataudų tankumo, tiesinių priklausomybių (R^2=0,9201–0,9604).

4. Kilpinių audinių pūko kilpos atsparumas ištraukimui priklauso nuo audinio sandaros, apdailos mikrokapsulėmis ir skalbimo proceso. Audinio padengimo MK procesas pūko kilpos atsparumą ištraukimui padidino 2,1 ir 1,5 karto, atitinkamai esant 5 ir 25 mm ištraukimo intervalui. Skalbimo procesas padidina pūko kilpos

atsparumą ištraukimui. Nustatyta, kad ištraukimo jėga po 25 skalbimo ciklų yra didesnė 3,9–3,4 ir 1,9–2,3 karto, palyginti su žaliais ir padengtais mikrokapsulėmis audiniais, kilpos ištraukimo intervalui esant 5–25 mm. Nustatyta priklausomybė tarp pūko kilpos ištraukimo jėgos ir rišiklio koncentracijos, aprašoma tiesinėmis lygtimis (R^2= 0,7620–0,7969)

5. Ramės / medvilninių kilpinių audinių sorbcinėms savybėms įtakos turi apdaila mikrokapsulėmis ir skalbimo procesas.

- Dinaminės absorbcijos procesas audiniuose, padengtuose mikrokapsulėmis, vyksta lėčiau nei žaliuose audiniuose. Analizuojant dinaminės absorbcijos pokyčius po skalbimo, pastebėta, kad iki 10 skalbimo ciklų absorbcija didėja, o po 15 skalbimų absorbcija ima mažėti. Nustatyta dėmės ploto pokyčio priklausomybė nuo stebėjimo trukmės aprašoma logaritminėmis lygtimis, kurių apibrėžties koeficientas R^2 iki 0,9847.
- Kilpinių audinių padengimas mikrokapsulėmis statinę vandens absorbciją sumažina 37,4 %.
- Vandens garų absorbcija po padengimo mikrokapsulėmis padidėjo 10,0 %, o po 25 skalbimo ciklų padidėjo 25,2 %, palyginti su žaliais audiniais.
- Atlikus audinio džiūvimo tyrimus, pastebėta, kad džiūvimo procesui daugiau įtakos turi skalbimo ciklų skaičius nei apdaila mikrokapsulėmis. Nustatyta priklausomybė tarp vandens likučio koeficiento ir džiūvimo trukmės, aprašoma logaritminėmis lygtimis (R^2 = 0,9400–0,9983).

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