Preliminary comparison of contact angle and wetting resistance of water-repellent treated cotton woven fabric

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Abstract. Filtration capability of textile media or the resistance towards the fluid penetration through different fabrics is depending on their surface repellence. The emission of condensed exhaled air on the outer layer of the half facemask from the FFP2 (EN 149) category indicates the termination of its protective properties. The overcoming by the fluid of the textile environment of the mask in its role of an artificial barrier goes through two stages. Initially, the aerosols in the exhaled air condense and meet the repulsion from the inner fabric. Later, due to the hydrodynamic pressure, the condensed droplets pass through the textile medium, overcoming the friction among the fibrous layers. Finally, the droplets irrigate the outer, front layer of the mask and are released into the environment with all the consequences. Closest to this physiological process is the hydrostatic pressure test method. The test of resistance to fluid penetration gives a final assessment of the property of the textile environment without clarifying the elements of the process. Experimental studies have shown similarities in the results of the penetration of the fluid through the textile medium and the initial repulsion of aerosols from the surface of the fibrous layer. The repulsion of water droplets is of predominant importance in relation to the general barrier capacity of the textile media. The main indicator of water repellence is the contact angle of the free drop on the fabric. The subject of this article is the comparison between the contact angle of wetting and the penetration of the fluid by means of standard test methods. The aim of the work is to explain and model the process of fluid penetration through protective masks, as well as to optimize the properties of repulsion and resistance.

Keywords: FFP2 masks, COVID-19, fluid filtration, contact angle.

1. Introduction
Technological developments in the last 18 months have allowed the identification of two main areas in the concept and production of protective face masks. During the preliminary achievements in the first direction of the masks, filtering functions were assigned. The technical conditions of these textile garments are given in 149: 2001 + A1: 2009 [1].
At the heart of the design, this type of mask allows airflow without interfering with normal inhalation and exhalation effort. Their functions are aimed at protecting the individual from the environment. These masks as a construction are designed to filter and retain micro particles, including micro-organisms with a size of about 3 microns.

The high requirements for filter masks and the difficult mass production caused the concept of the second direction of the barrier masks. Their technical conditions are given in the specification of the French standardization agency: AFNOR SPEC S76-001 / 2020 [2]. The requirement for these articles is aimed at inhibiting the fluid and associated micro particles on the direction of the directed flow. The purpose of barrier masks is to isolate the individual from the environment and to prevent the spread of infection.

Thus, two directions in the design of the masks were formed: filtration properties by retaining in the volume of the textile medium and repellent properties on the active surface of the medium. From the point of view of the general protection of the masks, their repellent ability has the task of initially and partially slowing down the flow. The study of the repulsion of a fluid with water or oil content presupposes the simultaneous test of air permeability [3], the resistance to wetting [4, 5, 6] and the surface resistance to a water drop. Figure 1 depicts the schematics for the various angles.

2. Materials and Methods

3. Cotton woven fabric

The main subject of the experimental work is a woven fabric with the trade name Filtro. The fibrous composition of the fabric is 100% cotton, the area mass is 157 g/m². The warp and weft threads are from a single ring yarn: Tt20x1, 100-Cotton. The warp density is 500 threads/dm and the weft density is 245 wefts/dm. The weave is Se3/1Z, and the reverse side with weft effect of the weaving crossings is the active surface of the fabric. In this unequal-faceted fabric structure, the reverse side has a relatively smoother surface than the striated face.

4. Fluid repellent treatment

In industrial conditions, the fabric is treated with different chemicals under different technological conditions, such as bath recipe, solution temperature and processing speed. The experimental work follows the algorithm of the factorial experiment. As a result, 8 variants were obtained, which were randomly distributed in the sequence of experiments. The purpose of the treatment is to increase the repellent properties of the fabric surface compared to applied fluids: air, aqueous solutions of mineral salts and oils of natural origin.

After many technological experiments for the water- and oil-repellent properties of the cotton fabric, a recipe with an optimal composition has been determined. The compounds that make up the recipe are based on Dimethyloctadecyl[3-(trimethoxysilyl)propyl]ammonium chloride used as a precursor, PhenOxyEthanol (C6H5OC2H4OH) used as an organic ingredient, as well as the modified silicon
dioxide ingredients. All ingredients of the recipe meet the requirements of OEKOTEX® Standard 100 and REACH, for safe production and application of the processed fabric. The chemicals are supplied by reputable manufacturers of textile auxiliaries such as Massimo Guarducci Srl, Nano-Care Deutschland AG, Creative Chemical Manufacturer GmbH and Textil Color AG. The arguments of the factor experiment relate to the temperature of the bath, the immersion time / rate of movement of the fabric and the temperature of drying and polymerization of the thin layer. The temperature of the bath varies from 50°C to 85°C. The speed of the fabric varies from 15 to 19.5 m/min. The drying and polymerization temperatures range from 120 to 160°C. The only peculiarity in the planning of the experiment is the uniformity of the machine mode for the seventh and eighth variants. For the eighth variant, the bath temperature is 65°C, the fabric speed is 18 m/min, the drying temperature is 145°C, but the fabric goes through double immersions in two consecutive bathtubs. This is the reason for getting the best results for the eighth option.

5. Characterization

Air permeability was measured using the apparatus DVT HG DLC, DEVOTRANS, Turkey [7], in compliance with the methodological requirements in the standard - Determination of the permeability of fabrics to air, ISO 9237: 1995 [3].

The resistance to water penetration through the fabric was measured with the HIDROTESTER 3H, FX 3000-III, № 851.06.08, TEXTEST AG, Switzerland [8].

Static contact angle measurements of the treated fabrics variants were performed using an Easy Drop DSA20E Krüss GmbH [9] drop shape analysis system (Germany) at 20 ± 0.2 °C. A sessile drop of deionized water with a volume of 10 μL controlled by a computer dosing system was deposited onto the fabric. The contact angles were calculated by computer analysis of the acquired images of the droplet. The data are average from 20 measurements for each sample, (Figure 2).

6. Results and discussion

Liquids come in contact with solid surfaces in a variety of applications, including medicine and agriculture. In all kind of applications, knowing the contact angle value gives a strong indication on the properties of the fabricated material. When a drop of water is placed on a solid, it will spread on the surface based on the intermolecular interactions between the solid and the liquid. Water contact angle immediately give an indication of the wettability of the solid surface. If the measured contact angle is above 90 degrees, the solid have poor wetting and is hydrophobic. It is important to determine the water contact angle of the cotton fabric that might be used for the preparation of face masks. The values of the angles of contact for all cotton fabrics were measured with distilled water drop. The representative images of the droplets of the fabric variant 7 were shown in Figure 2. This fabric was almost super hydrophobic with water contact angle of 140.6°±3.4° (Figure 2). The measured values for the other cotton fabrics’ variants were hydrophobic with water contact angles ca. 95°, (Table 1).

![Figure 2. Images of water droplets deposited on the surface of cotton fabric variant 7.](image-url)
Air permeability testing, water penetration tests, spray tests, water repellency and oil repellency test were performed as well. The test results are summarized in Table 1.

**Table 1.** Experimental results on repellent properties of the used cotton fabrics.

| Properties assessment      | EN ISO Dim | Fabric samples variants |
|----------------------------|------------|-------------------------|
| air permeability           | 9237 mm/s  | 159,7 131,9 129,1 107,5 101,7 82,8 76,7 61,1 |
| Water penetration — Hydrostatic pressure test | 811 mmH2O | 108 113 151 190 200 200 215 225 |
| Water repellency           | 14419 grade | 6 5 6 6 5 6 6 5 |
| Oil repellency             | grade      | 2 2 5 4 1 4 2 2 |
| Static contact angle       | degree     | 84,1 94,6 95,6 96,5 99,0 102,1 105,7 140,6 |

The measurement of the surface resistance by the method of the static contact angle gives a relatively more objective assessment than the method of the absorbent drop for a certain period.

In the way of obtaining the contact angle is comparable with the methods for measuring the air permeability or water penetration, due to the use of a measuring device and numerical evaluation of the property.

The convergence of the curves from the measurements of air permeability, water penetration and contact angle is due to the objective approach in the measurement and the detail of the numerical estimates of the indicators.

The significant advantage of the contact angle consists in the possibility of visualization and physical explanation of the acquired water-repellent property.

Figure 3 visualises the graphs of the measured properties for all eight variants.
7. Conclusions

Figure 3 clearly illustrates the correctly selected parameters of the technological mode for the presented eight characteristic variants of the factorial experiment. The practical realization of the presented experimental work was preceded by a long period of time in which the trial and error method was accepted as a determining principle. The final version shows the possibility of finding an optimal technological regime in which the necessary water and oil repellent properties of the fabric are achievable.

First of all, the optimization criterion is observed - the balance between air permeability and water penetration resistance. It is seen that the increase of the resistance against the penetration of the water flow is connected with the decrease of the air flow as well. Equilibrium as an optimization criterion consists in maximum filtration / repulsion of the liquefied fluid with minimum resistance to inhalation and exhalation.

Secondly, it was found that the resistance to wetting with a water or oil drop could not be represented by the changes in the 6-point scores shown in the graphs. They have a discrete nature and a very subjective way of measuring. From the graphs shown, no convergence can be established between the variation of water and air resistance, measured with instruments and estimated with direct quantitative indicators, and the corresponding dimension.

In this case, the static contact angle method compensates for the subjective methods and is equivalent in reliability and physical explanation to the hardware measurements.

The method of static contact angle is applicable in the process of technological optimization of fabrics for protective masks. Through the detailed assessment and the possible analysis of the change of the repulsive treatment, the layers in the construction of the mask can be distinguished and the industrial reproducibility can be improved.

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