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Investigation of the wettability of the hydrophobic textile after mechanical treatments

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Abstract. In this study, hydrophobic textile wettability was experimentally tested. The influence of the number of fatigue cycles and environment temperature on the wettability (water contact angle) of hydrophobic coated textile have been investigated. Two types of fatigue have been used to characterize hydrophobic surface after wearing: the flexing fatigue resistance test of the samples was carried out on bending machine IPK 2M and folding machine "Pegasil EL-15F". The evaluation was made by measuring the contact angle after 3000, 6000, and 7500 cycles (EN ISO 7854). Surface chemical processing was carried out in four different concentrations of chemical solutions. According to experimental results the sufficient coating conditions were ascertained.

Key words: wettability, hydrophobic effect, contact angle, cotton fabric, flexing fatigue.

1. INTRODUCTION

For many applications low wettability of a finished fabric is important and potential applications include rainwear, protective clothing [1,2], upholstery [3], and automotive interior fabrics. Low wettability is one of the most important requirements for these materials, which strongly depends on two properties: the surface free energy and the surface roughness [4]. Textiles are composed of fibres that provide inherent micro-scaled surface roughness. Consequently, it is easy to turn textiles hydrophobic by coating them with an appropriate hydrophobic reagent [5].

The method of water drop contact angle [6–13] is widely used for defining hydrophobic properties of waterproof textile materials. The basis of this method is that a particular quantity of liquid is instilled onto the observational surface and after a certain time the water drop contact angle is measured; according to the appointed contact angle, surfaces are classified into groups [14]. The larger the contact angle, the more hydrophobic the surface is considered. A surface is hydrophobic if the water drop contact angle is bigger than 90°; if this contact angle is smaller than 30° the surface is hydrophilic [15]. Water drop contact angle is used while measuring textile fibres having various hydrophobic coatings [4,11,12,16].

The size of the water drop contact angle depends on the properties of the surface under observation and on various effects that appear during the measurement [6,10]. Roughness of the surface and its chemical nature has the biggest influence on the angle size [13,17,18]. Products for special destination have to satisfy high requirements of transferring persistency, heat and humidity. To evaluate the durability of coated materials during their exploitation, the resistance against cyclic fatigue [19,20], and the effect of environmental temperature [21,22] is measured. Fatigue or cyclic ex-

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tension has impact on hydrophobic properties of coated materials. Even slight permanent fatigue can worsen mechanical behaviour of the material and change its properties as well as reduce the usage duration of the product. It has been estimated that after 9000 fatigue cycles 160–640 µm cracks appear and water permeability in the coated materials increases [2]. Cyclic extension opens microscopic pores of the textile materials that are coated with polyurethane coating and increases water permeability of that material about 20% [20]. During the exploitation, even very high quality composite materials grow old and weaken like all other materials. Even inconsiderable permanent fatigue can considerably reduce strength, change qualities of laminated leather, and shorten the duration of product exploitation [19]. Surface changes can be estimated using optical microscope and the material weight loss. Water drop contact angle reduces when the roughness of the material surface increases.

The review of scientific literature has shown that hydrophobic properties of materials are important to protective products. Depending on the particularity of the application, materials are affected by various mechanical treatments and these have different influence on the properties of the materials.

The aim of this research is to approve different finishing technologies effectiveness in the case of different chemical agents and different samples preparing and to investigate the influence of environment temperature at cyclic flexing on the hydrophobic properties of textile materials.

2. MATERIALS AND METHODS

In the present study we applied the twill weaving cotton fabric (surface density 310 g/m^2; average thickness 0.55 mm). Surface density of the investigated materials was estimated according to the standard LST EN 12127:1999 using digital scales KERN EG (error ±0.001 g). The thickness δ of the specimens was measured using thickness gage Schmidt (error ±0.01 mm), the pressure was 1.0 kPa. The coefficient of variation did not exceed 2%.

Four commercially available hydrophobic agents were selected for investigating the influence of the exploitation properties on the materials hydrophobicity. Their concentration is presented in Table 1.

Specimens were immersed into 4 different agent's liquids for 3 min at room temperature (23 ± 2 °C). According to manufacturer's recommendation, the samples were dried in two ways: by drying at room temperature for 24 h or using the convection oven E5CN at 120 °C for 20 min. Similar treatment of the samples has been used by other investigators [8,9,18].

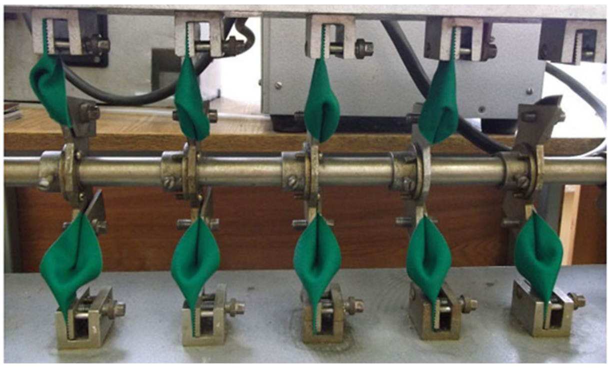
Table 1. The characteristics of investigated hydrophobic treatments

Code of treatment	BNT 1	BNT 2	BNT 3	BNT 4
Composition	$FSiO_2$ 5% + addition	$FSiO_2$, 5%	$FSiO_2$, 10%	10% silicone

To evaluate wettability, the method of drop contact angle measurement is used. One drop of distilled water is applied with a pipette on the surface of the investigated specimens and is photographed in 20 s using a chronometer. According to literature, it is optimal time during which the applied drop forms itself [16]. FujiFilm 14 was used; resolution was 42 288 × 3216 megapixels. The distance from the object glass to the specimen was 10 cm. Six tests were performed using each tested material and the coefficient of variation did not exceed 2.0%.

Cycle fatigue that imitates bending, flexing, and stretching was performed using the machine IPK 2M (Fig. 1a). According to the standard ISO 7854, textile

(a)



(b)

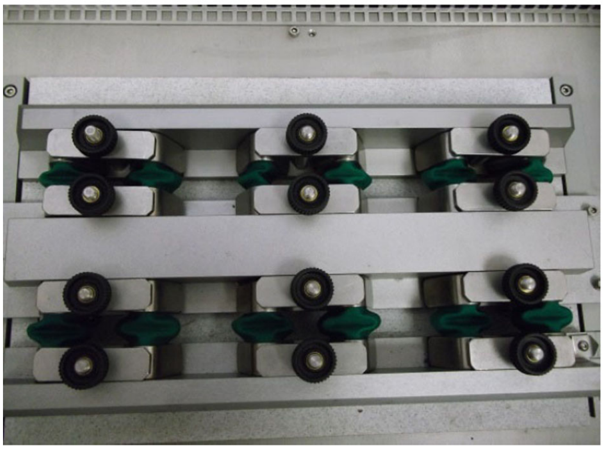


Fig. 1. Fixation of the samples in apparatus: (a) IPK 2M, (b) Pegasil EL-15F.

fabrics coated with rubber or plastic have to withstand 7500 cycles fatigue and remain unchanged. Thus in order to estimate fabric resistance against cycle fatigue the specimens were fatigued using the 3000, 6000, and 7500 cycle mode. The specimens were fatigued in wale and course directions.

Test specimens for the flexing test were cut from the materials in the shape of a square with the side 64 ± 1 mm. The test was performed according to the standard LST ISO 5423:1992. The fatigue test was carried out using a machine of flexing (Pegasil/ZIPOR, Portugal) with the aim to evaluate the occurrence of cracking in the area of greater flexion during the use of the product. The equipment is placed inside a cold chamber to adjust test temperature till –25 °C. The testing temperature was –5 °C (Fig. 1b). Using different equipment, the same number of flexing cycles was used: 3000, 6000, and 7500.

To analyse microstructure of fibres before and after treatment, a scanning electron microscope (SEM, Quanta 200 F) was used for the evaluation of the damage after the biggest number of flexing cycles. Microscopic images of the specimens after 7500 flexing cycles were compared with initial appearance of the specimens. Later on, the wettability was measured.

3. RESULTS AND DISCUSSION

3.1. Finishing conditions

In the first part of the research we studied the effect of specimens' drying, after impregnation, on the wettability. The initial contact angle was measured and wettability was estimated.

Based on the analysis of the treatment technology of cotton textiles with fluorochemicals, described in [9,17], with contact angle more than150°, it was deduced that application of particular agents by the immersion method may result in superhydrophobization of the textile surface. It was observed that environmental temperature by drying the specimens has slight influence on the contact angle. The biggest alteration of the contact angle was obtained for the T1 solution; the greatest value ($\alpha = 138.2°$) was obtained when the sample was drying in a convection oven at 120 °C for 20 min. These data agree with data in [8], where 10% concentration solution on mixed fiber composition was used. At room temperature, CA values of dried samples were till 5% lower than CA values of samples dried in the thermal oven (Fig. 2).

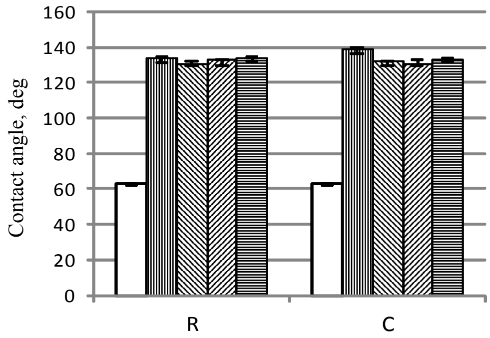


Fig. 2. Treated samples (☐ – initial, ▥ – T1, ▧ – T2, ▨ – T3, ☰ – T4) surface contact angle, when specimens were dried at room temperature (R) and in convection oven (C).

3.2. Flexing durability

With the increase of the number of flexing cycles, the value of CA decreases. On average, the total decrease is 12.8% in the case of solution T1 when samples were dried in the convection oven, and 11.7% at room temperature dried samples (Fig. 3).

As it is known, temperature is a very important parameter of the fatigue life of polymers [22]. In our experiments, with an increase in the number of flexing cycles, the value of CA decreases. On average, the total decrease is 11.4% in the case of T1 when samples were dried in the convection oven, and 8.2% in room temperature dried samples (Fig. 4). It is evident from Figs 3

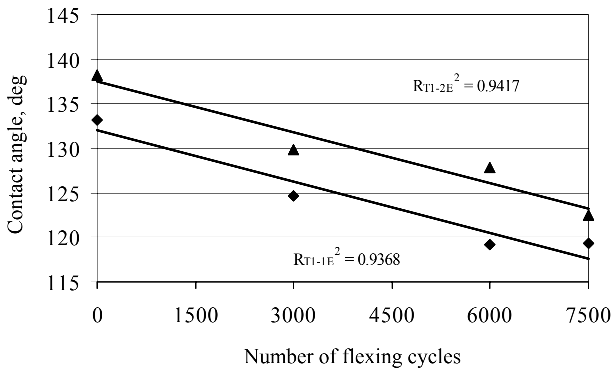


Fig. 3. Relationship between the number of flexing cycles and the contact angle, when flexing experiment was carried out at room temperature: ◆ – after hydrophobic treatment of the sample at room temperature; ▲ – after hydrophobic treatment of the sample in the convection oven at 105 °C.

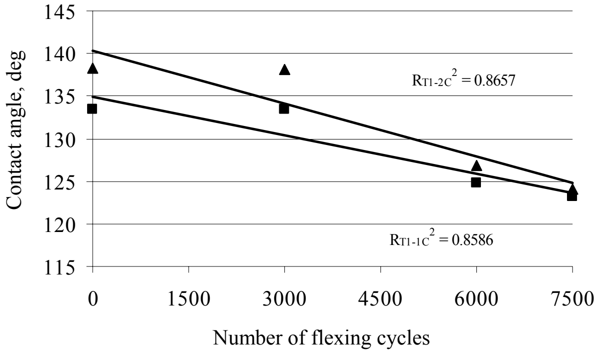


Fig. 4. Relationship between the number of flexing cycles and the contact angle, when flexing experiment was carried out at –5 °C: ■ – after hydrophobic treatment with sample drying at room temperature; ▲ – after hydrophobic treatment with sample drying in the convection oven at 105 °C.

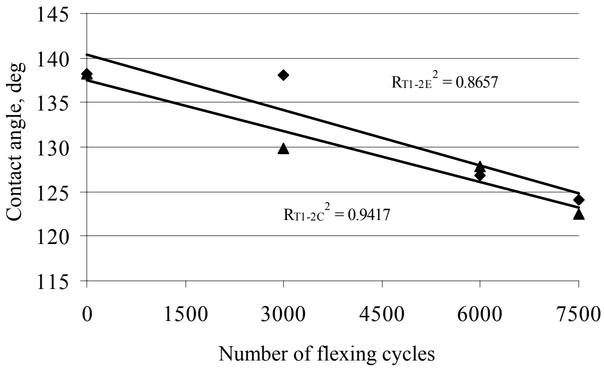


Fig. 5. Relationship between the number of flexing cycles and the contact angle after hydrophobic treatment of samples in convection oven at 105 °C: ◆ – flexing experiment was carried out at –5 °C; ▲ – flexing experiment was carried out at room temperature.

and 4 that when the number of flexing cycles increases, the values of CA decrease in all cases. When the flexing experiment was carried out at room temperature, the reduction of CA was even but when flexing experiment was carried out at –5 °C, the values of CA after 7500 flexing cycles became approximately equal.

Experimental results show that the environmental temperature has marginal influence on CA (Fig. 5). On the other hand, contact angle marginally increases when temperature of the fatigue environment decreases. High value of the coefficient of regression at temperature –5 °C ($R^2_{T1\text{-}2C} = 0.9417$) confirms this dependence.

As shown in Fig. 6, initial contact angle of materials for all types of solutions showed more close values of CA. It is known that increasing the concentration of the solution increases the water contact angle [21]. It was obtained that different concentration of the solutions T1 (5%) and T2 (10%) give very close values of CA. It is evident from Fig. 6 that 3000 flexing cycles do not influence CA of the treated materials. Greater decrease in CA occurs after 6000 cycles. Although the test water drop contact angle values decreased in different ways, after 7500 cycles the CA of all samples decreased from 8% to 10%.

Morphological changes as a result of hydrophobic treatment of cotton textile have been investigated using SEM. It may be mentioned that microscopic analysis showed that surface roughness appeared on the hydrophobic treated fibres. As can be seen from Fig. 7, more durable hydrophobic coating has been obtained when the specimens were dried in a thermal chamber at 105 °C.

The comparison of surface morphology of treated samples after flexing (Fig. 7) shows significant changes in fabric's structure. It is evident from Fig. 7 that after 7500 cycles, when samples were dried at room temperature, single defects in the flexing zones arise.

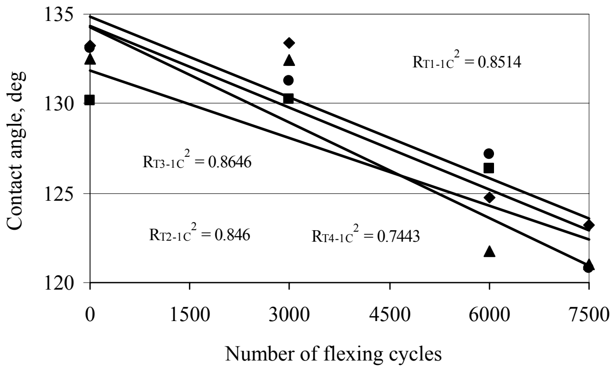


Fig. 6. Relationship between the number of flexing cycles and the contact angle when the flexing experiment was carried out at –5 °C in a cold chamber after hydrophobic treatment of the sample at room temperature: ◆ – T1, ■ – T2, ▲ – T3, ● – T4.

(a) (b) (c)

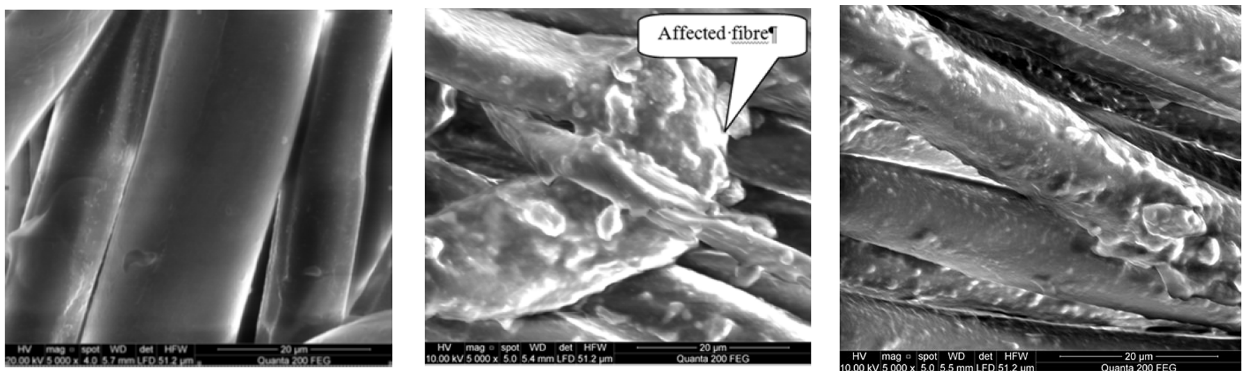


Fig. 7. SEM images of the coated materials: (a) initial; (b) after 7500 flexing cycles, when sample was dried at room temperature; (c) after 7500 flexing cycles, when sample was dried in a convection oven at 105 °C.

4. CONCLUSIONS

The experimental study presented in this paper was focused on the analysis of the hydrophobic treatment on cotton fabrics. The influence of the temperature on finishing materials, flexing fatigue, and cotton materials structure alteration was investigated.

According to the experimental results we may conclude the following:

- the environmental temperature has not a great influence on finishing specimens and contact angle, but hydrophobic coating has better durability when hydrophobic treatment specimens are dried in a convection oven at 105 °C;
- more effective is the T1 solution ($FSiO_2$ 5% + addition) than the T3 solution ($FSiO_2$ 10%);
- the linear relationships show that the increase of the number of the flexing cycles decreases the contact angle;
- fibre affected zone can be clearly seen in SEM images after 7500 cycles.

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Vetthülgava kanga märguvus mehaanilise töötlemise järel

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Vitalija Čepauskienė ja Virginijus Urbelis

Käesolevas töös katsetati vetthülgava kanga märguvust. Uuriti väsimustsüklite arvu ja keskkonnatemperatuuri mõju vetthülgavaks töödeldud kanga märguvusele (vee kontaktnurgale). Vetthülgava pinna kulutamisjärgseks iseloomustamiseks kasutati kaht tüüpi väsimusteime: katsekehade paindeväsimusteime tehti painutamismasinal IPK 2M ja voltimismasinal Pegasil EL-15F. Hinnang anti EN ISO 7854 kohaselt, mõõtes kontaktnurka 3000, 6000 ja 7500 tsükli järel. Kangapinda töödeli nelja erineva kontsentratsiooniga kemikaalilahusega. Katsetulemused kinnitasid kangapinna töötluse piisavust.