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Study on the Hydrophobicity and Antibacterial Activity of Silica Sol-Chitosan-HDTMS Treated Cotton Fabric Dipped in an Aquas Media

Hidrofobnost in protibakterijska aktivnost bombažne tkanine, obdelane s silicijevim solom, hitozanom in HDTMS iz vodnega medija

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Abstract

A hydrophobic surface with an antibacterial property has numerous uses, including self-cleaning, anti-sticking, anti-contamination, sports apparel, and wound healing/implant materials. The durability of the coating in an aquas media (pH 7.4) is a vital requirement for use in technical textile sectors, particularly in medical applications. In this study, we used silica sol, chitosan and hexadecyltrimethoxysilane (HDTMS) to create exceptionally hydrophobic surfaces with antibacterial properties on cotton fabrics. First, cotton fabric was treated with silica sol, which was produced by the hydrolysis and condensation of tetraethoxysilane (TEOS) in an alkaline environment. After that, chitosan was applied on the silica sol-treated fabric to add an antibacterial characteristic. The silica sol-chitosan-treated fabric was then given a hydrolysed HDTMS treatment to give a highly hydrophobic property. The hydrophobicity was assessed by measuring the water contact angle, while the AATCC-147 test protocol was used to assess the antibacterial property. The developed fabric exhibited a strong hydrophobic property. The fabric samples were immersed in an aquas media for 30 days to assess the coating durability by observing changes in hydrophobicity and anti-bacterial activity in terms of the zone of inhibition (ZOI). After 30 days of immersion in the aquas media, it was observed that the contact angle decreased from 151.7° to 129.5°, and the ZOI increased from 1 mm to 5 mm, which indicates an increase in anti-bacterial activity in relation to time of immersion. The wicking characteristics of coated and uncoated fabrics were also measured to determine how coating affects the wicking behaviour of fabric. EDS was performed to observe the coating stability for coated-dipped fabric samples after 30 days. SEM analysis was performed to examine the surface morphology, while FTIR was used to determine the surface functional groups after coating and changes after dipping in the aquas media. The developed hydrophobic cotton fabrics with anti-bacterial properties may help in the fabrication of natural biomaterials and other technical textile products.

Keywords: cotton, hydrophobic, antibacterial, silica sol, chitosan

Izvleček

Hidrofobna površina s protibakterijskimi lastnostmi ima številne uporabne lastnosti, vključno s samočistilnimi, proti sprijemanju in zamazanju, ki so pomembne za športna oblačila in materiale za celjenje ran ter vsadke. Obstojnost obdelave v vodi (pH 7,4) je bistvena zahteva za tehnične tekstilije, med njimi zlasti za medicinske tekstilije. Za izdelavo izjemno hidrofobne površine s protibakterijskimi lastnostmi na bombažni tkanini je bil v tej raziskavi uporabljen silicijev sôl, hitozan in heksadeciltrimetoksisilan (HDTMS). Najprej je bila bombažna tkanina obdelana s silicijevim sôlom, ki nastane s hidrolizo in kondenzacijo tetraetoksisilana (TEOS) v alkalnem okolju. Na tako obdelano tkanino je bil v naslednjem koraku za dosego protibakterijskih lastnosti nanesen hitozan. Sledila je naknadna obdelava s hidroliziranim HDTMS za dosego visoke hidrofobnosti. Hidrofobnost je bila ocenjena z merjenjem stičnega kota vode, protibakterijske lastnosti pa so bile določene z merjenjem cone inhibicije (ZOI). Obdelana tkanina je izkazala visoko hidrofobnost. Za oceno obstojnosti obdelave sta bili hidrofobnost in cona inhibicije (ZOI) določeni tudi na vzorcih, ki so bili za 30 dni potopljeni v vodni medij. Po 30 dneh potopa vzorcev v vodni medij se je stični kot vode zmanjšal s 151,7° na 129,5°, ZOI pa se je povečal z 1 mm, na 5 mm, kar kaže na povečanje protibakterijske aktivnosti s časom potopitve. Izmerjena je bila tudi vpojnost obdelanih in neobdelanih tkanin. S pomočjo analize EDS je bila proučena stabilnost obdelave na vzorcih tkanin. Analiza SEM je bila izvedena za proučevanje morfologije površine, FTIR pa je bil uporabljen za določitev funkcionalnih skupin na površini obdelanih vzorcev in kemijskih sprememb površine po namakanju v vodnem mediju. Razvoj hidrofobne bombažne tkanine s protibakterijskimi lastnostmi je lahko v pomoč pri izdelavi naravnih biomaterialov in drugih izdelkov iz tehničnih tekstilij.

Ključne besede: bombaž, vodoodbojnost, protimikrobnost, silika, hitozan

1 Introduction

Today, fabric made of natural fibres, such as cotton with hydrophobic and anti-bacterial properties, has many applications in different technical textile fields, in particular medical, industrial, military use, etc. The number of uses of hydrophobic surfaces in the technical field is increasing due to their self-cleaning, anti-sticking and anti-contamination properties [1, 3]. Natural textile materials, such as cotton, represent an excellent media for bacteria. Natural cotton fibres provide nutrients, oxygen and moisture to bacteria for their growth and multiplication [4–8]. Moreover, fabrics made of natural fibre yarn contain micropores inside the three-dimensional structure of the yarn. These micropores increase the chance of bacteria colony formation because bacteria can easily hide and proliferate inside the micropores [9–12]. The unfavourable tissue reaction of cotton biomaterial is higher than that in synthetic fibres, which declines after about a week [13]. This unfavourable tissue reaction is caused by the high hydrophilicity of cotton fibres because, after being implanted inside the body, their hydrophilic surfaces bind plasma proteins, such as coagulation factor XII, HMWK and prekallikrein [14, 15]. However, this issue can be avoided by generating surfaces that are extremely hydrophobic and have a contact angle of more than 120° [15–17]. Therefore, cotton fabrics require a hydrophobic coating with antibacterial properties for its use in medical, industrial, military and other similar purposes.

The hydrophobicity or hydrophilicity of a surface determines how liquids will interact with it. A hydrophobic surface can be achieved by changing the surface topology (roughness), by reducing reaction surface energy or through both [1, 18, 19]. The degree of wettability of a material can be determined by evaluating the water contact angle (WCA) between the solid and liquid phases. Based on the angle of contact between water droplets and the surface, a substance is categorized as either superhydrophobic (WCA > 150°), hydrophobic (90° < WCA < 150°) or hydrophilic (WCA < 90°) [1, 20]. Various methods are available for the fabrication of superhydrophobic surfaces on cotton, such as sol-gel, layer-by-layer deposition, plasma etching, chemical vapour deposition and nanoparticle deposition [18, 21–23]. Apart from other techniques, the sol-gel method, through the use of silica nanoparticles, has drawn the attention of researchers due to its biocompatibility, eco-friendliness and non-toxicity properties, and non-fluorinated nature with good experimental reproducibility [24, 25]. Zare et al. reported that silicone is extremely biocompatible when it interacts with host tissues because silicone is hydrophobic and has a low surface tension. It has good hemocompatibility and lowers the risk of encrustation when it comes into contact with body fluids [26].

Antibacterial properties on natural fibres can be developed by using natural, organic and inorganic antibiotics. Well-known natural antibiotics are chitosan and chitosan derivatives, while popular organic antibiotics are quaternary ammonium salts, guanidine, zwitterionic betaine compounds, peptide, etc. Similarly, inorganic antibiotics include Zn nanoparticles, Ti nanoparticles, Ag nanoparticles, etc. [27]. The most abundant polysaccharide found in nature is chitosan. Chitosan is a deacetylated derivative of the polysaccharide chitin, which is mostly found in crustacean exoskeletons and has received a lot of interest because of its versatility, non-toxicity, biodegradability and antibacterial characteristics [28–30].

Therefore, in this research, a hydrophobic surface with an antibacterial property was developed on scoured cotton woven fabric by using silica sol, HDTMS and chitosan. The silica sol was formed with the help of the sol-gel technique through hydrolysis and the condensation of tetraethoxysilane (TEOS) in alkaline conditions. First, the scoured fabric was treated with silica sol. The silica-sol treated cotton fabric was then coated with a chitosan solution to achieve an antibacterial property. After that, the chitosan-treated fabrics were coated with hydrolysed hexadecyltrimethoxysilane (HDTMS) to develop a highly hydrophobic surface through a reduction in surface energy.

Coating durability in an aquas media (pH 7.4) is important for application in medical use because post operative wounds generally take four to six weeks to heal [31]. For this reason, textile materials that are used for wound healing/implant materials will be in contact with physiological body fluid for a longer period (at least 30 days), during which time the average pH of physiological body fluid remains at 7.4 [32]. There is limited work on the exposure of silica sol, chitosan and HDTMS coated fabric in an aquas media (pH 7.4) for 30 days and its impacts on hydrophobicity and antibacterial activity.

The aim of this study was thus to develop a non-toxic, biodegradable hydrophobic fabric with anti-bacterial properties by using silica sol, chitosan and HDTMS, and to then study changes in the antibacterial activity and hydrophobicity of the surface when it is dipped in an aquas media (pH 7.4).

2 Experimental

2.1 Materials

The study was conducted using cotton woven fabric that was obtained from a local industry. The details of the fabric are presented in Table 1.

Table 1: Fabric details

Property	Value
Linear density of cotton warp (tex)	20.2
Linear density of cotton weft (tex)	30.1
Weave structure	plain
Fabric areal density (g/m^2)	110
Fabric density (EPCM × PPCM) (EPI × PPI)	26×17 (66 × 43)

The chemicals used for this study were TEOS (tetraethoxysilane), ethanol, NH_4OH, HDTMS (hexadecyltrimethoxysilane), chitosan (deacetylation degree of chitosan was ≥ 75.00%) and acetic acid. All the chemicals were purchased from Sigma-Aldrich. The cotton fabric was first de-sized using an acid de-sizing method to remove sized material and then scoured using sodium hydroxide (NaOH) to remove oily substances.

2.2 Methods

2.2.1 Synthesis of silica sol using sol-gel method

The alkaline hydrolysis and condensation of tetraethoxysilane (TEOS) in ammonium hydroxide (NH_4OH) and ethanol solution was used to make silica sol. A total of 5 ml of ammonium hydroxide (NH_4OH) solution was slowly added, with continuous stirring, into 100 ml of ethanol at 60 °C. The stirring was continued for 30 minutes. Subsequently, 6ml of tetraethoxysilane (TEOS) was slowly added into the solution, drop by drop, and stirring was carried out for 90 minutes to produce the silica solution.

2.2.2 Chitosan solution preparation

Chitosan solution was prepared by dissolving 2g of chitosan into 1000 ml of 2% acetic acid solution. The solution was then stirred for 2 hours.

2.2.3 Hexadecyltrimethoxysilane (HDTMS) hydration

Hexadecyltrimethoxysilane (HDTMS) (0.75 % V/V) was slowly added (drop by drop) into ethanol to

make the solution. The pH of the solution was maintained at 5.0 by applying acetic acid. Thereafter, the solution was stirred for 60 minutes to make an alkylsilanol solution.

2.2.4 Treatment of fabric

The scoured cotton woven fabric samples were dipped for 20 minutes in a silica sol solution. The samples were then padded using a laboratory padder with a wet pickup of 70% to 80% and dried for three minutes at 80 °C [33]. After that, the cotton fabrics were padded with a 2% chitosan solution. The padded fabrics were then dried at 80 °C for five minutes and cured at 140 °C for three minutes [34]. Subsequently, the cured samples were again dipped into HDTMS alkylsilanol solution for one hour and followed by drying at room temperature. Finally, the samples were cured at 120 °C for one hour [33]. The result was coated hydrophobic fabrics.

The durability of fabric coating was studied by immersing the fabrics in an aquas media (pH 7.4) for three days, 10 days and 30 days respectively. Changes in the contact angle and anti-bacterial property of the different dipped fabrics were then measured. The different fabric samples, before and after coating, are presented in Table 2. Moreover, the aquas media (pH 7.4) was prepared using distilled water and sodium carbonate. Sodium carbonate was added to distilled water to maintain the pH of media at 7.4.

Table 2: The different fabric samples before and after coating

Seq. no.	Sample code	Fabric
1	C1	Untreated cotton fabric
2	C2	Coated fabric (silica sol + chitosan (0.2%) + 0.75% (V/V) HDTMS)
3	C3	Dipped in aquas media for 3 days
4	C4	Dipped in aquas media for 10 days
5	C5	Dipped in aquas media for 30 days

2.3 Characterization technique used

Developed fabrics were characterized using the following techniques:

– **contact angle measurement**

The contact angles of different types of fabrics were measured to determine the surface wettability of the untreated, treated and dipped fabric. The contact angle as measured by using a Drop Shape Analyzer, Kruss, Germany.

– **determination of wicking properties**

A published method [35] was used to assess the wicking effect of coated and uncoated fabric. Fabric measuring 200 mm × 25 mm was cut for the purpose of evaluating wicking ability. A clamp was then equipped with a stainless-steel scale. After that, a glass reservoir was added to the frame's base. The fabric was also carefully controlled to avoid bending around its axis. The rise of liquid through the yarn of fabric was observed. The test was terminated when there was no indication that any liquid had been absorbed.

– **characterization of anti-bacterial property**

The antibacterial efficiency of coated fabric was evaluated using the AATCC-147 test protocol with *E. coli* bacteria [6]. A culture media was prepared using agar-agar (2%) and Luria broth (2%) in distilled water. The petri dish, tip, forceps, L rod and media were then sterilized in an autoclave for 15 minutes at 120 °C and a pressure of 1.05 kg/cm^2. The cultured media poured into petri dishes, and the petri dish, L rod, forceps, 1 ml tip box, pipette and fabric were again sterilized using UV sterilization for 15 minutes. A total of 100 µl of bacteria were dispersed in an agar media using an L rod after UV sterilization. The fabric was then placed in the centre of the petri dishes. The petri dishes were covered with paraffin paper. After this, the petri dishes were incubated for 24 hours at 37 °C. The zone of inhibition was determined to test antibacterial activity.

– **scanning electron microscopy (SEM) analysis**

The surface of the untreated and treated fabric was examined using a ZEISS Sigma 500 VP scanning electron microscope (Germany). Since cotton fibre is nonconductive, the fabric was coated with a thin film of gold before SEM measurements.

– **fourier infrared spectroscopy (FTIR) analysis**

Changes in the functional groups of untreated, treated and dipped fabric were observed using a BUKER ALPHA II FTIR spectrometer (Germany). The results were taken in a wavelength of 600 nm to 4000 nm.

– **energy dispersive X-ray spectroscopy (EDS) analysis**

EDS analyses of the uncoated, coated and dipped samples were performed to observe coating stability using an EDS, AMETEK ELECT PLUS device.

– **measurement of tensile strength**

The tensile behaviour of the yarns unravelled from coated fabric and from coated fabric dipped for 30 days were assessed using a Tinius Olsen Universal Testing Machine (UTM).The standard test norm ASTM D2256/D2256M 21 was applied to determine the tensile strength of the unravelled yarn.

– **measurement of tear strength**

The tearing strength of the uncoated and coated fabric was evaluated using the tongue (single rip) procedure (Constant Rate of Extension Tensile Testing Machine) according to standard test norm ASTM D2261.

– **measurement of stiffness**

The stiffness of the uncoated and coated fabric was measured according to the ASTM D1388-18 standard protocol.

3 Result and discussion

The procedure for forming a hydrophobic surface with antibacterial abilities on scoured cotton woven fabric is presented in section 2.2, while the potential chemical reactions are shown in Figure 1. Figure 1a illustrates how silica sol binds to cotton fabric through a condensation reaction between the hydroxyl groups of cotton and silica sol. Similar observations have also been document-

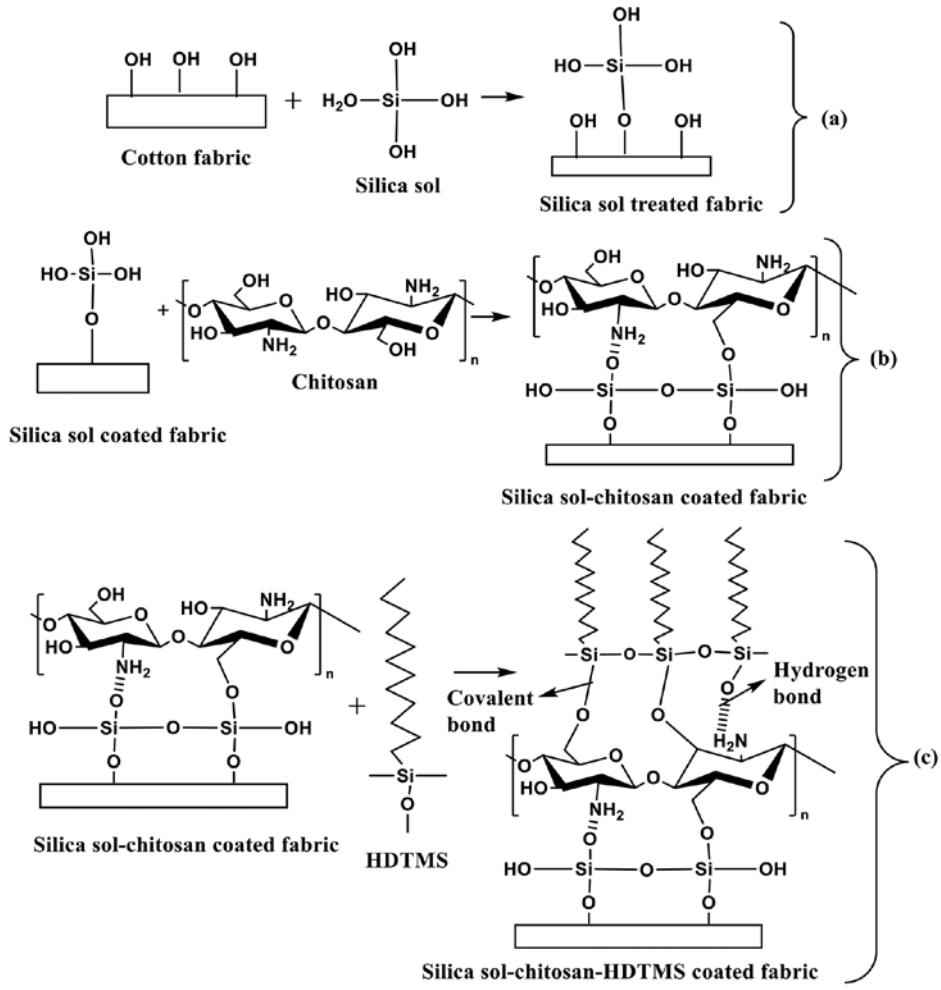


Figure 1: Potential reaction between: a) cotton fibre and silica sol; b) silica sol and chitosan; and c) chitosan and HDTMS

ed in available literature [33, 36]. The structure of chitosan contains hydroxyl and amine groups. Silica sol's hydroxyl group and chitosan's hydroxyl group established a covalent link, resulting in the silica nucleation. Although the amine group cannot create covalent bonds, it serves as an excellent hydrogen bonding partner. As a result, a hydrogen bond formed with the silica sol's hydroxyl group, as seen in Figure 1b, while a similar reaction was also observed by Budnyak [37]. Similar to the chitosan bonds with silica sol, HDTMS attached to chitosan through covalent and hydrogen bonds, as seen in Figure 1c [37].

FTIR spectra can be used to explain the binding mechanism. Figure 2 shows the FTIR spectra of materials that were uncoated, coated and dipped for 30 days. For hydrophilic groups (-OH groups and NH_2 groups), spectra in the range of 3200–3400 cm^{-1} was observed [38, 39]. After coating, there was a drop in the absorbance of spectra in the range of 3200–3400 cm^{-1}. This decrease may be the result of a condensation reaction among the hydroxyl groups of cotton and silica-sol, silica sol and chitosan, and chitosan and HDTMS, although the peak of -OH is still present even after coating, which indicates the presence of some hydroxyl group on the fabric surface even after HDTMS coating. The peak intensity in the range of 3200–3400 cm^{-1} was again raised after dipping the fabric in an aquas media for 30 days. This may be due to the breaking of hydrogen bond between hydroxyl group of HDTMS and NH_2 of chitosan, which results in an increase in hydrophilic groups (-OH and -NH_2). The breaking of bonds may be due to the prolonged direct contact of the samples with water. In water, hydrogen bonds break easily [40]. In addition, the unreacted OH group present on surface of coated fabric may absorb water molecules physically or by hydrogen bond [41, 43].

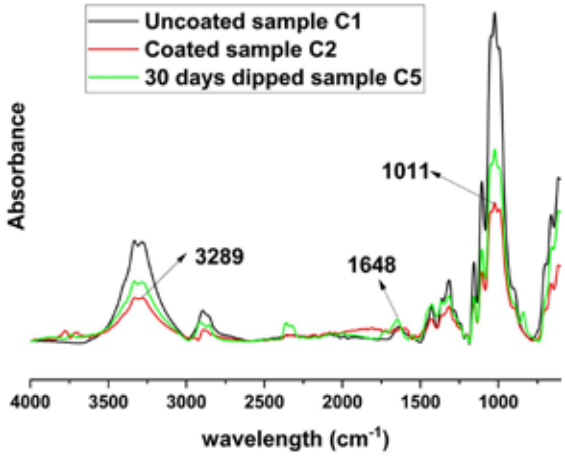
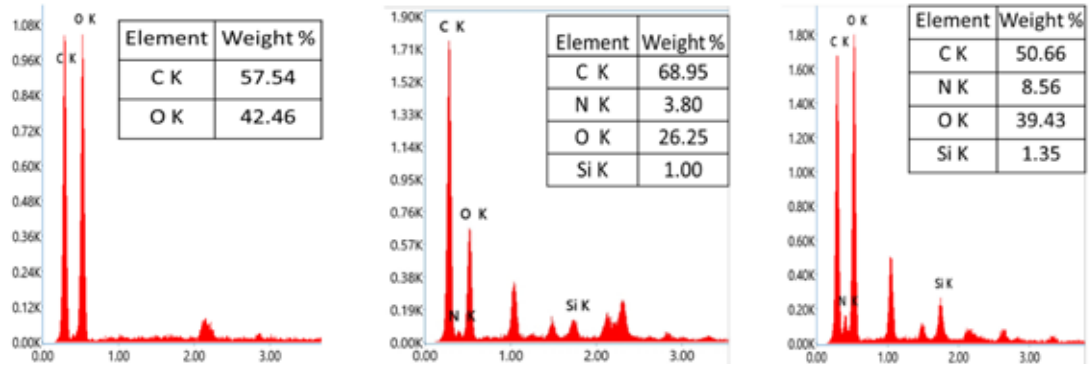


Figure 2: FTIR spectra of uncoated sample C1, coated sample C2 and sample C5 dipped for 30 days

Additionally, the spectra between 1600 cm^{-1} and 1700 cm^{-1} appeared for the sample dipped for 30 days, while the peak between 1600 cm^{-1} and 1700 cm^{-1} normally appeared for amine groups [34]. Moreover, the peaks in the range of 1600 cm^{-1} to 1700 cm^{-1} are further evidence that the hydrogen bond between HDTMS and chitosan were broken, exposing amine groups. The absorption Si-O-Si spectra of silica sol and HDTMS, and the C-O spectra of chitosan in the range of 1100–1000 cm^{-1} appear overlap with the untreated cotton C-O spectra (cellulosic bond), which is similar to the results of previously reported research [33, 43].

EDS analyses of uncoated sample C1, coated sample C2 and sample C5 dipped for 30 days were also performed to observe the stability of coating, and are presented in Figure 3. Only C and O atoms were detected on the surface of uncoated sample, while C, O, N and Si were detected for the coated sample and sample dipped for 30 days. Moreover, the presence



a) *b)* *c)*

Figure 3: EDX spectra of: a) uncoated sample C1; b) coated sample C2; and c) sample C5 dipped for 30 days

of N atoms and Si atoms on the surface after 30 days of dipping confirmed coating stability.

The hydrophobicity of the fabric surface was determined by measuring the contact angle of the sample surface using a drop shape analyser. Table 3 and Figure 4 present the contact angle values for fabrics C1, C2, C3, C4 and C5. Table 3 shows that the contact angle of untreated cotton fabric was not measurable because the water droplet vanished immediately from the fabric's surface as soon as contact with the fabric surface was made. This may be due to the large number of hydrophilic groups present on cotton fibres, fine pores in the yarn and water wicking through the yarn's capillaries, which are ultimately responsible for the extreme hydrophilicity of untreated fabric [44]. It is evident that after being treated with silica sol, chitosan and HDTMS the water contact angle of the coated cotton surface C2 reached a value of 151.7°, which indicates superhydrophobic nature of the surface [21, 45].

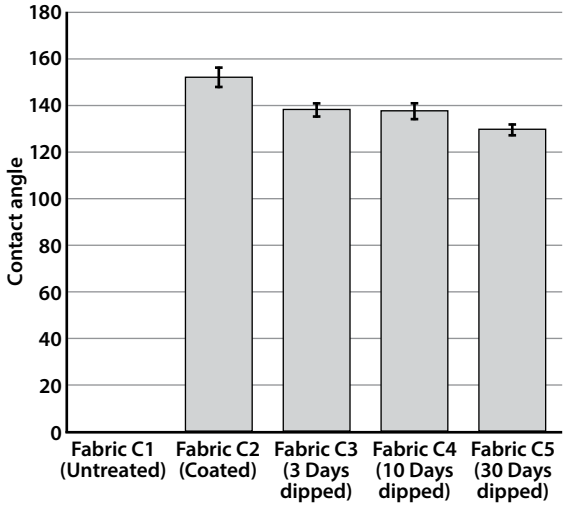
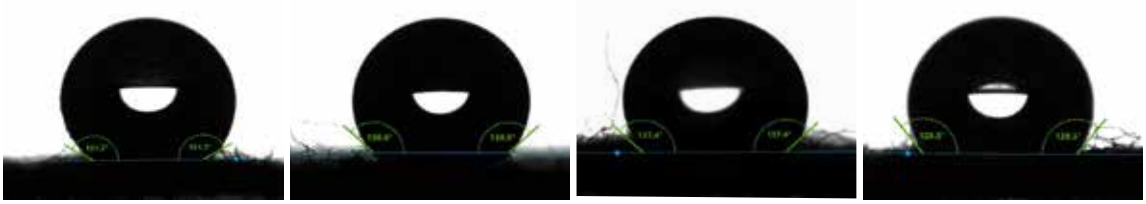


Figure 4: Water contact angle of untreated, treated and dipped cotton fabric

Table 3: Water contact angle of untreated, treated and dipped cotton fabric

Sample type	Contact angle (°)
C1	Not measurable
C2	151.7
C3	138.0
C4	137.4
C5	129.5

The sharp rises in contact angle may be due to the alteration of surface roughness of the fabric following the application of silicon sol, and a reduction in the surface energy of the cotton fabric surface due to subsequent treatment with HDTMS. Therefore, the combined effects of roughness and reduced surface energy lead to a highly hydrophobic surface [6, 33, 46]. The original tracing obtained from the drop shape analyser are presented in Figure 5 for the fabric surfaces of samples C2, C3, C4 and C5. It is evident from Figures 4 and 5 that the contact angle decreased significantly after three days of dipping in an aquas media i.e. 151.7° to 138°. Thereafter, the rate of reduction in the contact angle is relatively lower for the next 30 days. The contact angle was decreased after dipping in an aquas media. There are two probable reasons for the entire surface phenomena in this respect. First, the decrease in the contact angle after dipping was probably due to the cracks in the silica coating on the cotton fibre surface caused by the swelling of cotton. This is supported by earlier studies, which reported that the contact angle decreased after 25 to 30 washings, and the reduction may be due to cracks in silica film on the fibre surface caused by the swelling of cotton [33, 47]. The second reason is the absorption of water molecules by OH groups present on the coated



fabric C2 | *fabric C3* | *fabric C4* | *fabric C5*

151.7° | *138°* | *137.4°* | *129.5°*

a) | *b)* | *c)* | *d)*

Figure 5: Contact angle of: a) treated sample; b) sample dipped for three days; c) sample dipped for 10 days; and d) sample dipped for 30 days

surface during long-term direct contact with water [41, 42]. Furthermore, any material intended for use as a biomaterial must be biocompatible and must also have a contact angle greater than 120° for at least a week after implantation in order to stop this tissue reaction [13, 17]. This is because tissue reactivity normally subsides after about a week [13]. The developed cotton fabric exhibited a contact angle of above 120° even after 30 days of dipping in an aquas media. It is thus possible to use this developed fabric as a wound healing/implant material.

The wicking effect of uncoated and coated cotton fabrics (C1 and C2, respectively) were observed by measuring the wicking height. Figure 6 and Table 4 show the wicking height of cotton fabrics that are uncoated (C1) and coated (C2), respectively. It is evident from Figure 6 and Table 4 that cotton fabrics that are not coated exhibit wicking behaviour and have a wicking height of 8 cm ± 1 cm. This might be caused by the capillaries that exist within the structure of the yarn (intra-yarn spaces) and between the yarns (inter-yarn spaces) [48, 49]. Conversely, the cotton fabric treated with silica sol, chitosan and HDTMS displayed zero evidence of wicking.

Table 4: Wicking performance of uncoated and coated cotton fabric

Fabric type	Maximum wicking height (cm)
Uncoated cotton fabric sample (C1)	8 ± 1
Coated cotton fabric sample (C2)	0

The coating on the fabric surface developed an impermeable barrier by blocking the pores and capillaries inside the yarns and the fabric's structure [50]. As a result, the obstructions limited the capillaries' ability to work in coated fabrics. Moreover, the blocked pore/capillaries can be useful for medical applications because the bacteria can not hide and grow inside the yarn structure of coated fabrics where the pores are blocked.

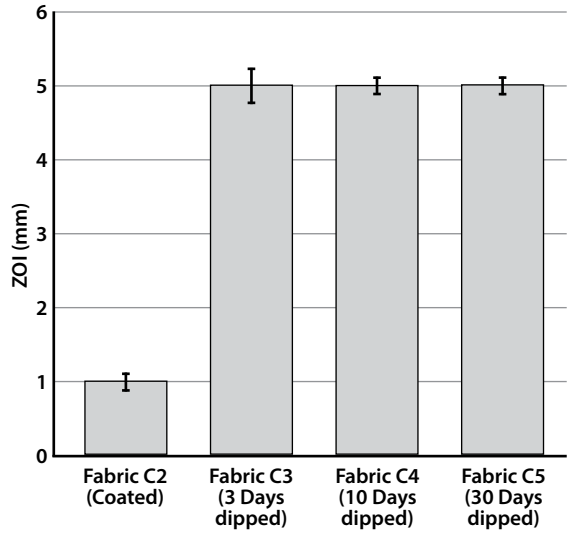
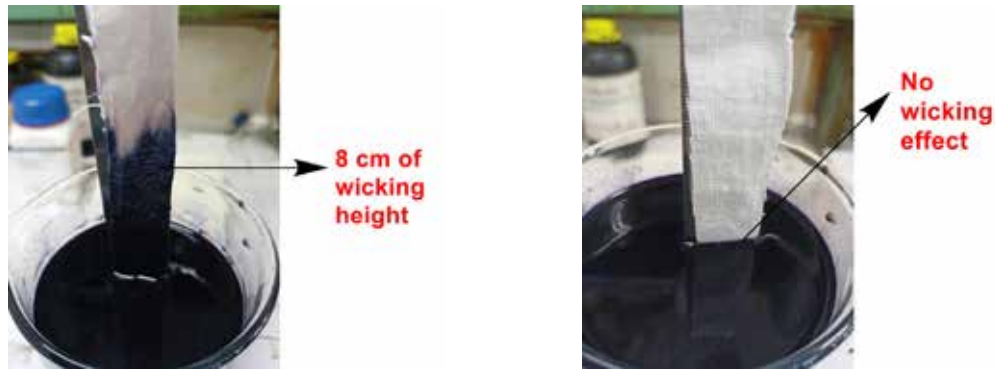


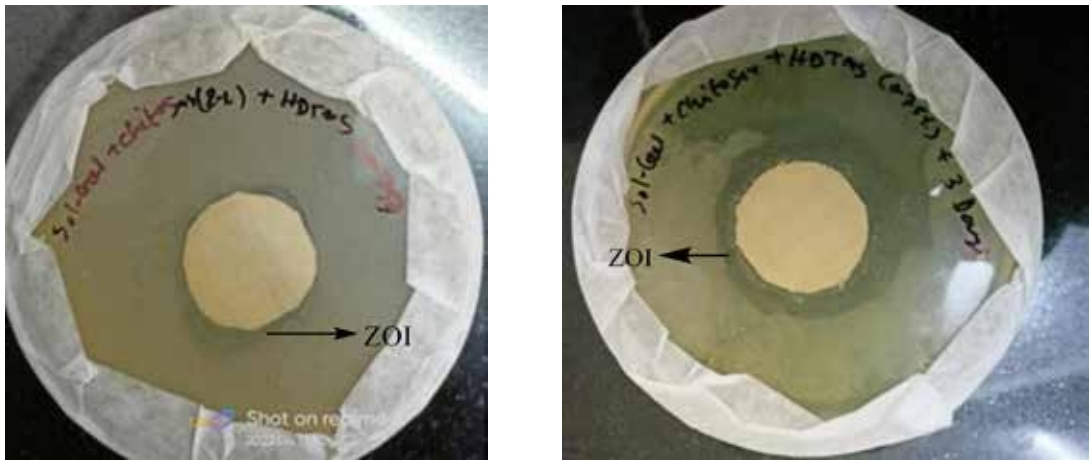
Figure 7: Zone of inhibition (ZOI) of treated and dipped fabric

The anti-bacterial activity of fabric C2, C3, C4 and C5 were measured with help of the agar-agar diffusion test method using *E. coli*. The zone of inhibition (ZOI) was calculated for all the samples and plotted in Figure 7. It was observed that the ZOI of fabric C2 (treated) was relatively low, at around 1 mm, while the ZOI increased in the case of dipped fabric C3, fabric C4 and fabric C5 relative to treated fabric C2. The ZOI of coated fabric sample C2 and dipped sample C3 are also shown in Figure 8. The



a) *b)*

Figure 6: Wicking of: a) uncoated cotton fabric (sample C1); and b) coated cotton fabric (sample C2)



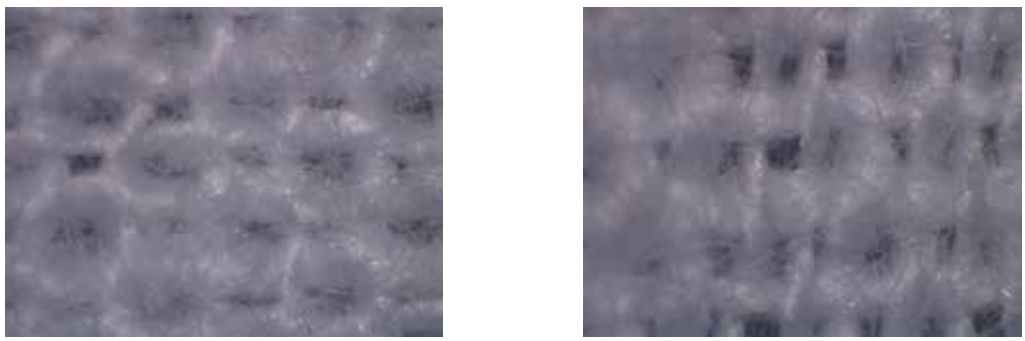
a) *b)*

Figure 8: ZOI of: a) treated fabric C2; and b) dipped fabric C3

increase in the ZOI of the dipped fabric samples (C3, C4 and C5) may be due to the breakage of the hydrogen bond between the -NH_2 of chitosan and the -OH of HDTMS. The hydrogen bond breaks due to long-term direct contact with water. Hydrogen bonds break easily in water [40]. The breakage of the hydrogen bond thus leads to the exposure of the NH_2 of chitosan. The exposure of NH_2 after dipping was also confirmed by FTIR, which showed the increased intensity of the NH_2 spectra at 1648 cm^{-1} (Figure 2). Moreover, when NH_2 was exposed to an aquas media, it became cationic in nature and thus killed bacteria [34].

However, both the coated fabric sample (C2) and dipped fabric samples (C3, C4 and C5) demonstrated antibacterial activity due to the presence of chitosan. There are two types of proposed mechanisms by which chitosan exhibits antibacterial property. In one method, the polycationic nature of the amino group (NH_2) in chitosan interferes with the metabolism system of bacteria by attaching to the cells of bacteria. In the other method, the chitosan binds with DNA to inhibit the synthesis of mRNA [34, 51]. Hence, the improved antibacterial activity of coated samples after dipping in an aquas media may be useful in addressing the antibacterial requirements of biomaterial [9–12].

The surface morphology of textile fabrics before and after coating are observed by using a Leica image analyser and SEM. The Leica image of fabric before and after coating are shown in Figure 9. The SEM images of uncoated sample C1, coated sample C2 and dipped sample C5 are shown in Figure 10. Higher roughness was observed on the SEM image of coated cotton fabrics C2 than on the untreated cotton fabric C1, which confirmed the attachment of chitosan and silicon nanoparticles. Moreover, the roughness of dipped sample C5 increased relative to coated sample. This may be due to the cracks in silica coating caused by the swelling of fibre during long-term direct contact with water [33].



a) *b)*

Figure 9: Leica images of: a) untreated fabric C1; and b) treated fabric C2

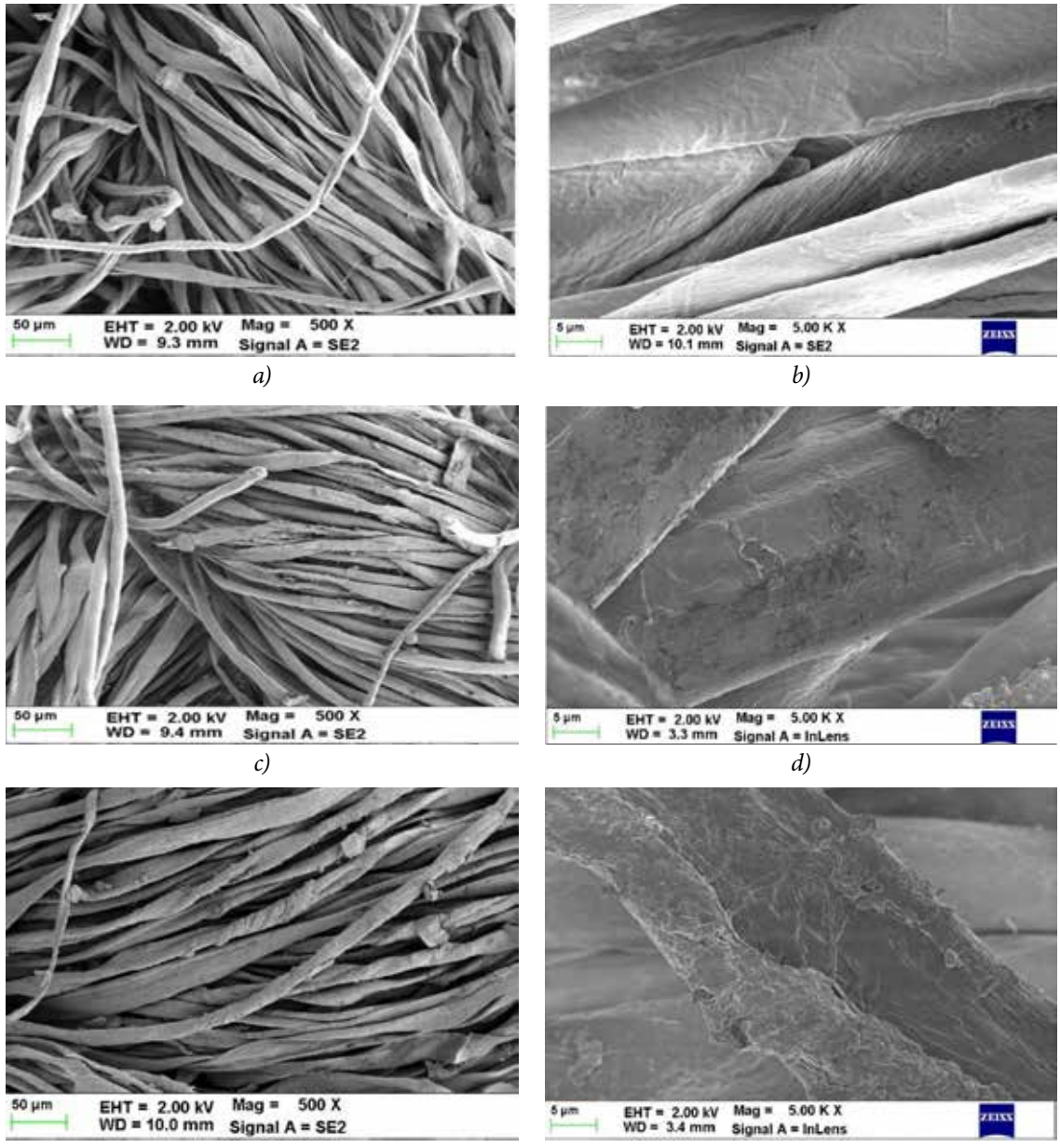


Figure 10: SEM images of: a) untreated fabric C1; b) untreated fabric C1 with higher magnification; c) treated fabric C2; d) treated fabric C2 with higher magnification; e) dipped fabric C5; and f) dipped fabric C5 with higher magnification

The tensile strength of yarn unravelled from treated fabric sample C2 and sample C5 dipped for 30 days was measured and is presented in Table 5. After 30 days of dipping, the yarn's tensile strength decreased by 6.4%. The degradation of cotton fibre in the aquas media may be responsible for this drop in tensile strength.

Table 5: Tensile strength of coated sample (C2); and sample (C5) dipped for 30 days (yarn)

Sample type	Breaking force (N)
C2	3.1 ± 0.3
C5	2.9 ± 0.4

The tearing strength of uncoated fabric sample C1 and coated fabric sample C2 were evaluated and are

presented in Table 6. It is evident from Table 6 that tearing strength decreased by about 2% after coating. Therefore, coating does not have a significant effect on tearing strength ($p > 0.05$).

Table 6: Tearing strength of uncoated and coated sample

Type of sample	Average tearing strength (N)
Uncoated fabric sample C1	15.34
Coated fabric C2	15.08

The stiffness of uncoated fabric sample C1 and coated fabric sample C2 was observed by measuring the bending length and is presented in Table 7. The bending length increased by 10% after coating. Therefore, the stiffness of the fabric is only marginally affected by coating ($p > 0.05$).

Table 7: Bending length of uncoated and coated sample

Type of sample	Average bending length (cm)
Uncoated fabric sample C1	1.8 ± 0.1
Coated fabric sample C2	2.0 ± 0.1

4 Conclusion

The developed cotton fabric exhibited a highly hydrophobic nature when coated with silica sol, chitosan and HDTMS, while the value of the contact angle of the treated fabric was around 151.7°. However, after three, 10 and 30 days of dipping in an aquas media, the contact angle decreased to 138°, 137.4° and 129.5° respectively, meaning a highly hydrophobic nature was maintained even after 30 days. Moreover, the treated fabric indicates zero wicking due to the blocking of pores. In terms of antibacterial activity, coated fabric demonstrated a low antibacterial activity with a ZOI of approximately 1 mm. However, the antibacterial activity was enhanced when the fabric was dipped in an aquas media for three, 10 and 30 days, with a ZOI of 5 mm. It is likely that the decrease in contact angle was due to cracks in the silica coating and the absorption of water molecules by -OH groups present on the coated surface. An increase in ZOI (1 mm to 5 mm) was seen as a result of the breakage of the hydrogen bond between chitosan and HDTMS, leading to the exposure of NH_2 groups of chitosan. The developed hydrophobic cotton fabric, with antibacterial capabilities even after 30 days of dipping in an aquas media, may be beneficial for developing natural biocompatible textile materials that may be used as wound healing/implant materials.

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Optimization of Knitted Fabrics for better Thermo-Physiological Comfort by using Taguchi-based Principal Component Analysis

Optimizacija toplotnega udobja pletiv z analizo glavnih komponent temelječo na Taguchijevi metodi

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Abstract

The water, air permeability and thermal resistance of fabrics are important attributes that have a significant impact on the thermal comfort properties of sportswear fabrics in different environmental conditions. In this work, terry and fleece fabrics were developed by varying the fibre content and mass per unit area of fabrics. Moreover, the thermo-physical properties of the developed fabrics, including air permeability, water vapor permeability and thermal resistance, were analysed before and after washing. The multi-response optimization of the thermal comfort properties of knitted fabrics was performed using principal component analysis (PCA) and the Taguchi signal-to-noise ratio (PCA-S/N ratio) to achieve optimal properties. It was determined that the selected parameters (fabric type, finishing, fibre content and fabric mass per unit area) had a significant effect on the thermal comfort properties of knitted fabrics. The PCA analysis showed that 100% cotton terry fabric before washing with an aerial weight of 220 g/m^2 had higher air and water vapor permeability value, but a lower thermal resistance value.

Keywords: fibre blends, fabric construction, heat and transmission properties of fabric, statistical analysis

Izvleček

Prepustnost za vodo, zračna prepustnost in toplotni upor pletiv pomembno vplivajo na toplotno udobje športnih oblačil v različnih okoljskih razmerah. V tej raziskavi so bila izdelana frotirna in flisna pletiva s spreminjanjem razmerja bombaža in poliestrskih vlaken ter spreminjanjem ploščinske mase. Pred pranjem in po njem so bile pletivom analizirane zračna prepustnost, prepustnost vodne pare in toplotni upor. Večodzivna optimizacija lastnosti toplotnega udobja pletiv je bila izvedena s pomočjo analize glavnih komponent (AGK) in Taguchijevega razmerja signal/šum

(razmerje AGK-S/N) za optimalne lastnosti. Ugotovljeno je bilo, da so izbrani parametri (vrsta pletiva, plemenitenje, vsebnost vlaken in ploščinska masa pletiva) pomembno vplivali na lastnosti toplotnega udobja pletiv. Analiza glavnih komponent je pokazala, da je neoprano 100-odstotno bombažno frotirno pletivo s ploščinsko maso 220 g/m^2 doseglo najvišje vrednosti zračne prepustnosti in prepustnosti vodne pare in najnižji toplotni upor.
Ključne besede: mešanice vlaken, konstrukcija pletiva, toplota, prepustnost, statistična analiza

1 Introduction

Knitted fabrics are widely acceptable textiles because of their good thermal comfort properties, availability in a wide range of products, and simple and low-cost manufacturing techniques [1]. These fabrics provide better freedom in body movements because their structure offers greater flexibility than woven fabrics. Knitted fabrics not only give the wearer a comfortable feeling during use, but also enable the designer to give a more fit look, which makes them ideal for use in sportswear, underwear and casual wear. Other characteristics that make knitted fabrics attractive are their moisture absorption and transmission ability and good handling properties.

Apart from development and advancement in the field of textiles, the rise of consumer awareness through different sources (social media, fashion shows and marketing campaigns by different brands), the dynamics of the clothing/fashion industry are changing at a rapid pace. Currently, clothing brands focus not only on new styles, but also target different aspects of clothing comfort [1]. Although comfort and aesthetics play an important role in the selection of a garment, comfort is becoming increasingly important because of different external factors (moisture, wind, temperature, and social and cultural influences) and internal factors (level of activity). In other words, clothing comfort can be defined as the absence of discomfort or displeasure. Clothing comfort can be categorized in different ways, such as thermo-physiological, sensorial, psychological and garment fit comfort [2]. Thermo-physiological comfort deals with heat and moisture transport properties through clothing, and depends on different factors such as fibre type, fibre content, shape of fibre, yarn characteristic, type of fabrication techniques and finishing treatments, as well as garment fit and size [3].

The air permeability (AP) of a fabric can be defined as the amount of air that passes through a fabric of 100 mm^2 in one second while maintaining a water head pressure difference of 10 mm [4]. The air permeability of fabric indicates how fabric will allow the movement of air through it [5, 6]. The air permeability of fabric relates to thermo-physiological comfort in different ways: air permeable material is likely to transmit water either in liquid or vapor form, while the thermal resistance of fabric is also closely related to the air trapped in the fabric structure. An earlier study [5] showed that increasing the porosity and stitch length of cotton fabrics resulted in an improvement in the thermal insulation properties of knitted fabrics. Thermal conductivity decreases with an increase in the loop length/stitch length of knitted fabric. It is understood that the thermal conductivity value of fibres is higher than the thermal conductivity of the entrapped air layer. The lower thermal conductivity of knitted fabrics from longer loop length or stitch length is the result of the high porosity of fabric [7, 8]. Fayala et al. further concluded that in cellulosic fabrics the surface characteristics and capillary structures of yarns have a significant effect on the thermal and air permeability of fabrics. Not only fibre content but also the fibre fineness, linear density of yarn, size and shape of pores also contribute significantly to the air permeability and thermal resistance of fabrics [9, 10]. In another study, Saha et al. [11] showed that fabric from 100% cotton demonstrated higher air permeability than other fabrics when the proportion of cotton was decreased and polyester content was increased because cotton has more amorphous regions than synthetic fibres, resulting in increased porosity.

The determination of evaporative heat loss under iso-thermal conditions is known as water vapor resistance (Ret). Water vapor resistance is determined as the differentiation of water vapor pressure between the two faces of a textile substrate divided by the resultant evaporative heat flux per unit area in the direction of the gradient. This capability of fabric or clothing to transmit water vapor plays a very important role in the thermo-physiological comfort of clothing. When the body stops sweating, the textile substrate next to the skin releases water vapors in the external environment to create a dry microclimate between the skin and textile fabric [11, 12].

Thermal resistance (R_{ct}) can be defined as the temperature difference between the two faces of a material divided by the resultant heat flux per unit area in the direction of the gradient. The thermal resistance of fabric is directly related to fabric density and thickness. When fabric density increases, the thickness of fabric also increases due to increased air gaps in the fabric structure, which ultimately increases thermal resistance [13]. Bivainyte et al. [14] investigated the impact of a double-layer knitted structure on the thermal transmission characteristics of fabric by using different types of materials, such as cotton, polyester, polyamide and polypropylene, and found that thermal resistance in double-layered knitted fabrics increases with an increase in the thickness of fabric. Due to the thicker fabric structure, more air is trapped, so the pores present within the fabric structure decrease, which ultimately increases thermal insulation. In another study, Saha et al. [11] used 100% cotton, 80% cotton/20% polyester and 60% cotton/40% polyester fibres content in fleece fabric, and found that the maximum thermal insulation was achieved by using 80% cotton/20% polyester because the polyester content made the fabric structure compact due to the presence of more crystalline regions than in 100% cotton fabric. Havenith [15] explained that heat insulation and water vapor resistance were increased with an increase in material thickness because of more air trapped in the thick fabric structure. A similar increase in the thermal resistance of fabric was found after washing, which resulted in more air trapping because the structure of fabrics became more compact after washing [16].

Jamshaid et al. investigated thermal comfort properties such as air permeability, thermal resistance and moisture management for both knitted and woven denim fabric. It was determined that knitted denim fabric demonstrated better thermal resistance, moisture management and air permeability values than woven knitted fabric [17]. Akbar et al. [18] studied the comfort properties of knitted denim. The samples of flax and polyester blends exhibited superior results in terms of air permeability and moisture management compared to samples made from polypropylene and cotton blends. Das et al. [19, 20] conducted a study on the development of advanced knitted structures for the base layer clothing of glacier regions. It was determined that the knitted structure developed from a blend of polyester/elastane (Lycra) and polypropylene/elastane (Lycra) has better thermal properties than pure polypropylene multifilament knitted fabric.

However, there is still a gap in current literature regarding the thermo-physiological comfort properties of different fibre content in the knitted structure of terry and fleece. Moreover, there is no such research available in which the effect of thermo-physiological properties for washed terry and fleece fabric has been explained. The objective of this study was to analyse the effect of knitted fabric type (terry and fleece) on thermo-physiological comfort properties such as air permeability, water vapor resistance and thermal resistance. Also analysed was the effect of different fibre content (cotton and polyester) and areal densities on thermo-physiological comfort properties of fabric before and after washing.

2 Materials and methodology

2.1 Materials

Used in this work were yarns differing in terms of the proportion of cotton (CO) and polyester (PES), which were provided by Masood Textile Mills Limited, Faisalabad, Pakistan. The details of these yarns are presented in Table 1. The chemicals and reagents used in the study were produced by Archroma, Pakistan. All the chemicals and reagents used were of analytical grade.

Table 1: Fibre content and yarn count used for developing knitted fabrics

Seq. no.	Yarn count (tex)	Fibre content
1	29.5	100% CO
2	29.5	80% CO/20% PES
3	29.5	60% CO/40% PES

2.2 Methodology

2.2.1 Fabric preparation

In this work, a total of 36 samples of terry and fleece fabrics were developed according to the combination of selected factor levels [fabric type: (terry and fleece), finishing: (before and after), fibre content: (100% CO, 80% CO/20% PES and 60% CO/40% PES) and fabric mass per unit area: (220, 240, 260)], and are presented in Tables 4 and 5 respectively. All samples were developed using a Mayer & Cie Relanit 4.0 circular knitting machine from Germany. Different machine settings, such as num-

ber of needles, diameter and needle gauge, were optimized to achieve the required areal densities (220, 240 & 260) of fabric samples (Table 2).

Table 2: Machine settings for developing different mass per unit area of fabrics

Seq. no.	No. of needles	Diameter of the cylinder (cm)	Needle gauge
1	1680	76.2	18
2	1872	76.2	20
3	2074	76.2	22

It is evident from Table 2 that as the fabric mass per unit area was increased from 220 g/m^2 to 260 g/m^2, the number of needles was increased to obtain more yarns to cover the required area in order to achieve the required values. The developed terry fabrics were converted into fleece fabric by abrading the surface of the fabric on a Gessner Napper Unipro 2006 raising machine. The thickness, stitch length and stitch density of terry and fleece fabrics, as well as mass per unit area are presented in Table 3.

2.2.2 Fabric processing

After fabric preparation, the samples were moved to the textile processing department of Masood Textile Mills Limited, Faisalabad, Pakistan for further processing:

– *semi-bleaching process*

This process was carried out to increase absorbency in the developed fabric samples. Knitted fabrics were semi-bleached at 110 °C for 55 minutes using Imacol C3G plus (1.5 g/L), Felosan RG-N detergent (0.6 g/L), RUCO-STAB OKP (0.33 g/L), Polipan conc. (0.50 g/L), NaOH flakes (2.50 g/L), and H_2O_2 50% (2.0 g/L).

– *neutralizing*

After the semi-bleaching process, neutralization was carried out to eliminate the presence of hydrogen, which can cause shade variations in the samples. All of the semi-bleached fabrics were neutralized at 55 °C for 42 minutes using CH_3COOH (2.0 g/L) and BT-88 catalase (0.2 g/L).

Table 3: Planned parameters and physical properties of knitted fabrics

Fibre content	Fabric type	Mass per unit area (g/m^2)	Thickness (mm)	Stitch length (cm)	Stitch density (cm^{-2})
100% CO	Terry	220	0.76	0.31	1364
100% CO	Terry	240	0.85	0.325	900
100 % CO	Terry	260	0.86	0.34	1080
80% CO 20% PES	Terry	220	0.71	0.305	1364
80% CO 20% PES	Terry	240	0.75	0.325	900
80% CO 20% PES	Terry	260	0.80	0.35	1080
60% CO 40% PES	Terry	220	0.72	0.305	1364
60% CO 40% PES	Terry	240	0.77	0.325	900
60% CO 40% PES	Terry	260	0.78	0.34	1080
100% CO	Fleece	220	0.74	0.31	1452
100% CO	Fleece	240	0.90	0.325	972
100% CO	Fleece	260	1.01	0.34	1160
80% CO 20% PES	Fleece	220	0.83	0.305	1452
80% CO 20% PES	Fleece	240	0.93	0.325	972
80% CO 20% PES	Fleece	260	0.97	0.35	1160
60% CO 40% PES	Fleece	220	0.84	0.305	1452
60% CO 40% PES	Fleece	240	0.87	0.325	972
60% CO 40% PES	Fleece	260	0.95	0.34	1160

– *dyeing*
Cotton/polyester (PES/CO) blended knitted fabrics were dyed using reactive dyes. First, the cotton fibres were dyed in a jet machine using reactive dyes. After the completion of the dyeing process, the unfixed dye was removed in a soaping process.

– *finishing process*
After dyeing, the samples were dried and stabilized through the finishing process in Monforts Stenter 6 chambers (80 m/minute, 50–250 °C, steam 2 bar) for the purpose of achieving shape-retention, crease-resistance and resilience properties.

– *washing*
Later, 18 knitted terry and 18 fleece fabric samples were washed to give them special effects. The washing was performed in a Tonello washing machine using pre-cleaning, nano-bubble technology and neutralization.

The liquor ratio of water and Felosan RGN was maintained at 1:20 (g/L) for pre-cleaning. The fabric samples were soaked in the solution at 70 °C for 5 minutes. The neutralization of knitted samples was then performed in an acidic medium.
Using nano-bubble technology, a mixture of sodium hypochlorite (NaOCl) (400ML/L) and water was used to discharge colours from the garment surface. Nano-bubble technology uses less water and chemicals. Flow rate was maintained at 16 min/L with a pressure of 2.3 bar for five minutes, not only to remove the colour but also to give a tone-down effect to fabrics This process was performed in a Tonello washing machine [21].

2.3 Characterizations of knitted fabrics

2.3.1 Mass per unit area

The mass per unit area of samples was determined according to the standard testing procedure set out in ISO 3801:1977. Fabric samples were cut using a circular cutter with an area of 100 cm^2. After cutting the required fabric samples, they were weighed on a scale, and the mean values of the mass per unit area of the fabrics were calculated.

2.3.2 Fabric thickness

The thickness of developed knitted fabrics was measured using a Kawabata Evaluation System. A 20 cm × 20 cm fabric sample was cut and positioned on a compression tester in accordance with ASTM D1777. A compression force of 50 g was applied to the samples at a compression rate of 0.0067 mm/s on a fabric area of 2 cm^2. Three readings of each sample at different places were recorded.

2.3.3 Air permeability (AP)

The air permeability of fabrics was determined using an SDL-Atlas M021A fabric air permeability tester according to the ASTM-D737 test method. Fabric samples with an area of 20 cm^2 were tested for air permeability at an air pressure of 100 Pa. Ten values ¸for each fabric (five from the face and five from the back) were recorded at different places to obtain average values.

2.3.4 Thermal resistance (Rct)

The thermal resistance of fabric samples was measured using a PERMETEST measuring device according to the ISO 11092:2014 test method. Polytetrafluoroethylene (PTFE) membrane was used to cover the measuring head of the PERMETEST device and was kept dry to determine thermal resistance under steady-state conditions.

2.3.5 Water vapor permeability (WVP)

The water vapor permeability index was measured according to the ISO BS 7209-1990 test method. It was calculated by expressing the water vapor permeability of the fabric as a proportion of the water vapor permeability of a reference woven fabric.

2.4 Experiment design

2.4.1 Taguchi technique

The Taguchi technique was used for the optimization of the air permeability, thermal resistance and water vapor permeability of fabrics. The different factors and their levels used in this study are presented in Table 4.

Table 4: Factors and levels used in this study

Factors	Level 1	Level 2	Level 3
A: Fabric type	A1-terry	A2-fleece	
B: Finishing	B1-before	B2-after	
C: Fibre content	C1-100% CO	C2-80% CO 20% PES	C3-60% CO: 40% PES
D: Fabric mass per unit area	D1-220	D2-240	D3-260

In this study, three responses (AP, R_{ct}, WVP) were taken for simultaneous optimization using Taguchi-based principal component analysis (PCA). The detailed experiment design with the corresponding mean values of responses can be seen in Table 5.

A Taguchi orthogonal design is a highly fractional orthogonal design that was used to reduce the number of experiment runs. Four factors, two with two levels each and the other two with three levels each, were taken into consideration in our study. An L36 (2^2 x 3^2) orthogonal array was constructed for these factors using Minitab 19 software. To meet the property of orthogonality, every pair of columns, each of possible pairs of elements, appears the same number of times. Each combination was repeated three times as shown in Table 5.

Table 5: Taguchi-based experiment design with mean experimental results

Seq. no.	Coded				Actual				Responses		
	A	B	C	D	Fabric type	Finishing	Blend % CO/% PES	Mass per unit area (g/m²)	Y1: AP (mm/s)	Y2: WVP (g/m² day)	Y3: R_{ct} (m²K/W)
1	1	1	1	1	Terry	Before	100/0	220	1660	86	0.0089
2	1	1	2	2	Terry	Before	80/20	240	403	90	0.021
3	1	1	3	3	Terry	Before	60/40	260	324	95	0.028
4	1	1	1	1	Terry	Before	100/0	220	1660	86	0.0089
5	1	1	2	2	Terry	Before	80/20	240	403	90	0.021
6	1	1	3	3	Terry	Before	60/40	260	324	95	0.028
7	1	1	1	1	Terry	Before	100/0	220	1660	86	0.0089
8	1	1	2	2	Terry	Before	80/20	240	403	90	0.021
9	1	1	3	3	Terry	Before	60/40	260	324	95	0.028
10	1	2	1	1	Terry	After	100/0	220	673	86	0.01
11	1	2	2	2	Terry	After	80/20	240	303	85	0.024
12	1	2	3	3	Terry	After	60/40	260	303	94	0.0335
13	1	2	1	2	Terry	After	100/0	240	661	85	0.012
14	1	2	2	3	Terry	After	80/20	260	300	85	0.027
15	1	2	3	1	Terry	After	60/40	220	323	94	0.028
16	1	2	1	2	Terry	After	100/0	240	661	85	0.012
17	1	2	2	3	Terry	After	80/20	260	300	85	0.027
18	1	2	3	1	Terry	After	60/40	220	323	94	0.028
19	2	1	1	2	Fleece	Before	100/0	240	533	84	0.022
20	2	1	2	3	Fleece	Before	80/20	260	293	85	0.0317
21	2	1	3	1	Fleece	Before	60/40	220	304	92	0.031
22	2	1	1	2	Fleece	Before	100/0	240	533	84	0.022
23	2	1	2	3	Fleece	Before	80/20	260	293	85	0.0317
24	2	1	3	1	Fleece	Before	60/40	220	304	92	0.031
25	2	1	1	3	Fleece	Before	100/0	260	344	83	0.026
26	2	1	2	1	Fleece	Before	80/20	220	303	88	0.027
27	2	1	3	2	Fleece	Before	60/40	240	290	91	0.0317
28	2	2	1	3	Fleece	After	100/0	260	242	82	0.0303
29	2	2	2	1	Fleece	After	80/20	220	240	84	0.0266
30	2	2	3	2	Fleece	After	60/40	240	267	87	0.034
31	2	2	1	3	Fleece	After	100/0	260	242	82	0.0303
32	2	2	2	1	Fleece	After	80/20	220	240	84	0.0266
33	2	2	3	2	Fleece	After	60/40	240	267	87	0.034
34	2	2	1	3	Fleece	After	100/0	260	242	82	0.0303
35	2	2	2	1	Fleece	After	80/20	220	240	84	0.0266
36	2	2	3	2	Fleece	After	60/40	240	267	87	0.034

2.4.2 Principal component analysis and signal-to-noise ratio (PCA-S/N ratio)

An approach combining principal component analysis (weighed by their eigenvalues) and Taguchi signal-to-noise ratio was used to systematize the goals of the novel responses and eliminate the correlation between the many responses. A six-step process for applying the combined PCA-S/N ratio is specified in reference material [22].

Step 1: Convert the original data from the Taguchi experiment into signal-to-noise ratio for responses using the appropriate equation, depending on characteristics that differ according to the nature of the problem under study and that may be categorized as larger-the-better, lower-the-better or nominal-the-best. The mathematical equations (1–3) for S/N ratios are presented below:

Larger-the-better

$$\frac{S}{N}(ratio) = -10\,log\,(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_{ij^2}}) \quad (1)$$

where
y_{ij} represents the i_{th} duplicate of j_{th} response and n represents S, i.e. the number of repetitions.

Lower-the-better

$$\frac{S}{N}(ratio) = -10\,log\,(\frac{1}{n}\sum_{i=1}^{n}y_{ij^2}) \quad (2)$$

Nominal-the-best

$$\frac{S}{N}(ratio) = 10\,log(\frac{\bar{y}^2}{s^2}) \quad (3)$$

where

$$\bar{y} = \frac{y_1 + y_2 + y_3 +, \ldots\ldots\ldots\ldots\ldots\ldots, y_n}{n}$$

$$s^2 = \frac{\sum_{i=1}^{n}(y_i - \bar{y})^2}{n-1}$$

Taguchi's signal-to-noise ratio was applied for air and water vapor permeability responses according to equation (1) and for thermal resistance according to equation (2).

Step 2: Normalize the signal-to-noise ratios:

$$Y_{ij} = \frac{y_{ij} - \min(y_{ij})}{\max\,(y_{ij}) - \min\,(y_{ij})} \quad (4)$$

Step 3: Perform factor analysis using MINITAB 18 statistical software according to the principal component method (PCA) to determine the number of factors to extract in a factor analytic study.

Step 4: Compute MRPI (Multi-Response Performance Index) using the principal component achieved via factor analysis (equations 5–7).

$$X_1 = P_1Y_1 + P_1Y_2 \quad (5)$$

$$X_2 = P_2Y_1 + P_2Y_2 \quad (6)$$

$$MRPI = W_1X_1 + W_2X_2 + W_3X_3 + \cdots \quad (7)$$

where
W_1 and W_2 represent the weights of particular principal components. P_1 and P_2 represent the principal components of factors 1 and 2 respectively.

Step 5: Describe the optimum factor and the combination of its level.
Improved product features are obtained via a higher performance index. The factor's effect and optimum level of each controlled factor can be assessed on the basis of the performance index. The factor's effect (FE) can be determined as (equation 8):

$$FE = \max(MRPI) - \min(MRPI) \quad (8)$$

Step 6: Perform an analysis of variance (ANOVA) to determine significant factors.

3 Results and discussion

3.1 Air permeability

The air permeability of terry and fleece fabrics with different fibre proportions and areal densities was investigated before and after the washing process. It is evident from Figure 1 that all the values of air permeability showed that all fabric samples allow the flow/passage of air through them. However, a significant difference in air permeability was identified between terry and fleece fabrics. It was

determined that the fabric structure has a significant effect on the air flow rate through the fabric. The results also indicated that fleece fabrics offered higher resistance to air movement through the fabric than terry fabrics because the fleece fabrics have pile or plush on their surface, which offered more resistance to air while passing through the fabrics.
Hady and Baky found that fleece fabrics showed higher air resistance due to the more random arrangement of raised fibres from the surface of fabric [23]. Similar results were also reported by Badr and Nahrawv who determined that the raising of fibres on the surface of fabric helped provide an effective barrier against the flow of air [24].
It is evident from Figure 1 that air permeability was decreased both in terry and fleece fabrics after washing. Similar results were also reported by Mavruz and Ogulata [25] who found that air permeability was decreased in knitted fabrics after washing because the structure of fabric becomes tighter. In another study, S. Vasile et al. [26] showed that air permeability decreased with an increase in the number of washes. Increasing the mass per unit area of the fabrics also resulted in a decrease in the air permeability of fabrics, as shown in Figure 1. Published literature [27–28] shows that increasing the mass per unit area of a fabric resulted in a reduction in the air permeability of fabrics because fabric thickness increases with an increase in mass per unit area. This also offers higher resistance to air flow because of a reduction in porosity between the yarns in the fabric. Machine needle gauge, stitch length and material content play an important role in achieving the required mass per unit area of a fabric [29–30]. It is evident from Table 3 that as the mass per unit area of terry fabric was increased from 220 g/m^2 to 260 g/m^2, the stitch length decreased because of the higher number of needles per unit area, which resulted in an increase in the amount of yarn in a particular area. A decreased stitch length ultimately results in a more compact structure, which causes a reduction in air permeability [31]. As stitch length decreased, more yarns became closer to each other, thereby decreasing pore spacing, which facilitated a higher resistance to air flow (9). As the polyester content in the yarn was increased, a gradual reduction in air permeability of knitted fabrics was observed because of the increased packing density of fibres. Nazir et al. [32] developed a mathematical relationship between yarn packing density and yarn count, spindle speed, twist per inch, yarn diameter and yarn hairiness (yarn packing density = 0.21 - 0.000293; yarn count = 0.00000013; spindle speed = + 0.00633; twist per inch = +0.645; yarn diameter = 0.0316). Increasing the polyester content in the yarn resulted in higher yarn packing density, which resulted in a reduction in the number of pores, which in turn might cause reduced air permeability values.

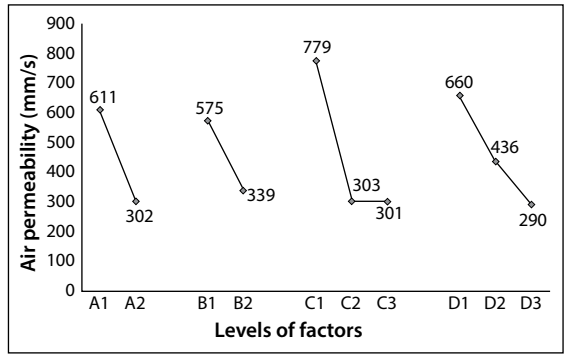

Figure 1: Air permeability of terry and fleece with different proportions of fibre content and mass per unit area before and after the washing process

3.2 Thermal resistance

The thermal resistance of terry and fleece fabrics with different proportion of fibre content and areal densities was determined before and after the washing process. It was established that different parameters, such as fibre content, yarn characteristics, fabric construction and finishing treatments, have a significant impact on the thermal characteristics of fabrics. It is evident from Figure 2 that fleece fabrics showed higher thermal resistance than terry fabrics. After washing, however, thermal resistance increased from 0.0238 m^{2}K/W to 0.0264 m^{2}K/W. A higher value of thermal resistance was found in fleece fabrics than in terry fabric because the fleece has a raised surface that resulted in the greater thickness of the fabric and trapped more air in its structure. As air is a good thermal insulator, the greater thickness of the fleece fabric resulted in higher thermal insulation than terry fabric [35]. Havenith [15] also reported similar results, as he showed that thermal resistivity increased as a fabric trapped more air in its structure.
As the mass per unit area of fabric was increased from 220 g/m^2 to 260 g/m^2, the stitch length also decreased, which resulted in a higher number of stitches in a particular area. The fabric thickness thus increased with an increase in the mass per

unit area of fabric [33–35]. Afzal et al. [16] and Mitra et al. [36] also reported similar results of thermal resistance in knitted fabrics. In this study, the thermal resistance of fabrics increased slightly after the washing process. This may be due to the fact that after washing, the fabric structure became more compact, which contributed to an increase in thermal resistance. However, Holcombe showed that washing did not have any impact on the thermal resistance of polyester/cellulosic fabrics [37]. Increasing the polyester content of the yarn, however, increased the thermal resistance of fabrics.

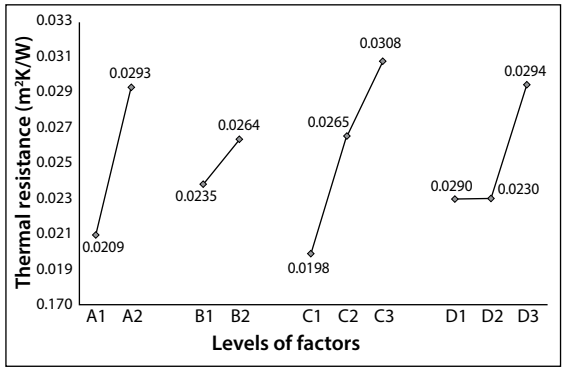

Figure 2: Thermal resistance of terry and fleece with different proportions of fibre content and mass per unit area before and after the washing process

3.3 Water vapor permeability

Water vapor permeability is a measure of how much vapor is transmitted through a material. It is evident from Figure 3 that terry fabric showed higher water vapor permeability than fleece fabrics, while both fabrics structures showed a decreasing trend of vapor permeability after the washing process.

Kandhavadivu et al. [38] reported that piled/fleece fabrics demonstrated a slower transfer of moisture than other knitted structures because of lower contact of fibre with moisture and increased thickness of material. It is evident from Figure 3 that after washing water vapor permeability decreased slightly, from 89 to 86 g/mm^2. This slight decrease in water vapor permeability may be attributed to an increase in fabric hairiness after washing, which offered more resistance to the flow of water vapors through the fabric thickness.

As mentioned earlier, the packing density of yarn increases with an increase in polyester content in the cotton/polyester blend, which resulted in a reduction in the inter-fibre spacing. Water vapor permeability is inversely proportional to the thickness of fabric; increasing the value of needle gauge decreases the stitch length of fabric. As a result, the GSM of fabric increases and the water vapor permeability of the fabric decreases [39, 40]. A decreasing trend in moisture vapor permeability was observed with an increase in the mass per unit area of fabrics as shown in Figure 3. Reducing the gaps in the fabric structure caused an increase in material thickness, which offered more resistance in moisture transportation from one side of fabric to the other.

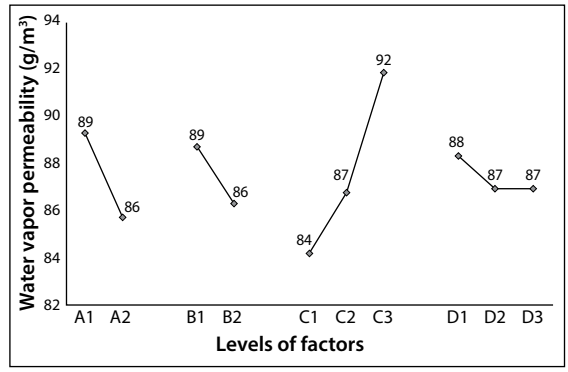

Figure 3: Water vapor permeability of terry and fleece with different proportions of fibre content and mass per unit area before and after the washing process

3.4 Optimization using principal component analysis combined with the signal-to-noise ratio (PCA-S/N ratio) method

The multi-response optimization of AP, WVP and R_{ct} was performed using principal component analysis (PCA). Initially, the S/N ratio values for the responses were determined as shown in Table 6. The S/N ratios were computed as larger-the-better for AP and WVP according to equation 1 and lower-the-better for R_{ct} according to equation 2. In the second step, normalized S/N ratios were calculated according to equation 4 for all the output variables to eliminate the probabilities of error and normalize several quality characteristics as presented in Table 5. In the third step, principal component analysis was performed using the results of normalized S/N ratios. The first principal component has an eigenvalue greater than one, and was considered for further analysis. The principal component analysis matrix is presented in Table 7. The data from Table 7 were used to calculate X1, X2 and MRPI according to equations 5–9, and the results are presented in Table 8.

$$\mathrm{MRPI} = 0.656(\mathrm{X}_1) + 0.323(\mathrm{X}_2) + 0.020(\mathrm{X}_3) \quad (9)$$

Table 6: S/N ratios of experimental results

Seq. No.	S/N ratios			Normalized S/N ratio			MRPI
	Y1: AP (mm/s)	Y2: WVP (g/m^2 day)	0.7630	AP	WVP	R_{ct}	
1	64.402	38.700	0.3931	1.000	0.331	0.000	0.7630
2	52.094	39.076	0.4414	0.268	0.633	0.639	0.3931
3	50.186	39.532	0.7630	0.154	1.000	0.854	0.4414
4	64.402	38.700	0.3931	1.000	0.331	0.000	0.7630
5	52.094	39.076	0.4414	0.268	0.633	0.639	0.3931
6	50.186	39.532	0.7637	0.154	1.000	0.854	0.4414
7	64.402	38.700	0.3931	1.000	0.331	0.033	0.7637
8	52.094	39.076	0.4414	0.268	0.633	0.639	0.3931
9	50.186	39.532	0.4480	0.154	1.000	0.854	0.4414
10	56.554	38.660	0.1792	0.533	0.299	0.084	0.4480
11	49.603	38.619	0.4016	0.120	0.266	0.739	0.1792
12	49.603	39.456	0.4193	0.120	0.939	0.988	0.4016
13	56.402	38.561	38.396	0.524	0.220	0.222	0.4193
14	49.542	38.599	31.361	0.116	0.250	0.827	0.1735
15	50.180	39.504	31.046	0.154	0.977	0.854	0.4337
16	56.402	38.561	38.396	0.524	0.220	0.222	0.4193
17	49.542	38.599	31.361	0.116	0.250	0.827	0.1735
18	50.180	39.504	31.046	0.154	0.977	0.854	0.4337
19	54.533	38.508	33.134	0.413	0.177	0.675	0.3415
20	49.303	38.577	29.981	0.102	0.233	0.946	0.1609
21	49.589	39.233	30.170	0.119	0.760	0.930	0.3420
22	54.533	38.508	33.134	0.413	0.177	0.675	0.3415
23	49.303	38.577	29.981	0.102	0.233	0.946	0.1609
24	49.589	39.233	30.170	0.119	0.760	0.930	0.3420
25	50.696	38.392	31.688	0.185	0.084	0.799	0.1644
26	49.603	38.856	31.361	0.120	0.457	0.827	0.2426
27	49.247	39.192	29.981	0.099	0.727	0.946	0.3184
28	47.658	38.288	30.367	0.004	0.000	0.913	0.0209
29	47.589	38.517	31.487	0.000	0.185	0.817	0.0760
30	48.490	38.752	29.355	0.054	0.373	1.000	0.1757
31	47.658	38.288	30.367	0.004	0.000	0.913	0.0209
32	47.589	38.517	31.487	0.000	0.185	0.817	0.0760
33	48.490	38.752	29.355	0.054	0.373	1.000	0.1757
34	47.658	38.288	30.367	0.004	0.000	0.913	0.0209
35	47.589	38.517	31.487	0.000	0.185	0.817	0.0760
36	48.490	38.752	29.355	0.054	0.373	1.000	0.1757

Table 7: Principal component analysis matrix

Seq. no.	Principal component	Eigenvalue	Proportion	Eigenvector
1	First	1.9689	0.656	−0.687 (y_{11}), 0.196 (y_{12}), 0.700 (y_{13})
2	Second	0.9702	0.323	0.196 (y_{21}), 0.976 (y_{22}), −0.096 (y_{23})
3	Third	0.0609	0.020	0.700 (y_{31}), −0.071 (y_{32}), 0.711 (y_{33})

Table 8: Main effects on MRPI

Seq. no.	Factors	Level 1	Level 2	Level 3	Max – Min
1	Fabric type (A)	7.8749	3.2319	-	0.5988
2	Finishing (B)	7.2074	3.8994	-	0.3039
3	Blend (C)	4.4865	2.4977	4.1226	0.1868
4	GSM (D)	4.7596	3.7256	2.6216	0.1905

Table 9: Results of ANOVA

Seq. no.	Factors	SS	df	MS	F-Test	Contribution (%)
1	A	0.5988	1	0.5988	64.43	38
2	B	0.3040	1	0.3040	32.71	18
3	C	0.1869	2	0.0934	10.05	12
4	D	0.1905	2	0.0953	10.25	12
5	Error	0.2788	30	0.0093	1.00	20
6	Total	1.3685	36			

The main effects on MRPI are presented in Table 8. The table shows that the controllable factors on MRPI value were, in the order of importance, A, B, D, C. The maximum MRPI value indicates better quality. The optimum parameters might therefore be set as A1, B1, C1 and D1.

The results of ANOVA are presented in Table 9. It was determined that fabric type was the most imperative factor, with a contribution of 38%, while finishing type was the second most important factor, with a contribution of 18%. The blend ratio and mass per unit area showed almost the same contribution, while error was determined to be 20%.

All the factors have a significant effect on the comfort properties of fabrics. However, factors A and B have a greater effect than C and D. The optimum response values might be set as A1, B1, C1 and D1, which corresponds to terry fabric, before washing, 100% cotton and 220 g/m^2 respectively. The results showed that this fabric had higher air and water vapor permeability values, but a lower thermal resistance value.

4 Conclusion

This study proposed a method for the multi-response optimization of knitted fabric thermal comfort properties for sportswear using Taguchi-based principal component analysis. Herein, terry and fleece knitted fabrics, with mass per unit area of 220 g/m^2, 240 g/m^2 and 260 g/m^2, were developed using a 29.5 tex (20's Ne) staple spun yarn with three blend ratios (100% cotton, 60% cotton/40 polyester and 80% cotton/20% polyester), while thermal comfort properties, including air permeability, water vapor permeability and thermal resistance, were analysed before and after fabric washing. It was observed that air permeability and water vapor permeability decreased and thermal resistance increased after washing. The multi-response optimization proposed optimum comfort properties for 100% cotton terry fabric with a mass per unit area of 220 g/m^2 before washing. ANOVA results showed that fabric type was the most critical factor affecting fabric comfort properties (38%), while finishing type (18%) had a comparatively smaller contribution to those properties. The blend ratio and mass per unit area (12%) were found to have a minimum effect on fabric comfort properties.

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Evaluation of Sawdust as a Sustainable Dye Source in Ethiopia

Ocena žagovine kot vira trajnostnega barvila v Etiopiji

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Abstract

Increasing eco-consciousness among consumers is creating an expanding niche market for textiles coloured using natural dyes. Natural dyes are derived from different parts of plants, animals (insects and invertebrates) and minerals. Although plant sources are common, a growing global population makes them compete with food crops. Hence, there is a need to investigate alternate avenues for procuring natural dyes. This research examined the feasibility of utilizing extractions of sawdust, a waste product of the wood furniture industry, as a natural colorant. Sawdust is an inevitable waste generated during the conversion of wood into consumer products such as furniture (tables, chairs, etc.), doors and windows. Sawdust, generated in significant amounts by timber mills, may be used in chipboard manufacture. However, the furniture industry disposes of sawdust as fuel or sometimes as communal waste. In this study, segregated sawdust of the most common woods was collected from Ethiopian furniture houses in Addis Ababa and Bahir Dar. Dyeing was attempted on cotton and wool fabrics using individual aqueous extractions. Different shades were obtained only on wool by simultaneous mordanting with mordants, such as copper sulphate, ferrous sulphate and potassium dichromate, using the exhaust dyeing method. Acceptable fastness to light, perspiration, rubbing and washing, as evaluated according to the relevant ISO standards, was obtained. It may be concluded that sawdust is a viable secondary source of natural dyes for textile coloration in Ethiopia and elsewhere.
Keywords: natural dyes, sawdust, mordanting, wool, cotton

Izvleček

Vse globlja ekološka ozaveščenost med potrošniki ustvarja čedalje večjo tržno nišo za tekstilije, obarvane z naravnimi barvili. Naravna barvila se pridobivajo iz različnih delov rastlin, živali (žuželk in nevretenčarjev) in mineralov. Rastlinski viri, ki se najpogosteje uporabljajo za te namene, so zaradi naraščajoče svetovne populacije namenjeni predvsem pridobivanju hrane. Zato je treba raziskati alternativne vire pridobivanja naravnih barvil. V tej raziskavi je bila kot vir naravnega barvila proučena možnost uporabe ekstrakta iz žagovine, ki je odpadek pohištvene industrije. Žagovina je neizogiben odpadek, ki nastane pri predelavi lesa v izdelke za široko porabo, kot so pohištvo (mize, stoli itd.), vrata in okna. Žagovina, ki v znatnih količinah nastaja v obratih za žaganje hlodov, se lahko uporablja pri proizvodnji ivernih plošč. Vendar pa pohištvena industrija odlaga žagovino tudi kot gorivo ali včasih kot komunalne odpadke. Za potrebe te študije je bila ločeno zbrana žagovina najbolj razširjenih gozdov iz etiopskih pohištvenih obratov v Adis Abebi in Bahir Darju. S posameznimi vodnimi ekstrakti so bile barvane bombažne in volnene tkanine. Različni barvni odtenki so bili po metodi izčrpavanja doseženi le na volneni tkanini s sočasnim čimžanjem s sredstvi, kot so bakrov sulfat, železov sulfat

in kalijev dikromat. Dosežene so bile sprejemljive obstojnosti na svetlobo, znoj, drgnjenje in pranje, ki so bile ocenjene v skladu s standardi ISO. Na podlagi izvedene raziskave je mogoče sklepati, da je žagovina uspešen sekundarni vir naravnih barvil za barvanje tekstila v Etiopiji in drugod.
Ključne besede: naravna barvila, žagovina, čimžanje, volna, bombaž

1 Introduction

Before the invention of synthetic dyes, humans used natural colorants from plants, animals, soil, insects and mineral resources. Those colorants were employed for the coloration of human and animal skins, hair, teeth, bones, all types of vegetable fibres (clothing) and woods in a wide range of colours [1, 2]. Since 1856, synthetic dyes have dominated textile coloration because of their low cost, ease of synthesis and application, and excellent fastness properties [3, 4]. Presently the use of synthetic dyes is estimated at around 10,000,000 tons per annum [5]. However, these synthetic dyes, derived from petroleum, not only destroy the environment during their synthesis, but also discharge effluents into water bodies and affect aquatic life [6–9]. Hence, there is growing interest in bringing natural dyes back to the textile sector [1, 10]. In this context, a variety of application methods and dye sources, such as beetroot, henna leaves, eucalyptus bark, tea leaves, turmeric rhizomes, Rubia tinctorum roots, Bixa orellana seeds, kola nut and walnut bark, have been investigated [2, 11–18]. Table 1 lists some common plants used as dye sources and colours obtained.

The consequence of an exploding global population is increased demand for food and living space. This results in intense competition between land required for food (agriculture), for shelter (housing) and for textiles (growing cotton and dye plants) [19]. Hence, any alternative natural dye source is highly attractive [20]. Several authors have proposed the use of by-products and waste products as potential dye sources. The various industries that have been considered include timber, food processing, beverage manufacturing, oil extraction and wine production. Such sources have the additional benefits of providing value addition and employment, and alleviating environmental pollution [10, 21–34].

Ethiopia has a rich history of textile production and coloration. The country is home to a plethora of plants that can potentially provide a rainbow of colours [35]. However, the problem of competition for land mentioned earlier is present here, as well. Getaneh [36] and Shuka [37] have reported on the variety and volume of the furniture industry in Ethiopia. Although the sector is quite disorganized, it produces a wide variety and sizable volume, catering primarily to the domestic market and occasionally to international customers. This research investigated sawdust from the Ethiopian furniture industry as an alternate source of natural dyes. The objectives were to assess the availability of sawdust (in terms of variety and volume), extract colorant from the most common varieties, apply them to textiles and determine the desirable properties of the dyed materials. The purpose of the study was to explore the possibilities of providing a value added product and employment, and creating a niche market.

Table 1: Example of plant dye source and colours obtained

Plant	Scientific name	Part used	CI Name/chemical group	Colour obtained
Burberry	Berberis *vulgaris*	branches and roots	C.I. Natural Yellow 18	yellow–brown
Canadian golden rod	Solidago *canadiensis*	buds, leaves	flavonoid dye	yellow–olive
Madder	Rubia *tinctorum*	roots	C.I. Natural Red 8	red–brown
Hollyhock	Alcea *rosea*	buds	anthocyan dye	brown–green
Privet	Ligustrum *vulgare*	berries	C.I. Natural Black 5	blue–green
Walnut tree	Juglans *regia*	green walnut, brown nut shell	C.I. Natural Brown 7	brown
Ash tree	Fraxinus *excelsior*	bark	flavonoid dye	beige–black
Sticky alder tree	Betula *alnus*	bark	gallotannin dye	beige–black

2 Materials and methods

Ready-for-dyeing plain weave cotton fabric from Bahir Dar Textile Share Company, Ethiopia and twill weave wool fabric from Bhuttico Industries, Himachal Pradesh, India was used as received.
Ferric sulphate, copper sulphate, potassium dichromate, sodium carbonate, sodium chloride, sodium hydroxide, hydrochloric acid and acetic acid were of laboratory reagent (LR) grade. Non-ionic standard soap from SDC was used in all trials.
Segregated sawdust, from the most common woods used, was sourced from different furniture manufacturers in Addis Ababa and Bahir Dar.

Colorant extraction

Aqueous extraction was carried out individually for sawdust from each variety of wood. An initial material to liquor ratio (MLR) (sawdust to water) of 1:20 was employed throughout. The mixture of sawdust and water was stirred thoroughly and the temperature increased to the boiling point. Extraction was continued for about 30 minutes, during which time the MLR was reduced to 1:15. After allowing the mixture to cool, the supernatant-coloured liquid was decanted, filtered, diluted to three times its volume and used for dyeing. The procedure was based on the work done by Trinidad [24] and Ismal [26].

Mordanting

Natural dyes generally have low affinity for textile materials and therefore require the use of mordants (metallic salts) for effective dyeing. Three methods, i.e. pre-mordanting (first treating with mordant and then dyeing), simultaneous mordanting (mordant and dye in the same bath) and post-mordanting (dyeing followed by mordanting), are widely practiced [1, 20]. In this work, simultaneous mordanting was the only method investigated. Individual mordanting processes, using ferric sulphate (5 g/L), copper sulphate (10 g/L) and potassium dichromate (2 g/L), were compared.

Cotton dyeing

Dyeing was carried out at a MLR of 1:30 using the diluted extract solution obtained in the previous step. Sodium hydroxide at 5 g/L and sodium carbonate at 2 g/L were used to obtain a pH of 9.5 in the dye bath. Dyeing was conducted for 30 minutes at 100 °C. Sodium chloride at 5 g/L and the appropriate mordant were added after 15 minutes to assist in exhaustion and fixation. This was followed by a warm rinse and two cold rinses in water to remove unfixed dye. Samples were air dried and conditioned prior to fastness assessment.

Wool dyeing

A MLR of 1:30, similar to cotton dyeing, was also used here. Solutions of 10% hydrochloric acid and 10% acetic acid were used in the dye bath. Half the acid was added at the beginning and the remaining added after 15 minutes, together with the mordant. Dyeing was carried out at 80 °C for a period of 30 minutes. After dyeing, the samples were subjected to a warm rinse and two cold rinses in water, and then air dried and conditioned.

Fastness assessment

Colour retention during use is an important requirement for textile materials. This involves assessing changes in colour due to exposure to light and when subjected to rubbing, both in dry and wet conditions. Moreover, there should be minimal alteration in colour during washing and no staining of other clothing being washed together. These properties were assessed according to the standard test methods listed in Table 2 and using the appropriate equipment.

Table 2: Test standards for fastness properties

Fastness to	Test method
Washing	ISO 105-C06:2010
Perspiration	ISO 105-E04:2013
Rubbing	ISO 105-X12:2016
Light	ISO 105-B02:2014

3 Results and discussion

Sawdust

A survey of furniture manufacturers in Addis Ababa and Bahir Dar determined that Eucalyptus camaldulensis (Eucalyptus Amharic Bahirzaf), Cordial Africana (Sudan teak Amharic Wonza) and MDF (medium density fibre wood) were the three most commonly used woods. Wonza was relatively expensive and thus lower quantities were used. The source was both indigenous and imported. Saw mills in Jimma, in the Oromia region, reportedly produce the highest furniture grade timber in

Ethiopia. The quantity of sawdust produced varied with the type of wood used and the design produced. No records were maintained in this regard, as sawdust is a waste product. Generated sawdust is not segregated and usually burnt as fuel by the workers. Separated samples of each wood type in the form of powder or shavings or small chips were collected. All samples were powdered in the lab prior to extraction.

Extraction

Each wood type yielded a different coloured solution after extraction. As can be seen in Figure 1, eucalyptus (a) produced pale yellow, MDF (b) medium brown and wonza (c) dark brown colours.

a) b) c)

Figure 1: Coloured solution from sawdust after extraction: a) eucalyptus, b) MDF and c) wonza

Dyeing

Cotton fabric was not coloured or was minimally stained, irrespective of the source and mordant combination used. The stain, if any, was lost during the rinsing process after dyeing. Hence, cotton was not dyed using sawdust extracts in this trial. This agrees with the theory that vegetable fibres (cellulosic) possess a low affinity for most natural dyes [1, 20]. These samples were therefore not subjected to further evaluation.

In comparison, wool fabric, being a keratinous animal fibre, was easily dyed using sawdust extracts. The hue obtained differed based on the mordant, with ferric sulphate (a) producing a reddish beige shade, potassium dichromate (b) a khaki shade and copper sulphate (c) a greenish shade. This is shown in Figures 2 to 4. Changes in the sawdust source affected the depth of shade in most cases. This reiterated the fact that mordant plays a critical role in deciding on the final shade of textile materials [38, 39].

Figure 2: Wool dyed using eucalyptus sawdust with different mordants: a) ferric sulphate, b) potassium dichromate and c) copper sulphate

Figure 3: Wool dyed using MDF sawdust with different mordants: a) ferric sulphate, b) potassium dichromate and c) copper sulphate

Figure 4: Wool dyed using wonza sawdust with different mordants: a) ferric sulphate, b) potassium dichromate and c) copper sulphate

Fastness

Table 3 contains the ratings of the dyed and or adjacent materials after being subjected to exposure to

Table 3: Fastness testing results

Sawdust source	Mordant	Fastness test ratings				
		Rubbing		Wash	Light	Perspiration
		dry	wet			
Wonza	Copper sulphate	5	4/5	GRADE 1 staining on wool	4–5	No staining or colour change
	Ferric sulphate	5	4/5	GRADE 1-2 staining on wool	5	No staining or colour change
	Potassium dichromate	5	5	GRADE 2 staining on wool	5	No staining or colour change
Eucalyptus	Copper sulphate	5	5	GRADE 2 Staining on wool	4–5	No staining or colour change
	Ferric sulphate	5	5	GRADE 1 staining on wool	5	No staining or colour change
	Potassium dichromate	5	5	GRADE1 staining on wool	5	No staining or colour change
MDF	Copper sulphate	5	5	GRADE 1 Staining on wool	4–5	No staining or colour change
	Ferric sulphate	4/5	5	GRADE 1 Staining on wool	5	No staining or colour change
	Potassium dichromate	5	4/4	GRADE 1-2 staining on wool	5	No staining or colour change

light, washing and rubbing. The results confirm that wool fabric dyed using sawdust extracts from eucalyptus, MDF and wonza possess acceptable fastness traits.

4 Conclusion

This research has identified a potential source of natural dye in the form of the sawdust produced in the Ethiopian furniture industry. A limited variety of shades can be produced by changing the mordant used. Although only wool was dyed, further work can be carried out to ensure cotton dyeing. A detailed cost analysis and the possibilities of commercialization, at least at the cottage-level, also need to be investigated. When commercializing, the environmental impact of metal salts (from unutilized mordants) in the effluent should also be considered.

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Cleaner Production of Bioactive and Coloured Cotton Fabric Using Euclea Divinorum Dye Extract with Bio-Mordants

Čistejša izdelava bioaktivnih in obarvanih bombažnih tkanin z uporabo izvlečka barvila Euclea Divinorum s pomočjo organske čimže

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Abstract

Coloured textile products are more marketable, and are therefore always in higher demand. This has increased the use of synthetic dyes in the textile industry, thus raising environmental pollution associated with synthetic dyes. Natural dyes have been shown to be suitable alternatives. However, the use of metallic mordants during dyeing means the process is not eco-friendly, hence the need to develop bio-mordants that can be used as alternatives to some toxic metallic mordants. In this study, the effects of bio-mordants on the dyeing properties of *Euclea divinorum* Hiern (Ebenaceae) dye extract were assessed using different mordanting methods on cotton fabric. Dyeing characteristics were evaluated in terms of colour fastness and colour strength. Antioxidant textile finishing properties of the natural dye on cotton fabric was determined using the 2,2-diphenyl-1-picrylhydrazyl radical (DPPH) method. The bio-mordants improved the colour strength for dyed cotton fabric from 0.612 to 0.863 and 0.911 for the cotton fabric mordanted with mango and rosemary, respectively. This study identified an important basis of suitable bio-mordants that can be applicable when dyeing cotton fabric with *E. divinorum* natural dye. In addition, the good antioxidant activity of 72.5% indicates that *E. divinorum* dye extract is a promising agent for the future development of bioactive, protective and health textile fabric.
Keywords: natural dye, bio-mordants, antioxidant activity

Izvleček

Obarvani tekstilni izdelki se dobro tržijo in po njih je vedno veliko povpraševanja. Zato so se sintetična barvila v preteklosti veliko uporabljala, to pa je vodilo tudi v povečano onesnaževanje okolja. Naravna barvila so primerna alternativa, vendar je raba kovinskih čimž pri barvanju z naravnimi barvili okolju neprijazna. Iz tega izhaja potreba po razvoju organskih čimž, ki bi lahko zamenjale strupene kovinske. V tej raziskavi so bili ocenjeni učinki organskih čimž na lastnosti obarvane bombažne tkanine z barvilnim ekstraktom iz Euclea divinorum Hiern (Ebenaceae) pri uporabi različnih metod čimžanja. Lastnosti obarvanja so bile ocenjene glede na barvno obstojnost in globino obarvanja.

Antioksidativne lastnosti bombažne tkanine, obarvane z naravnim barvilom, so bile določene z metodo 2,2-difenil-1-pikrilhidrazil radikala (DPPH). Organske čimže so izboljšale globino obarvanja obarvane bombažne tkanine z 0,612 na 0,863 oziroma 0,911 za tkanini, čimžani z mangom oziroma rožmarinom. Ta raziskava je pomembna osnova pri izbiri primernih organskih čimž za barvanje bombažnih tkanin z naravnim barvilom E. divinorum. Poleg tega dobra antioksidativna aktivnost 72,5 % kaže tudi na to, da je ekstrakt barvila E. divinorum obetavno sredstvo za nadaljnji razvoj tekstilij z bioaktivnimi, zaščitnimi in zdravilnimi lastnostmi.
Ključne besede: naravno barvilo, organska čimža, antioksidativno delovanje

1 Introduction

The textile dyeing process is among the leading polluting industrial processes due to use of the toxic synthetic dyes [1]. An environmentally friendly textile dyeing process can be achieved by substituting certain toxic synthetic dyes with natural dyes that have been shown to provide value added to textiles [2–4]. The process of manufacturing synthetic dyes, together with the associated textile application procedures, discharge toxic wastewaters that require costly resources for complete treatment, and thus find their way into the environment [5–7]. Environmental awareness has led to a recent shift to natural dyes, which in turn has stimulated increased research on natural dyes [8, 9].

Most natural dyes exhibit poor to moderate colour fastness on fabric, which has been a major limitation in their use in the textile industries. As a result, natural dyes are used together with mordants that help fix them on textile material. Commonly used mordants include metallic salts that fix dye molecules to the fabric through a combination that involves the dye molecules, the fabric molecules and the metallic ions that form insoluble precipitates [10]. Examples of metallic mordants are potassium aluminium sulphate, stannous chloride, potassium dichromate, copper sulphate, ferrous sulphate, and others [11].

The main purpose of metallic mordants during natural dyeing is to enhance the affinity of the fabric to the dye molecules since the metal ions form coordination complexes that allow the attachment of the dye to the textile fabric [12]. In addition, the use of metallic mordants during natural textile dyeing offers a wider spectrum of shades from a single natural dye extract [13]. The mordanting step in the natural dyeing process is very crucial, especially when dyeing cotton fabric because it lacks functional groups, such as carboxylic and amino acid groups present in other textile fabrics that act as positions where the dye molecules attach [10, 14]. On the other hand, most of these metallic mordants have been found to be harmful and a small amount of the metallic ion takes part in the fixation of the dye to the fibre, hence a huge percentage of it finds its way into the environment [15]. Global restrictions on production industries regarding the use of toxic substance with the aim of curbing increasing environmental pollution have stimulated research on the development of environment-friendly mordants that can be used as alternatives to poisonous metallic mordants [16].

Bio-based mordants are gaining popularity since they are obtained from nature and thus facilitate a suitable approach for making the natural dyeing process a part of green chemistry. Bio-mordants are basically natural substance that are rich in tannins or metal ions, and are mainly obtained from plants. Examples include tartaric acid, tannic acid [17], tamarind seed coat tannins [18], mango bark [19] extracts from myrabolan, pomegranate rinds, banana leave ash, rosemary plant, rhizomes of turmeric, bark of acacia, guava, etc. [7, 20, 21]. In order to lessen the environmental hazards caused by some metallic mordants, there is need to shift to bio-mordants, which will help in realizing the aim of producing ecologically coloured textile materials [22–24].

In addition to colours obtained from different natural dye extracts, functional textile finishing properties have also been achieved. These include antimicrobial [25–29], antioxidant [30, 31], deodorizing [32] and UV-protective properties [33–35]. The discovery of these additional functional properties of textiles brought about by natural dyes is important in the development of healthy and clean textile materials.

Antioxidant activity is one of the most significant properties of bioactive textile since it protects textile material from damage and safeguards the human skin from inflammation and aging due to oxidative stress caused by free radicals [36]. The human skin is continuously exposed to ionizing radicals, which are the primary cause of skin damage and the associated diseases [37]. Ultra-violet radiation is the

main source of free radicals in the environment, and accumulate to such a level that the antioxidants in the skin cannot neutralize them, leading to oxidative stress, which may cause skin cancer and other diseases [38].

Antioxidants, also referred to as free radical scavengers, are substances that react with free radicals and neutralize them, thus counteracting their harmful effects. Studies have shown that antioxidant molecules such as phenols and flavonoids have good anti-cancer activity against skin cancer [39]. The antioxidant activity of textile fabric is achieved through the deactivation of very reactive and destructive radicals in the environment, such as oxygen and nitrogen radicals [40]. It has been shown that natural dyes are suitable agents for achieving antioxidants properties in textile material because they are not toxic and do not irritate the skin [41–43]. Clothes are in direct contact with the skin. For this reason, the antioxidant activity of textile materials is important in the development of bioactive, healthy textile fabrics.

The antioxidant activity of *Euclea divinorum* aqueous root extract has been studied and was found to be between 74.5–82.5% DPPH (2,2-diphenyl-1- pic rylhydrazyl) [44]. The objective of this study was to determine the effects of bio-mordants on the dyeing properties of *E. divinorum* natural dye extract, and to explore its potential as an antioxidant finishing agent for cotton fabric. The durability of the antioxidant properties of textile materials after washing was also determined.

2 Material and methods

2.1 Materials

E. divinorum root bark was collected in Nandi County in Kenya (latitude 0° 01' 59.5" S and longitude 35° 3' 17.3" E). Commercially bleached, plain woven cotton fabric with 20 ends/cm, 13 picks/cm, 29.4 tex (Nm 34) warp count, 29.4 tex (Nm 34) weft count and a mass per unit area of 97.1 g/m^2 was purchased from a textile factory in Eldoret, Kenya, while 2,2 -diphenyl-1-picrylhydrazyl radical (DPPH) was used for antioxidant evaluation.

2.2 Extraction

E. divinorum root bark was washed and dried using sunlight, and then ground into powder form using an electric grinder. The natural dye as then extracted using distilled water. The extraction conditions used were: temperature of 84 °C, time of 146 minutes and M:L 7.5:100, as previously determined [45]. Conical flasks were used as extraction containers and a water bath was used to regulate the temperature. After extraction, the extracts were allowed to cool and filtered using filter paper.

2.3 Bio-mordanting

The mango (*Mangifera indica*) bark bio-mordant was extracted using the procedure described by [24], where extraction was performed at 90 °C for one hour using 75g/L of the sample in distilled water. Rosemary (*Rosmarinus officinalis*) was purchased from a local market, cut into small pieces, dried under the sun and then ground into powder. A mixture of 20g/L of rosemary powder in distilled water was used to extract the mordant at 100 °C for one hour [46]. Bio-mordanting was carried out using the pre-, meta- and post-mordanting methods. For the pre-mordanting method, the wet cotton fabric was immersed in the solution of the mordant using a material to-liquor ratio of 1:50 at 60 °C. Continuous stirring was maintained for one hour, followed by dyeing. In meta-mordanting, the wet cotton fabric was immersed in a flask containing the solution of the mordant and the dye extract using a material to-liquor ratio of 1:50 at 60 °C for one hour. For the post-mordanting method, the previously dyed cotton fabric was placed in flask containing the solution of the mordant using a material to-liquor ratio of 1:50 at 60 °C for one hour [24].

2.4 Dyeing

The cotton fabric was cut into equal sizes of 1g. Wetting of the fabric was performed using 5g/L of non-ionic detergent for 30 minutes prior to dyeing. *E. divinorum* aqueous dye extract was used to prepare the dyebath using a material-to-liquor ratio of 1:40 [1]. After dyeing, the dye bath was allowed to cool. The dyed samples were then washed with cold water to remove the unfixed dyestuff and subjected to soaping with a 2 g/L soap solution, followed by rinsing with water and air drying.

2.5 Colorimetric measurements

The colour characteristics of the dyed samples were measured with a Spectro -Flash X-rite SP62 spectrophotometer, using a D65 source of light and 10° standard observer. The CIELAB coordinates were measured. The un-dyed cotton fabric was used as

the blank. The relative colour strength (K/S) values were determined using the Kubelka–Munk equation (equation 1).

$$K/S = \frac{(1 - 0.01\,R)^2}{2 \times 0.01\,R} \quad (1)$$

where K represents the absorption coefficient, S represents the scattering coefficient and R represents the minimum reflectance of dyed substrate samples.

2.6 Colour fastness

The ability of cotton fabric to retain dye during washing and rubbing, and when exposed to light and perspiration was determined using the relevant standard colour fastness tests. These were conducted according to ISO 105-C02:1989, ISO 105 A02:1993, ISO 105-X12:2000 and ISO AATCC-2009 for washing, exposure to light, rubbing and perspiration fastness, respectively, with some changes, where the grey scale used to rate the colour fastness was between one and five, with five representing the best fastness [25].

2.7 Antioxidant activity

The antioxidant activity of the pure and dyed cotton fabric was evaluated using a DPPH radical scavenging assay [41]. A total of 2.54 cm^2 of the pure and dyed cotton fabric were separately immersed in a test tube containing 50mL solution of 2,2-diphenyl-1-picrylhydrazyl radical (DPPH) in methanol (0.15 mM) and mixed thoroughly. The samples were incubated in the dark at room temperature for 30 minutes. The absorbance of the solution was measured at 517 nm using a UV-Vis spectrophotometer. The percentage antioxidant activity was calculated according to equation 2.

$$A = \frac{A_{control} - A_{sample}}{A_{control}} \times 100\ (\%) \quad (2)$$

where A represents antioxidant activity, $A_{control}$ represents the initial absorbance of the DPPH solution and A_{sample} represents the absorbance of the remaining DPPH solution after incubation with the sample.

The durability of the antioxidant activity of the dyed samples was assessed by subjecting the dyed fabric to washing cycles, with the antioxidant activity determined after every washing. The washing tests were performed by putting the samples into a washing solution comprised of commercial detergent (2 g/L) with a material-to-liquor ratio of 1 : 50. The antioxidant activity was determined after the 1st, 5th and 10th washing cycles [41].

3 Results and discussion

3.1 Colorimetric analysis

The colour characteristics of the dyed samples were measured and are presented in Table 1. In terms of lightness (L^*), the mango and rosemary bio-mordants increased lightness from 63 to 66.33 and 67.27, respectively, providing lighter shades of brown. Similarly, the intensity of the colour increased as indicated by an increase in the value of C^*. The ability of mango and rosemary bio-mordants to form lighter shades indicates that they can be used as substitutes for metallic mordants that are used to brighten the colour of the dye on the fabric, such as alum and tin, an observation that has been reported previously in literature [46]. The polyphenols in the rosemary bio-mordants are responsible for the increased dye absorption by the cotton fabric, which leads to increased colour strength [20]. Pre-mordanting method for both mordants showed the best colour strength, as has been observed in other studies [19].

3.2 Colour fastness

Colour change in terms of washing and perspiration fastness was in the range of 4–5, except for the pre-mordanted fabric using mango and rosemary bio-mordants, which was 5 (Table 2). The pre-mordanting method allows the poly-phenols to attach to the cellulosic fibre then act as bridge between the fibre and the dye molecules, thus improving the ability of the dye to fix to the fabric, which in turn enhances the fastness properties of the dye [20]. The excellent light fastness throughout the dyeing process indicates that the dye-fabric complex formed is resistant to fading during the exposure to ultraviolet radiation [1]. Generally, the colour fastness of the cotton fabric dyed with *E. divinorum* dye extract was very good and is suitable for application in the textile dyeing industry.

3.3 Antioxidant activity

Antioxidant activity as a percentage is measured by the reduction of the absorbance of DPPH. When the phenolic hydroxyl donates a proton to the DPPH radical, the solution is decolorized and its absorbance is reduced [49]. The antioxidant activity of the assayed

Table 1: Colorimetric values and colour strength of the dyed and mordanted cotton fabric

Method	Mordant	L^*	a^*	b^*	C^*	$H°$	K/S	Shade
-	-	63.47	+4.63	+16.86	17.53	74.53	0.612	
Pre	Mango bark	67.27	+9.66	+21.01	23.13	65.32	0.863	
	Rosemary	66.33	+8.65	+18.80	20.72	65.19	0.911	
Meta	Mango bark	64.59	+11.82	+19.55	22.85	58.84	0.708	
	Rosemary	64.74	+10.65	+17.27	20.31	58.30	0.720	
Post	Mango bark	66.14	+9.97	+19.18	21.64	62.51	0.691	
	Rosemary	65.76	+7.26	+16.72	18.22	66.58	0.724	

Table 2: Colour fastness of the dyed fabric using different methods of mordanting

Method	Mordant	Washing fastness		Rubbing fastness		Perspiration fastness		Light fastness
		C.C [a)]	C.S [b)]	Dry	Wet	C.C	C.S	
	Without	4–5	5	5	5	4–5	4–5	5
Pre	Mango bark	5	5	5	5	5	5	5
	Rosemary	5	5	5	5	5	5	5
Meta	Mango bark	4–5	5	5	5	4–5	5	5
	Rosemary	4–5	5	5	5	4–5	5	5
Post	Mango bark	4–5	5	5	5	5	5	5
	Rosemary	4–5	5	5	5	5	5	5

[a)] colour change, [b)] colour staining

cotton fabric samples is shown in Table 3. Dyeing with the *E. divinorum* dye extract increased the antioxidant activity from 26.9% (undyed cotton) to 72.5% (dyed cotton), which can be attributed to the molecules adsorbed by the cotton fabric from the dye extract, which consequently imparts the radical scavenging activity into the fabric. The antioxidant activity of aqueous root extracts of *E. divinorum* was found to be between 74.5–82.5% DPPH [46]. It was also noted that the antioxidant activity of the fabric samples bio-mordanted with mango (82.4%) and rosemary (85.3%) was higher than that of the un-mordanted (72.5%) fabric, which could be due to the additional activity from the bio-mordant extracts that have been shown to have good antioxidant activity [50]. The mango bark extract comprises polyphenols that are responsible for the radical scavenging ability of the extract [48], [51].

The durability of the antioxidant activity after washing cycles was as indicated in Figure 1. A subsequent

Table 3: Antioxidant activity of the sample fabric

Sample	Antioxidant activity (%)
Undyed cotton	26.9
Dyed	72.5
Mango mordanted	82.4
Rosemary mordanted	85.3

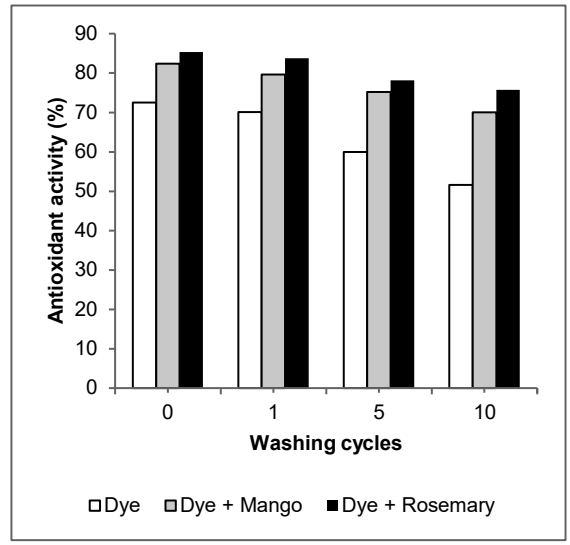

Figure 1: Antioxidant activity of dyed and mordanted cotton fabric after washing cycles

reduction in the antioxidant activity of the dyed samples was observed, and was found to be similar to what has been reported in other studies. However, the reduction in antioxidant activity became minimal after the 5th washing cycle, which is a significant attribute for this extract and can be ascribed to its good fastness properties [36]. The rate of reduction was lower in the mango and rosemary mordanted fabric than in the unmordanted fabric due to the enhanced fastness of the dye to the fabric [41]. The rosemary mordanted fabric showed good antioxidant durability (above 80%) after the 10th washing cycle), indicating that the elements responsible for bioactivity were not affected by the washing process and thus remained very active [36].

4 Conclusion

The bio-mordants improved the colour strength from 0.612 to 0.863 for the dyed cotton and to 0.911 for the cotton fabric mordanted with mango and rosemary, respectively. In addition, the bio-mordants modified the colorimetric values, resulting in different shades of brown colour on the cotton fabric. All the fastness properties in this case showed good results of 4–5 and higher. This study thus shows that bio-mordants provide the opportunity to make natural dyeing an eco-friendly process as suitable alternatives to toxic metallic mordants, and thus need to be exploited in the textile industry. The dye extract imparted the anti-oxidant activity onto the cotton fabric, which showed a good radical scavenging activity of between 72.5% and 85.3%. The antioxidant activities were found to be durable, even after several washing cycles. As a result, the dye extract is proposed as an agent for the future development of bioactive, protective and healthy textile fabrics.

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Mathematical Modelling of the Parameters of Braided Textile Tapes

Matematično modeliranje parametrov za izdelavo prepletenih tekstilnih trakov

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Abstract

Braided textile materials are widely used in many industries and agriculture. Braided tapes are used for domestic purposes, in the food industry, in construction, in medicine, in aircraft manufacturing, in electrical engineering, etc. Every braided product must correspond to a unique group of parameters and properties, depending on the initial manufacturing parameters. The production of braided tapes is still insufficiently explored. In the process of designing and manufacturing products with specified parameters, it is necessary to substantiate the formation of braided products. The manufacture of products with specific parameters and properties, and the creation of rational technological modes for that production represent urgent scientific issues to be addressed. One way to solve this problem is to conduct factorial experiments. This article thus presents the results of a factorial experiment, during which the following input parameters were determined based on preliminary studies: type of raw material, the linear density of raw materials and speed of removal of the product from the formation zone. The following were chosen as output parameters: breaking load, breaking elongation, the linear density of tapes, product width and the number of strands per 10 mm. The limits of factor variation were determined for four types of raw materials. Based on the results of the processing of the obtained experimental data, linear mathematical models were developed. The results of the verification of mathematical models indicated that they adequately describe the process of braiding tapes within the intervals determined by the conditions of the experiment. We thus established a connection between the factors of the braiding process and the properties of braided tapes.

Keywords: braided tapes, braiding process, mathematical modelling

Izvleček

Prepleteni tekstilni materiali se pogosto uporabljajo v različnih industrijah in kmetijstvu. V gospodinjstvu, živilski industriji, gradbeništvu, medicini, letalski industriji, elektrotehniki itd. uporabljajo prepletene trakove. Vsak prepleteni izdelek ima edinstvene lastnosti, ki so odvisne od začetnih proizvodnih parametrov. Sama izdelava prepletenih trakov je še vedno premalo raziskana. Pri načrtovanju in izdelavi prepletenih trakov s specifičnimi parametri je treba definirati način oblikovanja njihove strukture. Izdelava trakov in ustvarjanje gospodarnih tehnoloških postopkov povzročata znanstvene probleme, ki jih je treba premagati. Eden od načinov reševanja teh problemov je faktorski poskus. V članku so predstavljeni rezultati faktorskega poskusa, pri katerem so bili na podlagi predhodnih študij določeni vhodni (su-

rovinska sestava, dolžinska masa niti in hitrost odvajanja oblikovanega traku) in izhodni parametri (izbrane lastnosti končnega izdelka (traku): pretržna obremenitev, pretržni raztezek in dolžinska masa trakov, širina izdelka in število niti na 10 mm). Za štiri vrste izbranih surovin so bile določene meje variiranja faktorjev. Na podlagi rezultatov obdelave eksperimentalnih podatkov so bili razviti linearni matematični modeli. Rezultati preverjanja matematičnih modelov so pokazali, da ti ustrezno opisujejo proces izdelave prepletenih trakov v intervalih, določenih z eksperimentalnimi pogoji. Ugotovljena je bila povezava med dejavniki postopka prepletanja in lastnostmi prepletenih trakov.
Ključne besede: prepleteni trakovi, postopek prepletanja, matematično modeliranje

1 Introduction

The world is witness to the constant development and improvement of the design of braiding machines and their components. Modern technologies, including information technology, are being introduced in production practices. At the same time, there is a wide range of problems and certain issues which hinder further development that remain insufficiently explored. In addition, the number of scientific works relating to the processing of textile materials is small compared with other technologies.

In recent works, it has been determined that braided structures can be tailored for a particular application by choosing the right set of parameters in order to obtain the desired level of properties based on predictive modelling [1].

Problems relating to the mechanical aspects of the braiding process have been investigated for many years and are discussed below. There is a description and analysis of the forces acting in and around the braiding zone, yarn direction, yarn tension, take-off and braiding velocities, all of which affect the quality of braids. Also discussed are connections between the prediction of braiding angles and picks per length, depending on the take-off [2]. Works dedicated to development of 3D braiding technology can be singled out, as well as research regarding the development of 3D braiding technology in terms of fabric, braiding technology and equipment. [3, 4].

Interesting is the development and use of software. Computer programs have been developed that are capable of designing geometric models, including braiding products. These programs include WiseTex, TexMind Software and SolidWorks [5–7].

At the same time, the manufacturers of woven textile materials require modelling between the input parameters of the technological process and the physical and mechanical properties of the finished products.

Let us briefly consider the formation of a braided product. The thread forming the braided product moves simultaneously in two planes. The first movement is provided by the towing device, while the second is caused by the continuous movement of the spindle along its trajectory [2].

On preparatory equipment, raw materials (threads) are spun onto bobbins that are tucked into spindles. On the upper web of the machine, there is a system of horn gears with several notches into which the spindles are installed. The thread is unspun from the bobbin by a towing device and passes into the product formation zone – the place of braiding – where the threads of all systems form the product.

The trajectory of the spindle in the horizontal plane is called the move and is a set of connected alternating semicircles. Single-stroke and two-stroke are the most widely used machines.

In addition to the threads of the braiding system, braided tapes can include threads of the base system [8]. The warp threads occupy positions in the product along its axis inside the braid. The position of the braiding threads (Figure 1(a)) is characterized by the angle of inclination of the thread relative to the axis of the product (α), the number of strands per 10 mm and the characteristics of the weave repeat (*L*) [9]. The number of strands can be determined by the horizontal or vertical axis of a product. The number of strands along the horizontal axis is used very rarely; it is deemed sufficient to determine the number of strands along the vertical axis, on the track that is parallel to the axis of the product.

Conventionally, a weave repeat is the smallest number of threads forming a complete weave pattern [10]. By analogy with a braided structure, the weave repeat of a braided product is the smallest number of threads, in which the order of intersection of the threads of the braiding system is repeated [9].

Typically, the starting point, when determining the repeat (L) of the braided tape, is the point A_{n-1} (Figure 1(b)), which corresponds to the extreme

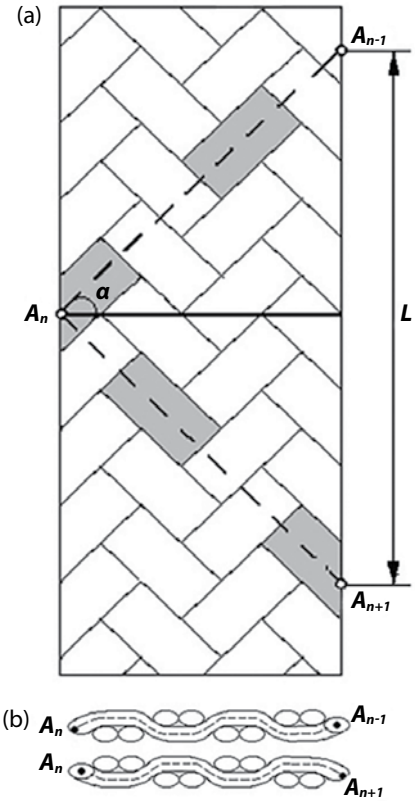

Figure 1: Schematic illustration of the structure of a braided product (a) and along its forming thread (b): angle to the axis of the product (α), distance of the pattern repeat (L), extreme points of the beginning (A_{n-1}) and end of repeat (A_{n-1}), and change of direction (A_n)

position that the thread axis can occupy. At the extreme points (A_{n-1}, A_n and A_{n+1}), the thread bends along the entire braid, changing the direction of movement to the opposite. Figure 1(b) presents a diagram of a section of a braided tape along the axis of the thread that forms it. In most cases, the threads in a braided product do cross not at a right angle, so cross-sectional images of the threads have the shape of an ellipse. For the convenience of depicting the section of the thread in the braid, we divide the repeat into two parts. All of the threads that form a braided tape cross twice in one repeat with other threads [11].

2 Methods

To study the mechanism of complex processes in the braiding of textile materials, it is necessary to establish the link between the factors of the process and the properties of the product, and present that link in a compact and convenient form with a quantitative estimation (in the form of a mathematical model). Traditionally, research methods in the textile industry are associated with experimentation. The methods of optimal experiment planning facilitate the use of mathematical apparatus, not only in the phase of processing the measurement results, but also for preparing and conducting research [12].
Four factorial experiments were carried out on a class 17 flat braiding machine (TP-17-3, produced by Tex-Inter Co. LTD). We chose four types of raw materials for the experiments: cotton thread (25 tex), polyester thread (34 tex), polyamide thread (29 tex) and fiberglass thread (68 tex). These types of raw materials are the most common used today in the production of braided tapes. Nevertheless, it should be noted that raw materials modified using modern methods can also be used to produce woven products [13, 14].
To study the process of braiding tapes using an experimental planning method based on preliminary studies, the following input parameters were determined: breaking load (N); breaking elongation (%); linear density (g/m); width (mm); and the number of strands per 10 mm (s/cm).
We chose the following factors to study braided tapes: type of raw material; equipment class; linear density of raw materials (tex); and product withdrawal speed from the formation zone (m/h).
All experimental tests were carried out according to current methods and standards [15–17].

3 Results

The limits of variation of the factors given in *Table 1* were determined for four types of raw materials and two classes of equipment.
Other parameters of the weaving process (such as the height of the braiding point, yarn tension and bobbin winding tension) during the experiment were within technological parameters.
The experimental plan and the obtained input parameters are shown in *Table 2*, where X_1 represents the linear density of the raw material (tex), X_2 represents product withdrawal speed from the formation zone (m/h), Y_1 represents breaking load (N), Y_2 represents breaking elongation (%), Y_3 represents linear density (g/m), Y_4 represents the number of strands per 10 mm (s/cm) and Y_5 represents product width (mm).

Table 1: Factors and their levels of variation in the braiding process

Type of raw material	Factors	Variation levels	
		-1	+1
Cotton thread	X_1 – linear density of raw material (tex)	25	100
	X_2 –product withdrawal speed from the formation zone (m/h)	57	142
Polyamide thread	X_1 – linear density of raw material (tex)	29	116
	X_2 –product withdrawal speed from the formation zone (m/h)	57	142
Fiberglass thread	X_1 – linear density of raw material (tex)	68	272
	X_2 –product withdrawal speed from the formation zone (m/h)	57	142
Polyester thread	X_1 – linear density of raw material (tex)	34	136
	X_2 – product withdrawal speed from the formation zone (m/h)	57	142

Table 2: Experimental plan and obtained input parameters in the braiding process

Type of raw material	Experiment number	Factor scores				Input parameters				
		Coded		Natural						
		X_1	X_2	X_1	X_2	Y_1	Y_2	Y_3	Y_4	Y_5
Cotton thread	1	1	1	100	142	238.3	19.2	1.84	2.80	7.6
	2	–1	1	25	142	43.15	9.8	0.40	3.70	4
	3	1	–1	100	57	218.3	23.6	2.02	6.58	8.9
	4	–1	–1	25	57	49.23	10.6	0.42	6.3	3.8
Polyamide thread	1	1	1	116	142	974.78	26.8	2.01	2.4	7.3
	2	–1	1	29	142	235.75	23.6	0.49	2.1	3.6
	3	1	–1	116	57	923.79	42.2	2.17	5.1	8.2
	4	–1	–1	29	57	242.64	18.8	0.50	6.2	4
Fiberglass thread	1	1	1	272	142	967.92	14.8	4.72	3	8.5
	2	–1	1	68	142	411.49	8.2	1.14	4.3	3.4
	3	1	–1	272	57	929.67	14	5.02	6.2	9.2
	4	–1	–1	68	57	421.3	10.2	1.25	5.8	4.8
Polyester thread	1	1	1	136	142	759.6	46.2	2.82	2.7	15.5
	2	–1	1	34	142	195.74	41.0	0.74	4	8.2
	3	1	–1	136	57	725.3	57.3	2.95	7.18	16.9
	4	–1	–1	34	57	193.39	37.4	0.72	7.36	8.7

The calculation of the coefficients of regression equations made it possible to obtain linear equations in encoded values. The resulting equations are given in *Table 3*, where X_1 represents the linear density of the raw material and X_2 represents the product withdrawal speed from the formation zone.

The model adequacy hypothesis was tested using Fisher's F-test. The results of the calculation of F-test values for the obtained linear models are shown in Table 4.

The calculated F-test values for the models range from 1.07 to 4.41, and do not exceed the table value of the F-test for a confidence interval of 0.95 and the number of degrees of freedom 1 and 16 equal to 4.49. Taking this into account, the hypothesis that the obtained models adequately describe the process of tape braiding was confirmed.

The significance of the coefficients of regression equations was verified by constructing a confidence interval. *Table 5* shows the results of the calculation of the confidence interval for each of the obtained models.

Table 3: Mathematical models of braided tape parameters obtained from the results of the experiment

	Type of raw material	Mathematic models
Breaking load (N)	Cotton thread	$Y_1 = 137.25 + 91.06X_1 + 3.48X_2$
	Polyamide thread	$Y_1 = 594.24 + 355.05X_1 + 11.03X_2$
	Fiberglass thread	$Y_1 = 682.60 + 266.20X_1 + 7.11X_2$
	Polyester thread	$Y_1 = 468.51 + 273.94X_1 + 9.16X_2$
Breaking elongation (%)	Cotton thread	$Y_2 = 15.80 + 5.6X_1 - 1.3X_2$
	Polyamide thread	$Y_2 = 27.85 + 6.65X_1 - 2.65X_2$
	Fiberglass thread	$Y_2 = 11.65 + 2.45X_1 - 0.45X_2$
	Polyester thread	$Y_2 = 45.48 + 6.28X_1 - 1.88X_2$
Linear density (g/m)	Cotton thread	$Y_3 = 1.17 + 0.76X_1 - 0.05X_2$
	Polyamide thread	$Y_3 = 1.29 + 0.80X_1 - 0.04X_2$
	Fiberglass thread	$Y_3 = 3.03 + 1.84X_1 - 0.103X_2$
	Polyester thread	$Y_3 = 1.808 + 1.078X_1 - 0.028X_2$
Number of strands per 10 mm (s/cm)	Cotton thread	$Y_4 = 4.844 - 0.156X_1 - 1.594X_2$
	Polyamide thread	$Y_4 = 3.95 - 0.2X_1 - 1.7X_2$
	Fiberglass thread	$Y_4 = 4.83 - 0.225X_1 - 1.175X_2$
	Polyester thread	$Y_4 = 5.31 - 0.37X_1 - 1.96X_2$
Product width (mm)	Cotton thread	$Y_5 = 6.075 + 2.175X_1 - 0.275X_2$
	Polyamide thread	$Y_5 = 5.775 + 1.975X_1 - 0.325X_2$
	Fiberglass thread	$Y_5 = 6.475 + 2.375X_1 - 0.525X_2$
	Polyester thread	$Y_5 = 12.325 + 3.875X_1 - 0.475X_2$

Table 4: Testing of the model adequacy hypothesis for braided tapes using the F- test

Parameter	Type of raw material	Variance of the adequacy of the mathematical model	Variance of the measurement of the parameter	Calculated F-test value
Breaking load	Cotton thread	170.04	38.95	4.37
	Polyamide thread	837.52	425.97	1.97
	Fiberglass thread	577.44	164.48	3.51
	Polyester thread	255.20	230.91	1.11
Breaking elongation	Cotton thread	3.24	1.80	1.80
	Polyamide thread	102.01	23.93	4.26
	Fiberglass thread	1.21	0.52	2.32
	Polyester thread	54.02	12.36	4.37
Linear density	Cotton thread	0.01	0.00	2.94
	Polyamide thread	0.01	0.00	1.17
	Fiberglass thread	0.01	0.00	3.52
	Polyester thread	0.01	0.00	2.74
Number of strands per 10 mm	Cotton thread	0.35	0.08	4.22
	Polyamide thread	0.49	0.11	4.31
	Fiberglass thread	0.72	0.17	4.35
	Polyester thread	0.31	0.09	3.35
Product width	Cotton thread	0.56	0.18	3.17
	Polyamide thread	0.06	0.06	1.11
	Fiberglass thread	0.12	0.09	1.42
	Polyester thread	0.20	0.12	1.64

Table 5: Confidence interval values for testing the significance of regression coefficients

Parameter	Type of raw material	Confidence interval value	
Breaking load	Cotton thread	3.323	5.857
	Polyamide thread	10.990	24.350
	Fiberglass thread	6.83	68.581
	Polyester thread	8.092	19.526
Breaking elongation	Cotton thread	0.714	1.107
	Polyamide thread	2.605	0.871
	Fiberglass thread	0.385	0.863
	Polyester thread	1.872	1.414
Linear density	Cotton thread	0.025	0.064
	Polyamide thread	0.037	0.031
	Fiberglass thread	0.027	0.221
	Polyester thread	0.024	0.046
Number of strands per 10 mm	Cotton thread	0.152	0.148
	Polyamide thread	0.180	0.108
	Fiberglass thread	0.217	0.166
	Polyester thread	0.163	0.180
Product width	Cotton thread	0.224	0.292
	Polyamide thread	0.126	0.170
	Fiberglass thread	0.156	0.174
	Polyester thread	0.187	0.194

A comparison of the absolute values of the coefficients of regression equations with the corresponding values of confidence intervals allows us to conclude that all the coefficients of regression equations are significant. The value and sign of the coefficient in the linear model, presented in encoded values, determine the influence of a particular factor on the parameter value.

4 Discussion

The value of the breaking load is most influenced by the linear density of the raw material. An an increase in linear density, within the interval determined by the conditions of the experiment and at a product withdrawal speed from the formation zone of 100 m/h, results in increase in the value of the indicator by 127–394%.

Figure 2 illustrates the breaking load response function of braided tapes made on a class 17 machine and the dependence of the breaking load on the linear density of the raw material at a fixed value of the product withdrawal speed from the formation zone (the factor varies within the intervals determined by the experiment).

An increase in the product withdrawal speed from the formation zone, within the intervals established by the conditions of the experiment, resulted in an increase in the breaking load by 5–20% (at a fixed value of the linear density of the raw material). It should be noted that tapes made of polyamide threads have the highest breaking load value, while tapes made of cotton threads have the lowest value.

The greatest influence on the value of the breaking elongation is exerted by the linear density factor of the raw material. An increase in that factor, within the values established by the conditions of the experiment, results in an increase in elongation by 32–178%. Figure 3 shows the planes of the response functions of the breaking elongation, as well as the dependency of the breaking elongation of braided tapes made from fiberglass thread and polyester thread at a product withdrawal speed of 100 m/h on the linear density of the raw material.

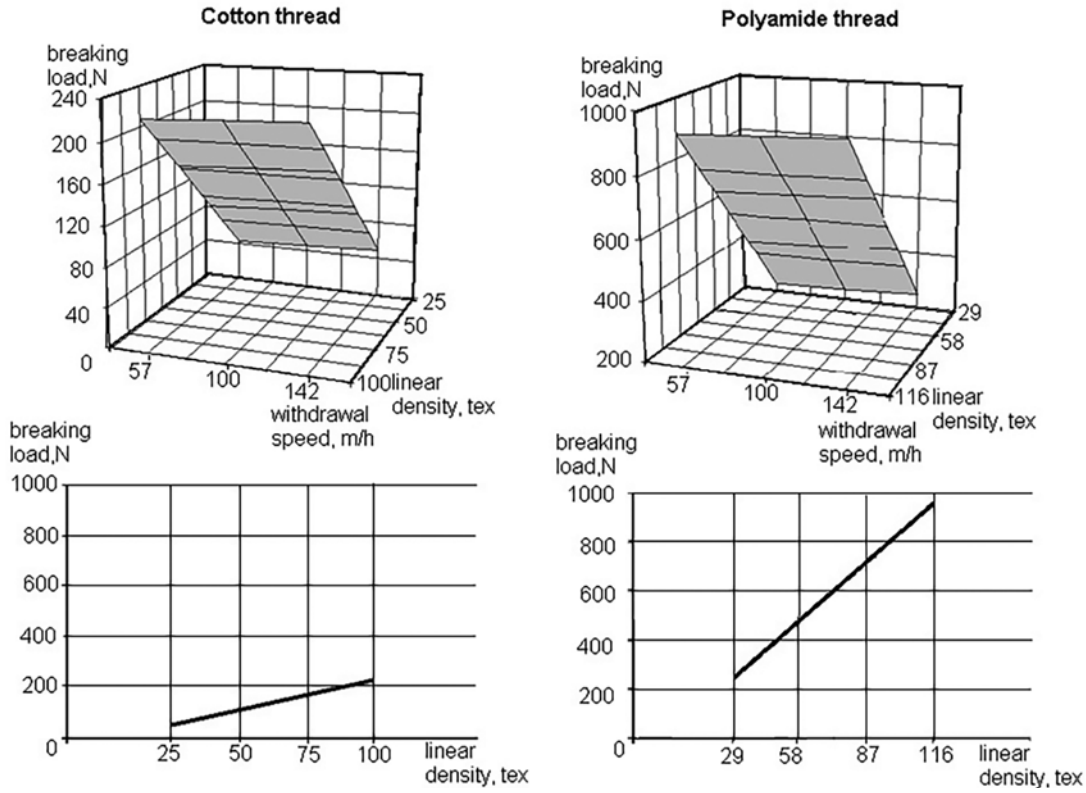

Figure 2: Planes of the response functions of the breaking load of tapes

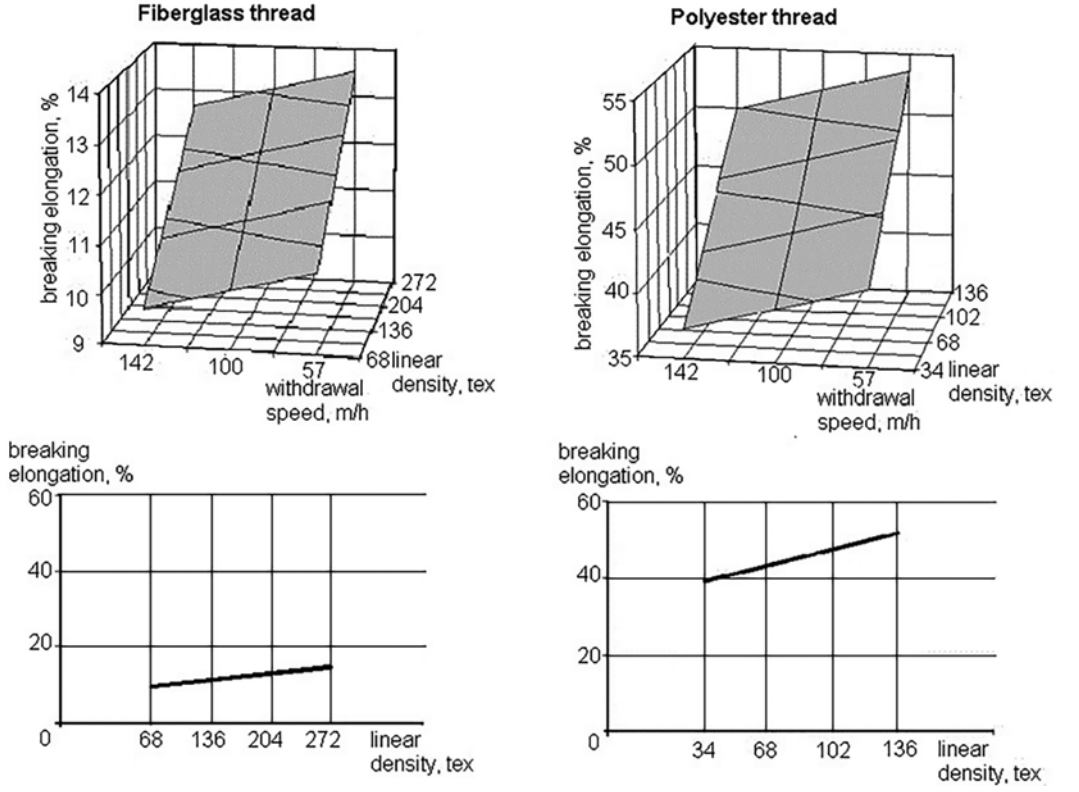

Figure 3: Planes of the response functions of the breaking elongation of tapes

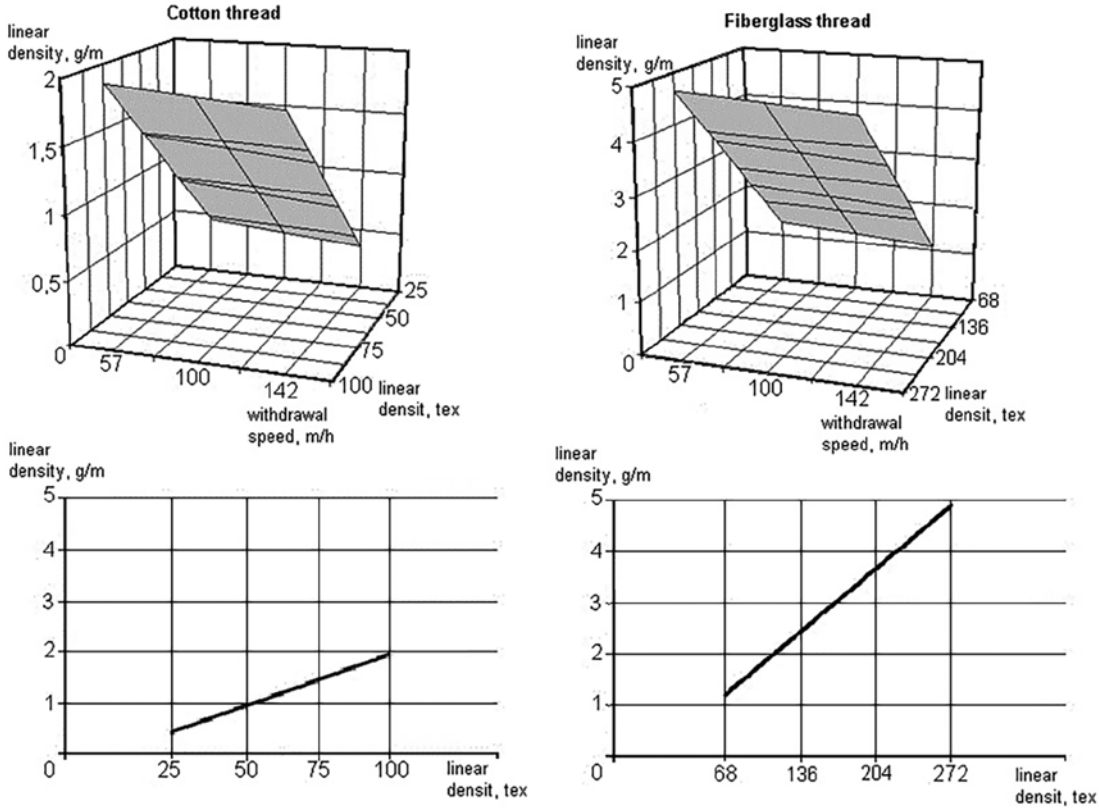

Figure 4: Planes of the response functions of the linear density of the tapes

An increase in breaking elongation by 7–19% is observed with a fixed value of the linear density of the raw material within the variation interval and a decrease in the product withdrawal speed from the formation zone.

The linear density of the product increases with an increase in the value of the linear density of the raw material factor by 295–390%. Figure 4 shows the planes of the response functions of the product linear density and the dependence of the linear density on the linear density of the raw material at a product withdrawal speed of 100 m/h. The graph is constructed according to linear equations characterizing the properties of tapes made from different types of raw materials.

The value of the linear density of the product decreases by 3–12% with an increase in the product withdrawal speed from the formation zone.

The greatest influence on the parameter of the number of strands per 10 mm of the product was exerted by the product withdrawal speed from the formation zone, an increase in which led to a decrease in the aforementioned parameter by 39–60%.A decrease in the number of strands per unit of length of the tape by up to 15% is observed up with an increase in the linear density of the raw material at a product withdrawal speed from the formation zone of 100 m/h.

The width of the tapes (Figure 5), *ceteris paribus*, increases by 87–123% with an increase in the linear density of the raw material and by 15% with a decrease in the product withdrawal speed from the formation zone. This indicator depends to a large extent on the linear density of the raw material.

5 Conclusion

Based on the data of full factorial experiments, linear models were developed that adequately describe the braiding process within the intervals determined by the conditions of the experiment. A connection was established between braiding factors and the properties of a braided product. Analysing the resulting linear models, we can determine the following. The value of the breaking load of the tapes is most influenced by the linear density of the raw material. An increase in linear density, within the interval determined by the conditions of the experiment and at a fixed product withdrawal

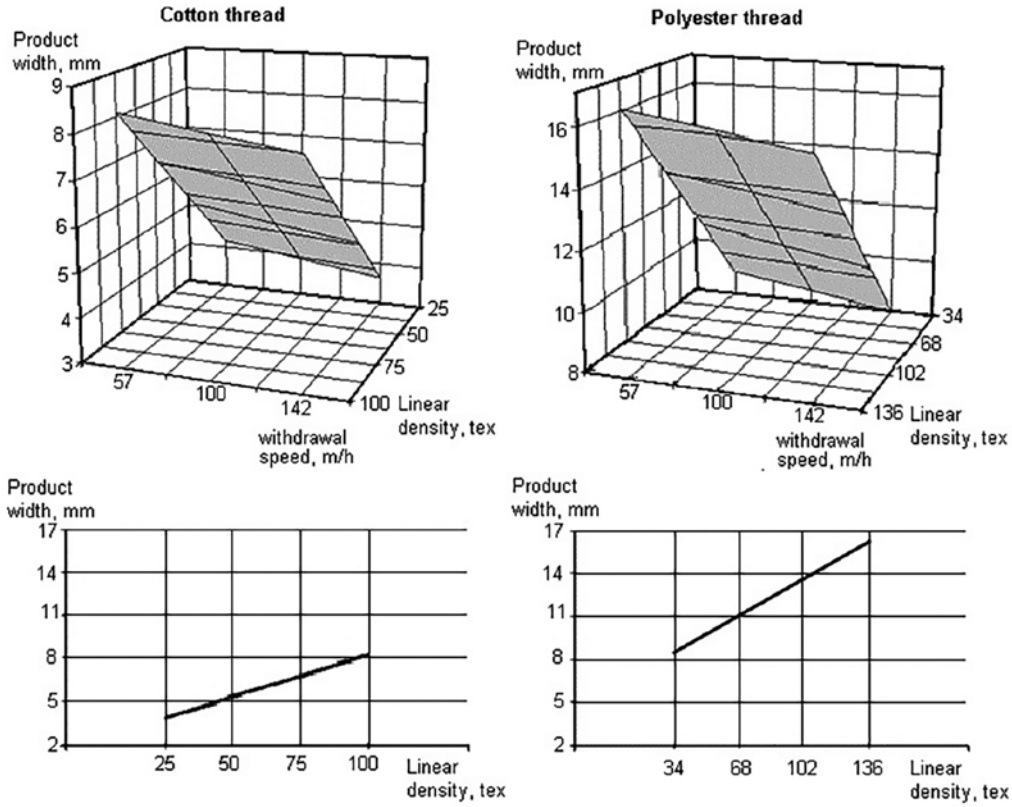

Figure 5: Planes of the response functions of the width of the tapes

speed from the formation zone within the interval, results in an increase in the value of the indicator by 127–394%. An increase in the product withdrawal speed from the formation zone results in an increase in the breaking load by 5–20%. The greatest influence on the value of the breaking elongation of the tapes is exerted by the linear density factor of the raw material, an increase in the value of which increases the elongation by 32–178%. An increase in breaking elongation of up to 19% is observed at a fixed value of the linear density of the raw material and a decrease in the product withdrawal speed.

The linear density of the tapes increases with an increase in the value of the linear density of the raw material by 295–390%. An increase in the product withdrawal speed from the formation zone results in a decrease in the value of the linear density of the product by up to 12%.

The greatest influence on the number of strands per 10 mm of tape was exerted by the factor of the product withdrawal speed from the formation zone, an increase of which led to a decrease in the parameter by 39–60%. A decrease in the number of strands per unit of length by 5–15% is observed with an increase in the linear density of raw materials.

The width of the tapes, *ceteris paribus*, increases with an increase in the linear density of the raw material by 87–123% and a decrease in the product withdrawal speed from the formation zone by 4–15%. The data obtained as a result of the studies carried out make it possible to optimally select the parameters of braiding and raw materials for the manufacture of products with the necessary properties at the design stage, which contributes significantly to improving the efficiency of the process of braiding textile materials.

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Adhesion of New Thermoplastic Materials Printed on Textile Fabrics

Adhezija novih termoplastičnih materialov, natisnjenih na tkaninah

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Abstract

Combining 3D printing, especially fused deposition modelling (FDM) as a material extrusion technique, with textile fabrics can lead to full-layer composites as well as partly reinforced textiles with different mechanical properties at different positions. While the combination of both techniques enables the production of new kinds of objects different from common fibre-reinforced matrices, the adhesion between both materials is still challenging and the subject of intense research activities. Besides well-known setup and printing parameters, such as the distance between nozzle and fabric or the extrusion temperature, material combinations, in particular, strongly influence the adhesion between 3D printed polymer and textile fabric. In this study, we investigate composites of woven fabrics from cotton (CO), polyester (PES) and a material blend (CO/PES) with newly developed thermoplastic materials for FDM printing, and show that depending on the FDM polymer, the adhesion can differ by a factor of more than four for different blends, comparing highest and lowest adhesion.
Keywords: 3D printing, fused deposition modelling (FDM), high-performance polymers, high-performance polyolefin, fibre-reinforced polymers

Izvleček

Kombinacija 3-D tiskanja, še zlasti modeliranja taljenega nanosa (FDM), in tkanine lahko vodi do izdelave laminiranih kompozitov, kot tudi do delne ojačitve tekstilij z različnimi mehanskimi lastnostmi na različnih predelih. Medtem ko kombinacija 3-D tiskanja na tkanino omogoča izdelavo novih vrst večslojnih materialov, ki se razlikujejo od navadnih z vlakni ojačenih matric, je adhezija obeh materialov še vedno izziv in predmet intenzivnih raziskav. Poleg dobro znanih parametrov nastavitev tiskalnika in parametrov tiskanja, kot je razdalja med šobo in tkanino ali temperatura ekstrudiranja, na oprijem med 3-D natisnjenim polimerom in tkanino močno vpliva predvsem kombinacija materialov. V tem članku so predstavljene raziskave kompozitnih materialov, izdelanih iz bombažnih tkanin, poliestrskih tkanin oziroma tkanin iz mešanice bombaž/poliester in na novo razvitimi termoplastičnimi materiali za tisk s tehnologijo FDM. Ugotovljena je bila več kot štirikratna razlika med najslabšo in najboljšo adhezijo glede na uporabljene polimere pri 3-D tisku in tkanine.
Ključne besede: 3-D tiskanje, modeliranje taljenega nanosa, FDM, visokozmogljivi polimeri, visokozmogljiv poliolefin, polimeri, z vlakni ojačeni polimeri

1 Introduction

In recent years, 3D printing has emerged from a technique that necessitated expensive equipment and was mostly used for rapid prototyping towards a technology that is easily available at low costs, and that is also used for the rapid printing of single objects, from spare parts to gimmicks. Amongst the various additive manufacturing techniques, material extrusion, in particular FDM printing, has found its way into companies and laboratories, schools and private households due to its ease of use, non-toxic or low-toxic materials and inexpensive printers. Nevertheless, the mechanical properties of such FDM printed parts are usually weaker than those of injection moulded objects, since the layer-wise production process leads to a strong anisotropy and often induces air voids, further reducing stability [1–3].

Commonly, two different approaches are reported in literature to improve the mechanical properties of FDM printed parts: on the one hand, new filaments with improved mechanical properties can be used, e.g. fibre-reinforced polymers [4-6]; on the other hand, macroscopic combinations with mechanically stronger materials are possible, such as textile fabrics. In the latter case, the adhesion between both materials is important to avoid the detaching of the polymer from the fabric under mechanical forces. When combining stereolithography (SLA) printed areas on textile fabrics, low-viscous resin can easily penetrate into even fine fabrics [7], while FDM printed objects often adhere better to thicker fabrics with relatively large pores [8, 9]. Besides the fabric structure, primarily the nozzle-fabric distance influences the adhesion due to form-locking connections [10, 11], while the printing bed temperature [12, 13], chemical pretreatments [14, 15] or thermal post-treatments [16, 17] can also influence adhesion. It has also been shown that soft thermoplastic materials printed with FDM often have a higher adhesion on textile fabrics than rigid ones and, amongst the latter, that poly(lactic acid) (PLA) demonstrates higher adhesion on examined textile fabrics than acrylonitrile butadiene styrene (ABS), while nylon has higher or lower adhesion than PLA, depending on the substrate [18, 19].

That is why this study investigates six newly developed FDM filaments, partly glass or carbon fibre-reinforced, invented by Grauts GmbH [20]. We describe the filaments, having different elastic properties, and show their strongly varying adhesion on three different textile fabrics.

2 Materials and methods

The textile fabrics used in this study are depicted in Table 1. The pure CO and PES fabrics have a similar structure, while the CO/PES woven fabric is thicker and has a higher mass per unit area. The latter value was measured using an SE-202 analytical balance (VWR International GmbH, Darmstadt, Germany), while the fabric thickness values were measured using a J-40-T digital thickness gauge (Wolf-Messtechnik GmbH, Freiberg, Germany).

Printing was performed using an Orcabot XXL Pro 2 FDM printer (Prodim, The Netherlands), enabling printing with an extrusion temperature of up to 285 °C. The printing parameters for all filaments were as follows: nozzle diameter: 0.4 mm; layer height: 0.2 mm; unheated printing bed; 100% linear infill in ± 45° orientation; two perimeters; printing speed: 30 mm/s; and z-distance between nozzle and printing bed: 0.5 mm. A relatively high z-distance was chosen because it is well-known that near the optimum z-distance, even the smallest deviations have a large impact, while around the setting where the nozzle just slightly touches the fabric, this impact is almost negligible [10]. Thus, the chosen z-distance facilitated the comparison of the slightly thicker CO/PES fabric with thinner fabrics. The extrusion temperatures were optimized in pre-tests and were set in the range 230 °C to 250 °C, as depicted in Table 2.

Table 1: Textiles used in this study

Sample	Material	Structure	Mass per unit area (g/m^2)	Thickness (mm)
CO	100% cotton	Plain weave	150	0.50
PES	100% PES	Plain weave	160	0.50
CO/PES	70% CO, 30% PES	Plain weave	205	0.65

Table 2: Thermoplastic materials for FDM processing and corresponding printing temperatures. HPP = high-performance polyolefin

Filament name	Material	Shore hardness	Extrusion temp. (°C)
PA+Carbon	Polyamide / 15% carbon fibres	75 D [a)]	250
Mid GF 1461	Polyamide / 15% glass fibres	80 D [a)]	235
Mid GF 1613	Polyamide / 15% glass fibres	93 D [a)]	230
HPP+GF 1443	HPP / 15% glass fibre	84 D [a)]	230
HPP 1444	HPP	52 D	235
HPP 1476	HPP	57 D	235

[a)] Values measured using a PCE-DD-D durometer (PCE Instruments, Meschede, Germany) on 3D printed parts with 100% infill; other Shore hardness values were provided by the manufacturer.

This table also describes the FDM printing polymers. These filaments were chosen, since they have strong mechanical properties and can withstand much higher temperatures than PLA and other common thermoplastic materials used in FDM.

Samples were designed according to DIN 53530, using Autodesk Fusion, as rectangles with an area of 150 mm × 25 mm and a height of 0.4 mm (i.e. two printed layers). Adhesion tests were performed using Sauter FH2K and Zwick-Roel Z010 universal test machines according to DIN 53530 and evaluated according to ISO 6133, procedure B, taking into account the median of the measured adhesion force peaks for each sample. Three specimens were investigated for each combination of filament and textile fabric. Microscopic images were taken using a Camcolms2 digital microscope.

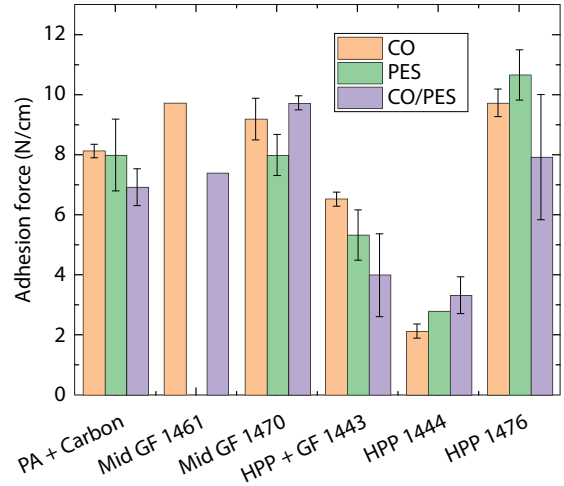

Figure 1: Adhesion forces between different textile fabrics (coloured) and novel FDM printing filaments

3 Results and discussion

The results of the adhesion force measurements are depicted in Figure 1. Since the z-distance is not optimized, the values are generally smaller than possible with these material combinations. The largely small error bars, however, indicate the reliability of the measured values.

Of all fabrics included in this study, the HPP 1444 filament shows the lowest adhesion. Much larger values are visible for HPP 1476, in particular, but also for the Mid GF 1461 and Mid GF 1470 glass-fibre reinforced filaments. It should be mentioned that Mid GF 1461 could not be printed properly on the PES fabric, so these values are omitted.

Unexpectedly, the CO/PES woven fabric, although thicker than the pure CO and PES fabrics, mostly shows smaller adhesion values than the others, while no large differences between CO and PES are visible. However, most of these differences are insignificant.

To evaluate these results, they can be compared with other 3D printing filaments reported in literature, using results that were measured for the nozzle just touching the textile surface. Some results found in literature (approximated for a z-distance identical with the fabric thickness) are given in Table 3.

As this comparison shows, the adhesion reached with the recently tested filaments is higher than some of the other results, but there are also adhesion forces more than twice the values measured in this study. One possible reason is that the new filaments tested in this study are high-temperature filaments, which should possibly be printed at even higher temperatures than in this study to reduce their viscosity during printing and enable deeper penetration into the textile fabrics under examination. Previous

Table 3: Values found in literature for adhesion forces, measured at or approximated for a z-distance identical to the fabric thickness. TPU = thermoplastic polyurethane (here with Shore hardness 86A), TPS = thermoplastic styrene (here with Shore hardness 67A and 79A)

Textile fabric	Fabric thickness (mm)	Filament material	Adhesion force (N/cm)	Ref.
Cotton woven	0.21	PLA	2	[10]
Polyester woven	0.19	PLA	0.5	[10]
Polyester woven	0.51	PLA	18	[10]
Polyester woven	0.51	ABS	3	[10]
Polyester woven	0.51	PA 6.6	11	[10]
CO/PES woven	0.45	TPU 2-86A	26	[17]
CO/PES woven	0.45	TPS1-67A	8	[17]
CO/PES woven	0.45	TPS2-79A	3	[17]
Cotton woven	0.49	PLA	8	[21]
Cotton woven	0.37	PLA	8	[21]
Cotton woven	0.39	PLA	16	[21]
Cotton woven	0.74	PLA	24	[21]
CO, PES, CO/PES	0.5–0.65	HPP 1476	8–11	This work

results from literature show that the shift in optimum z-distance is strongly correlated with the extrusion temperature [21], meaning future tests with increased printing temperatures are necessary.

On the other hand, previous studies showed that the textile fabrics used also played an important role, not only due to their pore dimensions, but also with respect to the fibre lengths in the fabrics, where hairy fabrics with long – and thus well-fixed – fibres in the yarn resulted in higher adhesion than fabrics from short-staple yarns, where the fibres are more easily pulled out of the yarn and thus cannot fix the imprinted polymer layers properly.

To investigate this possibility, Figure 2 shows microscopic images of the detached back of different polymers printed on the cotton fabric under investigation here. On the back of the black filaments, white cotton fibres are clearly visible, especially on the sample from the Mid GF 1470 filament. This matches the results of the adhesion tests where both Mid GF filaments gave high values on the cotton fabric. For HPP 1444, only very few fibres are visible, while a more in-depth look at HPP 1476 shows several fibres, but with optically reduced contrast on the blue filament. None of the images on HPP 1444 reveals more than a few fibres, which is in line with the finding that this filament has the lowest adhesion of all three textile fabrics. It should be mentioned, however, that here only small parts of the printed samples are depicted and that variations of the amount of fibres stuck on the polymer are visible for all samples.

While the filaments investigated here do not reach the maximum adhesion forces reported in previous studies, most of them (especially HPP 1476) show reliable adhesion on all three tested textile fabrics. Due to their good mechanical properties and high heat distortion temperature, compared with PLA, further experiments in combination with other fabrics, at higher extrusion temperatures and with optimized z-distance will be performed to enable the use of these filaments in composites for high-temperature applications or improved mechanical properties.

4 Conclusion and outlook

Six new filaments were FDM printed on different woven fabrics. Most of them showed reliable adhesion forces on PES, CO and CO/PES fabrics and fibre bundles attached to their back after separation during adhesion tests. The largest differences were found between two high-performance polyolefin materials, unexpectedly with the softest material (HPP 1444) having the lowest adhesion amongst the tested samples. The chosen substrates did not have a significant influence on adhesion to the thermoplastic materials printed on them.

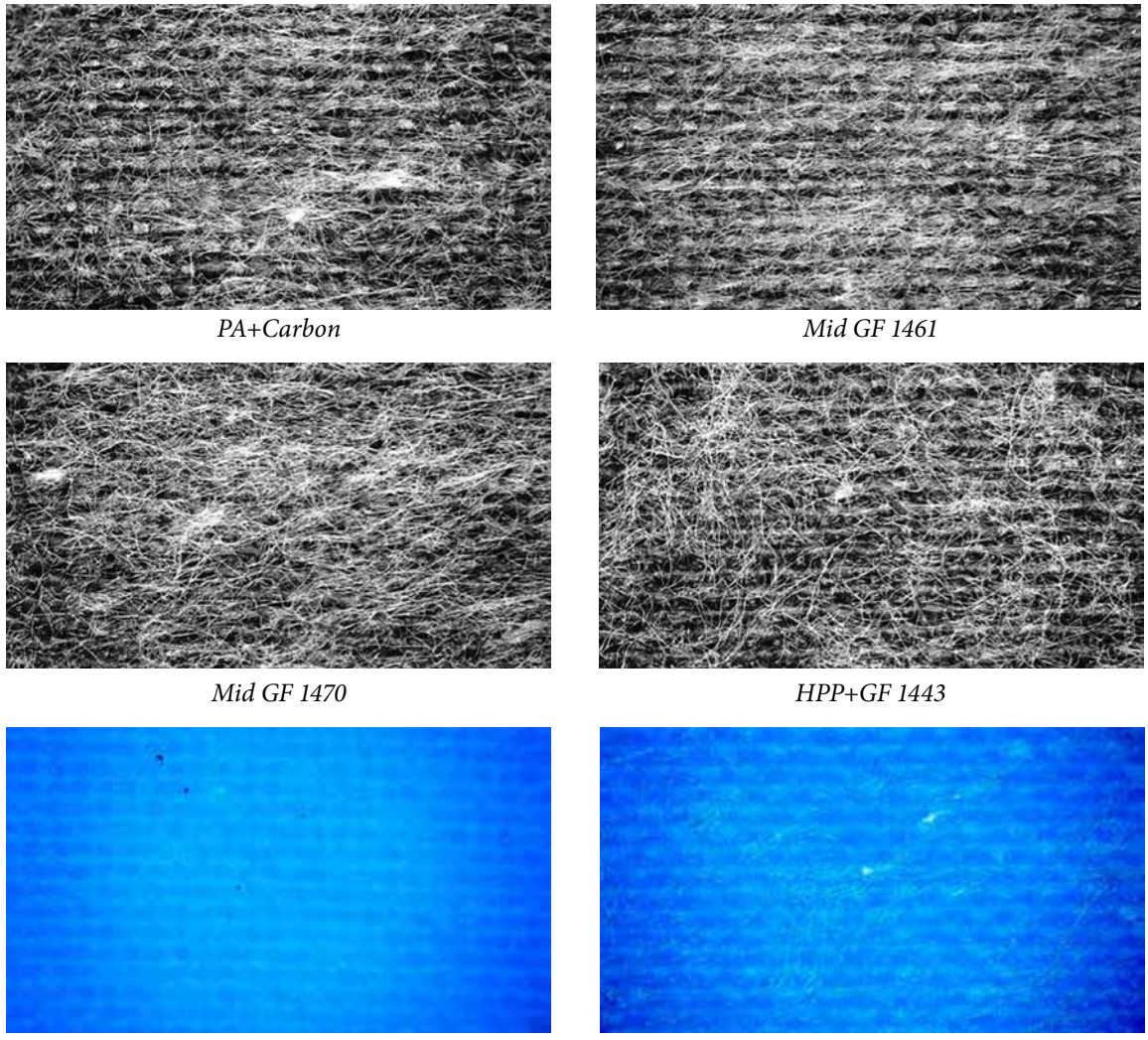

Figure 2: Back of the printed polymer, detached from cotton fabrics by adhesion tests according to EN 53530

Future tests will concentrate on optimizing the z-distance between fabric and printing nozzle, as well as the extrusion temperature, and on the investigation of more fabrics with different woven structures to further improve adhesion, so that applications, especially in high-temperature surroundings where PLA cannot be used, are enabled.

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Enhancement Reinforcing Concrete Beams Using Polypropylene Cord-Knitted Bars

Izboljšanje ojačitve betonskih nosilcev s pletenimi kompozitnimi palicami iz polipropilenskih vrvic

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Abstract

Currently, technical fabrics play a major role in many industries due to their multiple characteristics. The aim of this research was to utilize composite knitted bars to reinforce concrete beams. Six cord-knitted samples with two different polypropylene yarn counts (outer layer) and three different core materials were manufactured and immersed in a local epoxy material (Kemapoxy 150). Composite knitted bars were prepared in this way. Several tests were conducted for fabrics and knitted bar samples. All data were collected and analysed using two different tools: ANOVA test and radar chart area. Finally, three concrete beams with a varying number of cord-knitted bars (one bar, two bars and three bars) were produced. The results indicated that the differences in outer and core yarns for cord-knitted samples have a significant effect on several fabric and bar characteristics. The knitted bars with PP core yarn can be more beneficial for concrete that do does not require high stress, while the knitted bars using glass fibres and polypropylene (50% and 50% PE) as core materials are not appropriate for applications that require more flexibility and extensibility. Reinforced concrete beams were improved significantly with cord-knitted bars, taking into account the number of bars per area, which may cause the minimizing of flexure force through an increase in that number of bars per area.
Keywords: cord-knitted, cement, construction, mortar, strength

Izvleček

Tehnične tkanine so zaradi mnogoterih lastnosti pomembne v številnih panogah. Namen te raziskave je bil uporabiti pletene kompozitne palice za ojačitev betonskih nosilcev. Pletene vrvice, izdelane iz polipropilenskih prej v zunanji plasti in jedra iz združenih prej, so bile potopljene v lokalno dostopno epoksismolo Kemapoxy 150. Izdelanih je bilo šest vzorcev pletenih kompozitnih palic iz dveh polipropilenskih prej različne dolžinske mase in s tremi različnimi jedri. Opravljenih je bilo več testiranj pletenih vrvic in pletenih kompozitnih palic. Vse meritve so bile statistično analizirane (ANOVA) in prikazane s pomočjo polarnih grafikonov. Izdelani so bili trije betonski nosilci z eno, dvema oziroma tremi pletenimi kompozitnimi palicami. Rezultati so pokazali, da različne preje v zunanji plasti in jedru pomembno vplivajo na lastnosti pletenih vrvic in pletenih kompozitnih palic. Pletene kompozitne palice z jedrom iz polipropilenske preje so primerne za betonske nosilce, ki ne zahtevajo velikih obremenitev. Pletene kompozitne palice iz steklenih vlaken oziroma iz 50 odstotkov polipropilenskih in 50 odstotkov polietilenskih vlaken niso primerne za aplikacije, kjer sta zahtevani

večja upogibljivost in raztegljivost betonskih nosilcev. Pletene kompozitne palice so izboljšale učinkovitost armiranih betonskih nosilcev, pri čemer je bila ugotovljena največja upogibna sila pri uporabi dveh palic na betonski nosilec.
Ključne besede: pletenina, cement, gradbeništvo, malta, trdnost

1 Introduction

Recent advances in textile production technology have led to its use in many applications, such as medical, agricultural, aerospace, filtering, etc., due to its unique properties. These textiles are known as technical textiles [1]. Technical textiles are defined as "Textile materials and products manufactured primarily for their technical and performance properties rather than their aesthetic or decorative characteristics" [1].

The corrosion of steel bars (rebar) used for reinforcing concrete is a major factor in reducing the service lifetime of reinforced concrete constructions. Thus, the solution is to cover or replace rebars with non-corrosive materials [2]. Textile reinforced concrete is a new advanced composite material. It generally comprises alkali-resistant fibreglass (AR) or carbon fibres with cementitious matrix reinforcement, in contrast to steel reinforcement. The single fibres of glass or carbon AR can be positioned in textile in any direction, which results in the adopted perfect orientation of an applied load [3].

The use of polypropylene fibres (PP) in concrete increases the concrete's tension and compressive strength [4, 5]. PP fibres are hydrophobic and have a melting point of about 160 °C [6]. When used in concrete, the yarns of PP create pathways in the concrete to evaporate moisture. PP fibres have been used in concrete to reduce cracking, increase toughness and impact resistance, and thus improve the energy absorption capacity of the concrete [7–9]. Many studies have shown that adding a small amount of PP fibres to fresh concrete can significantly reduce plastic cracking in the early stages, while these fibres can also significantly limit surface cracking and aggregate settlement in fresh concrete, reducing the possibility of setting cracks [7, 10].

Textile composites are stiff materials (enhanced by fibres, yarns and different fabric materials) that have properties, such as light weight, flexibility, solid construction and toughness. They have been employed for structural or load-bearing applications due to their outstanding quality [11, 12].

Rebars made of glass fibre-reinforced polymers provide various advantages over standard reinforced steel, including a higher ratio, greater corrosion resistance, and greater fatigue load resistance. A lack of ductility, however, is one of the key drawbacks of GFRP rebars [13, 14].

Fibre-reinforced polymers (FRP) provide special benefits to address a variety of civil engineering issues in situations when traditional materials fall short of expectations. Unlike steel, FRP can withstand the corrosive effects of acids, alkalis, salts, and other similar hostile compounds, without being damaged by electrochemical degradation. FRP is widely recognised as a steel substitute in applications where steel is susceptible to the significant risk of corrosion due to its superior properties, including high tensile strength and corrosion resistance [13, 14]. Tarek Elsayed *et al.* suggested the use of aramid, carbon and glass fibres via pultrusion to produce local rebars [14].

Knitting techniques usually give fabrics unique performance characteristics due to their yarn-looping shape [15, 16]. Those types of knitted fabrics have been employed as textile reinforced concrete (TRC) for concrete reinforcement in some studies [17]. In the same context, the cord-knitted fabric is considered one type of knitted fabric that is produced in a tube shape with different structures using a very small diameter of a circular knitting machine. Inlay warp, weft, and core yarns can be added for cord construction, where the final applications of the cord-knitted fabrics are affected by tightness factors [18].

The available commercial materials for the TRC system were basalt, carbon, glass and polyphenylene fabrics with different construction. Mortar must have fine grains, good workability, plastic consistency, low viscosity (to facilitate application on steep or vertical surfaces) and sufficient shear strength (to keep the composite material from peeling away from the substrate) [19, 20].

Taking into account earlier studies, this research aimed to utilize PP fibres to design and produce bars as a composite technique with a cord-knitted fabric for reinforcing concrete beams (which is an economical material for this application) and evaluate its performance.

2 Materials and methods

Six cord-knitted samples were fabricated with a plain structure and constructed with two layers (outer and core). Two different yarn counts from PP yarn (133 tex and 267 tex) were used for the outer layer, while three different materials with the closest yarn counts were used for the core layer as presented in Table 1. Figure 1 illustrates the experimentally manufactured cord-knitted samples and cross section.

2.1 Manufacture of cord-knitted samples

A cord-knitting machine with a cylinder diameter of 6 mm, and eight needles was used to manufacture the six polypropylene cord-knitted samples. Figure 2 shows a cord-knitting machine and its specifications.

Figure 2: Cord knitting machine – internal cylinder diameter (cord diameter) = 6 mm
Quantity of needles: 8needles per diameter; machine speed: 800–1200 rpm; no creel needed and cones can be installed directly on the machine; automatic stop-motion system with tensioner configuration

2.2 Preparation of composite cord-knitted fabrics

Kemapoxy 150 (solvent-free, transparent epoxy) commercial resin from the company CMB was used for the preparation of composite bars as shown in

(a) *(b)*

Figure 1: Cord-knitted fabrics: a) produced PP cord-knitted fabrics, b) cord-knitted cross section

Table 1: Experimental design of manufactured cord-knitted fabrics

Sample code	Outer layer		Core layer	
	Material	Yarn count (tex)	Material	Yarn count (tex)
1	PP [a)]	267	PP [a)]	approx. 156
2	PP [a)]	267	GF [b)]	
3	PP [a)]	267	50% PP/50% PE [c)]	
4	PP [a)]	133	PP [a)]	
5	PP [a)]	133	GF [b)]	
6	PP [a)]	133	50% PP/50% PE	

[a)] polypropylene, [b)] glass fibre, [c)] polyethylene

Figure 3. In order to prepare Kemapoxy 150, component B (hardener) was added to component A (resin) and manually mixed for three minutes. The cord-knitted fabrics were then immersed in a mixture for one minute, removed and put on a flat surface. The air was then vacuumed from the fabrics to obtain a stiffer shape and rough surface after drying.

(a) (b)

Figure 3: Coated samples (a) by Kemapoxy (b)

2.3 Reinforcement of concrete beams with composite cord-knitted bars

The concrete mix was prepared with the mixer shown in Figure 4 using the proportions presented in Table 2. One blank concrete beam and three groups of concrete beams measuring 50 cm × 10 cm × 10 cm were then produced. The first group of concrete beams was reinforced with one prepared PP cord-knitted bar, the second group of concrete beams was reinforced with two prepared PP cord-knitted bars and the third group of concrete beams was reinforced with three prepared PP cord-knitted. Figure 5 shows the pouring of reinforced prepared beams and Figure 6 shows reinforced concrete beams with PP prepared bars.

Figure 4: Concrete mix

Table 2: Proportions of concrete mix

Cement (kg)	Water (kg)	Super plasticizer (L)	Sand (kg)	Coarse aggregate (kg)
325	146	4.8	765	1145

Figure 5: Pouring reinforced beams with PP prepared bars

Figure 6: Reinforced concrete beams with PP prepared bars

2.4 Testing and analysis

Standard tests were used to test cord-knitted fabrics, composite bars and reinforced concrete beams, as described below.

2.4.1 Fabric testing

According to standard methods, tests were conducted on cord-knitted fabrics to determine their stitch length [21], tightness factor [22–26], longitudinal weight [27], thickness [28], bending length [29] and tensile force [30]. All tests were carried out according to standard conditioning, as described in test method BS 1051 for textile testing, where all samples were placed for 24 hours at a temperature of 20 °C ± 2 °C and a relative humidity (RH) of 65 % ± 2 %.

2.4.2 Testing of composite cord-knitted bars

At the national research centre, all prepared composite samples were examined for the bars' longitudinal weight [27], diameter and tensile force [31], in accordance with standard methods to investigate any potential effects on the application of reinforced concrete beams.

2.4.3 Testing of reinforced concrete beams

One of the key metrics for describing concrete strength is flexure strength. It is a measurement of a beam's or slab's resistance to bending failure. According to the ASTM C293 standard test method (centre point loading) [32], flexure strength is tested in the lab of the Housing and Building National Research Centre by applying a point load to concrete beams in the middle of a span length. Figure 7 shows the flexure of a beam that has been reinforced with bars made from PP cord-knitted textiles.

Figure 7: Flexure point load test of reinforced beam with the PP cord-knitted bars

2.5 Data analysis

All results were collected and analysed using two different tools: an ANOVA test with a P-value of ≤ 0.05 was performed in order to identify the significant/insignificant effect of different variables on the production of bars, while a radar chart area was calculated and plotted in order to rank the samples and assign the preferable performance characteristics.

3 Results and discussion

3.1 Physical and mechanical characterizations

Cord-knitted samples were tested and tabulated to compare their characteristics, as shown in Table 3. The findings show that the cord-knitted bars with a core PP yarn (samples No. 1 and 4) achieved the lowest fabric thickness and fabric tensile force, as well as the highest tightness factor and elongation percentage relative to other cord-knitted samples. Furthermore, the cord-knitted bars with core glass fibres (GF) (samples No. 2 and 5) attained the highest fabric weight/length at one meter and the lowest elongation, while the cord-knitted bars with a core of 50% PP/50% PE (samples No. 3 and 6) achieved the highest fabric thickness, bending length and tensile force. The reason can be traced to variation in the density and elasticity of the core yarns, which are seen in several knitted-bar characteristics.

As a result of the above, prepared composite bars of cord-knitted samples were tested, and the findings are presented in Table 4. The findings show that the bar samples prepared with PP as a core material achieved the lowest weight, diameter and tensile force (N). On the other hand, the bar samples prepared with GF achieved the highest weight, medium diameter and tensile force, while the bar samples prepared

Table 3: Tests results of PP cord-knitted samples

No.	Stitch length (cm)	Tightness factor	Weight (kg)	Thickness (mm)	Bending length (cm)	Tensile force (N)	Elongation (%)
1	0.2	5.74	0.147	5.1	33.5	1120	32.77
2	0.228	5.03	0.163	5.8	40	1670	4.32
3	0.247	4.65	0.126	6.3	40	2190	18.73
4	0.251	6.47	0.189	6.4	32.5	1180	73.32
5	0.255	6.37	0.218	6.5	37	1290	5.25
6	0.286	5.68	0.167	7.3	42.5	2990	59.92

with 50% PP/50% PE achieved a medium weight, and the highest bar diameter and tensile force, emphasizing the unique and significant effect of core yarns on the characteristics of bar samples.

Table 4: Tests results of PP cord-knitted prepared bars

Sample no.	Bar weight (kg)	Bar diameter (mm)	Bar tensile force (N)
1	0.028	5.9	1800
2	0.033	5.9	2620
3	0.032	6.3	3370
4	0.031	6.5	1770
5	0.037	6.7	2240
6	0.034	7.3	3360

3.2 Significant/insignificant effect

In order to explain the effect of different variables in the production of samples, an ANOVA test with a P-value of ≤ 0.05 was performed, with the results shown in Tables 5 and 6. The results indicate that the difference in outer PP yarn count (133 tex and 267 tex) has a significant effect on the stitch length, weight and thickness characteristics of cord-knitted samples, and on the diameter characteristic of the prepared composite bars. Moreover, the results indicate that the variation of core yarns only had significant effect on the fabric weight of the cord-knitted fabric, and on the tensile force of the prepared composite bars.

Table 5: ANOVA results of PP cord-knitted samples

Characteristics	P-value	
	Outer count yarns	Core materials
Stitch length (mm)	0.030139 [a)]	0.077754
Sample weight (g)	0.008714 [a)]	0.028537 [a)]
Sample thickness (mm)	0.028714 [a)]	0.074176
Sample bending length	0.785166	0.098936
Sample tensile force (kN)	0.687811	0.135106
Sample elongation (%)	0.174346	0.176963

[a)] Significant effect

3.3 Ranking of samples

In order to determine preferable performance characteristics, radar chart areas were calculated and plotted for all samples, as shown in Figure 8 and Table 7. The results indicate that the cord-knitted sample No. 6 (PP 266 tex, 50% PP/50% PE core yarn) achieved the highest behavioural characteristics, while the cord-knitted sample No. 5 (PP 133 tex, GF core yarn) recorded the lowest. Moreover, the results indicate that the cord-knitted sample No. 1 with a core yarn PP and outer PP yarn count of 133 tex ranked highest among other samples with the same outer PP yarn count, while the cord-knitted sample with a core yarn PP and outer PP yarn count of 266 tex ranked second. We can thus conclude that the PP yarns had a significant effect on produced cord-knitted performance characteristics.

Table 6: ANOVA results of PP cord-knitted prepared bars

Characteristics	P-value	
	Outer count yarns	Core materials
Bars' weight (g)	0.31687	0.269231
Bars' diameter (mm)	0.020204 a)	0.088235
Bars' tensile force (kN)	0.364074	0.016863 a)

[a)] Significant effect

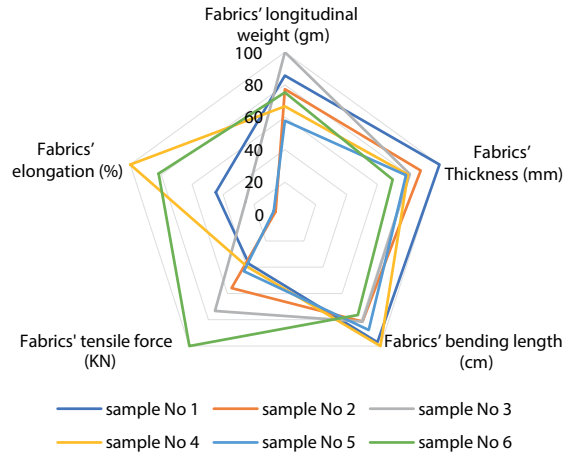

Figure 8: Radar chart for PP cord-knitted fabric samples

At the same time, radar chart areas for prepared composite bars were calculated and plotted, as seen in Figure 9 and Table 8. The result indicate that samples with 50% PP/50% PE core yarn ranked highest at different yarn counts of outer PP yarn, while samples with PP core yarn ranked lowest at different outer PP yarn counts, despite achieving a preferable rank among cord-knitted samples. This could be attributed to the decreasing tensile force of PP yarns compared with other materials (glass fibres and 50% PP/50% PE), an indication that its

Table 7: Radar chart for PP cord-knitted fabrics

Sample no.	Weight (%)	Thickness (%)	Bending length (%)	Tensile force (%)	Elongation (%)	Radar chart area	Rank
1	85.7	100	97.01	37.4	44.6	26070.41	3
2	77.2	87.9	81.2	55.8	5.89	18312.8	5
3	100	80.9	81.2	73.2	25.5	23823.32	4
4	66.6	79.6	100	39.4	100	26478.26	2
5	57.7	78.4	87.8	43.1	7.16	15159.14	6
6	75.1	69.8	76.4	100	81.7	30964.57	1

Table 8: Radar chart area calculation for prepared bars

Sample no.	Weight (g)	Thickness (mm)	Bar tensile force (kN)	Radar chart area	Rank
1	100	100	53.4	17911.6	5
2	84.8	100	77.7	19793.8	2
3	87.5	93.7	100	22784.7	1
4	90.3	90.8	52.5	15337.2	6
5	87.5	88	66.5	16778.8	4
6	75.7	80.8	99.7	18809.7	3

potential use in various applications does not require high stress. At the same time, the other materials may be inappropriate for applications requiring more flexibility and extensibility.

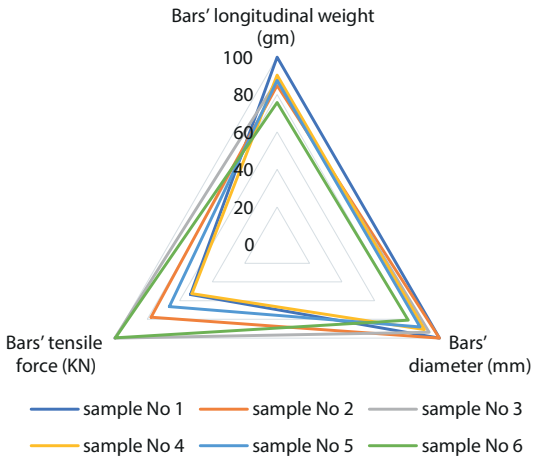

Figure 9: Radar chart for PP prepared bars

3.4 Results for reinforced concrete beams

Concrete beams reinforced with composite PP cord-knitted bars (PP 133 tex, PP core yarn) were produced. Three different composite cord-knitted bars (one, two and three bars) were applied to concrete beams, while a basic concrete bar without cord-knitted fabric was also tested. The flexure forces of basic and reinforced concrete beams were tested, as shown in Table 9. The results confirm that, although PP bars (PP core yarn, PP outer yarn) achieved the lowest tensile force (kN) as seen in Table 3, they enhanced reinforced concrete beams using varying numbers of cord bars, indicating that other prepared cord-knitted bars with altered cores yarn could further improve reinforced concrete beams. Additionally, the results indicate that the number of bars per area impacts the flexure force of reinforced concrete beams, as exceeding a certain limit might reduce flexure force, where beam No. 3 with three bars achieved the lowest force and beam No. 2 with two bars achieved the highest force.

Table 9: Flexure force results for concrete beams reinforced with polypropylene cord-knitted prepared bars

Sample no.	Number of bars	Reinforcing bar material	Flexure force (kN)
Basic	0	No reinforcing	10.5
Beam 1	1	PP	14.7
Beam 2	2	PP	15.7
Beam 3	3	PP	13.5

4 Conclusion

Based on the results reported in this study regarding the use of cord-knitted PP fabrics to reinforce concrete beams, the following main conclusions can be drawn:

- The variance of outer PP yarn count (133 tex and 266 tex) and core yarns has a significant effect on several cord-knitted fabric and bar characteristics.
- Although the cord-knitted fabric with PP outer and core yarns did not achieve the highest results (depending on radar area), it did have a significant effect on the samples' performance characteristics.
- According to the testing of knitted bars, the potential use of bars with PP core can be excluded for applications that require high stress. On the other hand, the knitted bars with core materials glass fibres and 50% PP/50% PE are not suitable for applications requiring more flexibility and extensibility.
- Cord-knitted bars enhanced reinforced concrete beams effectively, while the number of bars per area should be considered during the production of reinforced concrete beams, as exceeding a certain limit might reduce flexure force.

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