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Preparation of CO_2 adsorbing polyester fabric by treating it with titanium isopropyl alcohol

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Novel fabric that will absorb CO_2 in the environment has been produced by coating TiO_2 on the fabric surface of a polyester bedspread. The products are characterized by FTIR, EDX and SEM analyses. The parameters, such as the amount of titanium added to the bath and the temperature, are studied to specify maximum CO_2 absorption, which ultimately is set as 6 mL titanium source/L and 130ºC (% 95 ram). At the same time, various tests, such as pilling, abrasion, and durability of the fabric, are also performed. The fabric sample, thus produced, is also passed through the irritation tests performed by accredited laboratories.

Keywords: Bedspread, CO_2 adsorption, Polyester, Titanium oxide

1 Introduction

Besides factors such as heat, light, and noise, ambient air also affects people's health, comfort, and efficiency in indoor environments. Carbon dioxide (CO_2) is an important air pollutant that impairs indoor air quality. Typically, 0.03% of atmospheric air is CO_2 by volume. Therefore, it is impossible to get rid of CO2 in indoor environments. The continuity of breathing also causes CO_2 accumulation in indoor environments. The amount of carbon dioxide exceeding 1000 ppm in the environment causes respiratory diseases, headache, anorexia, eye, nose, and throat irritation, as well as concentration disorder.

Frequent and long-term breathing of over 2500 ppm carbon dioxide can lead to severe and irreversible health problems. It may cause permanent damage to essential organs, such as the eyes, heart, respiratory system, and brain. Waking up from sleep at night, failure to breathe comfortably, and decreased sleep quality are usually caused by CO_2 accumulating around the person during sleep (for those without any health problems). Therefore, if the CO_2 in the environment could be cleaned while sleeping, sleep quality would increase, enabling people to live a healthier life and be more productive atwork[1,2].

Many chemicals have been synthesized or produced for CO_2 adsorption. However, there is no study reported that shows CO_2adsorption by the final product in the textile sector. The most commonly known CO_2 absorbent chemicals in both industry and literature are molecular sieves, phthalocyanine compounds, silica, activated carbon, and titanium dioxide[1-4]. Phthalocyanine compounds are not preferred because they are expensive, whereas molecular sieve, activated carbon, silica, and solid TiO_2 cannot be applied to the fabric. However, different derivatives of TiO_2 can be applied to the fabric. In addition, TiO_2 is an excellent CO_2 absorbent material as compared to molecular sieve. The World Health Organization has made a statement about the use of TiO_2 in food, but did not have any statementaboutclothes[5].

TiO_2 is an inexpensive, stable and non-toxic chemical that acts as a catalyst with light. Since the first discovery of its photocatalytic effect, many studies have been conducted on this feature of TiO_2[6,7]. As in other pollutants, the oxidative process can equalize the CO_2 in the environment by using the photocatalytic feature of TiO_2[8-11]. The conversion of CO_2 to methanol in an aqueous or humid environment by photocatalytic effect is shown in the following equations[12]:

$$CO_2 + h\nu \longrightarrow {}^*CO_2^- \quad \ldots(1)$$

$$^*CO_2^- + H_2O \longrightarrow HCOO^* \quad \ldots(2)$$

$$HCOO^* + 1e^- \longrightarrow HCOO^- \quad \ldots(3)$$

$$HCOO^- + H^+ \longrightarrow HCOOH \quad \ldots(4)$$

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$$HCOOH + H^{+} + h\nu \longrightarrow HCHO + H_2O \quad \ldots(5)$$

$$HCHO + \tfrac{1}{2}\ H_2 \longrightarrow CH_3OH \quad \ldots(6)$$

It is not easy to apply TiO_2 directly to the fabric. However, its derivatives, such as titanium isopropyl oxide, can be coated on non-woven surfaces such as polyester. It has been reported in the literature that polyester is coated with metal oxides, such as TiO_2, ZnO, and ZrO. These studies show that metal oxides are attached to the polymer surface by physical absorption[13-15]. In wet chemical methods, coatings are generally attached to the surface by physical adsorption, that is, by van der Waals forces. The physical absorption mechanism is shown in Fig. 1.

In this study, titanium dioxide has been fixed as a thin film on the polyester textile surface to produce a CO_2-adsorbent and non-toxic fabric, that will prevent the accumulation of CO_2in the environment. The production of such a fabric will allow people to sleep more comfortably, improve both human health and sleep quality and increase work efficiency.

2 Materials and Methods

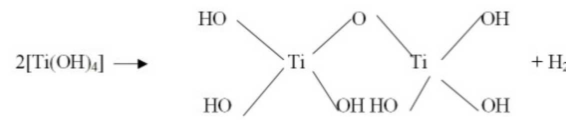
All chemicals were purchased in analytical purity; no further purification was made. In order to allow the factories to continue production for a certain period, gel and sol-gel solutions of titanium dioxide, which do not precipitate, were produced. Titanium isopropyl alcohol (TIPA) was doped with polyethylene glycol (PEG-600) in an isopropyl alcohol (IPA) medium.

Preparation of Bath

Approximately 5 g of PEG 600 was dissolved in 50 mL IPA at 60-70º by stirring for 30-45 min using a magnetic stirrer. Then 1 mL of pure TIPA was added to the solution and stirred for a few more minutes (at 1000 rubs). The solution was put in a tube. This sample was coded as T1. Then, various amounts of TIPA were added to the bath, provided that all the other components of the bath and all performed procedures remain the same. They were coded as follows: T2 (2 mL TIPA), T3 (4 mL TIPA), T4 (6 mL TIPA), T5 (8 mL TIPA) and T6 (10 mL TIPA). Following equations show that titanium isopropyl oxide hydrolyzes with water and TiO_2 is formed after the condensation reaction[16]:

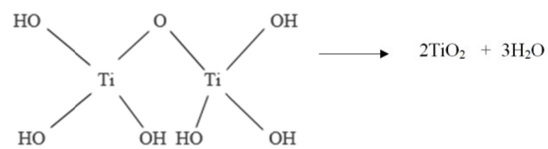
Hydrolysis:

$$(CH_3\text{-}CH(CH_3)\text{-}O)_4Ti + 4H_2O \longrightarrow Ti(OH)_4 + 4(CH_3\text{-}CH(CH_3)\text{-}OH) \quad \ldots(7)$$



…(8)

Condensation:



…(9)

At the same time, sol-gel solutions were mixed with water and kept for a minimum of 24 h.The physical mechanism of the coating process is given in Fig. 1, and the chemical mechanism is given in Eqs (7)-(9).Only the T2 solution did not precipitate and remained gel-like as desired.

These solutions were added to the chemical bath in different amounts for fixation. The acidifier and softener, used for fixing polyester fabric, were added to the bath along with the sol-gel solution. Then the fixing temperature was scanned for the optimum parameter (solution's TIPA content). The process given in Fig. 2 was simulated in a laboratory setting. The pickup value was kept constant at 95%. According to Fig. 2, the fabric is first sent to the chemical bath in standard textile production, containing wetting, softener, buffer, and catalyst chemicals. The resulting fabric is squeezed in the ram and sent to heat treatment.

Samples of 1 g weight were produced from the fabrics and studied forCO_2 adsorption analysis. In addition, the fabrics and solutions were characterized by SEM, EDX, and FTIR analyses.

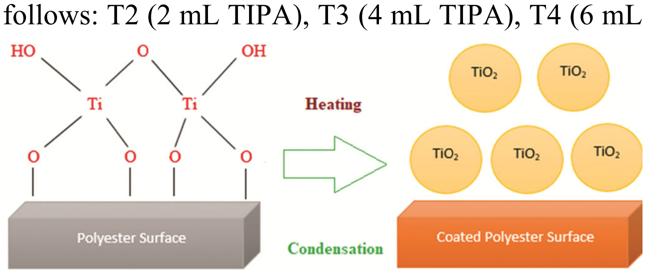


Fig. 1 — Coating mechanism of processes

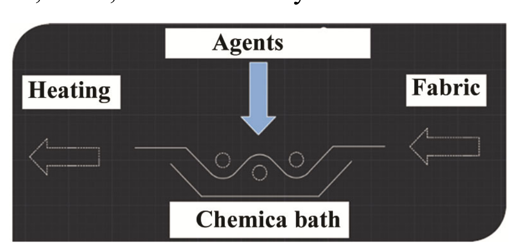


Fig. 2 — Simulation process

For SEM and EDX analysis, fabric samples were coated with Au/Pd at 45-angstrom thickness using a Polaron sc 7620 mini sputter coater device. Regarding liquid samples' analysis, imaging was performed by dropping it on a carbon film-coated copper grid and putting it on the device after drying. In FTIR analysis, Bruker Alpha, having a resolution of 4 cm x 1; equipped with a DTGS detector, performing 10 scans for each spectrum, was used to record vibration peaks.

Carbon dioxide adsorption analysis was carried out by Haliç Çevre, accredited in this field. The measurements were performed by the HLC-04 method (Internal Method) with CO_2 detectors that can perform measurements at ppm level. The measuring mechanism is shown in Fig. 3. As shown in Fig. 3, the sample is placed into the sample tube before the measurement. Then vacuum is applied, 5000 ppm CO_2 is supplied from the CO_2 tube to the measurement tube. After a while, the amount of CO_2 in the environment is read by the detector in ppm. Honeywell Multi RAE Lite (CO- CO_2-SO_2-NO_2-O_2)/M01C011732 was used as a sensor. Measurements were made at 21ºC, 50% relative humidity and 1007 hPa pressure.

The bedspread fabric produced with optimum parameters was also subjected to pilling and abrasion tests, which were carried out by Pro White Tester, using Martindale Test Method according to TS EN ISO12945-2 test standards. In addition, the burst resistance of the sample was tested according to TS EN ISO 13938-2 test standards. Colorfastness to rubbing was studied by rubbing the adjacent fabric to the test sample placed on the crockmeter.

3 Results and Discussion

As mentioned earlier, samples (T1-T6) with different amounts of TIPA, IPA and PEG-600, were kept under room conditions (24°C) for at least 24 h. Then, 20 mL of pure water was added, and the precipitation formation was visually checked (Fig. 1).

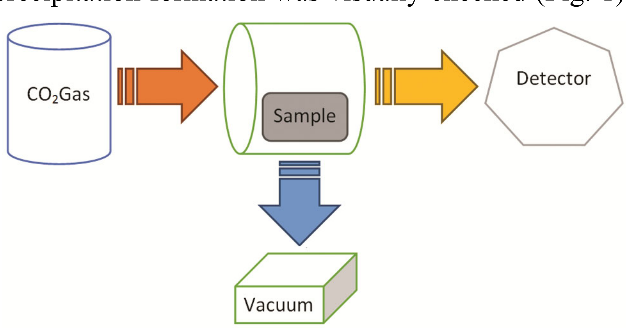


Fig. 3 — CO_2 adsorption measurement

The objective of this stage is to ensure that no precipitation occurs since the formation of precipitation after adding the solution to the aqueous medium of the bath is not desired in the production process. Titanium isopropyl oxide can be precipitated as TiO_2 by interacting even with water. Only T1 and T2 samples were observed without precipitation, and only T2 sample was obtained in the desired gel form. FTIR and EDX analyses of the T2 sample are given in Figs. 4 and 5 respectively.

The vibrations belonging to the –OH group can be seen at 3328 cm^{-1}. The band belonging to aliphatic –CH vibrations is observed between 2874cm^{-1} and 2976 cm^{-1}. Sharp and clear peaks corresponding to –CO, -C-O-C, -CH, and -C-OH vibration signals are observed in the 1313-1475 cm^{-1} range. Overtone bands of C-H and Ti-O vibration peaks are detected at 913-816 cm^{-1} (refs 17).In Fig. 4 (b), peaks other than titanium isopropyl oxide are detected.–C=C vibration peaks belonging to the aromatic ring are observed at 1715 cm^{-1},–OH peaks seen at 3000 cm^{-1} almost disappeared, and the vibration peaks of the –CH aliphatic group became more apparent at 2866 cm^{-1}. The high number of vibrational peaks of the aromatic ring and carboxylic ester at 1256-1365-1462 cm^{-1} is remarkable. In addition, a characteristic peak of Ti-O

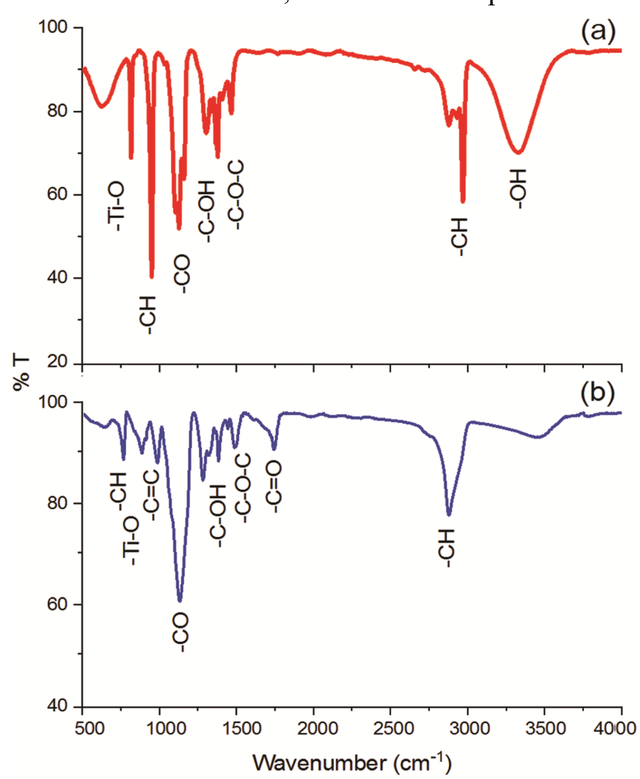


Fig. 4 — FTIR analysis of (a) T2 sol-gel, and (b) TiO_2 coated fabric surface

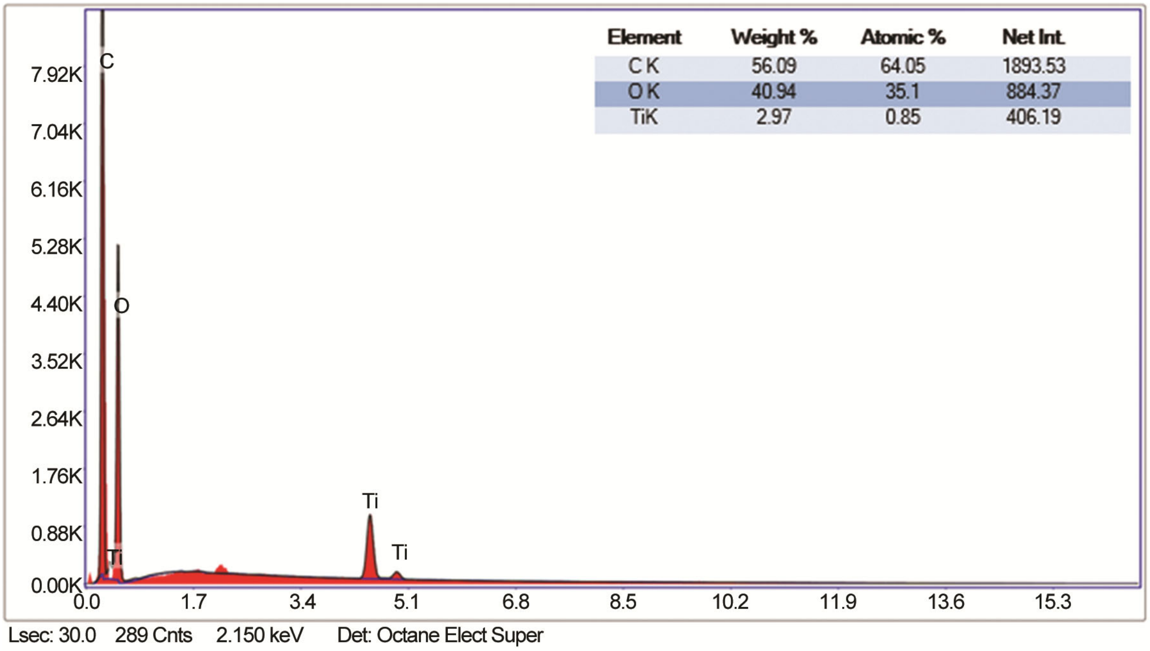


Fig. 5 — EDX analysis of T2 sol-gel

vibration is detected at 836 cm^{-1}. As expected, vibration signals of both PEG-600 and TIPA and IPA are observed in Fig. 4 (a). In addition to the presence of Ti-O in Fig. 4 (b), all characteristic peaks of polyester are detected[18]. Around 3% titanium is detected in EDX analysis. Due to the excessive amount of oxygen in the environment, we can get inference about the amount of TiO_2 in the environment from the amount of titanium. Thus, we observed the vibration peaks of TiO_2 at 816 cm^{-1}, which is attached to the fabric surface.

After setting the optimum sol-gel solution (sample T2), various amounts of this solution (2-4-6-8-10 mL sol-gel/1L), were added to the chemical bath of RAM for fixation. In the meantime, the heating process was continued at 130ºC to find the optimum temperature for the doping process. In other words, after doping the bath, fabric samples, squeezed with 95% pickup, were treated at a constant temperature of 130ºC, which is the standard production process used by the textile factory to produce polyester bedspread fabric. At the same time, other ingredients, such as 1mL/L acid modifier and 1mL/L softener were added to the bath with the tested amounts of T2 sol-gel.

EDX analysis results of the fabrics produced after doping the bath are given in Fig. 6. Instead of a detailed EDX analysis of each fabric sample (Fig. 5), we combined them in a single graph (Fig. 6), which clearly shows the amount of absorbed CO_2 and its relationship with the amount of TiO_2 on the surface.

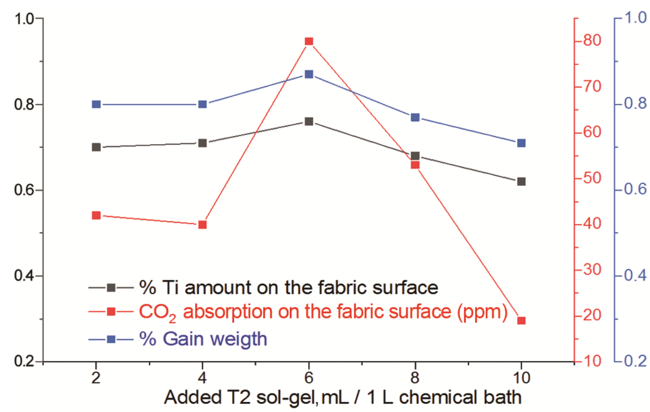


Fig. 6 — EDX analyis of Ti amount on fabric surface and the CO_2 adsroption on the fabric surface

Ti% adhering to the surface and consequently, TiO_2 amount increases, as the amount of TIPA added to the bath, is increased [the formation of TiO_2 is given in Eqs(5)-(6)]. However, TiO_2 stops adhering to the polyester surface when the doping agent exceeds 6 mL, and a decrease is observed in Ti %. Reaction mechanism is given below:

$$Ti(CH_3CH_3CHO)_4 + H_2O \longrightarrow Ti(OH)_4 + 4CH_3CH_3CHOH \quad ...(10)$$

$$Ti(OH)_4 \xrightarrow{heat} TiO_2 + 2H_2O \quad ...(11)$$

The high correlation between the amount of absorbed CO_2 and the amount of titanium oxide on the surface is not surprising (Fig. 7). According to Eqs (1)

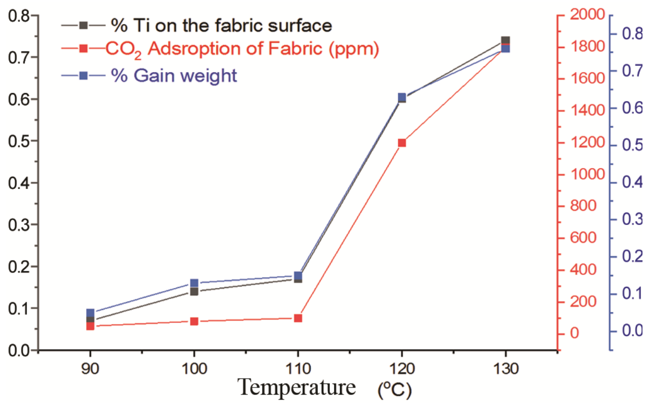


Fig. 7 — CO_2 absorption of fabric according to heat process

- (4), the factors that will increase the photocatalytic effect are light and catalyst[19].

The most exciting result is achieved after heating sample fabrics at different temperatures. The amount of TiO_2 attached to the surface is observed to increase as the temperature increases. Along with this, an incredible increase occurs with the increase in the amount of absorbed CO_2. TiO_2, which cannot adhere to the surface at low temperatures, can enter the inner layers of the polyester surface at high temperatures, which is closer to the softening temperature of polyester. It may be attached to the surface by chemical adsorption rather than physical adsorption, resulting from poor intermolecular interaction[19-21].

Figure 6 shows that as T2 solution is added to a one liter chemical bath, a very slight decrease is observed in the amount of titanium adhering to the surface. As the T2 solution added to a one liter bath is increased from 2 mL to 10 mL, the Ti% measured on the surface is found only around 0.7%.The amount of absorbed $CO_2$is found to be maximum when 6 mL of TIPA is added to a one liter bath, and the amount of titanium on the surface approaches 0.8%.

The Martindale method was employed to determine the abrasion resistance of the produced fabric; Pro White Test Device was passed over the surface of a circular test sample while an abrasive wool fabric with a specified force formed a Lissajous pattern. During this process, the test sample could easily rotate around the axis at the centre, perpendicular to the plane of the test sample. Visual evaluation of surface pilling was performed after this stage of the rubbing experiment. A circular test sample under a certain pressure was abraded with an abrasive (standard fabric) that abrades via reciprocating motion, creating a Lissajous pattern. The test results were performed according to TS EN ISO12945-2 Standards. The burst resistance of the standard sample with a diameter of 44.45 mm, tested by a bursting tester device under a maximum of 6000 N, is 350kP. The burst resistance of the sample produced with optimum parameters is 1100kPa.

The color fastness test is performed to check the resistance of the color when the knitted sample is rubbed in dry and wet form. The rub fastness test is carried out by rubbing the adjacent fabric to the test sample placed on the crockmeter. At the end of the test, the staining of the adjacent fabric was evaluated using the grayscale.

The pilling at 2000 rubs is evaluated on a scale of 1-5. The pilling value of the fabric sample dried at 130ºC, having 0.74-0.76% Ti on the surface is found between 4 and 5. The abrasion test result shows good abrasion resistance at 30000 rubs. The fastness is evaluated on the grayscale, where the minimum fastness is 1 and the maximum is 5. According to the test result from accredited laboratories measurement, the rub color fastness is found between 4 and 5, which indicates an insignificant slight color change or no change. As a result, the polyester bedspread fabric sample coated with 0.74-0.76% Ti on the surface and dried at 130ºC has passed physical tests. Burst resistance is found to be 1100kPa against the standard value of 350kPa.

The SEM image of the fabric produced according to the final parameters is given in Fig. 8. No wear or damage is observed in the yarn structure. Yarn thickness is observed to be 7.24 µm. It is due to the lack of homogeneity of the polyester polymer surface resulting from the processes, such as bath and RAM. Particles of 1µm and below are TiO_2 solid crystals, that adhere to the surface. They can be observed very clearly on the surface.

Figure 9 and Table 1 show the elongation-shear tests of the TiO_2 coated polyester fabric as compared to an uncoated polyester fabric. According to the results, the maximum stress sees a notable increase of 17.929 N/mm^2 after coating. The maximum applied force has decreased by 19.861 N. TiO_2 coated polyester fabric is elongated by 229.714 mm before shearing, whereas the uncoated fabric is elongated by 168.34 mm. The coated polyester mattress fabric is elongated by 459% of its size, and the uncoated polyester mattress fabric is elongated by 336%. These results show that the flexibility of the fabric has increased with the components in the bath coating TiO_2.

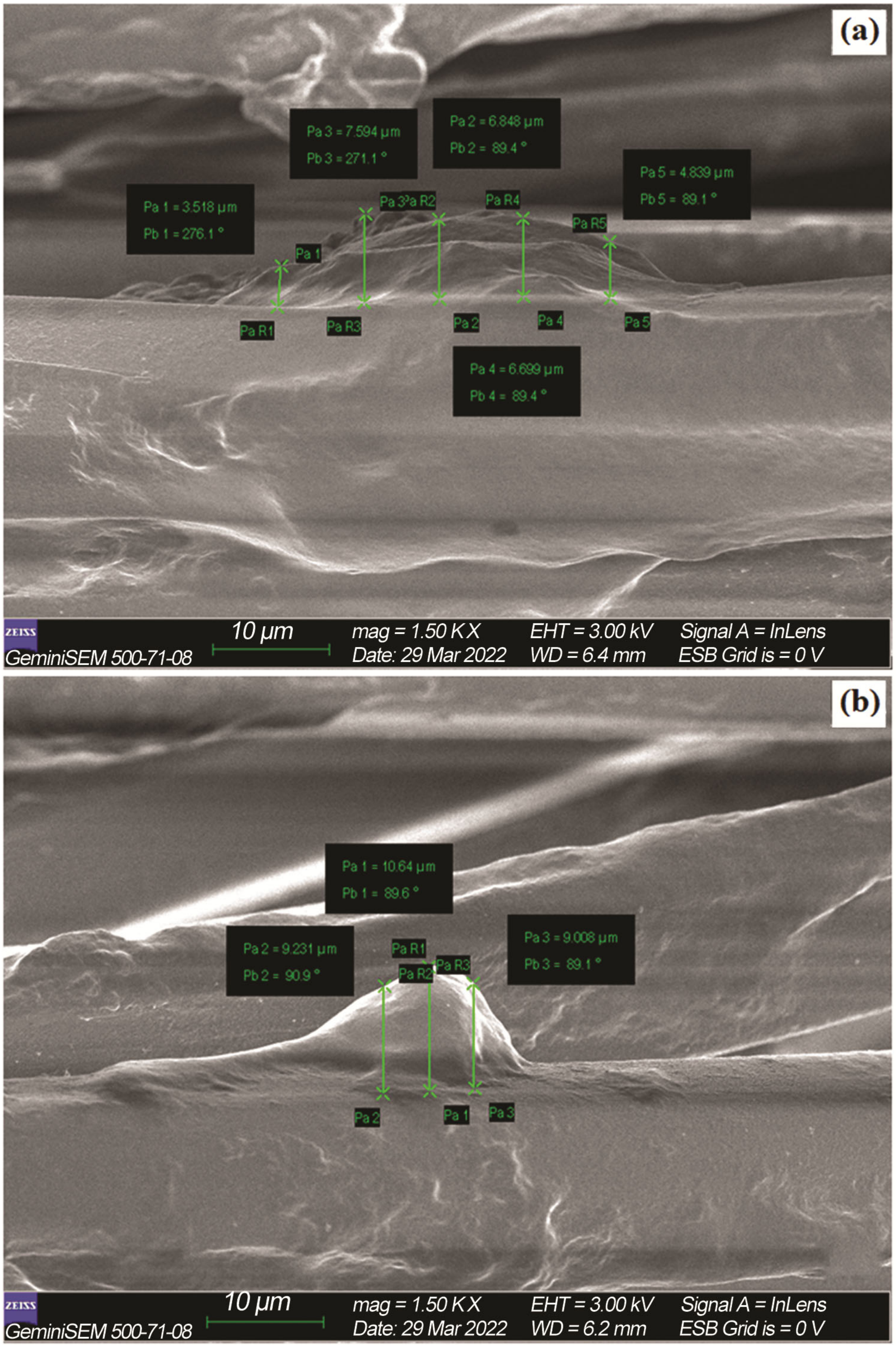


Fig. 8 — (a) 6-7 µm thickness coating of fabric produced at optimum parameters, and (b) 9-10 µm thickness coating of fabric produced at optimum parameters

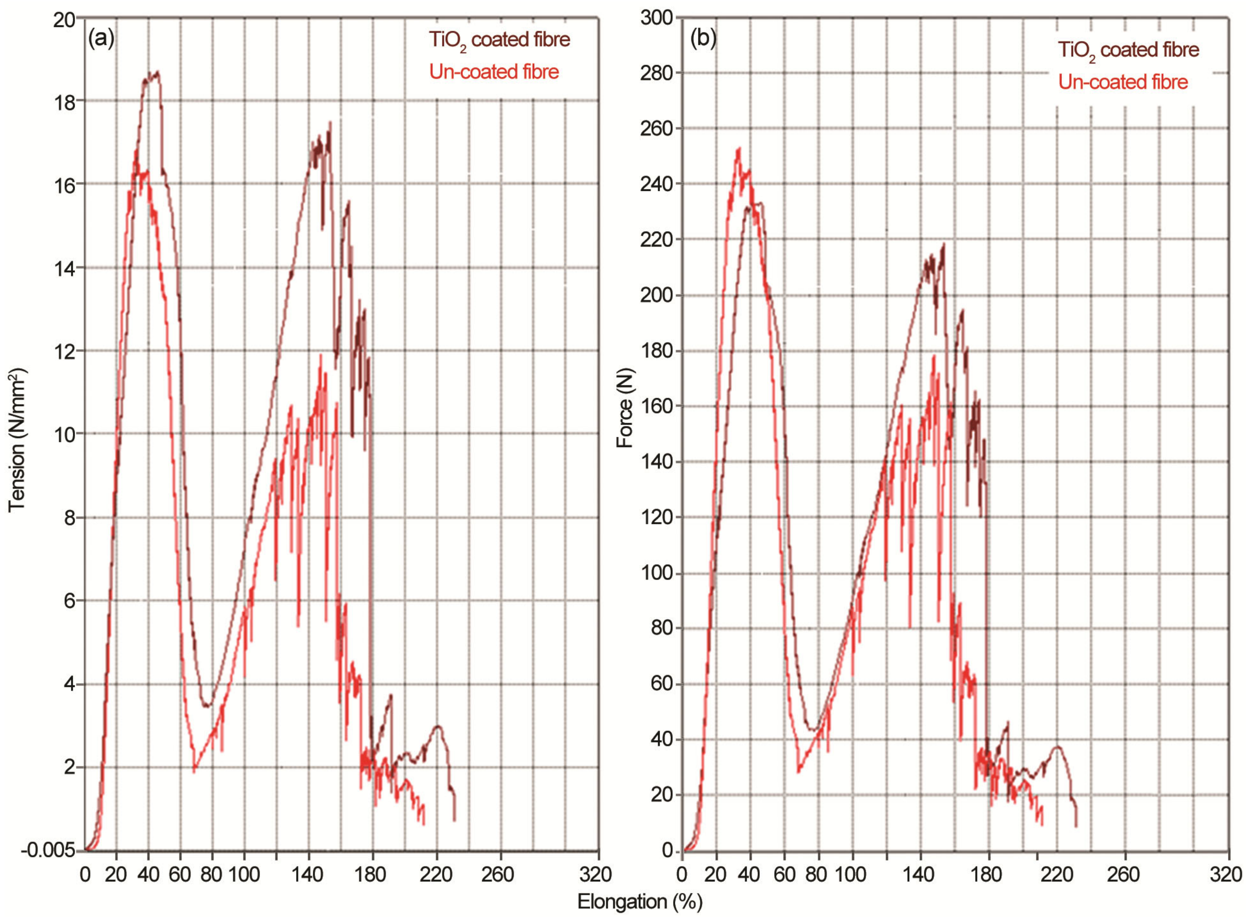


Fig. 9 — (a) Tension-elongation tests, and (b) force-elongation tests

Table 1 — Elongation-shear tests

Fabric	Max stress	Max force	Max elongation	Max elongation
	N/mm^2	N	%	Mm
Un-coated	169.087	253.630	336.629	168.314
Coated	187.016	233.769	459.428	229.714

4 Conclusion

The polyester bedspread surface has been coated with TiO_2 from a suitable titanium source. The CO_2 adsorption by produced fabric samples is measured. Optimum parameters are specified as 6 mL T2 solution/1 L bath and 130ºC fixing temperature. According to the pilling result, the 2000 rubs pilling value of the fabric is 4-5; the abrasion test result shows an abrasion resistance at 30000 rubs. The bursting strength is measured as 1100 kPa. Colorfastness to rubbing is measured in the range of 4-5. Accordingly, the fabric passed physical tests after TiO_2 coating as well. This study can produce a bedspread fabric that improves people's sleep quality, protecting human health and improving sleep quality. At the same time, bath components increase the fabric's flexibility. This study also defines a method for applying some chemicals to the fabric surface.

Further studies on the project are ongoing; the other parameters that the fabric should possess for producing a full textile product are under study. The samples passed the 24-48-72-h irritation test, carried out by the accredited laboratories of Hacettepe University Faculty of Pharmacy, by showing no sign of erythema and edema.

Acknowledgement

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